Microwave dielectric study of spin-Peierls and charge ordering transitions in (TMTTF)$_2$PF$_6$ salts

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We report a study of the 16.5 GHz dielectric function of hydrogenated and deuterated organic salts (TMTTF)$_2$PF$_6$. The temperature behavior of the dielectric function is consistent with short-range polar order whose relaxation time decreases rapidly below the charge ordering temperature. If this transition has more a relaxor character in the hydrogenated salt, charge ordering is strengthened in the deuterated one where the transition temperature has increased by more than thirty percent. Anomalies in the dielectric function are also observed in the spin-Peierls ground state revealing some intricate lattice effects in a temperature range where both phases coexist. The variation of the spin-Peierls ordering temperature under magnetic field appears to follow a mean-field prediction despite the presence of spin-Peierls fluctuations over a very wide temperature range in the charge ordered state of these salts.

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I. INTRODUCTION

The Fabre ((TMTTF)$_2$X) and Bechgaard ((TMTSF)$_2$X) series of charge transfer salts show a very rich sequence of competing ground states when either hydrostatic or chemical pressure is applied [1]. In the universal phase diagram of these series, the (TMTTF)$_2$X are Mott insulators that develop a charge ordered (CO) state [2,3]. This state is followed at lower temperature by either an antiferromagnetic N´eel or a lattice distorted spin-Peierls (SP) phase. This pattern turns out to be affected by pressure, anion X substitution, and to some degree by deuteration of the methyl groups. This is how the (TMTTF)$_2$X can be moved along the pressure axis with respect to the (TMTSF)$_2$X series for which metallic, antiferromagnetic and superconducting phases can be stabilized. In low pressure conditions, the quasi-one-dimensional (TMTTF)$_2$X salts thus appear as model correlated systems to study the interplay between spin, charge, and lattice degrees of freedom.

In several (TMTTF)$_2$X salts with octahedral anions X, the CO transition follows and in some cases coincides with the $4k_F$ charge localization [4,5,6,7,8]. The first evidences of this transition in X= SbF$_6$ and AsF$_6$ came from transport measurements [4,5,9]. The transition was dubbed “structureless”, because of the absence of any structural modification associated to it [5]. The CO character of the transition only comes much later from the low frequency dielectric response [6,10], and NMR experiments in which charge disproportionation in the unit cell was unveiled [7,11,12]. The CO character of the transition is also found from infrared spectroscopy measurements [13]. The CO transition is also accompanied by the onset of a ferroelectric state revealed by the divergence of the low frequency dielectric constant [14].

As regards to spin degrees of freedom, these are essentially decoupled from the progressive charge localization or the CO transition. However, in compounds like X= PF$_6$ in normal pressure conditions, a SP transition takes place at $T_{SP} \approx$ 18 K (hydrogenated) [15,16] and 13 K (deuterated) [17]. The spin singlet state that goes with the SP lattice distortion has been borne out by spin susceptibility [18,19,20], magnetic susceptibility [21,19,20], and NMR spin relaxation data [18,20,21]. In the PF$_6$ salt, the observation of X-ray 2$k_F$ diffuse scattering indicates the presence of lattice precursors of the SP transition below 60 K [17]: these open a spin pseudo gap [24], as exhibited by magnetic susceptibility and NMR data [18,20,21].

Finally, lattice expansion effects were recently observed at both CO and SP transitions of the PF$_6$ and AsF$_6$ salts [22,23]. Indeed, distinct lattice effects were found at $T_{CO}$ in the uniaxial expansivity along the interstack $c^*$ direction, signaling an active role of anion X lattice degrees of freedom in the stabilization of the CO ground state, and further clarifying the ferroelectric nature of the transition. It is also the $c^*$-axis expansivity that is the most strongly affected at the SP transition.

In this paper, we address the issue of interplay between charge, spin and lattice degrees of freedom by studying the 16.5 GHz complex microwave dielectric function of (TMTTF)$_2$PF$_6$ single crystals under magnetic field. Dielectric anomalies are observed at both the CO and SP transitions. The increase of the $T_{CO}$ scale and the drop of $T_{SP}$ are also confirmed for the deuterated compound as a negative shift on the pressure scale. Up to 18 Tesla,
the SP transition temperature varies quadratically with field, in fair agreement with the mean-field prediction obtained by Cross 23. Other magnetic fields effects on the polarizability of the system are found and ascribed to the interaction between CO and SP orderings. Finally, we report the existence of thermal relaxation effects in the deuterated compound near the SP transition.

II. EXPERIMENT

Hydrogenated (H$_{12}$) (TMTTF)$_2$PF$_6$ single crystals were grown from THF using the standard constant-current (low current density) electrochemical procedure. The synthesis of the fully deuterated TMTTF-d$_{12}$ (D$_{12}$) was attempted using the procedure of Wudl et al. 29 for the preparation of 3-chloro-2-butanone-d$_7$. However, this awkward very poorly described procedure led in our hands to traces, if any, of the desired chloroketone. Therefore, we worked out a more practical and shortened procedure to prepare this compound: perdeuteroacetoin was first prepared from perdeuteriobiacetyl [30] according to the literature 31 and the procedure for the conversion of allylic alcohols into chlorides 32 applied to this hydroxy ketone. To the cooled mixture of the latter with a slight excess of PPh$_3$, hexachloroacetone in sulfolane was added dropwise. The mixture stirred at room temperature for two weeks; a flash distillation of the reaction mixture followed by distillation at atmospheric pressure led to the desired chloroketone with a (up to) 50% yield. Starting from this deuterated chloroketone, TMTTF-d$_{12}$ was obtained using usual procedures and the corresponding deuterium incorporation-levels estimated by NMR to be 97.5%.

The crystals have the shape of a needle oriented along the chain axis a, from which were cut short slabs of typical dimensions 2.4x0.8x0.4 mm$^3$ for H$_{12}$ and 1.5x0.4x0.07 mm$^3$ for D$_{12}$. We used a standard microwave cavity perturbation technique 33 to measure the complex dielectric function $\varepsilon^* = \varepsilon' + i\varepsilon''$ along the a axis. A copper cavity resonating in the TE$_{102}$ mode was used at 16.5 GHz. The organic slab is inserted in a mylar envelope and immobilized by thin cotton threads to prevent any stress or movement during thermal cycling. The envelope is glued on a quartz rod to allow its insertion in the cavity and the precise orientation of the slab along the microwave electric field. Following the insertion of the sample, changes in the relative complex resonance frequency $\Delta f/f + i\Delta(1/2Q)$ (Q is the cavity quality factor) as a function of temperature are treated according to the depolarization regime analysis after substraction of the envelope contribution. With this microwave technique, the slab is associated to a prolate ellipsoid and absolute values can only be measured within 30% of accuracy. A magnetic field up to 18 Tesla could be applied along the $\varepsilon^*$ axis.

III. RESULTS AND DISCUSSION

When the temperature is decreased from 300 K, the dielectric function of both H$_{12}$ and D$_{12}$ salts shows a similar behavior: the real part $\varepsilon'$ decreases smoothly with a faster rate below 100 K before its saturation at low temperatures; the imaginary part $\varepsilon''$ rather increases first, reaches a maximum below 100 K and decreases rapidly toward zero at low $T$. These temperature profiles are presented below 120 K in Fig. 1. The features observed below 100 K are sharper for the D$_{12}$ compound than for the H$_{12}$ salt, because of the diffusive character of the transition identified of the relaxor type 34. The determination of $T_{CO}$ is more difficult for the H$_{12}$ salt, because of the diffusive character of the transition identified of the relaxor type 34. By looking at the temperature derivative of both parts of the dielectric function (see inset of Fig. 1 top panel), we deduce the value $T_{CO} \approx 63$ K, in agreement with previous works 37. Contrary to previous dielectric measurements on the same compound 10, however, our microwave data clearly show anomalies in the low temperature range at $T_{SP}$, which will be analyzed later in
Earlier analysis of the dielectric response measurements performed at lower frequencies ($10^3-10^7$ Hz) [10, 14], showed that the $\varepsilon'(T)$ data can be qualitatively understood by assuming the existence of critical slowing down near a phase transition. The maximum of $\varepsilon''(T)$ was used to obtain an effective temperature dependent relaxation time $\tau(T)$. In a similar way, we propose to model the frequency dependence of the dielectric function by using a monodispersive Debye relaxation term to get the temperature dependence of the relaxation time at a single frequency $\omega$ from the ratio

$$\tau(T) = \frac{\varepsilon''(T)}{\omega[\varepsilon''(T) - \varepsilon_{\infty}]}$$  (1)

The temperature profile $\tau(T)$ deduced from Eq. 1 is not very sensitive to the exact value chosen for the high frequency dielectric constant $\varepsilon_{\infty}$, which was fixed to 2.5 [33]. The $\tau(T)$ curves deduced for both salts are shown in Fig. 2 on a log-log scale. These relaxation time curves are quite different from the data collected at lower frequencies [10]: not only are the absolute values smaller by 4 orders of magnitude at 100 K, but the temperature variation is the opposite below the CO transition. Indeed, for the H$_{12}$ salt, although $\tau(T)$ increases below 120 K, it presents a maximum around 80 K and decreases rapidly by more than one order of magnitude down to 40 K. Below, $\tau(T)$ keeps decreasing, but with a different slope. Between 80 K and the lowest temperature reached at 2 K, $\tau(T)$ has practically decreased by 3 orders of magnitude, while the low frequency data rather show an increase of the same size with only a change of slope near $T_{CO}$ [10]. A kink is observed at $T_{SP}$ followed by a small variation of the decreasing rate. If one looks at the relaxation time for the D$_{12}$ salt in Fig. 2, it shows a similar behavior from 120 to 40 K, but the maximum in $\tau(T)$, which is sharper, occurs just below $T_{CO}$. For $T < 40$ K, the decrease is less pronounced than for the H$_{12}$ salt due to an additional contribution to the dielectric function, and which will be discussed in more detail below.

FIG. 2: (Color online) Temperature dependence of the relaxation time $\tau(T)$ obtained from Eq. 1.

The discrepancy between microwave and low frequency data for the H$_{12}$ salt may be linked to the nature of the relaxor ferroelectric state and to the four orders of magnitude difference in frequency. The microwave experiment is likely to be sensitive to the spatial variation of polar order taking place at relatively short length scale, which is characterized by a short relaxation time. As we move further below $T_{CO}$ on the temperature axis, these polar fluctuations decrease in size yielding a decrease of the dielectric constant and of the corresponding losses, as depicted in Figures 1 and 3.

As regards to the increase of $T_{CO}$ by deuteration, it has been shown recently that the collective displacement of counteranions X$^-$ is directly coupled to the charge modulation along the stacks [26] and that the stabilizing potential grows not only with the size of the anion but also upon deuteration [17]. The increase of $T_{CO}$ revealed by the microwave experiment on a deuterated crystal is consistent with this picture.

Let us examine now the anomalies appearing in the SP state. We show in Fig. 3 the dielectric function of the H$_{12}$ salt below 30 K. In zero magnetic field, the real part $\varepsilon'$

FIG. 3: (Color online) Temperature dependence of the the dielectric function near $T_{SP}$ for the H$_{12}$ salt in 0 (red), 12 (blue) and 18 (green) Tesla. The black line is the $a + bT^3$ fit.
increases abruptly below 16.5 ± 0.1 K and then decreases slightly as we move sufficiently down in temperature. To the increase in \( \epsilon' \) corresponds a peak in the imaginary part \( \epsilon'' \). The latter decreases to zero with a larger slope. These anomalies are consistent with the onset of the SP long-range order at \( T_{SP} \approx 16.5 \) K \cite{17}. This is also compatible with the application of a magnetic field, which depresses \( T_{SP} \) down to 12.5 K ± 0.2 at 18 Tesla. Except for a small temperature interval where critical SP fluctuations enhance the dielectric constant, the magnetic field has no noticeable effect above \( T_{SP} \), where \( \epsilon' \) follows a perfect \( T^3 \) power law up to 30 K, as indicated by the black line extrapolated toward the very low temperature domain (top panel). The microwave absorption is very low in this temperature range and this yields larger imprecisions on \( \epsilon''(T) \) when the field is applied. Nevertheless, we found a \( T^2 \) power law with \( x \) lying in the 1.4-1.6 interval for \( T > T_{SP} \). We have subtracted these power laws extrapolated to zero to obtain the contribution of the SP state to the dielectric function at low temperatures, \( \Delta \epsilon' \) and \( \Delta \epsilon'' \), shown in Fig. 4.

The anomaly shown by \( \Delta \epsilon' \) as we dip into the SP ordered region reveals an increase of polarizability in the presence of the lattice distortion. This is compatible with a reduction of the amplitude of the CO order parameter as the SP order sets in, an effect also consistent with previous NMR and optical measurements on the cousin SP compound \((\text{TMTTF})_2\text{AsF}_6\) \cite{12, 13}. It is worth noting that the amplitude of the variation shown by \( \Delta \epsilon' \), as a result of SP ordering, is at the most 1% of the background value exhibited in Fig. 3. While the anomaly indicates that the lattice distortion does alter the charge ordered state of \((\text{TMTTF})_2\text{PF}_6\), the relative smallness of the effect may explain the difficulty for charge sensitive probes to detect any reduction of CO ordering by the SP state in this material \cite{13}.

The rapid increase of the dielectric response below \( T_{SP} \) suggests a temperature dependence of \( \Delta \epsilon' \) governed by the criticality of the SP order parameter. A log-log plot analysis of the \( \Delta \epsilon' \) data reveals indeed that the critical behavior can be reasonably fitted with a power law \( \Delta \epsilon' \propto (T_{SP} - T)^\beta \). Using the value \( T_{SP} \) obtained from the maximum slope of \( \Delta \epsilon' \), the exponent \( \beta \approx 0.36 \) is extracted (dashed line of Fig. 3). This non mean field value is consistent with the \( \beta \) expected for a SP—one-component—order parameter in three dimensions.

Considering now the magnetic field dependence, \( T_{SP} \) is found to be progressively depressed under field. The transition’s width increases due to a larger temperature interval of SP critical fluctuations. However, in contrast to other spin-Peierls systems like CuGeO\(_3\) \cite{36}, or Peierls systems \cite{37}, for which the order parameter is independent of magnetic field in the low temperature limit \cite{35}, \( \Delta \epsilon'(T \to 0) \) is here field dependent. The fact that for \((\text{TMTTF})_2\text{PF}_6\), the SP lattice distortion occurs in the CO—ferroelectric state may be responsible for the slight increase of the low temperature polarizability in the presence of a field. As lattice effects are involved in both transitions, it appears difficult, however, to predict how both types of order interact and are modified by a magnetic field.

As reported in neutron experiments \cite{17}, we also observe a reduction of the SP transition temperature upon deuteration. However, thermal relaxation effects are observed at microwave frequencies at low temperatures for the \( \text{D}_2 \) salt. By deuteration the decrease of internal pressure increases the volume of the anion cavity delimited by the methyl groups. These deuterated groups can thus move more freely and are probably the cause of the anomalous relaxation effects. The dielectric function of this salt is shown below 30 K in Fig. 5. The temperature dependence of both parts of \( \epsilon' \) is quite different from the ones shown in Fig. 3 for the \( \text{H}_2 \) salt. Although we can identify features related to the SP transition, that is a sudden slope variation on \( \epsilon' \) and a sharp peak on \( \epsilon'' \) at 13.1 K, there is clearly another mechanism contributing to the dielectric function in this temperature range. This contribution is critically dependent on time as evidenced by the two curves appearing in Fig. 4 taken respectively 2 and 24 hours after the first cooling down to 2 K. In the inset, we present the time dependence of \( \Delta \epsilon' \) at \( T = 3 \) K, just after cooling the sample from 40 K. Two
FIG. 5: (Color online) Temperature dependence of the dielectric function near $T_{\text{SP}}$ for the D$_{12}$ salt: 2 hours (red) and 24 hours (blue) waiting time at 3 K. Inset: time dependence of $\Delta\epsilon'$ at 3 K.

Time scales are clearly observed (insert of Fig. 5): a relatively fast one during the first half-hour and a very slow one for which saturation is obtained approximately after 48 hours. Similar effects with decreasing amplitude are observed up to 40 K. These thermal relaxation effects related to deuterated methyl groups prevent any analysis of the SP contribution to the dielectric function similar to the one given in Fig. 4 for the hydrogenated samples. These effects also mask the real temperature dependence of the relaxation $\tau(T)$ below 40 K in the CO state (Fig. 2). Nevertheless, our microwave data confirm the decrease of $T_{\text{SP}}$ by roughly 30% upon deuteration [17] and its magnetic field dependence $T_{\text{SP}}(H)$ could be studied up to 18 Tesla and compared with the hydrogenated salt.

The magnetic field dependence of the SP transition temperature is known to fit a quadratic variation [36, 39] in relatively low fields as predicted theoretically [28]. In the hydrogenated (TMTTF)$_2$PF$_6$ salt, NMR and magnetization measurements have indeed revealed some quadratic dependence [22], but the entire range of low field values for $T_{\text{SP}}(H)$ was not available. From our field dependent microwave data, the phase diagram including the D$_{12}$ and H$_{12}$ salts is presented in Fig. 6 and compared to the mean-field prediction (continuous and dash lines) obtained by Cross [28].

$$T_{\text{SP}}(H) = \frac{T_{\text{SP}}(0)}{1 - c \left( \frac{g\mu_B H}{4\pi T_{\text{SP}}(0)} \right)^2},$$

(2)

where the $g$-factor is fixed at 2 for (TMTTF)$_2$PF$_6$ [40]. For the H$_{12}$ salt, the fit gives $c \simeq 15.3$, and $c \simeq 12.9$ for the deuterated salt which is relatively close to the predicted value of 14.4, in comparison to other systems like CuGeO$_3$, where pronounced deviations are found [41].

IV. CONCLUSION

In (TMTTF)$_2$PF$_6$ salts, we found that both the charge ordering and spin-Peierls transitions can be studied from measurements of the microwave dielectric function. In the gigahertz frequency range, the technique is sensitive to short-range polar order, which yields a decrease of the dielectric constant below $T_{\text{CO}}$ due to a rapid reduction of these ordered regions and of their relaxation time. The microwave data confirm not only the relaxor character of the CO transition in the hydrogenated salt, but also its stabilization upon deuteration, which increases $T_{\text{CO}}$ by 33%. Contrary to systems with no CO ordering, dielectric anomalies are also observed in the SP state for both salts, with a 20% reduction of $T_{\text{SP}}$ upon deuteration. The opposite effects of deuteration on $T_{\text{SP}}$ and $T_{\text{CO}}$ are compatible with an effective negative shift on the pressure axis.

Thermal relaxation effects have been detected in the deuterated salt, which have impeded a complete analysis of the SP ordering. These SP anomalies confirm an indirect coupling between charge, lattice and spin degrees of freedom due to intricate lattice effects in a temperature range where both the SP and CO ground states coexist. A quadratic magnetic field dependence of the SP transition temperature appears to follow the mean-field prediction despite the existence of one-dimensional lattice fluctuations over a wide range of temperature in (TMTTF)$_2$PF$_6$. 
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