Electronic properties of tetrathiafulvalenium-tetracyanoquinodimethanide (TTF-TCNQ)*

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Several important experimental results on tetrathiafulvalenium-tetracyanoquinodimethanide (TTF-TCNQ) suggestive of collective many-body effects are summarized and measurements of the dc conductivity are discussed.

In response to the preceding comment by Thomas *et al.*,[1] we wish to briefly summarize several important experimental results on tetrathiafulvalenium-tetracyanoquinodimethanide (TTF-TCNQ) suggestive of collective many-body effects and to comment on the measurements of the dc conductivity.

It has long been known that a one-dimensional (1D) metal is intrinsically unstable to the formation of charge- or spin-density waves.[2-5] The chainlike stacking[6] of molecules in the TCNQ salts with the implied π-electron overlap was suggestive of highly anisotropic properties and, in a rough sense, of quasi-one-dimensionality. Understanding of experimental systems which are electronically one dimensional came with quantitative measurements of the anisotropy of the optical[7-13] and transport[14-19] properties of 1D conductors. The observation in polarized optical reflectance of a plasma edge for $\vec{E}$ parallel to the conducting axis and a flat frequency-independent reflectivity through the far ir for $\vec{E}$ perpendicular to the conducting axis implies that even though the conductivity perpendicular to the chain is finite, this transverse transport does not arise from metallic behavior; i.e., $\omega_p^{\parallel}\tau_{\parallel} > 1$ and $\omega_p^{\perp}\tau_{\perp} < 1$, where ω_p and τ are the plasma frequency and scattering time. Moreover, the measurements of the magnitude of the transverse components of the conductivity tensor yield an estimated mean free path $l_{\perp}$ of order 10^{-3} of a lattice constant, whereas in the parallel direction the mean free path as estimated from the dc or the optical conductivity near the plasma edge implies metallic propagation. Thus $l_{\parallel} > a_{\parallel}$ and $l_{\perp} << a_{\perp}$, where $a_{\parallel}$ and $a_{\perp}$ are the lattice constants parallel to and perpendicular to the principal conducting axis. The implication is that the interaction of the electrons with the lattice is sufficiently strong to localize electrons on individual chains with diffusive hopping between chains. This localization[20] converts a metal with an anisotropic band structure into an array of 1D metallic chains.

TTF-TCNQ (Ref. 21) is a one-dimensional metal at high temperatures which undergoes a metal-insulator transition[22,23] at 54 K to a high-dielectric-constant[15] nonmagnetic[22,24] ground state.[25] Recent x-ray scattering[26,27] and elastic-neutron-scattering[28] studies showed the existence below 54 K of a low-temperature incommensurate superlattice having a periodicity of $3.4b$ in the chain direction. The structural results established the existence of the charge-density-wave ground state arising from the 1D Peierls instability and determined $2k_F = 0.295b^*$. Careful studies[27-29] of the transition region from 38 to 54 K provided detailed information on the three phase transitions[30] observed on going from the ordered superlattice to the 1D conductor. In the conducting state above 54 K the x-ray results showed[26,27] 1D diffuse scattering consistent with a 1D lattice distortion or a phonon anomaly. Inelastic-neutron-scattering studies of the phonon spectrum revealed a Kohn anomaly at $0.295b^*$ at room temperature[31,32] which becomes stronger[32] with decreasing temperature. The constant value of $2k_F$ leads to a temperature-independent charge transfer[33] of 0.59 electrons from donor to acceptor. These structural studies, therefore, established the existence of the Peierls distortion and the associated giant Kohn anomaly, and provided detailed information on the temperature dependence.

The electronic properties that distinguish TTF-TCNQ from ordinary metals are revealed by studies of the temperature- and frequency-dependent conductivity and dielectric function, $\sigma_1(\omega)$ and $\epsilon_1(\omega)$, from dc through the ultraviolet. The early measurements of the four-probe dc electrical conductivity from our laboratory[23] showed a strong increase with decreasing temperature and suggested intrinsic normalized peak values at 58 K as high as 500 with an absolute value of order 10^6 $(\Omega\,\mathrm{cm})^{-1}$. Schafer *et al.*[34] suggested these early results arose in part from inherent difficulties in the measurement technique for small anisotropic samples. Detailed studies[14]

using both the conventional four-probe technique and the Montgomery configuration led to the development of an internal signature of measurement problems due to inhomogeneous current flow and showed that spurious peaks can be determined directly via structure in $\sigma^b(T)$ arising from the temperature-dependent anisotropy. This test has been verified in detail experimentally[14,35] on TTF-TCNQ, through computer models and resistance network studies, and has been used successfully by others.[36]

Extensive studies[14,37] of the b-axis dc conductivity and materials properties of TTF-TCNQ showed that the quasi-one-dimensionaltiy leads to stringent requirements on material purity, crystal perfection, quality of contacts, and special care on the part of the experimenter in handling delicately sensitive crystals. We measured the temperature dependence of the dc conductivity for over 400 TTF-TCNQ single crystals. The physical dimensions of approximately 200 of these were measured in order to determine absolute values: In the initial work, when sample perfection was not studied, and crystal-growth techniques and sample purification methods were developing, the room-temperature conductivity averaged over the first thirty samples was 325 ± 150 $(\Omega\ \mathrm{cm})^{-1}$. Most of these early crystals showed peak values between six and ten times the room-temperature value. As techniques of crystal growth, chemical preparation, and sample mounting and handling improved, the conductivity results showed a corresponding change. The average room-temperature conductivity measured on 65 samples midway in our studies was 500 ± 180 $(\Omega\ \mathrm{cm})^{-1}$; the normalized peak values ranged from slightly below 10 to above 100. The average room-temperature value for 30 samples measured in a subsequent group was 550 ± 150 $(\Omega\ \mathrm{cm})^{-1}$. In our final studies, procedures were changed to select for measurement only those crystals judged superior by optical reflection and transmission microscopy. The average room-temperature conductivity over the last 15 samples measured was 660 ± 130 $(\Omega\ \mathrm{cm})^{-1}$. The time evolution of the room-temperature data suggests an intrinsic room-temperature value of approximately 800 $(\Omega\ \mathrm{cm})^{-1}$. There is a clear correlation between the increased sample quality (due to improved materials, contacts, and sample handling procedures) and the room-temperature values. For the final 60 samples studied, the normalized peak values are listed in Table I.

We note that it is not a question of a few special samples; over 30% of the samples listed in Table I showed peak values greater than 20 times $\sigma^b(300\ \mathrm{K})$. Therefore, both the absolute value (discussed above) and the peak distribution (Table I) are higher than observed in other laboratories[1] [data from other groups yield average room-temperature values from 300 to 500 $(\Omega\ \mathrm{cm})^{-1}$ and correspondingly low peak values].

TABLE I. Normalized peak values of the b-axis dc conductivity for the last 60 samples measured.

$\sigma^b(58\ \mathrm{K})/\sigma^b(300\ \mathrm{K})$	Number of samples
10–15	15
15–20	26
20–25	9
25–30	3
30–100	4
100–150	3

Our experimental results for pure samples[14] show that the maximum conductivity occurs at 58 K, whereas less-pure samples[37] yield lower peak values with maximum conductivity occurring at higher temperatures.[38] Thus, impurities lead to resistive effects and thereby limit the intrinsic conductivity.

These extensive dc studies lead to the following conclusions: (i) peak normalized values of order 100 with absolute magnitude approaching 10^5 $(\Omega\ \mathrm{cm})^{-1}$ are intrinsic; (ii) crystal purity and perfection are of crucial importance; (iii) "typical" peak normalized values of 15 or less are a consequence of materials preparation and/or sample handling.

Contactless microwave measurements[15,39] of the b-axis conductivity are in good agreement with the dc results. These microwave-loss measurements were analyzed using closed-form analytical solutions for highly anisotropic metals in the surface-impedance regime. The measurement technique and analytical solutions have been tested by measuring the temperature-dependent anisotropic conductivity of graphite ($\sigma_\parallel/\sigma_\perp > 10^3$) and $2H-TaSe_2$ ($\sigma_\parallel/\sigma_\perp \gtrsim 10$) with results in agreement with the reported dc values.[40] The microwave results[39] on TTF-TCNQ show 8 of 16 carefully chosen crystals exhibiting $\sigma^b(58\ \mathrm{K})/\sigma^b(300\ \mathrm{K})$ values from 25 to 80 and the temperature dependence from sample to sample showing a series of nested curves quantitatively similar to the dc results. The extreme sensitivity to crystal perfection, purity, and handling observed in the dc experiments were even more obvious in the microwave studies; for example, exposure of a sample to laboratory atmosphere for just a few hours was sufficient to decrease $\sigma^b(58\ \mathrm{K})/\sigma^b(300\ \mathrm{K})$ by a factor of 2.

Given the demonstrated sensitivity of the dc and

microwave transport to purity and crystal perfection one might ask if the intrinsic peak conductivity is even higher than 10^5 $(\Omega\,\mathrm{cm})^{-1}$. Our opinion is that this is not the case. The absence of higher measured values coupled with the estimated oscillator strength from ir studies[10] suggests an upper limit for $\sigma^b(58\ \mathrm{K})$ of about 10^5 $(\Omega\,\mathrm{cm})^{-1}$.

The initial suggestion[23] of viewing the behavior of TTF-TCNQ in terms of collective many-body effects associated with the soft-phonon Peierls instability[2-5] was based principally on the relatively large and strongly temperature-dependent dc conductivity in the context of earlier studies[41,42] of metal-insulator transitions in other quasi-1D TCNQ systems. The existence of high metal-like conductivities in quasi-1D systems as evidence for many-body effects remains under continued active study. For example, Bychkov,[43] Berezinsky,[44] and Gogolin, Mel'nikov, and Rashba[45] have shown that for independent electrons in a strictly 1D system containing impurities or defects, the conductivity should decrease as the temperature is lowered, approaching zero with a temperature dependence determined by the ratio of the impurity and phonon scattering rates, $\tau_{\mathrm{imp}}/\tau_{\mathrm{ph}}(T)$. Large conductivity peaks with normalized values of 50–100 and absolute values approaching 10^5 $(\Omega\,\mathrm{cm})^{-1}$ are therefore difficult to understand on the basis of single-particle scattering, yet are insufficient to demand by themselves an explanation in terms of collective effects since σ_{dc} represents only the zero-frequency limit of $\sigma_1(\omega)$. Data over the full frequency range as obtained from dc, microwave, ir, and optical studies reveal that there is an energy gap and an extremely narrow zero-frequency mode in $\sigma_1(\omega)$. These results, together with the structural studies summarized above, show that TTF-TCNQ is not a simple metal, and that collective effects are dominant features of the electronic properties.

In the conducting regime above 58 K there is an energy gap[9-11] ($E_g \simeq 0.14$ eV) with a small residual conductivity in the gap region.[9-11,46] The results of recent far-infrared studies[46] of polarized reflectance from single-crystal mosaics of TTF-TCNQ at 100 K are shown in Fig. 1. The dc and microwave data are included with uncertainty in absolute value obtained from the discussion given above; $\sigma^b(300\ \mathrm{K}) = 700 \pm 100$ $(\Omega\,\mathrm{cm})^{-1}$ and $\sigma(100\ \mathrm{K})/\sigma(300\ \mathrm{K}) = 10 \pm 1$. The solid curve is from a best fit to the $\vec{E} \parallel \vec{b}$ reflectance using a single Lorentzian oscillator centered at zero frequency and a residual conductivity in the far ir of 50 $(\Omega\,\mathrm{cm})^{-1}$.[46]

The full frequency dependence[7-11] of $\sigma_1^b(\omega)$ (Fig. 1) shows a maximum at 1000 cm^{-1} with a smooth Drude-like frequency dependence observed at higher frequencies. The corresponding behavior[7-11] of $\epsilon_1^b(\omega)$ (Fig. 1) is like that of a semiconductor with transitions across the energy gap sufficiently strong to give negative values between 1000 and 6000 cm^{-1}. At lower frequencies ϵ_1^b is positive with magnitude of approximately 100, consistent with the measured oscillator strength across the gap (0.14 eV) as obtained from the plasma frequency (1.2 eV); $\epsilon \simeq 1 + \omega_p^2/\omega_g^2 \simeq 100$. The large zero-frequency conductivity drives ϵ_1^b negative in the far ir.

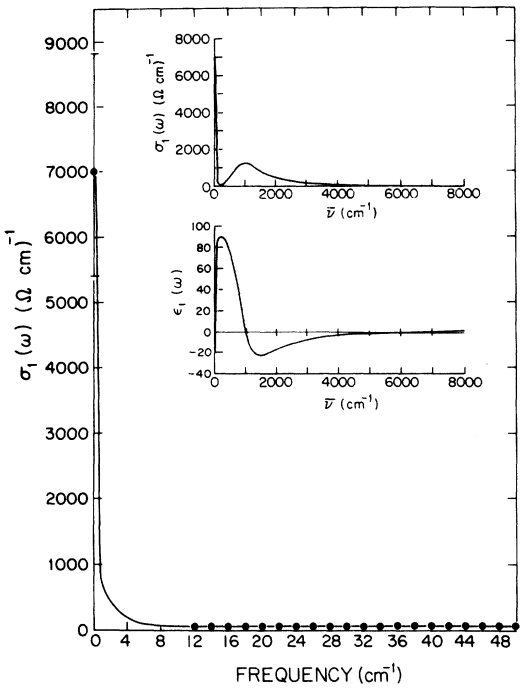

FIG. 1. Frequency dependence of $\sigma_1(\omega)$ below 50 cm^{-1} at 100 K. Insert: Sketch of full frequency dependence of $\sigma_1(\omega)$ and $\epsilon_1(\omega)$ at 100 K (see Refs. 9–11 and 46).

The energy gap remains as the dominant feature in $\sigma_1(\omega)$ and $\epsilon_1(\omega)$ in the insulating state at 4.2 K.[7-11,46] However, measurements of the b-axis reflectance in the gap region[46] below 50 cm^{-1} reveal an optical mode centered at approximately 2 cm^{-1}, suggesting that the oscillator strength responsible for the conductivity above 54 K is shifted to finite frequency in the insulating state.

Low-temperature microwave measurements[15,47-49] (10 GHz) yield the unusually large value of $\epsilon_1^b = 3500$ much greater than the single-particle contribution observed in the ir due to virtual excitations across the energy gap. Thus, the low-temperature dielectric constant is indicative of additional oscillator strength at very low frequencies and is quantitively consistent[46] with the low-temperature far-ir observations described above.

The experimental behavior of TTF-TCNQ is currently being pictured in terms of the phenomenological model of a Peierls-Fröhlich condensation[50-52] of the conduction electrons with a high mean field scale temperature.[9-11,24,51] In this model, the electronic properties are dominated by charge-density-wave fluctuations associated with the Peierls instability in which a phonon mode is driven soft[53] by the divergent response function of the 1D electron gas at $q = 2k_F$. The Peierls instability thus would lead to the metal-insulator transition near 54 K and the periodic superlattice distortion in the low-temperature semiconducting state. Above 54 K, in the fluctuation regime, the observed energy gap (0.14 eV) is identified as the Peierls gap with the condensate moving to contribute to the high dc and microwave conductivity. Below 54 K, the condensate is pinned[51,54] by high-order commensurability, interchain coupling or defects leading to the observed incommensurate superlattice, and to a shift of the collective-mode oscillator strength into the ir with an associated conversion of the dc conductivity into a large low-frequency dielectric constant of the form[51]

$$\epsilon_1 = 1 + \frac{2}{3}\,\frac{\omega_p^2}{(2\Delta)^2} + \frac{\Omega_p^2}{\omega_{pin}^2}.$$

The middle term arises from single-particle oscillator strength ω_p^2 associated with the Peierls energy gap 2Δ; the final term is the contribution from the pinned Fröhlich mode with oscillator strength Ω^2 and characteristic pinning frequency ω_{pin}.

The observation of the incommensurate superlattice[26-28] is of fundamental importance, for it implies that the phase of the distortion is arbitrary and only becomes fixed by higher-order effects or the relatively weak interchain coupling. As a result, so long as[55,56]

$$k_B T > [\xi(T)/b]V_0(\varphi),$$

where $\xi(T)$ is the longitudinal coherence length, b is the lattice constant, and $V_0(\varphi)$ is the amplitude of the periodic pinning potential, the fluctuating charge-density wave can move to form a current-carrying state. On the other hand, the effect of impurities as potential pinning centers has been discussed[57] with the suggestion that in the presence of such centers the fluctuations will be resistive.[58] This important theoretical question has not yet been settled. Experimentally,[14,37] however, impurities do give rise to resistive fluctuations leading to a lower peak conductivity occurring at a higher temperature.

Owing to the lack of long-range order and the presence of intrinsic dissipative processes, persistent currents are not expected, and thus a Peierls-Frölich condensate is not a superconductor. However, the extremely weak pinning forces (ω_{pin} of order a few wave numbers) observed in TTF-TCNQ and the corresponding weak interchain coupling suggest the possibility that under proper conditions the pinning might be suppressed leading to a wider fluctuation range and higher conductivity.

A broken symmetry conduction mechanism arising from a (nearly) phase-independent charge-density wave would represent the only example yet found in nature of collective electron transport which is different from pairing superconductivity. The experimental evidence for collective transport in these 1D materials is accumulating and is a principal reason for their interest. Continued experimental studies of TTF-TCNQ and related derivatives are in progress to further test the generality of these ideas and provide a firm experimental basis for the development of the microscopic mechanism.

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