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Infrared-active vibrational modes of heavily doped "metallic" polyacetylene

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A direct comparison of the infrared-active vibrational (IRAV) modes in heavily doped, lightly doped, and photoexcited *trans*-$(\mathrm{CH})_x$ is presented. The infrared absorption spectrum of heavily doped *trans*-$(\mathrm{CH})_x$ exhibits doping-induced IRAV modes and an onset of electronic absorption which are substantially red shifted with respect to the corresponding absorptions observed at light doping levels and which have an unusually strong temperature dependence. Analysis of the data leads to the following conclusions: For heavily doped *trans*-$(\mathrm{CH})_x$. *all* the doping-induced charges are involved in the generation of the IRAV modes in the "metallic" state; the pinning of the π-electron charges which cause the IRAV modes has virtually disappeared; and there is an energy gap (or pseudogap) in the excitation spectrum with magnitude $\sim 1500\ \mathrm{cm}^{-1}$, or ~ 0.2 eV. Thus, although many of the most fundamental issues associated with metallic polyacetylene at high doping levels remain unresolved, the existence of a pseudogap and IRAV modes demonstrate that it is not a simple metal.

I. INTRODUCTION

As a result of the bond alternation caused by the Peierls distortion, *trans*-$(\mathrm{CH})_x$ is a semiconductor with a twofold-degenerate ground state. This degeneracy allows for the possibility of nonlinear excitations in the form of solitonlike bond-alternation domain walls, each with an associated electronic state at the center of the energy gap. Since the energy for creation of such excitations is less than that required for electrons and holes within a rigid structure, solitons are expected to be formed when charges are added to *trans*-$(\mathrm{CH})_x$ via charge-transfer doping or photoexcitation. The creation of spinless charged solitons and the associated self-localized gap states in polyacetylene have been confirmed through a variety of detailed experimental studies.[1]

The intense infrared activity induced by charged solitons is due to the localized phonon modes caused by local deviations from the symmetry of the perfectly dimerized pattern in the vicinity of the structural "kinks."[1] The infrared-active localized phonons associated with charged solitons arise from the the symmetrical vibrational modes (Raman-active modes) and are red shifted with respect to these Raman modes.[1,2] Three principal localized infrared-active vibrational (IRAV) modes associated with the uniform translation of charged solitons are observed in *trans*-$(\mathrm{CH})_x$; one for each of the Raman-active modes which are coupled to the π electrons[1] (weaker modes arising from coupling to nonuniform translations of the charged kinks have also been predicted[3] and observed[4]). The oscillator strength of these modes increases linearly with the doping concentration.[5] The existence of the IRAV modes and the excellent agreement of the experimental results with theoretical predictions has provided detailed confirmation of the importance of the electron-phonon coupling in polyacetylene.[1,4]

At intermediate doping concentrations (a few percent), *trans*-$(\mathrm{CH})_x$ shows a moderate electrical conductivity, characteristic midgap states, and IRAV modes, but no significant Pauli magnetic susceptibility; the combination suggests a significant contribution to electrical transport by mobile charged solitons.[1,6(a)] As the doping level increases beyond the critical concentration ($\sim 5-6\%$), there is an abrupt onset of a temperature-independent spin susceptibility which has been interpreted as the Pauli magnetic susceptibility of a normal metal.[6] This interpretation would imply the closing of the Peierls energy gap and the disappearance of bond alternation. However, the characteristic IRAV modes are known to persist into the "metallic" regime with enhanced oscillator strength that continues to grow linearly with doping concentration.[5]

As far as we know, the existence of enhanced IRAV modes, which are red shifted with respect to the corresponding Raman modes in a system such as polyacetylene, requires either the existence of localized phonons associated with the coupling of nonuniform charge distributions to the lattice[1] (as in the case of dilute solitons) or coupling of the uniform phase motion of an incommensurate charge-density wave to the symmetric Raman modes[7] (the latter can be viewed as a coherent translation of a dense soliton or polaron lattice). Of these, only the polaron lattice would appear to be consistent with a "metallic" state which exhibits a temperature-independent susceptibility and an electrical conductivity that approaches that of copper.[8] However, for the polaron lattice, the calculated IRAV-mode intensities are too weak by many orders of magnitude in comparison with the experimental results. Thus, the origin of the enhanced IRAV-mode intensities in heavily doped polyacetylene remains an important unresolved problem.

In this paper, we report the results of a series of experimental studies which concentrate on the infrared activity of heavily doped *trans*-$(\mathrm{CH})_x$. The infrared absorption spectrum exhibits doping-induced IRAV modes which are substantially red shifted with respect to the corresponding absorptions observed at light doping levels and which have an unusually strong temperature dependence. A direct comparison of the IRAV modes in heavily doped, lightly doped, and photoexcited *trans*-$(CH)_x$ is presented. This comparison leads to the conclusion that in heavily doped *trans*-$(CH)_x$, the pinning parameter associated with the IRAV-inducing charges is extremely weak (comparable to that for photoexcitations), and that the pinning becomes even weaker at lower temperatures. The weak pinning of charges on the polymer backbone in the metallic regime indicates a reduction of the Coulomb binding energy to the counterions; the IRAV-inducing charges appear to be essentially free for translational motion. Finally, we have reexamined the photoinduced absorption previously reported for heavily doped *trans*-$(CH)_x$. Our conclusion is that there is no genuine photoinduced signal; the spectral changes observed during laser illumination are entirely due to heating.

II. EXPERIMENT

A thin film (~2000 Å) of polyacetylene was polymerized directly on a KBr substrate using the traditional Shirakawa method. The as-made film was then isomerized at 180 °C for 40 min under high vacuum ($<5\times10^{-6}$ Torr). No *cis*-$(CH)_x$ component was detected in the infrared spectrum after the isomerization. Chemical doping was carried out by exposing the thin film to iodine vapor for varying periods of time. At each level of doping, the iodine doped sample was pumped under high vacuum for about an hour (to ensure equilibrium) before recording the spectra. The doping concentration was monitored by careful measurement of the IRAV frequencies and by comparison of the observed frequencies with the data reported by Ehrenfreund *et al.*[9] The doping level for the final measurements was estimated to be approximately 6%, just beyond that required for the sharp onset of the red shift of the 1400-cm^{-1} mode.[9]

Infrared absorption spectra were obtained with an IBM (Bruker) Fourier transform interferometer by direct measurement of infrared transmittance of the chemically doped *trans*-$(CH)_x$ thin film. The spectral resolution was set to 4 cm^{-1}. The photoinduced absorption measurement was carried out by using the standard 10-s difference technique[10] in conjunction with an optical pumping by using an Ar^+-ion laser for both heavily doped and pristine *trans*-$(CH)_x$. The temperature of the sample was controlled by mounting a sample on a liquid-nitrogen-cooled cold finger.

III. RESULTS

At dilute doping concentrations, the three characteristic IRAV modes[1] at 930, 1288, and 1400 cm^{-1} were found along with an increasing absorption at higher frequencies due to the electronic transition to the soliton midgap level located at ~0.7 eV. After doping to saturation into the metallic regime, each of the doping-induced IRAV modes was red shifted (at room temperature) to 559, 1284, and 1373 cm^{-1}, respectively, and the broad electronic absorption peaked at ~0.55 eV (~4500 cm^{-1}) at room temperature. Upon cooling to 80 K the IRAV modes were red shifted still further, to 487, 1280, and 1362 cm^{-1} (see Fig. 1).

In the heavily doped sample, the oscillator strength of each of the IRAV modes and that of the electronic absorption increased significantly upon cooling to 80 K, as shown in Fig. 1. Because the sum rule of the total conductivity has to be satisfied, this unexpected temperature sensitivity of the oscillator strength must come either from a reduction of the dc conductivity at low temperatures, or it must imply a genuine reduction of the dynamic mass. Note that in Fig. 1, the absorption strengths of the IRAV modes are comparable to that of the electronic absorption (although the oscillator strengths in the IRAV modes are, of course, much less than that in the electronic transition).

Figure 2 shows a direct comparison of the infrared absorption spectrum of the heavily doped sample (at 80 K) with that of the photoinduced absorption spectrum of pristine *trans*-$(CH)_x$ (at 80 K). The photoinduced absorption spectrum was obtained from the same sample used subsequently for the doping study. The sample was pumped with the 2.7-eV (456 nm) Ar^+ laser line at 50 mW/cm^2; signal averaging over a 12-h period was required to obtain the excellent signal-to-noise ratio. The photoinduced absorption spectrum is characteristic of that reported earlier[1,4]; three photoinduced IRAV modes were found at 518, 1280, and 1364 cm^{-1}, respectively, with an associated electronic absorption which onsets at ~0.2 eV and peaks at ~0.45 eV. The two absorption spectra in Fig. 2 are essentially identical except that the spectral features (especially the electronic absorption) of the heavily doped sample are somewhat broader. This broadening in the doped sample is not surprising; it can be attributed to distribution in local site energies arising from the counterions.

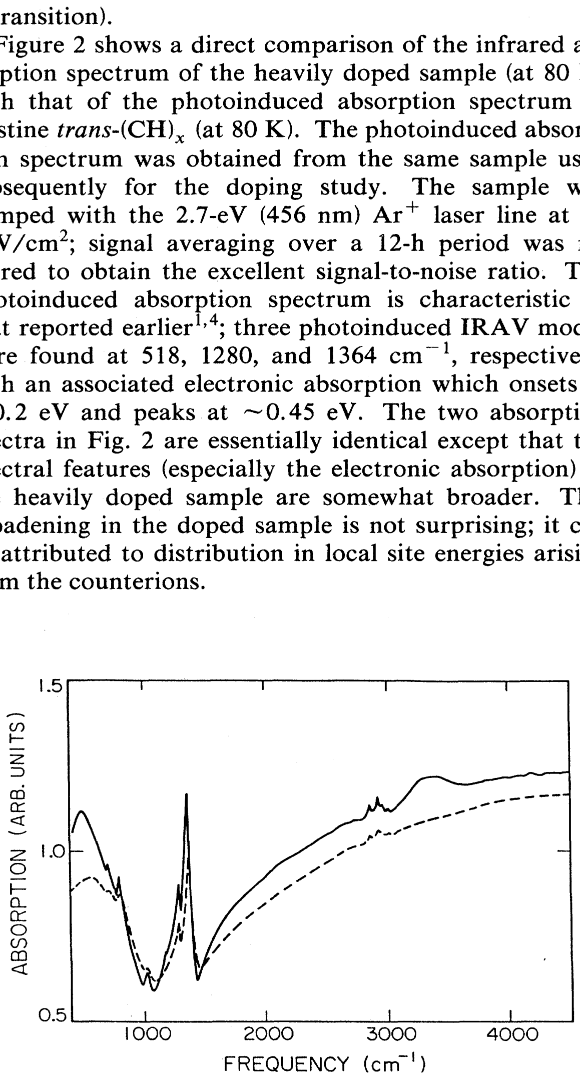

FIG. 1. The absorption spectrum of *trans*-$(CH)_x$ heavily doped with iodine. The solid curve was obtained with the sample at 80 K; the dashed curve was obtained with the sample at 300 K. The doping level was estimated to be approximately 6%, just beyond that required for the sharp onset of the red shift of the 1400-cm^{-1} mode.

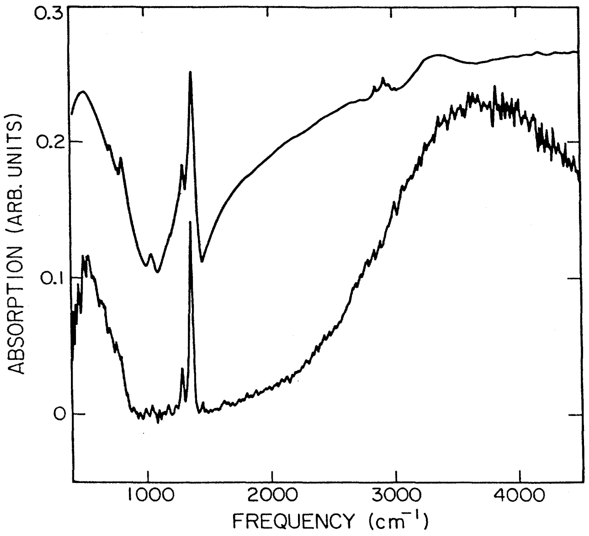

FIG. 2. A direct comparison of the infrared absorption spectrum of the heavily doped sample (at 80 K) with that of the photoinduced absorption spectrum of pristine *trans*-$(CH)_x$ (at 80 K).

IV. DISCUSSION

A. Photoinduced absorption of heavily doped polyacetylene

Our attempt to identify the nature of charged excitations in heavily doped *trans*-$(CH)_x$ via the photoconversion process discussed by Ehrenfreund *et al.*[9] failed because of the extreme temperature sensitivity of the infrared spectrum (see Fig. 1). As shown in Fig. 3, the thermal difference spectrum is essentially identical with the spectral changes which occur during laser illumination; the lower plot shows the normalized difference spectrum $(\alpha_{80\ K}-\alpha_{300\ K})/\alpha_{80\ K}$ and the upper spectrum is the illumination-induced spectral change of heavily doped *trans*-$(CH)_x$ at 80 K. Our conclusion from Fig. 3 is that there is no genuine photoinduced signal in heavily doped polyacetylene. The spectral changes observed during laser illumination are entirely due to sample heating and arise from the temperature sensitivity demonstrated in Fig. 1. We conclude, therefore, that the report[9] of photoinduced absorption in heavily doped polyacetylene is incorrect.

B. Comparison of the spectra

The absorption spectrum for heavily doped polyacetylene shown in Figs. 1 and 3 is not consistent with that expected for a simple metal in which the Peierls energy gap has closed as a result of the disappearance of bond alternation.

(i) The IRAV modes are essentially pure absorption; there are no Fano-like interference effects[11] which are indicative of the presence of a large continuum background absorption.

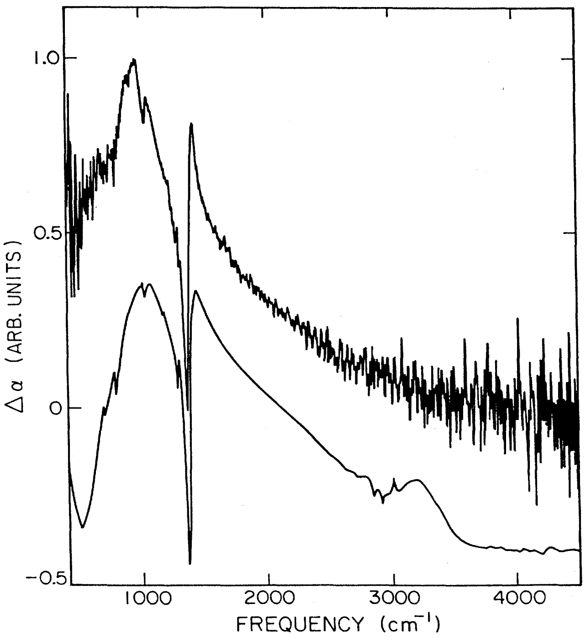

FIG. 3. The thermal difference spectrum is compared with the spectral changes which occur during laser illumination; the lower plot shows the normalized difference spectrum, $(\alpha_{80\ K}-\alpha_{300\ K})/\alpha_{80\ K}$ and the upper spectrum is the illumination-induced spectral change of heavily doped *trans*-$(CH)_x$ at 80 K.

(ii) There is no sign of the free-carrier Drude absorption expected for a metal even at frequencies as low as 450 cm^{-1}.

Although the free-carrier contribution which extrapolates to the measured $\sigma(0)$ must be in the far infrared below 450 cm^{-1}, most of the π-electron oscillator strength remains in the broad absorption band above 0.2 eV. We conclude that there is a gap (or a pseudogap where α is small but nonzero) in the π-electron excitation spectrum with magnitude of about 1500 cm^{-1} (≈ 0.2 eV). The existence of pseudogap in the excitation spectrum was also inferred earlier from analysis of the reflectance of heavily doped polyacetylene.[5(b)]

Figure 2 shows the remarkable similarity between the doping-induced absorption found with heavily doped *trans*-$(CH)_x$, and the photoinduced absorption observed in pristine samples. Not only are the same IRAV spectral features observed, they have almost identical frequencies.

The amplitude mode formalism due to Horvitz[2] has been remarkably successful in explaining the one-to-one correspondence between photoinduced and doping-induced IRAV modes (at dilute doping concentrations) and in relating them to the resonant Raman modes in conducting polymers. In this respect, polyacetylene has been a model compound; the IRAV modes are accurately described by the amplitude mode formalism through solutions to the following equation:

$$\sum_{n=1}^{3} \frac{\lambda_n}{\lambda} \left[\frac{(\omega_n^0)^2}{(\omega)^2-(\omega_n^0)^2} \right] = -\frac{1}{1-\alpha_p} , \qquad (1)$$

where λ_n are electron-phonon coupling constants associated with each of the resonant Raman modes with $\lambda = \sum_{n=1}^{3} \lambda_n$ and ω_n^0 are bare-phonon frequencies. Both λ_n and ω_n^0 are adjustable parameters. The phenomenological pinning parameter α_p directly measures the degree of pinning of the charged excitations (solitons, polarons, and bipolarons) in the system.

At dilute doping levels, each soliton is bound to a counterion by the attractive Coulomb potential. As a result, the charged solitons are expected to be pinned (to the counterions). Since there are no pinning centers in the pristine sample (except for impurities and chain defects introduced during the sample preparation process), one anticipates that the pinning parameter will be close to zero for photogenerated charged solitons. This is in qualitative agreement with the experimental results. At dilute doping concentrations, an excellent fit was obtained for all doping-induced IRAV modes with $\alpha_p = 0.238$, consistent with relatively strong pinning. For the photoinduced IRAV modes in the pristine sample, an excellent fit was obtained with $\alpha_p = 0.079$, consistent with weak pinning of the photoinduced solitons. These fits yielded the three bare frequencies ($\omega_1^0 = 1230$, $\omega_2^0 = 1310$, and $\omega_3^0 = 2040$ cm^{-1}) previously found for *trans*-(CH)$_x$ from the resonant Raman (ω_n^R) scattering data[2] ($\omega_1^R = 1065$, $\omega_2^R = 1291$, and $\omega_3^R = 1460$ cm^{-1}) and the electron-phonon coupling constants $\lambda_1/\lambda = 0.090$, $\lambda_2/\lambda = 0.017$, and $\lambda_3/\lambda = 0.893$. The latter are slightly different from those obtained in the original work[2] ($\lambda_1/\lambda = 0.07$, $\lambda_2/\lambda = 0.02$, and $\lambda_3/\lambda = 0.91$). The finite pinning parameter for photogenerated charged solitons ($\alpha_p = 0.079$) is not understood. Although it may be due to impurities or defects in the polymer, this seems unlikely since the same pinning parameter is obtained from samples from different laboratories and with significant differences in sample preparation techniques. Thus, a pinning parameter of $\alpha_p = 0.079$ appears to be characteristic of free charges, i.e., of solitons which can exhibit essentially free translational motion along the polyacetylene chains.

Although the amplitude mode formalism can be consistently applied only at dilute concentrations, the similarity of the two spectra shown in Fig. 2 suggests that the analysis should be extended in a phenomenological manner to the spectrum obtained in the heavily doped regime. Detailed analysis of the data using Eq. (1) yields $\alpha_p = 0.129$ at room temperature and $\alpha_p = 0.071$ at 80 K for heavily doped samples in the "metallic" regime. We have attributed the temperature dependence to that of the pinning parameter (rather than, for example, to the ω_n^0) since a single pinning parameter provides a good fit at both temperatures. It is significant that the pinning has weakened dramatically in "metallic" *trans*-(CH)$_x$ to a value even smaller than that appropriate to the photogenerated charged solitons.

The temperature dependence of the pinning strength α_p of heavily doped *trans*-(CH)$_x$ is surprising. Moreover, there is an increase in the oscillator strengths as the temperature is lowered. Both of these changes would seem to be in contradiction with the experimentally observed decrease in the dc conductivity as the temperature is lowered. However, transport measurements of even the best polyacetylene samples[12,13] show a very weak temperature dependence, suggesting that the macroscopic transport phenomena are still dominated by the sample morphology.

One might expect that even in the case of a simple metal (with no energy gap and no bond alternation) the Coulomb potentials from the counterions in doped *trans*-(CH)$_x$ would lead to nonuniform charge density and thereby allow the Raman modes to become infrared (ir) active. However, the observed IRAV modes are not at the resonant Raman frequencies, ω_n^R, but red shifted from them; for the three modes this red shift is $(\omega_1^R - \omega_1) \approx 580$ cm^{-1}, $(\omega_2^R - \omega_2) \approx 10$ cm^{-1}, and $(\omega_3^0 - \omega_3) \approx 100$ cm^{-1}. Thus, in the case of a simple metal, the large red shift of all three IRAV modes with respect to the Raman modes in a manner which can be described in terms of a single extremely weak pinning parameter would not be expected to result from the counterion Coulomb potentials.

The increase in ir oscillator strength at low temperatures shown in Fig. 1 suggests a corresponding reduction in the dc conductivity (in agreement with experiment[14]), since the sum rule of the total conductivity must be satisfied. Alternatively, a genuine reduction in the dynamic mass would also cause an increase in the oscillator strength of IRAV modes. Since even the lowest-frequency IRAV mode is at an energy much greater than $k_B T$, we feel that the latter is unlikely.

V. CONCLUSION

Is "metallic" polyacetylene a metal? The "metallic" regime is characterized by the following three important aspects of the data.

(1) The IRAV modes are absorption bands; there is no indication of Fano-like antiresonances studied in the one-dimensional charge-transfer salts.[11] In addition, since there is no indication of free-carrier Drude absorption, the intraband contribution to $\sigma(\omega)$ must be in the far infrared[15] below 450 cm^{-1}. Therefore, there is a gap in the excitation spectrum (or a pseudogap where α is small but nonzero) with magnitude of about 1500 cm^{-1} (≈ 0.2 eV).

(2) The intensities of the IRAV modes increase linearly with the dopant concentration with essentially the same slope as observed at more dilute concentrations.[5] This implies that *all* the doping-induced charges are involved and that the IRAV in the "metallic" state are not due to a small number of residual inhomogeneities (or nonuniformities) in the charge distribution.

(3) The pinning of the π-electron charges which cause the IRAV [i.e., *all* the charges, see (2)] has virtually disappeared.

These three conclusions are not consistent with the excitation spectrum of the simple metal which would result if the Peierls gap had been reduced to zero (there would be no gap and no IRAV modes). The existence of a pseudogap and demonstration of extremely weak pinning of a charged dense soliton or polaron lattice are particularly important in the context of recent results[8,14] which indicate that the dc electrical conductivity reaches metallic levels with values approaching that of copper in the best samples.

The observed weak pinning of the doping-induced charges in "metallic" *trans*-$(CH)_x$ can be understood as having its origin in the self-consistent screening of the Coulomb potential of the dopant counterion by the high density of mobile charges. However, although the value inferred from analysis of Fig. 1 is extremely weak, it is not zero (as would be expected for a sliding charge-density wave). We note in this context that in the case of a dense soliton (or polaron) lattice, the Goldstone mode would be the $q \rightarrow 0$ limit of an entire spectrum of acoustic modes. For a *charged*-soliton (or polaron) lattice, these modes would be shifted to a finite frequency by the long-range Coulomb interaction. Thus, the finite frequency for the lowest IRAV mode may result from the Coulomb interaction in a charged-soliton (or polaron) lattice, rather than being an indication of finite pinning.

As noted above, the free-carrier contribution which extrapolates to the measured $\sigma(0)$ must be in the far infrared below 450 cm^{-1}.[15] Nevertheless, most of the π-electron oscillator strength remains in the broad absorption band above 0.2 eV. The overall curve for $\sigma(\omega)$ is similar to that for the sliding incommensurate charge-density wave in tetrathiafulvalene-tetracyanoquinodimethane (TTF-TCNQ) first proposed on the basis of ir measurements on polycrystalline films[16] and subsequently verified through single-crystal reflection experiments.[17]

Based on these facts alone, one is led to consider the possibility that "metallic" polyacetylene is, perhaps, not a metal but a small-gap incommensurate charge-density-wave system and that the electrical transport in "metallic" *trans*-$(CH)_x$ may involve a coherent transport mechanism (e.g., a sliding charge-density wave, etc). The small value of the pseudogap in the excitation spectrum ($\sim$0.2 eV) is consistent with that expected for an incommensurate charge-density wave[1,18] driven away from the twofold-commensurate state of the neutral polymer by charge-transfer doping.

The speculation that heavily doped *trans*-$(CH)_x$ might be a sliding dense lattice of charged solitons would have to be somehow made consistent with the clear onset of a temperature-independent spin susceptibility observed at doping concentrations above about 6 mol % for both *n*-type and *p*-type doped samples.[6] A possible resolution of this apparent contradiction would be that an incommensurate spin-density wave is involved (rather than a charge-density wave), since in such a case there would be a temperature-independent spin susceptibility (analogous to that of an antiferromagnet). However, one would expect a much weaker coupling to the lattice in the case of a spin-density wave. Thus, the very intense and strongly red-shifted IRAV modes are not consistent with a spin-density-wave mechanism.

An alternative which appears to be in agreement with the essential experimental facts is the polaron-lattice model.[12] The polaron lattice is certainly consistent with the observed susceptibility. In the case of a polaron lattice,[12] the IRAV modes would be red shifted from the Raman modes provided that the pinning is weak. Although the intensity of the IRAV modes was calculated to be much too small, this calculation ignored the effect of the counterions;[19] the counterion Coulomb potentials may lead to sufficient nonuniformity in the charge density to yield the observed IRAV mode intensities.[13] For the polaron lattice, $\sigma(\omega)$ would have two contributions with a "gap" in between: (i) a free-carrier contribution corresponding to the mobile carriers in the lattice of polaron-like distortions; (ii) an interband contribution; for hole polarons the transition is from the filled π band to the Fermi level in the lower polaron band; for electron polarons, the transition is from the Fermi level in the upper polaron band to the empty π^* band. Since the data imply that only a small fraction (of order the doping fraction, *y*) of the π-electron oscillator strength is involved in (i), the free-carrier scattering lifetime τ would have to be extremely long.[20] For a dc conductivity of 10^5 S/cm, the scattering time for the carriers in the polaron lattice at room temperature would have to be $\tau \sim 2 \times 10^{-13}$ s. Although the intrinsic values (due to phonon scattering) may be even greater than this,[20] such extremely long scattering times for a partially crystalline material with a structural coherence length of order 100 Å would be quite remarkable.

In summary, despite a decade of research, many of the most fundamental issues associated with "metallic" polyacetylene at high doping levels remain unresolved. The existence of a pseudogap and IRAV modes demonstrate, however, that it is not a simple metal.

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