

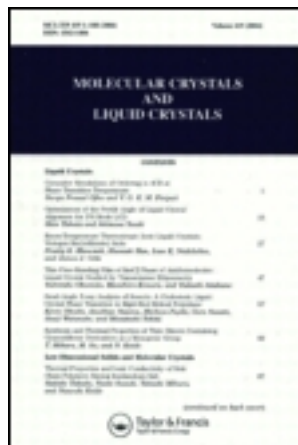
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Spin and charge excitations in polyacetylene

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SPIN AND CHARGE EXCITATIONS IN POLYACETYLENE

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Spin and charge excitations in pristine and AsF_5 -doped polyacetylene have been investigated to resolve the issue of whether or not topological solitons have been observed. We present experimental results for both materials suggesting that the simple model from which the soliton-based interpretations have been derived is inapplicable. In pristine *trans*-polyacetylene, both protonated and deuterated, conclusions are deduced from spin-echo and continuous-wave EPR about the spin diffusion rate, its temperature dependence and anisotropy that contradict predictions of the soliton theory. In AsF_5 -doped *cis*-polyacetylene, we conclude from magnetic and transport measurements that the doping is very inhomogeneous, the doped and undoped parts being largely distinct with the doped regions metallic. The conductivity changes previously interpreted as arising from a transition within the doped region are attributed to a change in the topology of these regions, *i. e.* the crossing of a percolation threshold to a regime in which the metallic regions are no longer isolated. Other experiments for and against the soliton interpretation are also discussed.

1. INTRODUCTION

Polyacetylene has recently attracted considerable interest because of evidence for the presence of two novel kinds of quasi-particles: mobile deformations resembling kink solitons and carrying charge 0 and spin $1/2$, proposed to account for the magnetic properties of pristine *trans*-polyacetylene,¹ and similar deformations carrying charge 1 and spin 0, proposed to account for the electrical and magnetic properties of doped polyacetylene.^{2,3} These solitons are compelling predictions of a model^{4,5} that assumes long uninterrupted polymeric chains and, in the case of the doped material, homogeneously distributed dopant ions. However, the actual morphology (a tangle of fibrils each consisting of a sequence of overlapping platelets)⁶ and the presumably complex chain arrangements within a platelet (especially after isomerization from the *cis* isomer, prepared originally, to the *trans* isomer, in which the kink solitons are argued to occur) raise serious doubts about the applicability of the simple model.

To see, in each case, if kink solitons are the source of the observed properties, we have performed a variety of experiments to test crucial predictions of the soliton model. In section 2, devoted to the pristine material, we summarize three kinds of evidence on the spin dynamics derived from spin-echo and continuous-wave EPR experiments and find them in direct contradiction with the predictions of the soliton theory.⁷ We do not, however, try to explain these results in terms of another model. In section 3, we summarize a number of properties of AsF₅-doped (CH)_x, especially our results for certain magnetic and transport properties, and show them to be more compatible with the aggregation of dopant ions to form soliton-free metallic regions rather than with a homogeneously doped material in which soliton excitations play a key role.⁸

2. PRISTINE POLYACETYLENE

In this part we shall present spin-echo and continuous-wave EPR measurements on *trans*-(CH)_x and its deuterated analog *trans*-(CD)_x. However before presenting the results, we review the predictions of the soliton theory^{4,5} that are related to our measurements:— 1) The on-chain diffusion coefficient ν_D should be $\sim 10^{14} \text{ sec}^{-1}$ at room temperature. This result was obtained^{9,10} by using the relationship for one-dimensional Brownian motion of domain walls in contact with thermal phonons derived by Wada and Schrieffer.¹¹ 2) The on-chain and off-chain spin diffusion rates should be entirely different because the kink soliton, a topological excitation, cannot hop between chains. The

off-chain spin diffusion rate ν_1 , which must therefore arise from electron or hole hopping, was determined¹² from the transverse hopping conductivity to be 10^7 sec^{-1} giving rise to an anisotropy ratio for the spin-diffusion rates of 10^7 .

Electron spin diffusion rates may be determined from measurements of proton spin-lattice relaxation rates, T_{1p} , and also from measurements of phase-memory times, T_M , in electron spin-echo experiments. Nechtschein *et al.*¹³ have interpreted their T_{1p} measurements as confirming the one-dimensional nature of the diffusion and the large ν_D expected from the soliton picture. This interpretation, however, requires knowing or assuming values for both the contact and dipolar electron-nuclear coupling strengths. The diffusion rates can be extracted from T_M measurements without knowing the various coupling strengths if the one-dimensional nature of the diffusion is assumed. Since the coupling strengths are not known for polyacetylene, we have used the spin-echo method to measure the diffusion rates. To resolve one remaining ambiguity, we have also used linewidth results from continuous-wave EPR experiments.

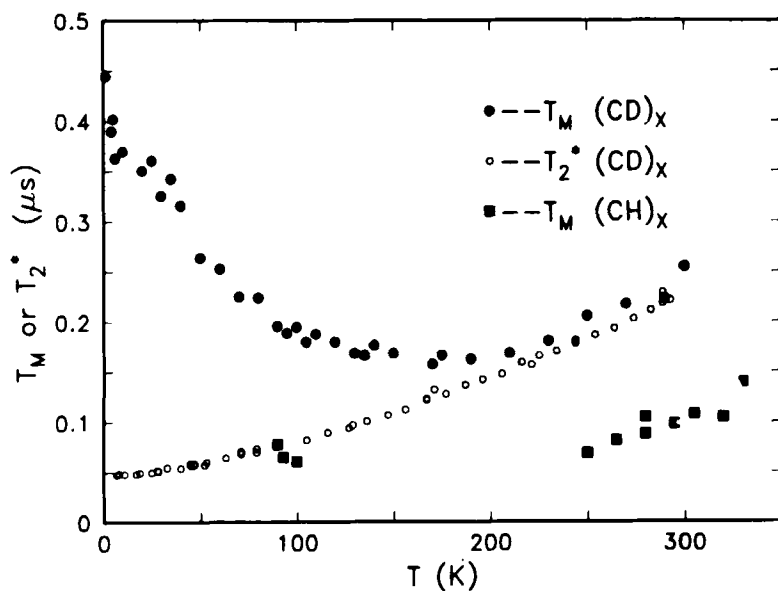


FIGURE 1 Phase-memory time T_M and inverse half-linewidth T_2^* vs. temperature in $(CH)_x$ and $(CD)_x$. All $(CD)_x$ measurements are on the same sample.

Figure 1 shows T_M , from the spin-echo experiments, and T_2^* , the inverse of the half-linewidth in the continuous-wave EPR spectrum, both as functions of the temperature T , measured on a single sample of *trans*-(CD)_x. For comparison, a partial set of T_M measurements on *trans*-(CH)_x is also shown. We concentrated on (CD)_x because the larger values of T_M allowed greater accuracy. We see for (CD)_x that while T_2^* is a monotonically increasing function of temperature, T_M has a minimum of ~ 160 nsec at $T = T_{\min} \approx 150$ K. The minimum separates the motional-broadening regime ($T < T_{\min}$) from what is commonly termed the strong-narrowing regime ($T > T_{\min}$). Below T_{\min} , the width T_M^{-1} of the spin packets broadens with increasing temperature, while the CW linewidth ($\sim T_2^{*-1}$), which is largely determined by the inhomogeneous distribution of local fields, narrows with increasing temperature. Above T_{\min} , both T_M^{-1} and T_2^{*-1} narrow as T increases, T_M being approximately equal to T_2^* as both approach T_2 , the inverse linewidth neglecting inhomogeneities.

The observation that $T_M > T_2^*$ at temperatures $T < T_{\min}$ shows that in this temperature range the spin diffusion rate is smaller than the spread of local-field values (in frequency units). An upper limit on the latter of 10^9 sec^{-1} may be obtained for (CD)_x from a calculation of the electron-electron dipolar broadening in (CH)_x.⁹

A quantitative evaluation of the spin diffusion rate as a function of temperature was performed⁷ using the Gaussian spectral-diffusion model and a one-dimensional soliton diffusion in which all electrons are assumed to diffuse with the same mechanism characterized by the same parameters. From this model, we evaluated ν_D/N^2 (N being the half-width of the kink soliton) which we call ν_D' . The results are shown in Fig. 2 for several assumed anisotropy ratios ranging from 5 to 10^6 . A striking feature is that ν_D' ($= \nu_D/N^2$, N being the half-width of the kink soliton) increases with temperature, rapidly for $T > 40$ K. This result is contrary to the result inferred from the temperature dependence of the proton relaxation rate by Kume *et al.* in these proceedings. It is also contrary to theoretical predictions for the soliton mobility when only soliton-phonon interactions are invoked to limit the mobility.

As seen in Fig. 2, the maximum value of ν_D' at room temperature is $2 \times 10^9 \text{ sec}^{-1}$, so that if one assumes the conventional kink soliton half-width, then one concludes that at room temperature $\nu_D \leq 10^{11} \text{ sec}^{-1}$ (and at T_{\min} its value is consistent with the earlier bound of 10^9 sec^{-1}). This upper limit is more than three orders of magnitude lower than expected from the first of the soliton-theory predictions, mentioned above, and from the reported NMR measurements.¹³

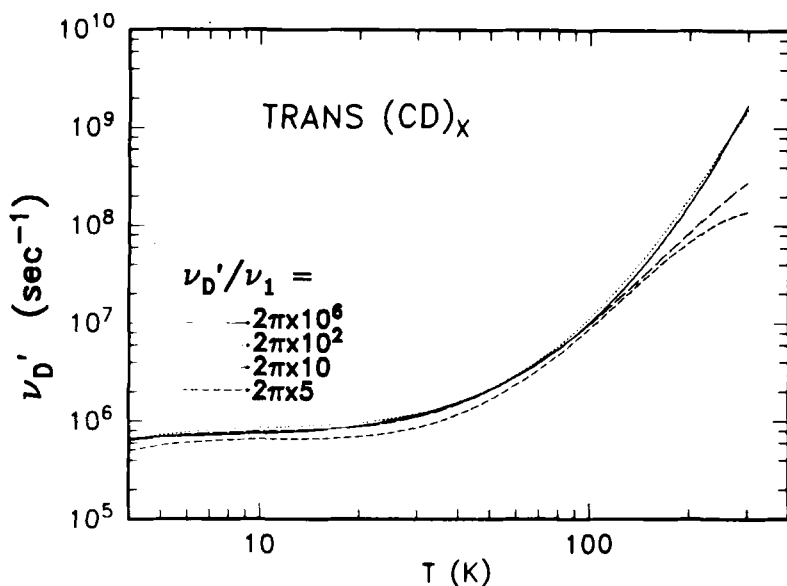


FIGURE 2 1-D spin-diffusion rate ν_D' ($\nu_D' = \nu_D / N^2$) vs. temperature in *trans*-(CH)_x as determined from measured value of T_M for several assumed values of anisotropy. ν_1 is the off-chain hopping rate.

We summarize our results: 1. The on-chain spin diffusion rate, as evaluated from spin-echo measurements (assuming a kink soliton width of 15 sites) is less than 10^{11} sec^{-1} , not equal to 10^{14} sec^{-1} . 2. The spin diffusion rate increases with increasing temperature, especially for $T > 40 \text{ K}$. 3. The ratio of on-chain to off-chain spin diffusion rates (assuming an off-chain rate of 10^7 sec^{-1}) is less than 10^4 , not equal to 10^7 . Thus observed spin dynamics of pristine *trans*-polyacetylene as determined from spin-echo and CW EPR experiments contradict the predictions of the soliton model in at least two important ways.

3. DOPED POLYACETYLENE

A. Spin Susceptibility

The idea that the charge carriers in doped (CH)_x are spinless kink solitons was first proposed by Weinberger *et al.*¹⁴ to explain an

apparent absence of any Pauli susceptibility in their static susceptibility experiments for less than $\sim 1\text{--}2\%$ AsF_5 concentration. Because of the uncertainties associated with the big diamagnetic correction they had to apply, and because of the large background Curie tail when the *trans*-(CH) $_x$ is obtained by thermally-induced isomerization, we decided to measure the spin susceptibility directly with EPR in doped *cis*-(CH) $_x$, i.e. in material that had not been isomerized thermally but which was presumably isomerized by doping (see below). Originally, we reported on two dopant concentrations.¹⁵ Subsequently, we have extended the concentration range and refined the sample characterization.⁸

In Fig. 3 we show the temperature dependence of the spin susceptibility of *cis*-(CH) $_x$ when doped with different levels of AsF_5 (the actual dopant species is probably AsF_6^-) in the temperature range $100 < T < 300$ K. For comparison, the susceptibility of pristine *cis*-(CH) $_x$ is also shown in this figure. It is clearly seen that doping in the concentration range $0.003\text{--}0.04$ dopant ions per carbon atom (what we shall call $0.3\text{--}4.0\%$ doping) *increases* the spin susceptibility and that the spin susceptibility generated by doping has hardly any temperature

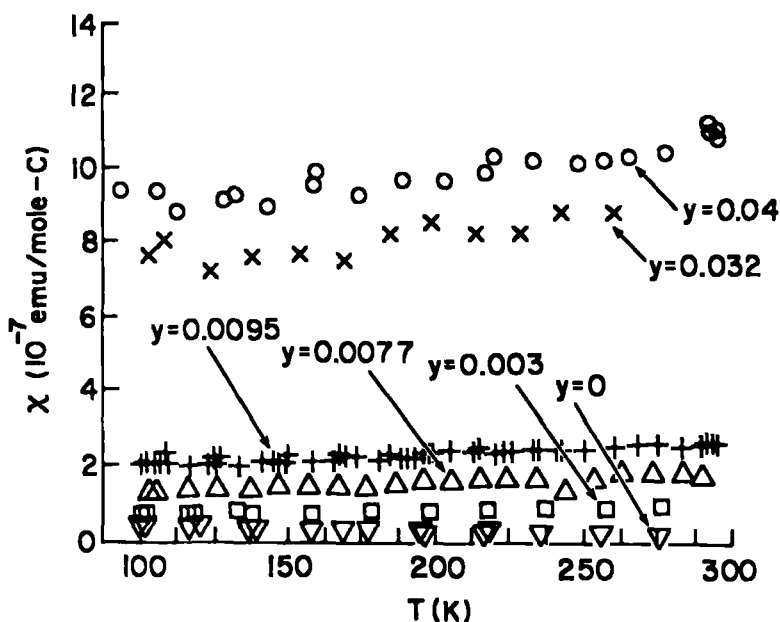


FIGURE 3 Spin susceptibility as a function of temperature for AsF_5 -doped *cis*-(CH) $_x$ for various dopant concentrations y .

dependence in this temperature range, even for the lowest concentration of dopant (0.3%). Furthermore, in Fig. 4, we see that the magnitude of the room-temperature susceptibility is proportional to the dopant concentration over a broad range of concentrations (0.3–12%), its variation with concentration showing no anomalous behavior near 1% where the electrical conductivity increases so abruptly.

In our view, the occurrence of a temperature-independent spin susceptibility is incompatible with doping that is homogeneous. With homogeneous doping, at least for the lower concentrations ($\sim 0.3\%$), the carriers (holes) would not contribute a Pauli susceptibility. Either they would occupy localized states pulled from the band and contribute a Curie-like susceptibility, or they would occur in trapped, spinless kink-soliton states and contribute no spin susceptibility at all. Since the susceptibility is Pauli-like, banding apparently occurs. Thus, the impurity states must overlap even for the lowest dopant concentration, when the *average* impurity separation along the polymer chain is ~ 300 sites. Since the overlap at such separations is negligible, the doping must be very inhomogeneous, suggesting the existence of metallic regions or domains of relatively high dopant concentration separated by semiconducting regions of low or negligible dopant concentration. Further-

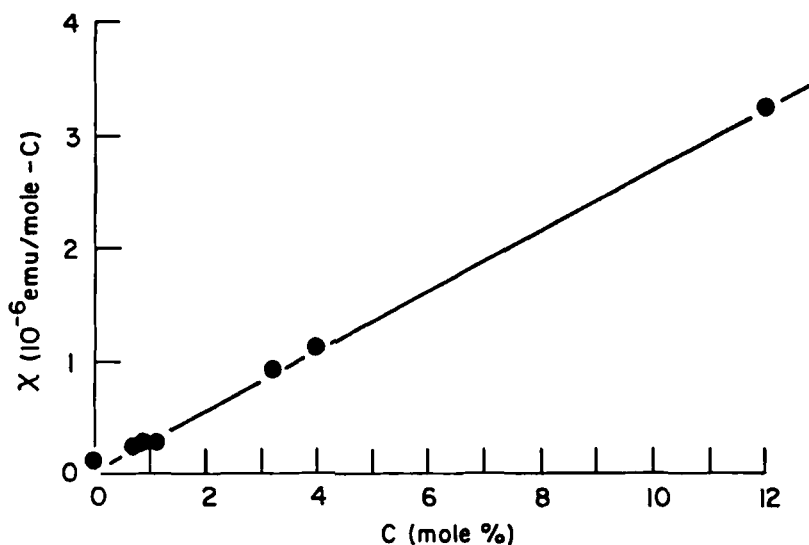


FIGURE 4 Room-temperature spin susceptibility of AsF_5 -doped $\text{cis}-(\text{CH})_x$ as a function of dopant concentration y . The value of the spin susceptibility for the 12% dopant concentration was taken from Ref. 14. This value was measured by the Slichter-Schumaker technique.

more, the proportionality between the spin susceptibility and the impurity concentration suggests that the distribution of transfer integrals is concentration-independent. This would indeed be the case if the metallic domains all had similar dopant concentrations. The effect of changing the concentration would be to increase the size of these domains and/or to increase their number until a kind of percolation threshold is reached and the metallic domains are sufficiently close to form, effectively, a metallic continuum. The crossing of such a threshold, though expected to have a considerable effect on the transport properties, should have no effect on the magnitude or temperature dependence of the spin susceptibility. Just such behavior is observed at about 1% doping where a transition is seen in the conductivity¹⁰ but no anomaly is seen in the magnetic properties.

Since the Pauli susceptibility we observed was in doped *cis*, not in doped *trans*, it might be argued that these results are not relevant to doped *trans*-(CH)_x, that doping in the *trans* isomer is homogeneous, and that soliton carriers have indeed been observed there. To test this possibility directly, we measured the spin susceptibility with EPR in a sample of AsF₅-doped *trans*-(CH)_x having 0.5% dopant concentration and were able to see an appreciable Pauli susceptibility despite the much larger Curie background. Later measurements on very slowly doped *trans*-(CH)_x at two concentrations between two and three percent were also very close to the straight line in Fig. 4. We also noted that the dc conductivity vs. T in the doped *trans* and doped *cis* samples were similar, hardly compatible with soliton conduction in the former and an entirely different conduction mechanism in the latter.

The similarity of the properties of doped *cis* and doped *trans* properties was not surprising. There are several reasons to believe that doping (CH)_x causes isomerization: a second-moment analysis of the proton NMR line,¹⁶ the low-temperature specific heat,¹⁷ resonance Raman spectra,^{18,19} and ¹³C NMR.²⁰ There is, nevertheless, considerable difference of opinion as to how much doping is required to isomerize most of the sample, the proton NMR indicating less than 1%, the Raman and ¹³C NMR indicating more than 5%.²¹ We call attention specifically to the ¹³C NMR experiments in which the Stuttgart group studied the Pauli susceptibility indirectly by looking for a Knight shift of the ¹³C NMR line in AsF₅-doped (CH)_x at room temperature. While they found no evidence for a Pauli susceptibility below 7% dopant concentration, in contrast to our results and in apparent confirmation of the soliton interpretation, they also found evidence for very little *cis-trans* conversion below this dopant concentration. Thus the apparent absence of Pauli susceptibility in the range 1–7% dopant concentration, where the conductivity is metallic and the material is

mostly *cis*, would imply that the "spinless carriers" occur in the *cis* isomer where there can be no mobile kink solitons.²² If the carriers are solitonic excitations, they must not be kink solitons but polaron-like solitons (analogous to a kink-antikink bound pair^{23,24}). Furthermore, to be spinless these polarons must carry two charges. Since such doubly-charged polarons should be accompanied, for energetic reasons, by singly-charged polarons with spin 1/2 which, it is claimed, are not observed, we are more inclined to believe that the appearance of little isomerization is evidence for inhomogeneous doping, that for some unexplained reason (possibly the large linewidth in the metallic regions), the observed ¹³C NMR comes mainly from the nonmetallic regions where the doping, the isomerization and, therefore, the spin susceptibility are all negligible.

Finally, we should comment on the recent results of Ikehata *et al.*²⁵ at the University of Pennsylvania, in which the Pauli susceptibility of *trans*-(CH)_x doped with AsF₅ in a very slow and elaborate way was measured with EPR at 10MHz (Schumacher-Slichter technique) and also found to be negligible up to 7% dopant-ion concentrations. Originally, it was suggested that this was evidence for more homogeneous doping than had been achieved in our earlier experiments. Subsequent experiments on doped *trans*-(CH)_x prepared both at IBM San Jose and jointly with the Penn group have shown that the differences between the IBM and Penn results arise not from differences in material preparation but from differences between experimental effects at 10 MHz and 10 GHz. Whether these differences reflect some genuine frequency dependence or are associated with the Dysonian line shapes at 10 GHz is under investigation. It should be noted, however, that for $y < 0.03$ the line at 10 GHz is only weakly Dysonian.

B. Electrical Conductivity

If metallic domains are formed on doping, the electrical transport properties should reflect this in several ways. As an indication of what is to be expected, we may consider the case of granular metals, where genuine metallic grains are dispersed in a dielectric medium. This physical situation has many similarities to the picture suggested here of metallic domains in a semiconducting medium. The theory and experiments for granular metals,²⁶ which are in remarkable agreement, show that there can be enormous differences in observed conductivity depending on whether one is above or below the percolation threshold at which the metallic grains become sufficiently connected to form unbroken paths through the material. Sheng, Abeles and Arie²⁷ have shown that at sufficiently low fields and at concentrations below the percola-

tion threshold, the conductivity is dominated by the energy required to produce a pair of oppositely charged grains from a pair of neutral grains and the probability of a carrier tunneling between these grains. This leads to a temperature dependence of the form

$$\sigma \sim \exp(-AT^{-1/2}) \tag{1}$$

which is observed in granular materials at low fields and which also appears to describe $(\text{CH})_x$, as was shown in Ref. 15 and as we show again in Fig. 5.

At these concentrations and sufficiently low temperatures, granular metals are also predicted to show a characteristic non-ohmic behavior at high fields given by^{28,26}

$$R_H = R_{H_0} \exp(E_0/E) \tag{2}$$

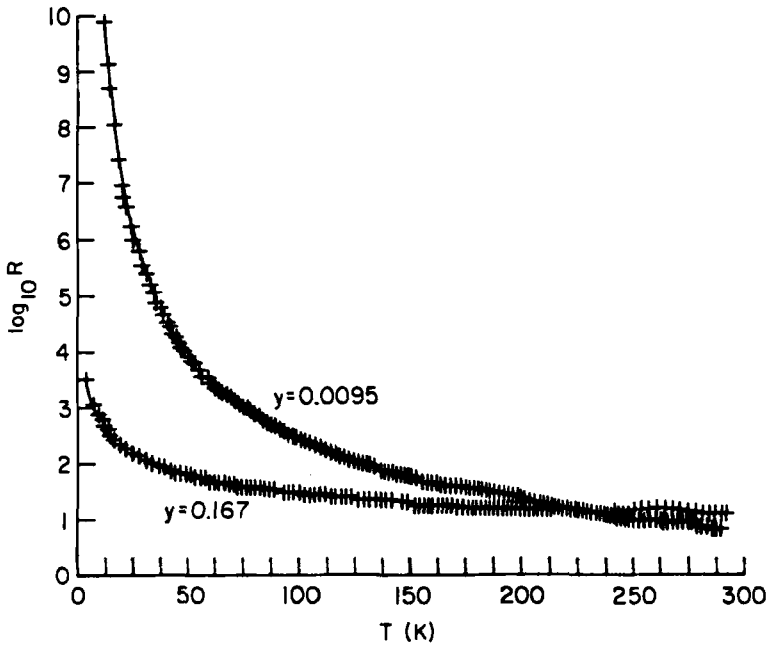


FIGURE 5 Resistivity (marked as +) as a function of temperature for AsF_5 -doped $\text{cis}-(\text{CH})_x$ with 0.95% and 16% dopant concentration y . The solid curves are best fits using equations (1) and (2) respectively.

with E_0 independent of temperature. Results of low-temperature measurements by Mortensen *et al.*²⁹ on *cis*-(CH)_x doped with AsF₅ to a concentration of 0.5% are reproduced in Fig. 6. The approach to a single straight line at high fields is in agreement with this predicted behavior.

The frequency dependence of the ac conductivity below the percolation threshold is also consistent with the metallic-domain picture. Originally, experiments at room temperature showing no frequency dependence up to 500 MHz in iodine-doped (CH)_x were interpreted as a disproof of the metallic-domain hypothesis, at least with iodine doping.³⁰ However, recent experiments on AsF₅-doped samples for a variety of concentrations and to temperatures as low as 10 K show unmistakable frequency dependence. The results for three dopant concentrations are shown in Fig. 7. It is seen that at higher temperatures, the data for different frequencies but the same dopant concentra-

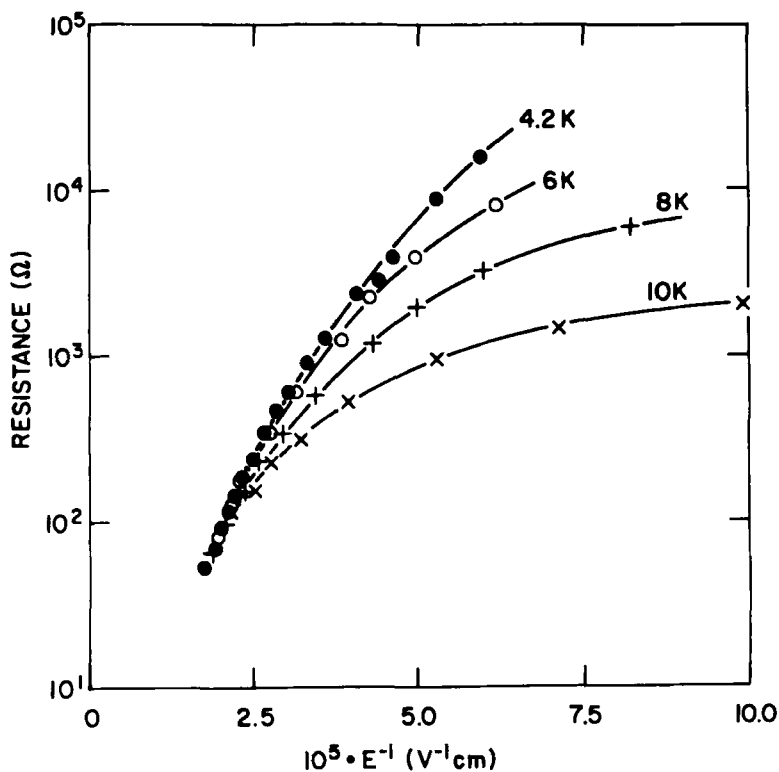


FIGURE 6 Log of Resistance vs. $1/E$ for AsF₅-doped *cis*-(CH)_x with 0.5% dopant concentration for several temperatures below 10 K.

tion can all be described by Eq. (1) (straight lines in Fig. 7), but at lower temperatures there are deviations which are more pronounced at higher frequencies, as expected. For comparison, we have included analogous data for two concentrations of gold in germanium, a prototypical granular-metallic system. The similarity to the behavior of AsF₅-doped *trans*-(CH)_x is evident.

For concentrations well above the percolation threshold, one still should not expect the conductivity to have a metallic temperature dependence for two reasons: 1) The metallic domains in (CH)_x are highly disordered, which at sufficiently low temperatures^{3†} or

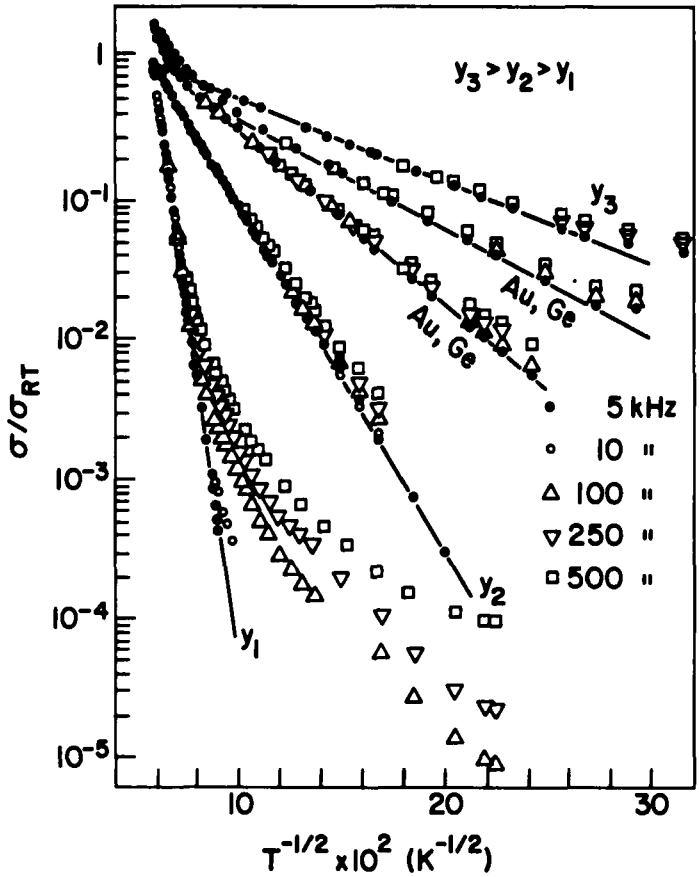


FIGURE 7 AC-Conductivity vs. $T^{-1/2}$ for AsF₅-doped *trans*-(CH)_x at various frequencies and dopant concentrations. For comparison, data for two samples of the granular metal Au in Ge are also shown.

concentrations¹² should lead to a temperature dependence characteristic of variable-range hopping in three dimensions:

$$\sigma \sim \exp(-A'T^{-1/4}) \quad (3)$$

2) Even above the percolation threshold, there might still be a need for tunneling through a potential barrier, either between the fibrils or between the platelets of which they are constituted. However, between such large metallic regions, the charging-energy effect²⁷ should be negligible. In this case, the tunneling will still be affected by any modulation of the potential barrier by thermal fluctuations, as analyzed by Sheng.³² The characteristic temperature dependence of this effect is

$$\sigma \sim \exp[-T_1/(T+T_0)] \quad (4)$$

In Fig. 5, the results obtained for a sample doped with 16% AsF₅ are shown to be fit remarkably well with relation (3). A fit of similar quality with relation (4) was demonstrated by Sheng.³² From these data, one can not choose between these two mechanisms. In any case, there is no need to invoke a semiconductor-to-metal transition within the platelets or fibrils to explain the different temperature dependence and magnitude of the conductivity below and above the apparent transition.

C. Summary

We have given evidence and arguments for the following picture: 1) The doping of *cis*-(CH)_x is inhomogeneous for all concentrations of dopant that we have studied (0.3–4%), the doped and undoped parts of the polymer forming separate phases. 2) The doped parts form metallic-like clusters exhibiting a Pauli spin susceptibility. 3) At low dopant concentrations, the dc conductivity, which was previously interpreted to be activated or three-dimensional variable-range hopping as in a normal semiconductor, possibly with kink solitons as carriers, or to be *via* tunneling between charged and neutral kink solitons,¹² involves conduction between isolated metallic particles dispersed in a dielectric, as described by Sheng, Abeles and Arie. 4) At high dopant concentrations, the conductivity, which was thought to be that of a disordered metal or degenerate-band semiconductor, appears to be essentially that, but with conduction along percolation paths connecting such regions, and hopping (*i. e.* tunneling) between the fibrils, as analyzed by Sheng. 5) The semiconductor-to-metal transition, which was previously inter-

puted as arising *within* the doped region, results from a change in the topology of these regions, *i. e.* it is the crossing of a kind of percolation threshold to a regime in which the metallic regions nearly touch to form a continuum.

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