Low-temperature thermopower of $(SN)_x$

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Measurements of the thermoelectric power of $(SN)_x$ are reported over the temperature range 0.15–4.2 K for magnetic fields of 0–15 kOe applied parallel and perpendicular to the fiber axis. Above 1 K, the thermopower is large, negative, and dominated by phonon drag, which indicates that electrons contribute more to the electrical conductivity than do holes, and that the phonons are predominately scattered by the electrons. As the temperature is decreased below 1 K in zero field, an increase in the thermopower is observed which is attributed to the Kondo effect. This increase is terminated at the superconducting transition temperature, where the thermopower drops to zero. With the application of a magnetic field, a complicated behavior is observed below 1 K which is attributed to a combination of the quenching of superconductivity, superconducting fluctuations, magnetic impurities, and the Kondo effect.

I. INTRODUCTION

Polymeric polysulfurnitride, $(SN)_x$, is an unusual material from several aspects. Crystals are formed by the solid-state polymerization of S_2N_2,[1,2] and consist of bundles of $(SN)_x$ fibers with diameters on the order of 100 Å.[3,4] The fibers are composed of aligned polymer strands in crystalline form. The electronic properties are quite anisotropic[5–8] due both to the microscopic structure (which gives higher conductivity along the polymer strands) and to the arrangement into fibers. Recent experiments have shown that the latter effect is dominant in causing the anisotropy.[9,10] In addition $(SN)_x$ has been shown to be superconducting with a transition temperature, $T_c \simeq 0.3$ K.[11] It has extremely anisotropic critical fields[10,11] resulting from the fibrous morphology. Although the band structure is quite three dimensional,[12–17] it has been suggested that the small diameter of the fibers may lead to quasi-one-dimensional superconductivity in the temperature (T) regime where the coherence length $\xi(T)$ is large compared to the fiber diameter.[18,19] The reduced dimensionality leads to large superconducting fluctuations above T_c.

The electrical conductivity of $(SN)_x$ has shown a resistivity (ρ) minimum at low temperatures which is very sample dependent, the general feature being that "high-quality samples" have less of an increase in ρ as the temperature (T) is lowered below the minimum than low-quality samples.[20] This has led some authors to discuss a Kondo effect in $(SN)_x$,[5] especially in light of the presence of low-temperature magnetic susceptibility measurements which indicate the presence of localized magnetic moments.[21]

In this paper we report measurements of the thermopower (S) of $(SN)_x$ over the temperature range 0.15–4.2 K in the presence of a magnetic field (H) which was varied in magnitude (0–15 kOe) and orientation relative to the fiber axis. The motivation was to further elucidate the role of superconducting fluctuations, localized magnetic moments, and the electron-phonon interaction in $(SN)_x$. Thermopower is a zero-current measurement so that the superconducting fluctuations can be probed in the absence of possible critical current or heating effects. Several investigations have also shown the sensitivity of thermopower to the presence of magnetic impurities.[22,23] Because of competition between effects based on magnetic impurities and those based on superconductivity, the use of a magnetic field variable in magnitude and orientation was an essential diagnostic tool in our experiments. During the runs the same apparatus was used for measuring the thermal conductivity (K) of $(SN)_x$.[24] There is an important connection between the two experiments, as the phonon-drag contribution to S, discussed below, indicates that electron-phonon scattering may be the dominant scattering mechanism for the phonons above 1 K. Brief accounts of the work reported here have been presented elsewhere.[25]

II. EXPERIMENTAL DETAILS

The thermopower measurements were performed in vacuum outside the mixing chamber of a dilution refrigerator. With the same setup we were also able to measure K, ρ, and the upper critical field, H_{c2}, (as a function of angle and temperature) of this sample. The $(SN)_x$ needle used in this experiment had approximate dimensions of $4.9 \times 0.12 \times 0.09$ mm^3 and a resistivity ratio $\rho(300)/\rho(4.2) \sim 18$. The $(SN)_x$ sample was provided by R. L. Greene and G. B. Street of IBM San Jose. It showed the same T_c and H_{c2} behavior as that reported for other samples.[10,11]

Here we present a brief description of the experimental setup; a more detailed report will be given elsewhere.[26] Four electrical and thermal contacts were made to the $(SN)_x$ crystal as in the conventional four-probe resistivity measurement technique. The connections to the sample were made by evaporating gold contacts, wrapping 25-μm-diameter Cu wire around these contacts and applying silver paste. We have found that evaporated Au films produce our lowest contact resistance to $(SN)_x$. Heat was supplied through one of the end contacts with the opposite end connected to a temperature controlled plate, which acted as a thermal ground. Copper wires from the two inner contacts were thermally anchored to separate Cu pads for the temperature measurements. Thermal guards were used to keep these pads at the same temperature as the sample, i.e., to insure that no heat flowed in the leads used to measure the sample temperature. The temperature difference between the two inner contacts was measured with carbon resistance thermometers. The thermoelectric voltage was measured across the same inner contacts. A temperature difference of $\Delta T/T \leq 3\%$ was used. At low temperatures it was therefore necessary to measure signals as low as 3×10^{-10} V. To accomplish this we used a signal averager to average over several heat pulses. Superconducting wire leads were used from the Cu pads for the electrical measurements. Since the Cu pads were at the same temperature as the inner contacts of the $(SN)_x$, there was no thermal gradient along the Cu wires. Thus, there was no contribution from these wires to the measured thermoelectric voltages. Further evidence for the neglect of the copper connections comes from the fact that no voltage was observed when the $(SN)_x$ was superconducting.

Errors at low T are dominated by the uncertainty in S, whereas at high-T errors are mainly in the measurement of T. Characteristic errors are shown by error flags on the figures.

III. EXPERIMENTAL RESULTS AND DISCUSSION

In Fig. 1 we have plotted S as a function of T in the range 0.15–4.2 K for zero magnetic field. There are three main features of Fig. 1; (i) a rapid increase (negative) above 1 K, (ii) a minimum and subsequent rise between 0.8 and 0.3 K, and (iii) a drop to zero at 0.3 K. The sign, magnitude, and T dependence of S fit fairly well onto previous measurements by several authors at $T>4.2$ K.[5,8,27,28] As T is increased above 4.2 K these authors found an increase in S with a maximum (negative) value at ~20 K. They associate this behavior with a phonon-drag peak. Two of the main features of Fig. 1 are qualitatively well understood, the phonon-drag contribution for $T>1$ K and the superconducting transition at

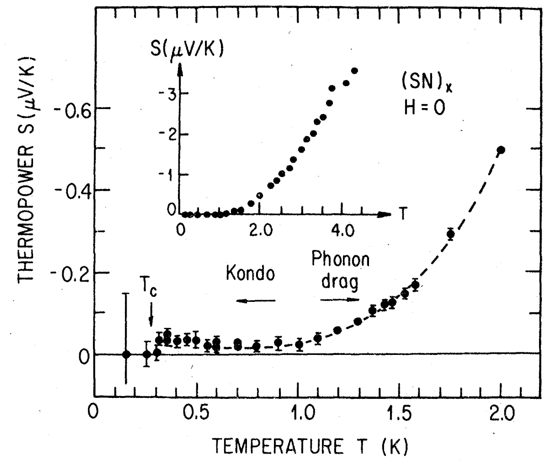

FIG. 1. Thermopower of $(SN)_x$ in zero magnetic field as a function of temperature. The phonon-drag tail is notable above 1 K. Near 0.3 K the superconducting transition is seen. The increase between 1 and 0.3 K is attributed to the Kondo effect.

$T=0.27$ K. These aspects are discussed in more detail below.

Band-structure calculations[15] and magnetoresistance measurements[9] indicate that $(SN)_x$ is a two carrier metal with $n_e \simeq n_h \simeq 2.1\times10^{21}/\text{cm}^3$, where n is the concentration and the subscripts e and h refer to electrons and holes, respectively. The thermopower of such a system is usually separated into a diffusion term S_d and a phonon-drag term S_l. The diffusion term for electrons can be written in the form[29]

$$S_d^e = \frac{-\pi^2 k^2 T}{3|e|}\left[\frac{\pm D_e(\epsilon)}{n_e} + \frac{\partial \ln\mu_e(\epsilon)}{\partial\epsilon}\right]_{\epsilon_F}$$

$$= \frac{\mp C_e}{n_e|e|}\left[1 \pm \frac{n_e}{D_e(\epsilon)}\frac{\partial \ln\mu_e(\epsilon)}{\partial\epsilon}\right]_{\epsilon_F}, \tag{1}$$

where $D_e(\epsilon)$ is the electron density of states, ϵ is the energy, μ_e is the mobility, C_e is the electron specific heat, ϵ_F is the Fermi energy, and the upper sign is taken. The result for holes is obtained by making the replacement $e\rightarrow h$ and taking the lower sign. In its simplest form, the two-carrier phonon-drag term for electrons becomes[30]

$$S_l^e = \mp\frac{C_l}{3n_e|e|}\left[1+\frac{\tau_{pe}}{\tau_{ph}}+\frac{\tau_{pe}}{\tau_{p\bar{c}}}\right]^{-1}, \tag{2}$$

where C_l is the lattice specific heat, τ_{pe} is the mean time for scattering of phonons by electrons, τ_{ph} is the mean time for scattering of phonons by holes, and $\tau_{p\bar{c}}$ is the mean time for scattering of phonons by all other processes not involving these carriers. The same formula holds for the phonon-drag effect on holes by making the replacement $e \rightarrow h$ and taking the lower sign in Eq. (2). The full diffusion and phonon-drag terms are then combined using the relations[30]

$$S_d = \frac{\sigma_e S_d^e + \sigma_h S_d^h}{\sigma_e + \sigma_h} \tag{3}$$

and

$$S_l = \frac{\sigma_e S_l^e + \sigma_h S_l^h}{\sigma_e + \sigma_h} , \tag{4}$$

where σ_e and σ_h are the electrical conductivity of the electrons and holes, respectively. Since, at low T, $C_l \propto T^3$ and it is usually found that $S_d \propto T$, the thermopower is often presented as

$$S = AT + BT^3 , \tag{5}$$

where the first term is attributed to S_d and the second to S_l.

In order to separate the two terms it is conventional to plot S/T vs T^2, as is done in Fig. 2 for $1 < T < 4.2$ K. Over this range quite good agreement with Eq. (5) is found. The least-squares fit to Eq. (5) is shown by the solid line. The slope, which determines the phonon-drag term, is well defined by the data, whereas the $T=0$ intercept, which gives the band term, is seen to be poorly defined, but in any case very small. From a least-squares fit of our data for $T > 1$ K we find $A = 0.008 \pm 0.027$ μV/K^2 and $B = -0.0433 \pm 0.0027$ μV/K^4. Here we have used the value of one standard deviation as the error limits.

The most prominent feature seen in Fig. 2 is that S is dominated by phonon drag in the range $1 < T < 4$ K. Here we discuss the implications for the phonon-electron interactions. Combining Eqs.

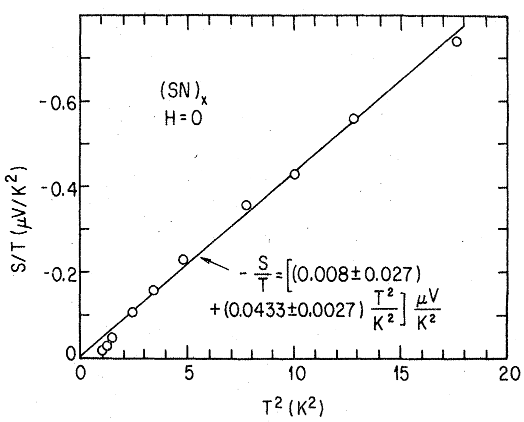

FIG. 2. Thermopower of $(SN)_x$ divided by temperature as a function of temperature squared for $1.0 \leq T \leq 4.2$ K. The line is at least-squares fit of the data. A large phonon drag and a negligible electronic term are the main features.

(2) and (4) and using $n_e = n_h = n$ gives

$$S_l = -\frac{C_l}{3n|e|}\left[\left(\frac{\sigma_e}{\sigma_e+\sigma_h}\right)\left(1+\frac{\tau_{pe}}{\tau_{ph}}+\frac{\tau_{pe}}{\tau_{p\bar{c}}}\right)^{-1} - \left(\frac{\sigma_h}{\sigma_e+\sigma_h}\right)\left(1+\frac{\tau_{ph}}{\tau_{pe}}+\frac{\tau_{ph}}{\tau_{p\bar{c}}}\right)^{-1}\right] . \tag{6}$$

Substitution of the measured[31] $C_l = (8.8 \pm 0.4) T^3$ μJ/gK^4, the density of 2.32 g/cm^3, and $n = 2.1 \times 10^{21}$ cm^{-3} calculated[15] from the volume of the electron (or hole) pockets relative to that of the Brillouin zone yields $C_l/3n|e| = 0.020 T^3$ μV/K^4, which is to be compared with our experimental $S_l = (0.0433 \pm 0.0027) T^3$ μV/K^4. This result indicates that the bracketed expression in Eq. (6) is 2.17. The fact that this figure is approximately twice its maximum value of one should not be taken too seriously given the approximations of the theory. What it does suggest strongly is that the bracket is dominated by its first, or positive, term, and that this term is close to the maximum value it can assume. Several consequences follow from this result. First, from the magnitude the sign of S_l it is seen that the phonon-drag thermopower is dominated by electrons. Furthermore, for the bracket to have a value close to the maximum possible, $\sigma_e >> \sigma_h$, i.e., the low-temperature electrical conductivity is dominated by that of the electrons. These phonon-drag measurements do not allow one to identify the dominant electron scattering mechanism. Nevertheless, it is worth pointing out that the observed phonon "bottleneck" rules out the electron-phonon interaction as a means of dissipating electron momentum.[32] Other evidence indicates that the main scattering of electrons is by imperfections. This can be seen from the near independence of ρ on T in Fig. 7.

In contrast, the scattering of phonons is due predominantly to electrons. This follows because the bracket in Eq. (6) can be close to its maximum possible value only if $\tau_{pe} << \tau_{ph}$ and $\tau_{pe} << \tau_{p\bar{c}}$. Since the phonon-phonon and phonon-impurity scattering rates are small, we expect large phonon mean free paths in the range $1 < T < 4$ K. These conclusions are supported by the thermal conductivity of $(SN)_x$.[24]

It is worth noting that if the value of n deduced from x-ray spectra[4] ($n = 3 \times 10^{22}$ cm^{-3}) had been used, the observed phonon-drag term would have to be ~15 times smaller than that which we observe. We also point out that our partition of the conductivity between electrons and holes differs somewhat from that deduced from work on the magnetoresistance[5] of $(SN)_x$, where electron and hole mobilities $\mu_e = 610 \pm 60$ cm^2/Vs and $\mu_h = 430 \pm 40$ cm^2/Vs were found using calculated values[15] for $n_e = n_h$, the calculated plasma tensor,[15] and a single, isotropic scattering time for all carriers. These assumed conditions are much more restrictive than those we have used, and, in fact, determine the ratio μ_e/μ_h independently of the measured magnetoresistance. Therefore, we do not feel the difference between their results and ours is a serious contradiction. Since, unlike the magnetoresistance, the electron and hole contributions to S_l are of opposite sign, we believe our result $\sigma_e >> 2.3\sigma_h$ is the preferred one. The analysis presented here is based on a model which ignores the moderate anisotropy of the bands in $(SN)_x$. Not enough is known of this and the anisotropy in the scattering rates to permit a more detailed analysis at this time. In particular, there is no microscopic explanation of why $\sigma_e >> \sigma_h$ and $\tau_{pe} << \tau_{ph}, \tau_{p\bar{c}}$.

In contrast to the huge phonon-drag term, the $T=0$ intercept on Fig. 2 indicates a very small diffusion contribution to S. Although not enough is known about the mobility term to evaluate its importance, it is instructive to calculate the maximum magnitude of S_d, $|S_d^{\max}|$, by assuming the mobility term is unimportant. Since the contributions from electrons and holes have opposite signs [Eq. (1)], we have

$$|S_d^{\max}| = \frac{\sigma_e C_e}{\sigma n_e |e|} \quad . \tag{7}$$

By substituting[31] $C_e = (0.83 \pm 0.09)T$ mJ/mole K^2, $\sigma_e/\sigma = 0.7$, and $n = 2.1 \times 10^{21}$ cm^{-3}, we calculate $|S_d^{\max}| = 0.087T$ μV/K^2. Our observed value, $S_d = (0.008 \pm 0.27)T$ μV/K^2 is much smaller. This difference can be due to either or both of two factors: (a) cancellation of the contributions from electrons and holes, or (b) cancellation by the mobility terms.

We will now consider the temperature region $T < 1$ K. The dominant structure seen in this region on Fig. 1 is a sharp drop in $|S|$ at about 0.3 K. We associate this behavior with the superconducting transition. The transition temperature measured resistively on this crystal is 0.275 K where we have taken the midpoint of the transition, the width of which is about 50 mK. In thermopower measurements the transition occurs between 0.30 and 0.32 K. The thermopower in the superconducting state should be zero due to the vanishing entropy in the pair condensate.[33] Within our experimental error, the thermopower below T_c is zero for our sample.

In order to confirm that the reduction of $|S|$ below 0.3 K is due to superconductivity, data showing S as a function of magnetic field for $T = 0.250$ K are plotted in Fig. 3. Since the critical fields are strongly anisotropic,[10] we have shown the dependence of S on H for fields both perpendicular and parallel to the polymer and fiber axis ($H_\perp$ and $H_\parallel$, respectively). The arrows shown on Fig. 3 indicate the midpoint of the superconducting transition measured resistively for $H_\perp$ and $H_\parallel$. It is clear from this figure that S shows the superconducting transition as a function of H in much the same way as ρ does.

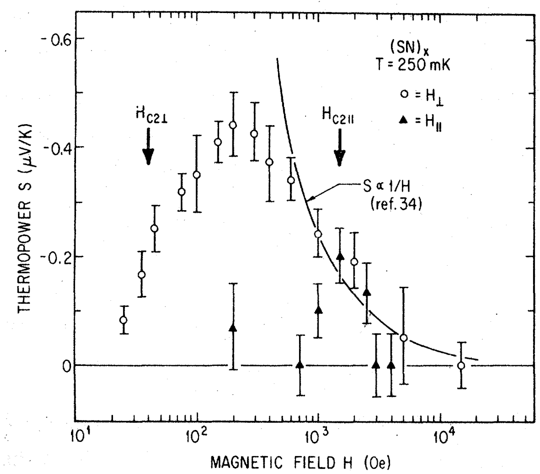

FIG. 3. Thermopower of $(SN)_x$ at $T=250$ mK as a function of magnetic field for fields both parallel and perpendicular to the crystal axis. The solid line shows the $1/H$ behavior expected of the Kondo effect at high field. The resistive critical fields correspond to the quenching of the thermopower.

What is very unusual about the H dependence is that $|S|$, once the superconductivity has been quenched, is considerably higher than $|S|$ observed above T_c in zero field (compare Figs. 1 and 3). In addition we see that as $H_\perp$ or $H_\parallel$ is increased above H_{c2}, $|S|$ reaches a maximum and then decreases to a very low value at $H = 15$ kOe. Since for $T < T_c$ the H behavior is isotropic for high fields, we believe that we are observing spin rather than orbital

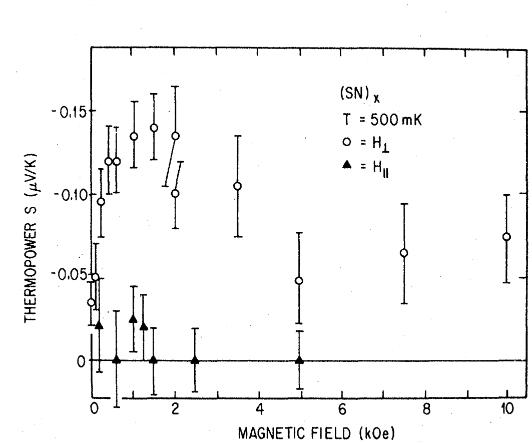

FIG. 4. Thermopower of $(SN)_x$ at $T=500$ mK as a function of both parallel and perpendicular magnetic fields. This is typical of the data obtained for $T > T_c$.

effects at this low temperature. The magnetothermopower is not merely reflecting an orbital Ettingshauser-Nernst effect[32] (similar to orbital magnetoresistance) which would be highly anisotropic. The perpendicular magnetic field dependence of thermopower is also seen to fit a $1/H$ form rather than H^2.[34]

Now let us consider the behavior of S in a magnetic field well above T_c. This is shown for $T = 500$ mK in Fig. 4. There it is seen that the thermopower is nearly independent of $H_{\parallel}$, whereas it shows the same general field dependence in a perpendicular field as for $T < T_c$. The significant differences are that above T_c, as H is increased starting from zero, $|S|$ begins with a nonzero value, increases to a maximum, and then decreases to a nonzero value. In Fig. 5 we have plotted the maximum thermopower, S_{max}, as a function of T for $H_{\perp}$. The value of $H_{\perp}$ at which this maximum occurs is shown in Fig. 6. From Fig. 5 it is seen that S_{max} increases as T is decreased for $T < 0.5$ K.

We attribute the low-T increase of $|S|$ to the interaction of the conduction electrons with localized magnetic moments, which can be quenched by the application of a magnetic field.[35] This occurs when the impurity magnetization is saturated by the magnetic field. At 250 mK, the value of $H_{\perp}$ needed to eliminate the magnetic contribution to S is about 5 kOe (Fig. 3). If we assume a g value of 2, this corresponds to a Zeeman splitting which is $2.7kT$, as expected.

The increase in $|S|$ with decreasing T (at T below the phonon-drag peak, Fig. 1) has been observed in many conventional metallic systems with magnetic impurities and is associated with the Kondo effect.[22,23] Calculations of the H and T dependence of the thermopower have been reported.[32] In the high-temperature regime the temperature dependence is logarithmic and the field dependence is $1/H$.[34] While we do not have sufficient temperature data to compare with this dependence, we have plotted a $1/H$ curve in Fig. 3 and see reasonable agreement.

The resistance (R) of our $(SN)_x$ crystal is plotted as a function of T in Fig. 7. There it is seen that R increases with decreasing T. This behavior has been seen in many of the previous resistivity studies, some of which have discussed it in terms of the Kondo effect.[5] As a rule of thumb most authors suggest that more perfect crystals have less of a resistivity increase at low temperatures.[20] Moreover, Kahlert and Seeger[28] have observed that samples which show a positive magnetoresistance can be bent to introduce defects and when remeasured give an initial negative magnetoresistance. Also by irradiating $(SN)_x$ the magnetic susceptibility has been seen to go from a Pauli to a Curie-Weiss law.[21]

The question naturally arises as to what are these local moments, which are clearly present from low-temperature magnetic susceptibility studies.[21] One possibility, suggested by the quality and bending studies, is the localization of electrons on broken

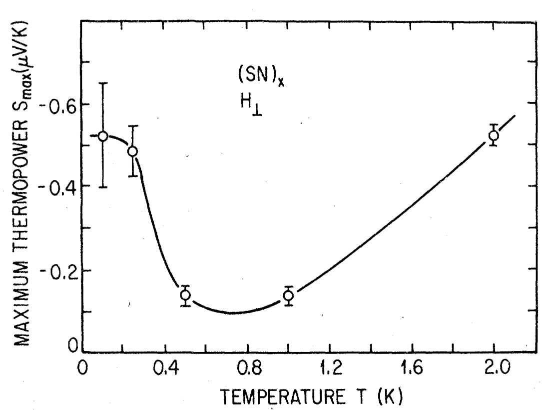

FIG. 5. Maximum thermopower of $(SN)_x$ for an applied $H_{\perp}$ as a function of temperature. The large value at low T is attributed to magnetic impurities. The solid line is a guide to the eye.

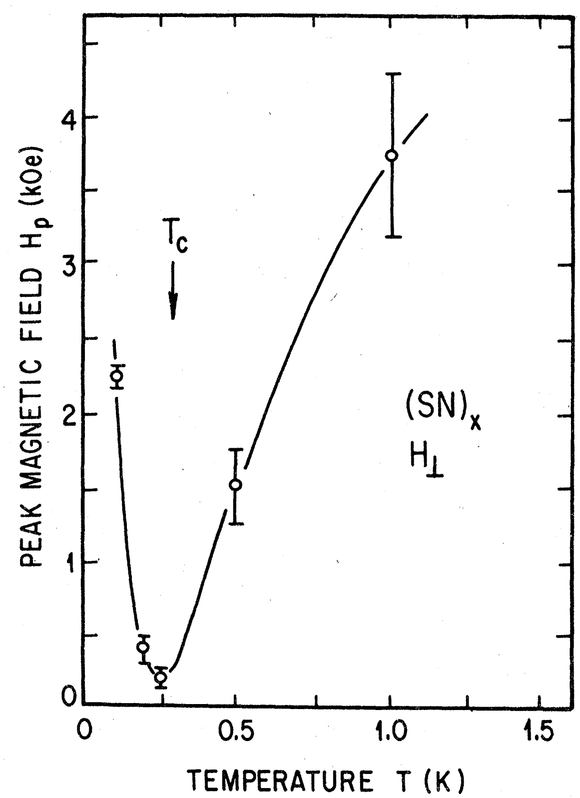

FIG. 6. Value of $H_{\perp}$ at which the maximum thermopower of $(SN)_x$ occurs as a function of temperature. Note that the field at maximum thermopower is minimum near T_c as measured resistively. The solid line is a guide to the eye.

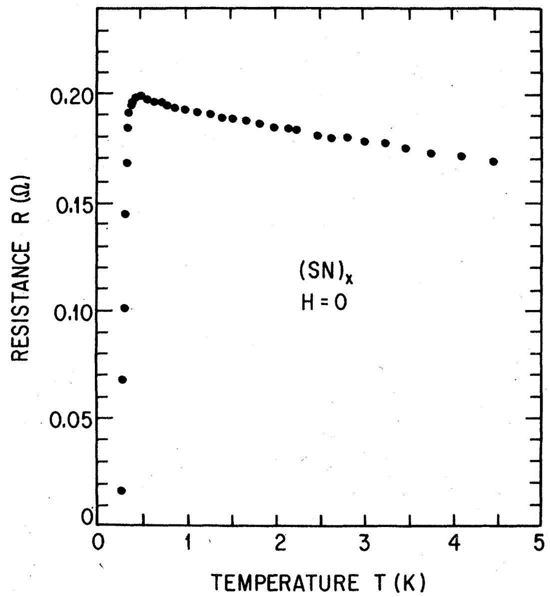

FIG. 7. Resistance of our $(SN)_x$ sample as a function of temperature in zero magnetic field. The superconducting transition temperature is 0.275 mK. The increase in resistance as the temperature is lowered at $T > T_c$ is attributed to the Kondo effect.

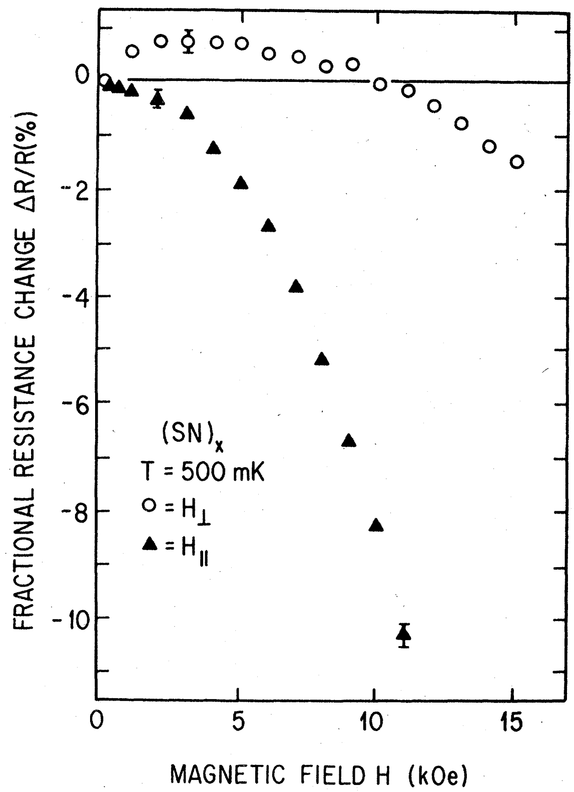

FIG. 8. Magnetoresistance of $(SN)_x$ at $T = 500$ mK. Note for parallel fields the magnetoresistance is always negative and gets increasingly larger in magnitude. For perpendicular fields it starts negative, goes slightly positive, then goes negative again. Representative error bars are shown.

bonds, caused by fractures on the polymer chains. In this case the interaction of the local moments with the conduction electrons may arise via a form of superexchange.

In further comparing Figs. 1 and 5 we note that for a finite $H_\perp$ the thermopower is larger than for zero field even at temperatures considerably above T_c. Below T_c we associate the magnetic field dependence with a combination of the quenching of superconductivity and the Kondo effect. Above T_c the situation is considerably more complicated. The large angular dependence of S above T_c (Fig. 4) is strong evidence that the increase in the peak magnetic field, $H_\perp$, shown in Fig. 6 is not due to localized magnetic moments. Civiak *et al.*[19] have shown that the resistance difference (between zero field and a magnetic field sufficient to quench the residual superconductivity) can be fit to an Aslamazov and Larkin expression for one-dimensional fluctuations.[18] However, they also imply that there is negligible magnetoresistance at 4.2 K. Several other authors have investigated the magnetoresistance and find a variety of effects. Beyer *et al.*[9] report a small positive magnetoresistance, a larger negative magnetoresistance, and a large positive magnetoresistance, as H is increased to 75 kOe. In magnetoresistance measurements from 0 to 15 kOe on our sample we have found both positive and negative resistance changes from $T > T_c$ to $T \simeq 2$ K (Fig. 8). For perpendicular fields the general behavior is an initial resistance increase followed by a larger decrease. For $H_\parallel$ the magnetoresistance is negative in the whole temperature range and varies roughly as $-\alpha H^2$. The effects are anisotropic and the interpretation at present is ambiguous.

We do not have enough data points directly above T_c to be able to compare with the theoretical treatment of Maki[36] for the fluctuation thermopower in a one-dimensional superconductor. We see in Fig. 6, however, that the field required to maximize the thermopower has an unusual temperature behavior. Since H at S_{max} is a minimum at T_c, we believe this behavior may be related to superconductivity.

In conclusion, from our study of the thermoelectric power of $(SN)_x$ we have shown that: (i) for $T > 1$ K the phonon scattering is dominated by the electron-phonon interaction, (ii) thermopower confirms the superconducting transition at 0.3 K, and (iii) there is strong evidence for local moments which may produce a low-temperature Kondo effect. There is also some tentative evidence of superconducting fluctuations above T_c, but considerably more work is needed to separate out several magnetotransport effects.

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