$(SNBr_{0.4})_x$: A superconductor of increased dimensionality

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We have measured some of the superconducting properties of $(SNBr_{0.4})_x$. The critical temperature is found to be enhanced over that of $(SN)_x$ by approximately 20%. The transition region is greatly narrowed. The perpendicular upper critical field is linear with temperature over a wide domain, attaining a value of about 400 Oe at absolute zero, and totally lacking the upward curvature shown by $(SN)_x$. The parallel field is not greatly changed. On the basis of these and other measurements, it is shown that $(SNBr_{0.4})_x$ is a "three-dimensional" superconductor due to improved coupling between the $(SN)_x$ fibers. The parallel field appears to be paramagnetically limited. We believe that spin-orbit scattering is significant, despite the small atomic numbers involved, because of severe disorder and boundary effects.

I. INTRODUCTION

A great deal of interest in recent years has focused on the problem of anisotropic superconductivity. Three years ago, the sulfur-nitrogen polymer $(SN)_x$ was found to be a type-II superconductor below 0.3 K,[1] and subsequent studies of its magnetic critical field[2,3] revealed a high degree of anisotropy. Moreover, a study of the resistive transition to the superconducting state[4] indicated that low-dimensional fluctuations are prominent. These and many other aspects of $(SN)_x$ are discussed in several general review articles.[5,6] No generally satisfactory explanation of these phenomena has yet been proposed. The situation is similar with the superconducting layered compounds, in which there has been a more concentrated experimental[7] and theoretical effort.[8]

Attempts have been made to produce chemical analogues of $(SN)_x$,[9] partly with the hope of enhancing the superconductivity. These efforts resulted in the discovery that $(SN)_x$ could be modified by reaction with halogens,[10,11] with the most widely investigated compound to date being $(SNBr_{0.4})_x$ (SNBr, for short). In this paper, we report the first measurements of various superconducting properties of this material.

We undertook these experiments in an effort to understand the nature of the bromination process. That is, we wanted to see if we could tell, by its effect on the superconductivity, where the bromine resides and in what chemical form. Our results indicate that SNBr is a more nearly three-dimensional superconductor than is $(SN)_x$, and that its properties are probably "intrinsic" as opposed to being dominated by the fibrous morphology of the samples. This requires that the bromine be present between the fibers in a form which *increases* the coupling.

II. EXPERIMENTAL DETAILS

Most of the $(SN)_x$ crystals used for these experiments were prepared in our laboratory by methods previously described[12] and their various properties have been discussed elsewhere.[1,5] These samples were taken from the same batches on which earlier studies from our laboratory were based, and had aged somewhat. The crystal surfaces were slightly tarnished. Data were obtained for seven crystals, by means of four-probe conductivity measurements along the highly conducting b crystal axis. The average room-temperature conductivity and resistivity ratio (ρ_{300}/ρ_4) before bromination were $(2.1 \pm 0.7) \times 10^3$ $(\Omega\,\mathrm{cm})^{-1}$ and 18 ± 5, respectively. After bromination, these became $(1.1 \pm 0.3) \times 10^4$ $(\Omega\,\mathrm{cm})^{-1}$ and 8 ± 2. Note that the low-temperature conductivity increased after bromination only by a factor of 2.

Superconductivity data were first obtained on pristine $(SN)_x$ crystals. These were then brominated to $(SNBr_{0.4})_x$ by a method detailed elsewhere[10] and finally were run again. This procedure enabled us to make direct comparisons of the superconducting critical temperature, transition widths, and critical fields on a given crystal both before and after bromination.

All the experiments were run in a commercial helium dilution refrigerator on a copper holder down to 30 mK. The thermometry had an accuracy of ± 2 mK. The actual temperature of the samples could have been different due to various causes, as follows. Since the data were taken by resistance measure-

ments, Joule heating was a possibility, and in fact, the current dependence of the transition temperatures was found to be quadratic. The current was held small enough that the shift was 3 mK or less at a temperature of 30 mK and less than 1 mK at 100 mK; the data were corrected to reflect this effect. No significant change in the shape of the transition as a function of current was observed. Due to the fact that the samples were not perfectly anchored thermally to the thermometers, gradients could be generated during sweeps in temperature. This was checked by taking a series of quasistatic readings and the error introduced was found to be less than 1 mK. Finally, sweeps of magnetic field could induce eddy currents in the copper holder and cause heating. Very low-sweep rates or even static measurements were required to hold this error below 1 mK. We estimate the overall temperature accuracy at ± 3 mK for the data reported here.

The critical-field measurements were taken using a fixed superconducting solenoid capable of producing 60 kOe in the vertical direction with 1% accuracy and an electromagnetic Helmholtz coil arrangement good for 100 Oe with 5% accuracy in any horizontal orientation. Due to the sensitive angular dependence of the critical field close to the highly conducting axis (parallel direction) of the samples and the difficulty in aligning the samples versus the supermagnet to better than a few degrees, it was necessary in attempting a measurement of the parallel field to correct the field orientation with the Helmhotz coils. Even then, only one sample was positioned well enough to permit taking direct parallel data at the very lowest temperatures. For $T \gtrsim 0.5 T_c$, however, the parallel fields were measurable on all the samples. The measurements of field versus angle were taken on this same crystal; the accuracy in angle is estimated as $\pm 0.2°$. In perpendicular fields, where alignment is much less critical, the angular accuracy was $\pm 3°$.

For all the data involving superconducting transitions, we defined the transition point as the value of field or temperature where the resistance reached half its normal value. While this is a questionable definition, as will be discussed later, the qualitative trends of the data, for example, the curvature versus temperature of the perpendicular critical field of pristine $(SN)_x$, are not significantly altered by assuming some other transition point.

III. RESULTS

Representative results of our experiments are depicted in Figs. 1–5. A unified discussion of these data will be given in Sec. IV. The pristine-sample data are all consistent with the earlier work of Azevedo *et al.*[2] and Greene *et al.*[1] Figure 1 shows a typical $(SN)_x$ transition in zero field both before and after bromination. Note the dramatically reduced transition width and lack of a precursive high-temperature tail for SNBr. The relative size of the enhancement in the transition temperature upon bromination depends on how the transition is defined. Sticking strictly to the midpoint yields a figure of 10%. Taking an extrapolation of the steepest transitional slope to zero resistance yields 20%. The choice of definition will be discussed later. No hysteresis was observed in any transitions of any of the samples investigated, whether in zero or finite magnetic field, implying that all are of second order.

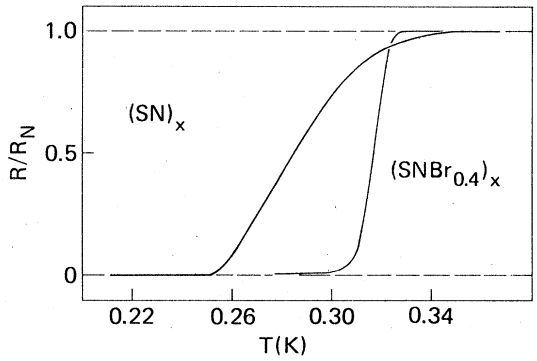

FIG. 1. Normalized four-probe resistive transitions in zero-magnetic field for a single $(SN)_x$ crystal both before and after bromination.

Figure 2 shows the perpendicular critical field of the crystal both before and after bromination. The temperature axis is calibrated in terms of reduced temperature (T/T_c) in order to facilitate comparison of the two curves. Note that the upward curvature of the pristine field has completely vanished upon bromination, yielding a substantial domain where the field is linear with temperature. This striking differ-

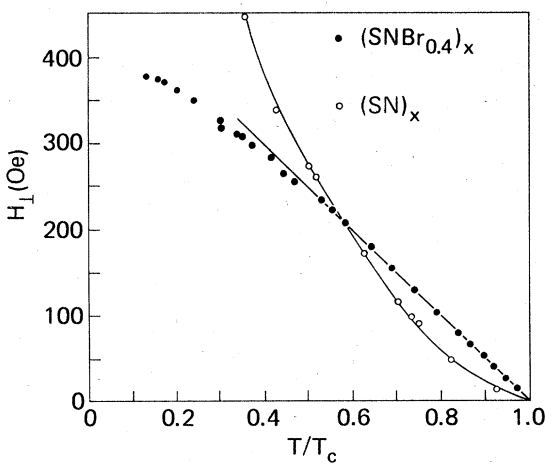

FIG. 2. Critical field oriented perpendicular to fiber axis vs reduced temperature for a single crystal both before and after bromination. Solid lines are visual guides.

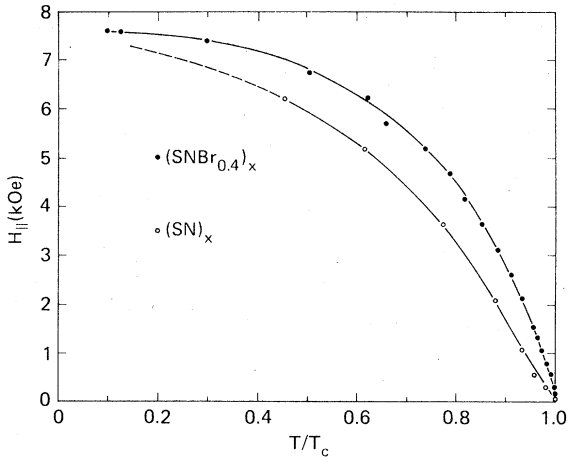

FIG. 3. Parallel critical field vs reduced temperature. Solid lines are visual guides. Dashed line is an extrapolation of data, following that of Azevedo *et al.* (Ref. 2).

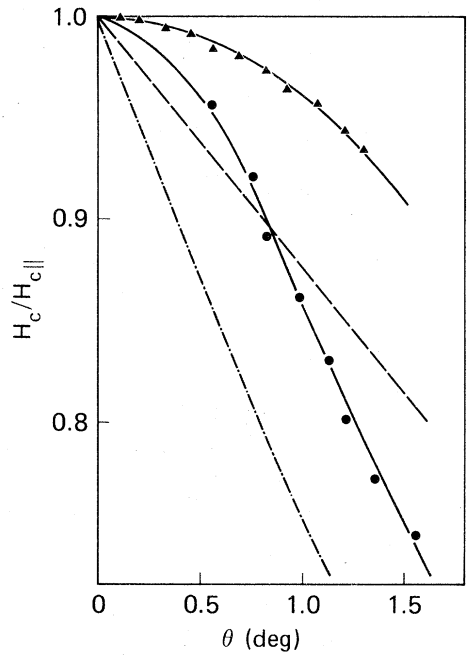

FIG. 4. Normalized critical field of SNBr as a function of small angle with respect to fiber axis. Data taken at 33 mK (circles) and 281 mK (triangles). Solid lines are zero parameter fits to Eq. (1). Dashed and dot-dash lines are the predictions of the thin-film formula at 281 and 33 mK, respectively (see text).

ence in behavior is of signal import in considering dimensionality of the material and will be referred to in our discussion. Numerically, the zero-temperature perpendicular critical field is extrapolated to be about 400 G, about an order of magnitude larger than the thermodynamic critical field for a type-I superconductor with a transition temperature of 0.3 K.

In Fig. 3 is seen the parallel critical field versus reduced temperature, both before and after bromination. The magnitude and qualitative shape of the two curves are similar. The detailed behavior shown near T_c is a characteristic only of this particular sample, and should not be regarded too strictly. Other samples behaved differently, including some with a small region of upward curvature. We believe this to be due to sample imperfections, our operational transition definition, and the extreme sensitivity near T_c of the critical-field magnitude to small angles. The magnitude of the parallel critical field of $(SNBr_{0.4})_x$ at low temperatures averaged about 8 kG. Note that this exceeds the Pauli-limiting field $H_P = 18.4 T_c \approx 5.9$ kOe. In fact, the measured field exceeds the paramagnetic limiting field for all $T < T_c$.

In Fig. 4, we have plotted the variation of the critical field of a SNBr crystal versus small values of the polar angle θ at 33 mK and at 281 mK. The data at 281 mK is incomplete and thus the value of $H_{c\|}$ was determined with an error of 3 or 4%. In order to plot the data in the format chosen, we assumed a particular value (3480 Oe) for $H_{c\|}$; the error involved in this procedure is too small to compromise our basic conclusions. The solid and dashed curves represent theoretical models to be discussed later. We also measured the azimuthal dependence of the perpendicular critical field and found it to vary smoothly, with two maxima in a 360° rotation and a peak-to-peak amplitude variation of less than 20%. This small azimuthal anisotropy shall be neglected in our discussion of the data: we shall assume axial symmetry. Information on the orientation of these maxima with respect to the crystal symmetry directions was not obtained.

Finally, in Fig. 5, we have plotted a normal-state property, the perpendicular resistivity, from 10 to 300 K both before and after bromination. These data are

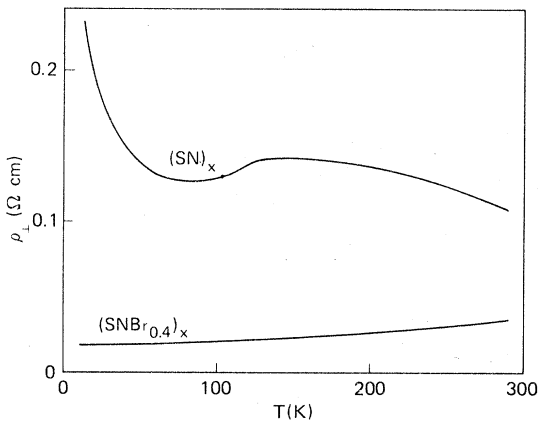

FIG. 5. Perpendicular normal dc resistivity vs temperature for a single crystal both before and after bromination.

included because they are relevant to the thesis of this work, supporting a model of increased coupling between fibers upon bromination. The pristine data have been reported previously[13,14] but this is the first time a continuous curve for SNBr has been obtained.[15] We used both four-probe and Montgomery[16] configurations in taking these data and they were in agreement. The room-temperature conductivity was enhanced by a factor of 3 upon bromination, and was 12 times better at 10 K; moreover, the brominated material showed a metallic temperature dependence, albeit with a very low resistivity ratio of about 2.

IV. DISCUSSION

From the experimental data, it is apparent that the bromination process radically alters the superconducting properties of $(SN)_x$. Whereas pristine $(SN)_x$ appears to be of low dimensionality but is not well described by any model, SNBr can be treated by anisotropic three-dimensional Ginzburg-Landau (GL) theory. In order to show this, we shall consider each piece of data in turn.

The resistive transition in pristine (SN_x) was investigated by Civiak *et al.*[4] They concluded that zero- or one-dimensional fluctuations of the type predicted by Azlamazov-Larkin theory are consistent with the data. There has been some doubt of this result among investigators in the field,[17] since the fits are restricted in range. An improvement has been obtained by an analysis[18] which does not presume a value for T_c, which was taken at the transition midpoint in Ref. 4, or for the dimensionality of the system. In fact, T_c obtained as a product of this treatment is seen to fall well below the midpoint close to the intersection of the extrapolated line of maximum slope with the line of zero resistance. This definition lowers T_c by about 10%. The high-T precursive tail is a signature of the low-dimensional transition. That this tail is missing in the SNBr data is an indication of increased dimensionality along with the overall sharpening. Then what is responsible for the width that remains after bromination? Sample imperfections are undoubtedly responsible. SNBr is known to be significantly more disordered than $(SN)_x$.[10,11] In fact, whereas the crystal structure of $(SN)_x$ has been determined, the severe disorder has thwarted similar attempts on SNBr. We conclude, therefore, that the wider transition in pristine $(SN)_x$ is almost completely due to low-dimensional fluctuations and not to sample disorder or inhomogeneities, since these effects must be amplified rather than reduced upon bromination.

The enhancement in T_c is also consistent with this interpretation. Fluctuations would be expected to reduce T_c from the mean-field value.[19] Indeed, it would seem that there should be very little fluctuation-induced depression of T_c in SNBr. However, there has been some indication in the literature that the mean-field transition temperature T_{c0} in pristine $(SN)_x$ may even exceed 1 K.[4,20] If this is the case, then either bromination depresses T_{c0} or some other as yet undetermined pair-breaking mechanism is present in both $(SN)_x$ and SNBr.

The most striking confirmation of increased dimensionality arises from the perpendicular critical field $H_{c\perp}$. Pristine $(SN)_x$ shows a persistent upward curvature also found in the two-dimensional (2-D) superconductors.[7] This has completely disappeared in SNBr and is replaced by a highly linear behavior, which has been known to occur only for a field perpendicular to a film or for a 3-D superconductor. Both H_c and the polar angle dependence of H_c in SNBr are most consistent with the 3-D model.

Consider a system which can be described as an anisotropic 3-D superconductor over some temperature region close to T_c. Suppose for simplicity that the system is axially symmetric. Then GL theory[21] predicts

$$\left(\frac{H\cos\theta}{H_{\parallel}}\right)^2+\left(\frac{H\sin\theta}{H_{\perp}}\right)^2=1 \quad . \tag{1}$$

This assumes that only orbital pair breakers are active. In Eq. (1), $H_{\parallel}=\phi_0/2\pi\xi_{\perp}$,[2] and $H_{\perp}=\phi_0/2\pi\xi_{\parallel}\xi_{\perp}$, where $\phi_0=2\times10^{-7}$ Oe cm^2, $\xi_{\parallel}$ is the GL coherence length along the high-conductivity b axis, and $\xi_{\perp}$ is the (average) coherence length in the transverse direction. The GL coherence length may be expressed as $\xi=\xi(0)(1-t)^{-1/2}$ where $t\equiv T/T_c$. The critical field is therefore linear in temperature near T_c with derivatives

$$\frac{H_{\parallel}}{dT}=\frac{\phi_0}{2\pi\xi_{\perp}^2(0)\,T_c} \quad , \qquad \frac{dH_{\perp}}{dT}=\frac{\phi_0}{2\pi\xi_{\parallel}(0)\,\xi_{\perp}(0)\,T_c} \quad . \tag{2}$$

Using Eqs. (2) and the slopes from Figs. 2 and 3 we can estimate the coherence lengths, obtaining $\xi_{\parallel}(0)\approx2000$ Å and $\xi_{\perp}(0)\approx100$ Å for SNBr. Since $H_{c\perp}$ for $(SN)_x$ does not show a clear linear region, the applicability of 3-D GL theory is questionable. However, following Ref. 2, we arbitrarily choose a slope and come up with $\xi_{\parallel}(0)\approx3000$ Å and $\xi_{\perp}(0)\approx140$ Å.

An independent estimate of these parameters can be obtained on the basis of microscopic theory. Noting that the mean free path $l\ll\xi_0$ for both materials in both directions,[1,2] we can use a dirty-limit formula relating the coherence length to the heat capacity, normal resistivity, and critical temperature[22]: $\xi_i(0)\approx10^{-6}\,(\rho_i\gamma T_c)^{-1/2}$ in cgs units. Using[23] $\gamma\approx400$ erg cm^{-3} K^{-2}, we obtain $\xi_{\parallel}(0)\approx2500$ Å and $\xi_{\perp}(0)\approx65$ Å for SNBr, $\xi_{\parallel}(0)\approx2000$ Å and

$\xi_\perp(0) \approx 20$ Å for $(SN)_x$. There is good agreement between these values and the ones above in the case of SNBr, but not $(SN)_x$. We therefore conclude that SNBr is a 3-D superconductor, while $(SN)_x$ is not.

What are the origins of anisotropy in either material and why is one different from the other? We have not mentioned until now the fibrous morphology of the samples. The fibers in $(SN)_x$ average 60–70 Å in diameter, but are reduced in size to 20–30 Å upon bromination.[10] This morphology has already been shown to affect the properties of $(SN)_x$, including the critical field.[5] The same possibility must be considered for SNBr. In such a situation, the properties could be considered to be extrinsically determined and therefore would not contain information on the intrinsic nature of the material. By the term intrinsic, we mean these effects are not dominated by nonmetallic transport between fibers; intrafiber disorder scattering is therefore considered to be "intrinsic."

Consider first the situation in $(SN)_x$. There the fiber diameter, d, easily exceeds the "microscopic" $\xi_\perp$. In the case $d \gg \xi_\perp$, we would expect effects at the fiber boundaries to be insignificant and the critical field to be intrinsic. While the band structure is anisotropic and can produce an anisotropic critical field,[1] it must be rejected as the explanation here: The band structure is not 1-D but 3-D, so the superconducting properties should be 3-D. But we have seen that low-dimensional fluctuations dominate the $(SN)_x$ transition. Moreover, not only does $H_{c\perp}$ fail to be linear with temperature near T_c as predicted by 3-D GL theory, but the angular dependence of Eq. (1) is not followed.[2] We are therefore forced to conclude that the intrinsic $\xi_\perp > d$, but that fiber boundary effects determine the observed value. This idea is supported by the fact that $\xi_\perp$ calculated from the resistivity is 20 Å, much less than a fiber diameter, whereas $\xi_\perp$ from $H_{c\parallel}$ is 140 Å. On the other hand, the $(SN)_x$ behavior is not that of completely isolated fibers. Dirty-limit GL theory for a single fiber with intrinsic isotropy and $\xi \gg d$ predicts[24]

$$\left(\frac{H}{H_\parallel}\right)^2 (\cos^2\theta + 2\sin^2\theta) = 1 , \tag{3}$$

where $H_\parallel = 2\sqrt{2}\phi_0/\pi d\xi(T)$. This predicts $H_\parallel/H_\perp = \sqrt{2}$ at all temperatures and $H_c \sim (1-t)^{1/2}$, neither of which is close to matching the $(SN)_x$ data. It was found in Ref. 2, in fact, that the angular dependence of H_c for $(SN)_x$ well below T_c fits the Tinkham thin-film formula[25] (see Fig. 4). But the resistive transition in $(SN)_x$ does not fit the expected two-dimensional pattern[4] and the temperature dependences of the critical fields do not follow those predicted for a thin film. It is apparent that $(SN)_x$ must be treated in a more sophisticated manner. Josephson coupling between fibers has been postulated,[26] but the theoretical models are still incomplete and somewhat unsatisfactory. In particular, the upward curvature of $H_\perp$ and the angular dependence have been treated only qualitatively.

The situation in SNBr is entirely different, since 3-D GL theory seems to be valid: The resistive transition is not fluctuation dominated; the angular dependence in Fig. 4 follows Eq. (1); and the critical-field temperature dependence near T_c is linear. Note that $\xi_\perp$ is much larger than a fiber diameter, indicating that fiber boundaries are less important than in $(SN)_x$ and that the properties may even be intrinsically determined. If this is the case, the intrinsic anisotropy is given by $\xi_\parallel/\xi_\perp \approx 60$. This value is not unreasonable, considering the Fermi-velocity anisotropy $v_{f\parallel}/v_{f\perp} \approx 9$ of $(SN)_x$, and the much higher degree of disorder in the transverse direction, especially in SNBr, so that $l_\parallel > l_\perp$. Indeed, it is possible that SNBr is intrinsically more anisotropic than $(SN)_x$ but extrinsically less so. In any case, the fibers appear to be much more strongly coupled in SNBr than in $(SN)_x$. This is also indicated by the change in $\rho_\perp$ upon bromination shown in Fig. 5 and by the Meissner-effect experiments of Dee *et al.*,[3,27] which show a nearly complete diamagnetism in SNBr and only a fractional effect in the pristine material.

It should be noted that we are discussing in this paper "upper" critical fields. $(SN)_x$ and SNBr are type-II materials with partial Meissner effects. We have abstained from labeling fields as "H_{c2}" for two reasons: Firstly, the distinction becomes blurred when size effects are significant. Secondly, there is a possibility that H_{c3} would be a more appropriate label for the parallel field, i.e., surface effects could be important. But all this is speculative at this point.

We must now confront the fact that $H_\parallel(0)$ in *both* $(SN)_x$ and SNBr exceeds the Pauli-limiting field[28] $H_{Po} = 18.4 T_c$ (kOe) by about 50%. Indeed, the paramagnetic limit is surpassed in *both* cases for all $T < T_c$. Further, while pure paramagnetic pairbreaking should result in a first-order transition, the lack of hysteresis in our data strongly implies that the observed transitions are second order. Therefore something is reducing the strength of the paramagnetic pair-breaking effect. In the case of very dirty superconductors such as we have here, spin-orbit scattering usually has been found to be the responsible agent.[29] If the interaction is strong enough, it would explain both the order of the transitions and the magnitude of the parallel critical field. The atomic numbers of S (16), N (7), and Br (35) are very low, however, and one ordinarily would assume spin-orbit scattering to be inconsequential for such small values of Z on the basis of experience with typical metallic alloy superconductors.[30]

The difference here is that the heavy structural disorder in the perpendicular direction (the fiber boundaries) leads to very high-scattering rates compared to most alloys. This in turn enhances the spin-orbit

term for a given value of Z. Consider SNBr. From optical data and dc conductivity at low temperature[5] $\tau_\perp \approx 10^{-14}$ sec. Assuming that most of the spin-orbit scattering occurs on bromine and that[30] $(\tau/\tau_{so}) \approx Z/137$,[4] we obtain $\tau_{so}^{-1} \approx 4 \times 10^{11}$ sec^{-1}. This actually compares quite well with the rate necessary to produce the observed critical-field enhancement[8]:

$$\tau_{so}^{-1} \approx 4 \times 10^{11} T_c \left(\frac{H_c}{H_{Po}}\right)^2 \text{ sec}^{-1} \quad . \tag{4}$$

Applying $T_c = 0.31$ K, $H_{Po} = 5.7$ kOe, and $H_c = 7.5$ kOe to Eq. (4), we have $\tau_{so}^{-1} \approx 2 \times 10^{11}$ sec^{-1}!

The situation in $(SN)_x$ requires a slightly different analysis, because one based on the experimental[5] $\tau_\perp \approx 10^{-16}$ sec may not be valid; the interfiber nonmetallic transport could be anomalously affecting this number. A more confident approximation is obtained by dividing the calculated average Fermi velocity of 10^8 cm/sec into the scale over which perpendicular structural disorder occurs, say 10 Å. Then $\tau_\perp \sim 10^{-15}$ sec and, assuming sulfur sites dominate the spin-orbit scattering $\tau_{so}^{-1} \approx 2 \times 10^{11}$ sec^{-1} again! We do not mean to imply that these numbers are actually so accurate, but they do indicate that our hypothesis is quite reasonable.

V. CONCLUSIONS

In conclusion, our measurements of T_c, ΔT_c, $H_{c\parallel}$, $H_{c\perp}$, and $\rho_\perp$ indicate that SNBr is a three-dimensionally coupled superconductor while $(SN)_x$ is not. This statement is supported by the results of numerous other experiments as well. The reason for the increased dimensionality is an improved metallic coupling between fibers. We do not know how the inclusion of bromine achieves this result, but it is reasonable to suppose that at least some of the bromine must lie between fibers and that the valence states on the bromine overlap significantly with the itinerant $(SN)_x$ states. This excludes the possibilty of many Br_2 molecules lying between the fibers,[7] but is consistent with the hypothesis of linear asymetric Br_3^- ions residing in these regions.[31]

We have also shown that spin-orbit scattering accounts for the fact that the Pauli limit is exceeded in both $(SN)_x$ and SNBr. Furthermore, the temperature dependence of the critical-field anisotropy is consistent with orbital pair breaking close to T_c for fields in either direction, but with the parallel critical field becoming paramagnetically limited (including spin orbit) as $T \to 0$. However, a detailed calculation of a critical field under these conditions remains to be done. Also, the question of whether surface effects are important for $H_{\parallel}$ must be considered.

Finally, our data indicates that while $(SN)_x$ is not 3-D, neither is it a simple 1-D or 2-D system. It appears that there may be dimensional cross over as a function of temperature.[26] This is analogous to the behavior of the layered superconductors,[7] but a detailed theory satisfactorily fitting the data has yet to be presented.

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