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Superconductivity and magnetic ordering in $Bi_3Sr_{1-x}Eu_x$

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We have studied the magnetic and superconducting properties of the system $Bi_3Sr_{1-x}Eu_x$ ($0 \leq x \leq 1$). Bi_3Sr is a strong-coupling superconductor ($T_c = 5.68$ K; $\lambda \approx 1.6$) and Bi_3Eu orders antiferromagnetically at 7.8 K. In the solid solution of the two binaries we found a region where coexistence of superconductivity and a spin-glass-type magnetism may exist at low temperature.

I. INTRODUCTION

During the past 20 years quite a number of investigations have been published on the possibility of a coexistence of superconductivity and magnetism.[1,2] The problem has generally been approached by studying the properties of a solid solution of a magnetic and a superconducting compound.

Usually, the transition temperature of a superconductor is quite drastically lowered by small amounts of magnetic ions.[3] If the interaction between magnetic ions is neglected and in cases where crystal-field effects are of no significance, this lowering of the transition temperature is calculated by means of the theory of Abrikosov and Gor'kov (AG).[4]

In order to have a reasonable chance to observe such a coexistence, the interaction between the magnetic ions and the conduction electrons should be weak. This is generally the case for rare-earth ions. Expecially suited for such an investigation are the Gd^{3+} or Eu^{2+} ions since the magnetic moments of these ions are usually quite stable and localized, and because (on account of their *S* ground state) splittings due to the cubic crystal field of the superconducting matrix can be neglected.

Up to now, mainly Gd^{3+} ions have been investigated, while, as far as we know, Eu^{2+} as a magnetic ion has rarely been the subject of research yet: La:Eu,[5,6] $LaSn_3$:Eu,[7] and $SnMo_6S_8$:Eu.[8] In the first two cases Eu^{2+} also lowers the transition point strongly [≈2 K/(at.% Eu) for the lanthanum matrix and ≈0.5 K/(at.% Eu) for $LaSn_3$]. However, in these cases the Eu^{2+} replaces a formal trivalent ion (La^{3+}) so that additional screening charges around the magnetic ion will occur which might perhaps influence the spin-exchange scattering between the conduction electrons and the 4*f* electrons of the magnetic impurity.

In the third case there are no such difficulties because Sn is in the divalent state. However, the density of *d* states on the Eu site is very low here, so T_c is scarcely influenced and no long-range magnetic ordering has been found.

To avoid the screening difficulties we choose a superconducting matrix with a bivalent ion: Bi_3Sr ($AuCu_3$ type). The superconductivity of the pure intermetallic compound was briefly mentioned in Ref. 9 with a transition temperature of $T_c = 5.62$ K, a result which was confirmed by our samples. There is little difference between the ion radii of Sr^{2+} and Eu^{2+}, so that stable Eu^{2+} can be expected in this compound. Moreover, in the course of our investigations we found that the system $Bi_3Sr_{1-x}Eu_x$ (at least at room temperature) retains its crystal structure in the entire range $0 \leq x \leq 1$, and that the lattice constant changes only by 1%.

The goal of our research was to study the superconducting properties of Bi_3Sr and the magnetic properties of Bi_3Eu in order to investigate the solid solution of the two and look for a possible coexistence of the two phenomena.

In the following we report on the sample preparation and on our investigations of the magnetic, superconducting, and caloric behavior of this system.

II. SAMPLE PREPARATION

Samples of about 5 g of the substances $Bi_3Sr_{1-x}Eu_x$ were melted in an arc furnace under 400-Torr argon (99.999%, chemically purified) on a water-cooled Cu plate, and homogenized. This was possible without any particular disturbance of the weighed-in stoichiometry. The purity of the original substances used was: 99.999% for Bi (Balzers), 99.9% for Eu (Metals Research) and 99% for Sr (Metals Research). Melting together Bi with the alloy $Sr_{1-x}Eu_x$, corresponding to the chosen concentration, caused a strong chemical reaction and sometimes small particles of the sample were flung away; very careful handling of the melting process, therefore, was necessary. For all samples mentioned here, the total loss of weight was always less than 1%, so the errors in concentration can be assumed to be of no significance. Debye-Scherrer diagrams at $T = 290$ K showed Cu_3Au structure over the entire range $0 \leq x \leq 1$. The lattice constant decreases monotonically from Bi_3Sr to Bi_3Eu which indicates the complete mixing capability of the substances. Weak additional lines in the Debye-Scherrer diagram, assigned to free Bi, are probably caused by oxidation of the sample surface.

For our conductivity measurements we used rods ($1.2 \times 1.2 \times 12$ mm^3) cut from the interior of the melted sample and mechanically polished. Similar but smaller rods were used for the magnetization measurements. The specific heat, finally, was determined from a bigger sample (≈12 g).

Because they corrode relatively fast, all samples were stored in evacuated glass ampoules after their preparation until the actual measurements were performed.

III. EXPERIMENTAL RESULTS

A. Measurements of the magnetic susceptibility

Magnetization measurements of the samples took place in a vibration magnetometer of the Foner type[10] in the temperature range from 2 to 300 K, applying an external field intensity from 0.1 to 1 T. In each case this field was strong enough to destroy the superconductivity. Undoped Bi_3Sr is diamagnetic in the above temperature range (Fig. 1).

The susceptibility passes a minimum at $T \approx 50$ K and increases strongly towards low temperatures. We believe this strong increase to be due to iron impurities in the strontium. By subtraction of a

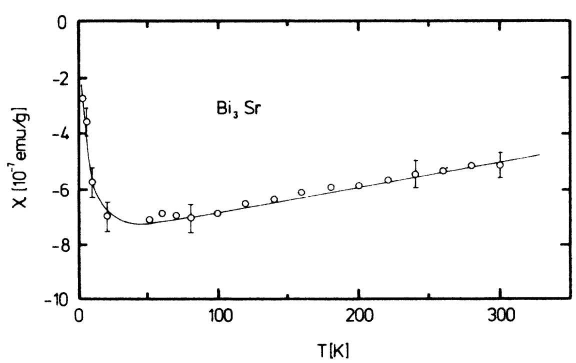

FIG. 1. Magnetic susceptibility vs temperature for Bi_3Sr.

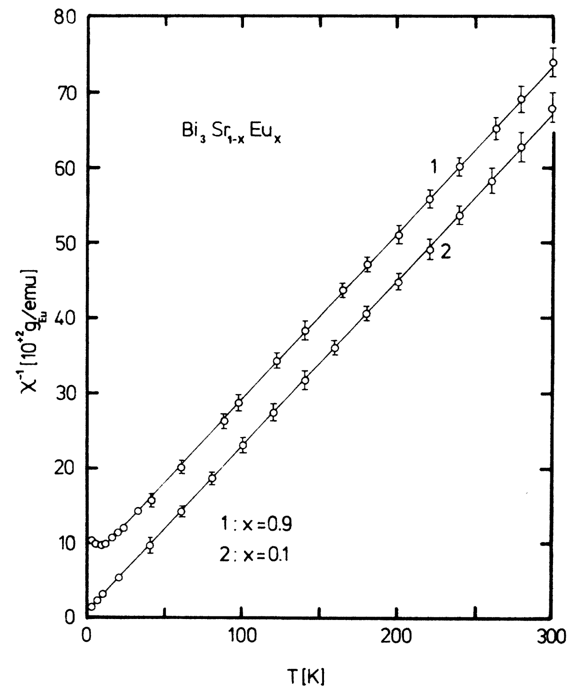

FIG. 2. Inverse susceptibility per gram Eu vs temperature showing a Curie-Weiss-law for two representatives of the solid solution $Bi_3Sr_{1-x}Eu_x$ ($x = 0.1$ and $x = 0.9$).

suitable paramagnetic Curie susceptibility $\chi = C/T$ the low-temperature increase can just be compensated. To this correction then correspond ≈890 at. ppm Fe^{3+} in strontium which is in good agreement with the producer's specifications. The thus corrected susceptibility of the undoped Bi_3Sr remains diamagnetic in the entire temperature range, even after subtraction of the core susceptibilities.[11] It was therefore not possible to determine a value of the conduction electrons' Pauli susceptibility.

All susceptibility values of the Eu-doped samples were corrected for diamagnetism of the undoped sample. At high temperatures all corrected susceptibilities follow a Curie-Weiss law with negative Θ_p. In the entire concentration range $0 \leq x \leq 1$ the Θ_p values can be described by the following linear relation: $\Theta_p = -36x$ K. The straight lines $\chi^{-1} = f(t)$ run parallel (Fig. 2) and provide the same magnetic moment per Eu ion for all concentrations: $\mu = (7.8 \pm 0.2)\mu_B$ for samples annealed at 500 °C in vacuum for one week. Unannealed samples yielded about 6% smaller μ values. We think this difference is due to a certain amount of Eu^{3+} present in the unannealed samples. Only europium-rich samples yield, at low temperatures, characteristic deviations from a Curie-Weiss law which we attribute to the onset of a long-range magnetic or-

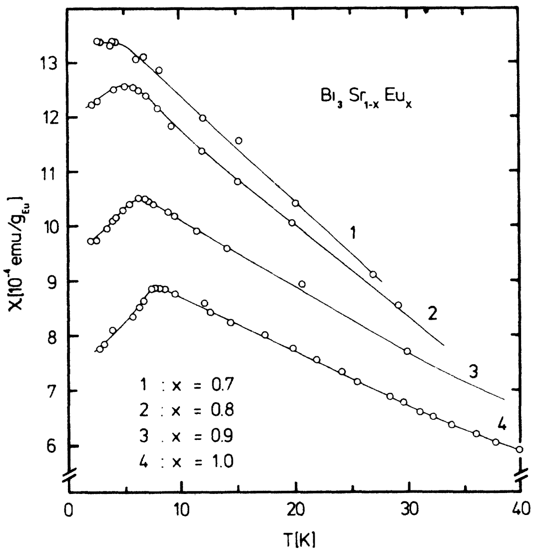

FIG. 3. Susceptibility per gram Eu vs temperature for $Bi_3Sr_{1-x}Eu_x$ ($x = 0.7$, 0.8, 0.9, and 1).

dering of antiferromagnetic nature (Fig. 2). If it is really a matter of simple antiferromagnetic ordering of the Eu ions this uncertainty will be cleared up by neutron-diffraction investigations in the near future. In Fig. 3 the low temperature behavior of χ is shown for several samples. We denote the temperatures where the susceptibility has a maximum by T_M.

B. Measurements of the superconducting transition temperature T_C

The T_c values were determined from the curve of the dc electrical resistance $\rho = \rho(T)$. In some cases these values were verified by ac measurement, by measuring the temperature dependence of the self-induction of a coil containing the sample. Both methods yielded the same T_c values. In most samples the T_c points are clearly defined ($\Delta T_c \approx 0.2$ K for 90%–10% change of the ρ values); only samples with high europium concentration ($0.3 < x < 0.4$) show somewhat larger ΔT_c ranges (up to 0.7 K). The above mentioned annealing process influences T_c strongly. The T_c values of the annealed samples with $x = 0.3$ are 2 K lower than those of the unannealed sample. Here, too, Eu^{3+} without a total magnetic moment seems to have been present before the annealing process. The heat treatment has no influence on T_c in undoped Bi_3Sr. The observed T_c lowering amounts in the range of smaller Eu concentrations ($x \geq 0.1$), to -0.28 K/(at.% Eu) and, thus, is surprisingly small. An Abrikosov-Gor'kov curve, fitted to this value, yields the critical concentration where superconductivity no longer should occur at the value $x_{cr} = 0.53$. The transition temperatures for the samples with $x > 0.1$, however, are clearly below the AG curve. Table I lists the measured transition temperatures T_c, the corresponding ΔT_c ranges and the T_c values expected according to an AG curve.

At 1.4 K, the lowest temperature for transition point measurement within reach of our equipment, the sample $Bi_3Sr_{0.6}Eu_{0.4}$ no longer became superconducting.

C. Resistivity measurements

The measurements of the specific resistivity of Bi_3Sr samples show a large ratio of $\rho(300\text{ K})/\rho(10\text{ K})$ of about 235. The residual resistivity at 10 K is about 0.3 $\mu\Omega$ cm, which is very low for an intermetallic compound. The resistance of Eu-rich samples shows a break in the $\rho(T)$ curve (Fig. 4). The temperatures belonging to this break correspond exactly to the magnetic ordering temperatures T_M from Sec. II. For the sample with $x = 0.7$, where no peak could be determined in the susceptibility (Fig. 3) the $\rho(T)$, however, showed a clear break and a corresponding T_M could be ascertained. All measurements were done by an ordinary four-point method.

D. Specific heat

The specific heat C was measured on samples having the concentrations $x = 0$, 0.3, 0.5, and 1. The samples were measured both as cast and after a heat treatment of 200 h at 500 °C in evacuated quartz ampoules. The annealed samples had T_c

TABLE I. Superconducting critical temperatures T_c in the series $Bi_3Sr_{1-x}Eu_x$. ΔT_c indicates the width of the resistivity transition. For a comparison we give the critical temperatures calculated from AG theory.

X	0	0.025	0.05	0.1	0.2	0.3	0.35
T_c (K) (expt.)	5.68	5.54	5.34	5.00	3.94	2.56	1.60
ΔT_c (K)	0.05	0.07	0.1	0.2	0.4	0.6	
T_c (K) (AG theory)	5.68	5.54	5.34	5.02	4.19	3.35	2.90

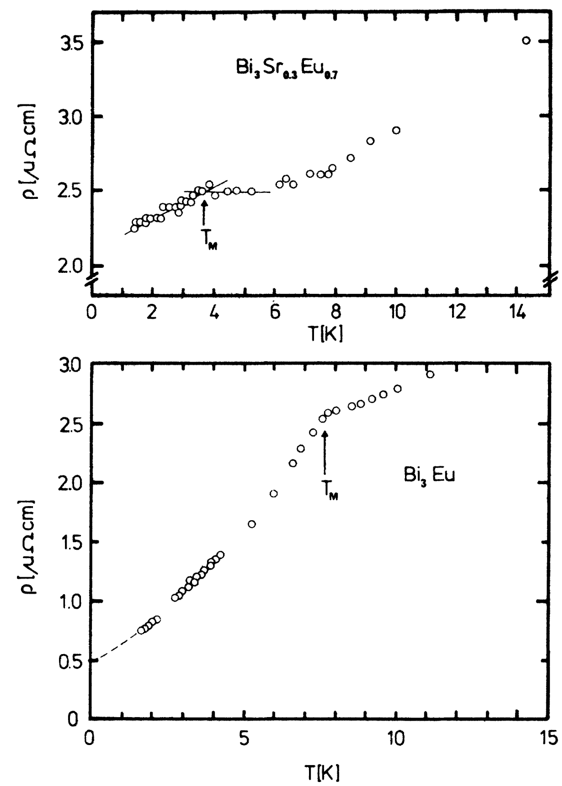

FIG. 4. Resistivity vs temperature for Bi_3Sr and $Bi_3Sr_{0.3}Eu_{0.7}$. T_M shows the magnetic ordering of the samples.

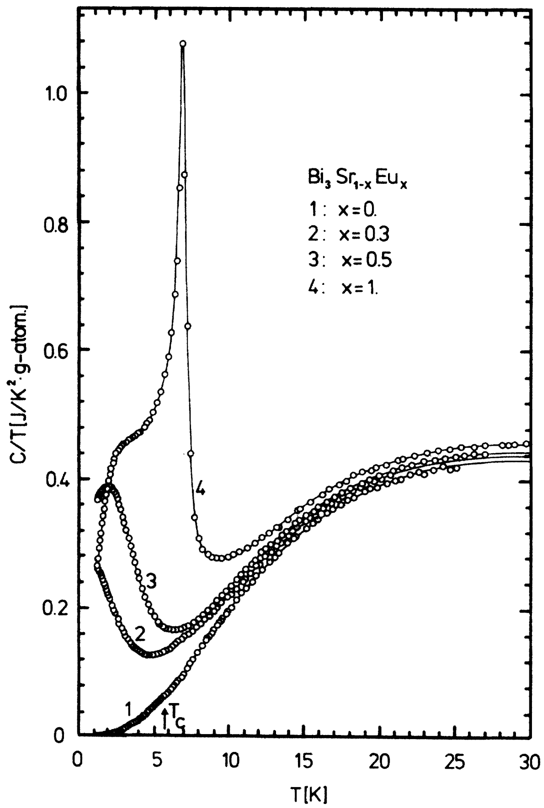

FIG. 5. Specific heat for $Bi_3Sr_{1-x}Eu_x$ ($x=0$, 0.3, 0.5, and 1). T_c shows the superconducting transition in Bi_3Sr.

values of 5.68, 3.7, and 1.45 K for $x=0$, 0.3, and 0.5, respectively. These values are higher than the ones for the corresponding small samples in Table I and indicate a higher concentration of Eu^{3+} ions. The measurements were performed between 1.2 and 30 K using a heat-pulse calorimeter.[12] The results for the annealed samples are shown in Fig. 5.

Due to the strong lattice and magnetic contributions to C, the superconducting transition could only be seen in the Bi_3Sr sample (T_c).

To analyze the normal specific heat, we write in the usual way,

$$C=\gamma T+C_D. \quad (1)$$

C_D is the lattice specific heat assuming a Debye spectrum with a temperature-dependent $\Theta_D(T)$. γ was determined using the condition that the entropy change between T_c and 0 K must be the same in the superconducting and in the normal state and $\Theta_D(0)$ was determined from the slope of the low-temperature specific heat ($T \ll T_c$). Thus we obtain

$$\gamma=0.9\pm0.1\ \mathrm{mJ/K^2\,g\text{-}atom},$$

$$\Theta_D(0)=115\pm7\ \mathrm{K}.$$

The jump in the specific heat ΔC at T_c is

$$\Delta C/\gamma T_c=2.7\pm0.5.$$

The function $\Theta(T)$ obtained using Eq. (1) is shown in Fig. 6. From the temperature dependence of Θ

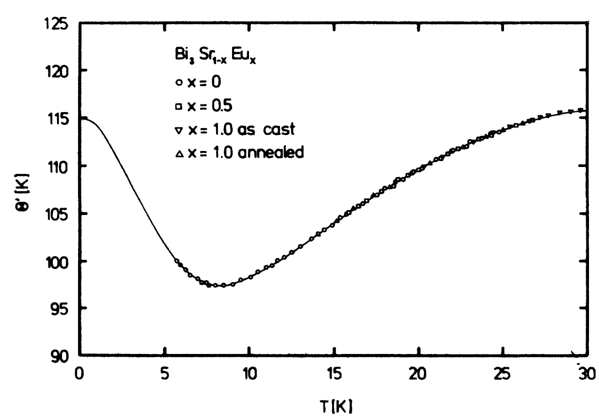

FIG. 6. Effective Debye temperature Θ' (corrected for mass change) vs temperature for $Bi_3Sr_{1-x}Eu_x$ ($x=0$, 0.5, and 1).

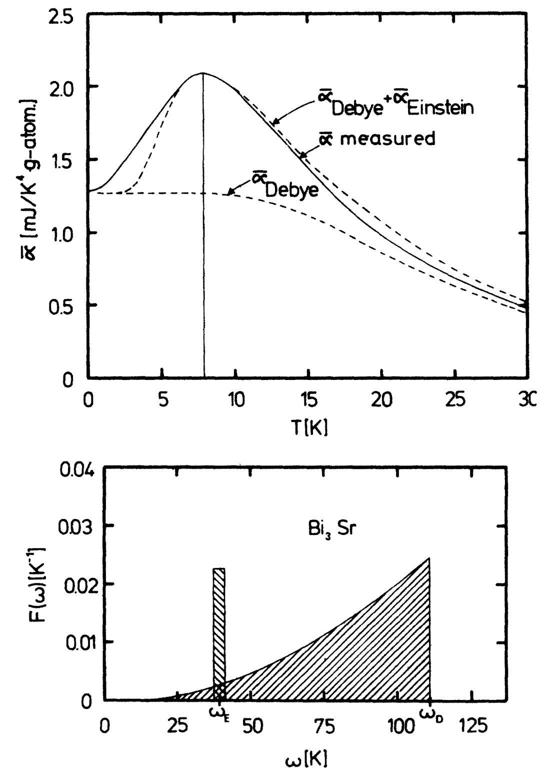

FIG. 7. (a) Measured and calculated mean slope $\overline{\alpha} = (1/T^2)(C/T - \gamma)$. (b) Tentative model for the phonon density of states of Bi_3Sr.

it is possible to construct a crude model for the phonon density of states $F(\omega)$. Junod *et al.*[13] have shown that such a minimum in $\Theta(T)$ is characteristic of an Einstein peak in $F(\omega)$. From the position and the depth of the minimum in $\Theta(T)$, the position $\omega_E = 39$ K and the relative weight $D = 0.1$ of the Einstein peak can be determined. This leaves us with a Debye frequency $\omega_D = 111$ K.

The function $F(\omega)$ is then assumed to have the form

$$F(\omega)=\frac{D}{1+D}\left(\delta(\omega-\omega_E)+\frac{3}{D}\frac{\omega^2}{\omega_D^3}\right), \quad (2)$$

and the specific heat becomes

$$C=\gamma T+\frac{1}{1+D}\left[C_D\left(\frac{\omega_D}{T}\right)+DC_E\left(\frac{\omega_E}{T}\right)\right]. \quad (3)$$

In Fig. 7 are shown the calculated [using Eq. (3)] and measured values of $\overline{\alpha} = (1/T^2)[(C/T) - \gamma]$ [$\overline{\alpha} \sim 1/\Theta_D^3(T)$ in the domain where the T^3 law is valid]. We see that the assumed form of $F(\omega)$ gives a reasonable fit to the experimental results. The small discrepancy that remains is due to the final width of the peak, neglected in Eq. (2). A pure Debye spectrum does not give a maximum in $\overline{\alpha}$. We should point out here that this analysis shows clearly the existence of a peak in the phonon density of states at $\omega \approx 39$ K and it also gives with a good accuracy its relative weight. However the peak at ω_D is artificial and the form of $F(\omega)$ at high ω may be quite different. For comparison we note that pure Bi has a first peak in the phonon density of states at $\omega \approx 40$ K and a second peak at about $\omega \approx 150$ K.[14]

On the other hand, Pb, which has quite similar superconducting properties to Bi_3Sr, has two peaks in the phonon density of states roughly at the same position as in our model for Bi_3Sr.[15]

In the samples containing Eu we see a magnetic transition, characterized by the maximum in $C(T)$ for $x = 0.5$ and $x = 1.0$ (Fig. 5). The corresponding transition temperatures are 2 and 6.9 K, respectively. These values correspond reasonably well with the transitions seen in resistivity and susceptibility.

The small differences are probably due to the non-negligible concentrations of Eu^{3+} ions in the large samples used for specific heat measurements. The apparent coexistence of superconductivity and

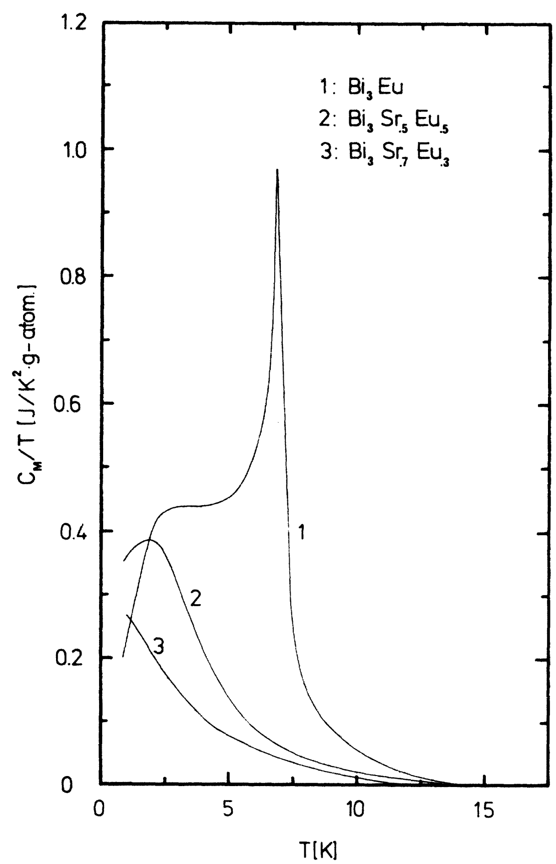

FIG. 8. Magnetic contribution to the specific heat of the annealed $Bi_3Sr_{1-x}Eu_x$ samples ($x \approx 1$, 0.5, and 0.3).

TABLE II. Comparison between measured and calculated entropy of the system $Bi_3Sr_{1-x}Eu_x$ ($x = 0.3$, 0.5, and 1).

	T_M (K)	measured entropy	calculated entropy
$Bi_3Sr_{0.7}Eu_{0.3}$	<1.2	1.06 ± 0.08	1.30
$Bi_3Sr_{0.5}Eu_{0.5}$	2.0	1.80 ± 0.02	2.16
Bi_3Eu (annealed)	6.9	3.60 ± 0.03	4.32
Bi_3Eu (as cast)	6.9	3.28 ± 0.03	4.32

magnetic order in the $x = 0.5$ sample is probably due to inhomogeneities in the distribution of the Eu^{3+} ions in connection with internal strains which in those samples are difficult to remove. To find the magnetic contribution to the specific heat we have to subtract the lattice (and conduction electron) specific heat. As can be seen in Fig. 5, there is a change in this lattice contribution as the concentration of Eu is increased. This change corresponds, however, just to a variation in the Debye temperature due to the change of atomic mass.

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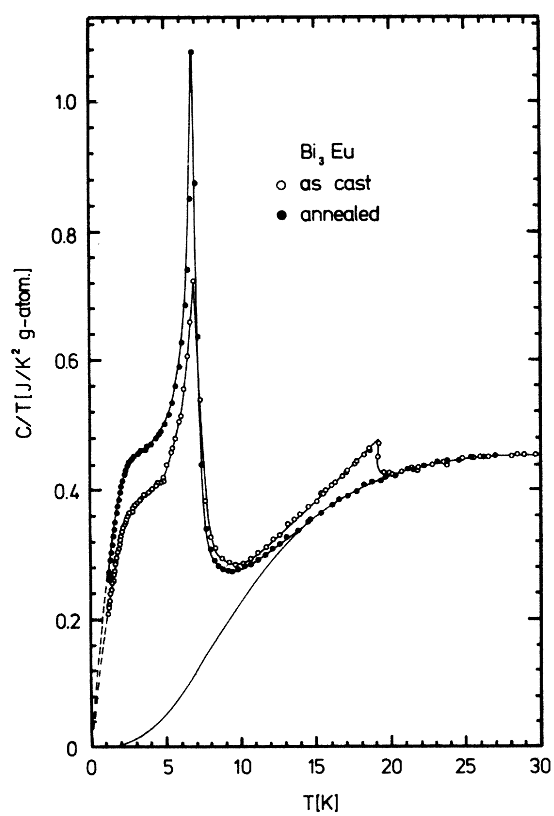

FIG. 9. Comparison of the specific heat of "as cast" and annealed Bi_3Eu samples.

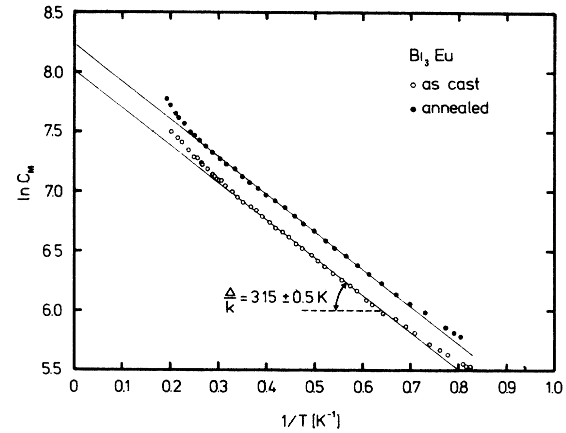

FIG. 10. Logarithmic plot of C_M vs $1/T$ for Bi_3Eu at low temperature (as cast and annealed samples).

$$\Theta'_D(T) = (M/M')^{1/2}\Theta_D(T) , \quad (4)$$

values of Θ'_D for the different samples (calculated in the temperature region where the magnetic contributions are negligible) become identical (see Fig. 6). We therefore take as lattice contribution the specific heat of Bi_3Sr with the Debye temperature corrected by relation (4). The magnetic contributions to C/T are shown in Fig. 8.

In Table II we compare the experimentally determined entropy change from high temperatures down to $T = 0$ with the theoretical value

$$S = 0.25xR \ln(2S + 1) .$$

The fact that the experimental values are lower than the theoretical ones can be explained by the presence of Eu^{3+} ions. This interpretation is consistent with the susceptibility and T_c results. The large amount of Eu^{3+} (17%) in the annealed Bi_3Eu sample is probably due to the large size of this sample as compared to the smaller samples used in the χ, ρ, and T_c measurements. The relatively high value of T_c in the $x = 0.3$ and $x = 0.5$ samples support this interpretation.

In the as cast samples we found a lower total magnetic entropy than in the annealed samples; this is probably due to a higher concentration of Eu^{3+} ions. At about 19 K we observed an anomaly in $C(T)$ as well as an anomaly in the resistivity, both disappearing completely upon annealing (Fig. 9). We interpret this as being due to an enhanced magnetic interaction in certain domains, perhaps caused by inhomogeneous stress.

The low-temperature magnetic specific heat of Bi_3Eu shows an exponential temperature behavior $C_M \approx e^{-\Delta/kT}$ as expected from a spinwave model.[16,17] The experimental value for the spin-wave gap is $\Delta/K_B = 3.15$ K (Fig. 10). At temperatures near T_M it is possible to fit C_M to a T^3 law.

IV. DISCUSSION

A. Bi_3Sr

From the experimental value of γ we find for the phonon-enhanced density of states $N(0) = 0.2$ states/eV atom spin. The electron-phonon interaction parameter λ is, as shown below, found to be ≈ 1.6 and this results in a band density of states $N_{BS}(0) = N(0)/(1+\lambda) \approx 0.08$ states/eV atom spin), where BS stands for band structure. This very low value of N_{BS} is consistent with the measured negative susceptibility (Fig. 1).

To determine the value of λ we use the modified MacMillan equation proposed by Allen and Dynes[18]:

$$T_c = \frac{f_1 f_2 \omega_{\log}}{1.20} \exp\left(-\frac{1.04(1+\lambda)}{\lambda - \mu - 0.62\mu^*\lambda}\right), \tag{5}$$

where f_1 and f_2 are factors of the order 1, and depend on the shape of the function $\alpha^2F(\omega)$. $\omega_{\log}$ is defined by

$$\omega_{\log} = \exp\left(\frac{2}{\lambda}\int_0^{\alpha}\frac{d\omega}{\omega}\,\alpha^2F(\omega)\ln\omega\right).$$

To determine $\omega_{\log}$ we assume $F(\omega)$ to have the shape suggested by the specific-heat measurements and following Birnboim[19] we assume $\alpha^2F(\omega) \sim (1/\omega)F(\omega)$. This gives $\omega_{\log} \approx 40$ K. For the quantity $\langle\omega^2\rangle^{1/2}$ needed to calculate the factors f_1 and f_2 we find $\langle\omega^2\rangle^{1/2} \approx 60$ K. Assuming $\mu^* = 0.1$ we find from Eq. (5) $\lambda \approx 1.6$. The different parameters obtained for Bi_3Sr are thus relatively near to those found for Pb.[18] The interpretation of Bi_3Sr as a strong coupling superconductor is further supported by the high value found for $(\Delta C/\gamma T)$ (2.7 ± 0.5). This value is close to the one found for Pb (2.71),[20] but far from the BCS value of 1.43. For the parameter η defined by

$$\eta \equiv M\langle\omega^2\rangle\lambda\,,$$

we obtained 1.8 (eV/Å^2).

There are of course some uncertainties about the exact form of $\alpha^2F(\omega)$. If we for instance assume $\alpha^2F(\omega) \sim F(\omega)$ we find $\omega_{\log} = 68$ K, $\langle\omega^2\rangle^{1/2} = 74$ K, $\lambda = 1.1$, and $\eta = 1.8$ (eV/Å^2). The value of λ depends thus strongly on the choice of $\alpha^2F(\omega)$. This suggests that this form is unimportant for superconductivity and as noted by Allen and Dynes,[18] the important parameter in determining T_c is η. Since $\eta = N_{BS}\langle I^2\rangle$ this means that the low value of N_{BS} is compensated in this system by a large value of $\langle I^2\rangle$ to produce a reasonable high value for η. $\langle I^2\rangle$ is the average of the electron-phonon matrix element over the Fermi surface. Our magnetization measurements on Bi_3Sr show that this compound is very close to a type-I superconductor. H_{c2} at 1.6 K was determined to be 0.092 T, and the estimate of the thermodynamic critical field was 0.053 T. This is rather unusual for an intermetallic compound and is due to the very low density of states at the Fermi surface combined with the equally low residual resistivity.

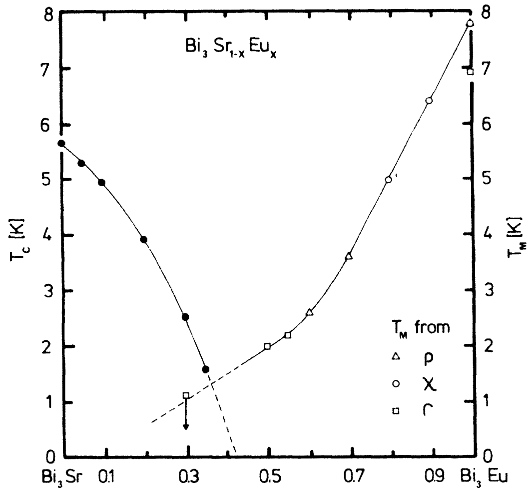

FIG. 11. Phase diagram for $Bi_3Sr_{1-x}Eu_x$ showing the superconducting critical temperature T_c and magnetic ordering temperature T_M as a function of concentration x.

B. Samples containing Eu

Our specific heat, resistivity, and magnetic measurements proved that Bi_3Eu and the Eu-rich samples show a long-range magnetic order of antiferromagnetic nature. As expected, the ordering temperature decreases linearly with decreasing Eu concentration. This is illustrated for the whole system by the phase diagram of Fig. 11. However, when we approach $x \approx 0.6$ we find deviations from this line and this may indicate a transition from a long-range order to a spin-glass-type region. In fact, we observe that in the Eu-rich samples the short range order contributions to the specific heat set in below 15 K (Fig. 8). As we decrease the concentration of magnetic ions this temperature hardly changes, so the magnetic transition is smeared out, and at $x \approx 0.3$ the situation is similar to the one found in $Ru_2Ce_{1-x}Gd_x$ for $x \approx 0.10$–0.15.[21-23] C_M/T increases with decreasing T_c, showing increasing short-range order but above 1.2 K no long-range order occurs. This behavior can be understood as a result of a nonuniform distribution of magnetic ions. We expect a magnetic order in this concentration range to be of a spin-glass type.

The existence of correlations between spins is also reflected in the critical temperature T_c which above $x = 0.1$ drops below the Abrikosov-Gor'kov curve (Table I).

So far, we have not observed any coexistence of superconductivity and antiferromagnetism in this system. Figure 11, however, shows clearly that there is a domain of possible coexistence around $x = 0.35$ and $T < 1$ K. Further investigations are now underway to explore this particular domain.

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