Specific heat of A-15 and bcc $Mo_{0.4}Tc_{0.6}$

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The specific heat of the A-15 phase $Mo_{0.4}Tc_{0.6}$ and of the allotropic bcc (A-2) phase of the same composition prepared by a rapid-quench technique has been measured between 4 and 20 K. The A-15 allotrope has an electronic-specific-heat coefficient γ of 3.3 mJ/(g-atom K^2) and a Debye temperature of 271 K. The bcc phase has a γ of 6.8 mJ/(g-atom K^2) and a softer lattice, with a Debye temperature of 258 K. The energy gaps of both structures are appreciably higher than those of any other known superconductors, with $2\Delta/kT_c$ values of 6.5 for the A-15 and 6.2 for the bcc compared with the previously known maxima of 4.8 for Nb_3Sn and 4.6 for Hg. In addition, the specific-heat discontinuity at T_c for the A-15 $Mo_{0.4}Tc_{0.6}$ is the largest yet observed, with the electronic superconducting specific heat C_{es} equal to $5.4\gamma T_c$.

I. INTRODUCTION

As reported recently,[1] $Mo_{0.4}Tc_{0.6}$ can be prepared in two different allotropes, A-15 and bcc. Thus, this system joins the list of other A-15's which have been prepared as bcc materials at the same composition, e.g., Nb_3Au,[2] and Nb_3Al.[3] These allotropes of $Mo_{0.4}Tc_{0.6}$ are considered unique since the susceptibility measurements[1] show the superconducting transition temperatures T_c are essentially identical at 13.4 K for both, although the bcc transition is somewhat broader. For the other known allotropic A-15, bcc pairs, the bcc phase has a significantly lower T_c; for Nb_3Au 11.0 vs 1.2 K; for Nb_3Al, 18.6 vs 3.1 K.

Thus, it was thought interesting to investigate the properties of both allotropes of $Mo_{0.4}Tc_{0.6}$ by low-temperature specific-heat measurements, to gain a better understanding of the factors responsible for maintaining the T_c of both phases approximately equal.

II. EXPERIMENTAL

A. Sample preparation

Finely divided molybdenum powder (99.9+% purity) and technetium metal powder (99Tc from Oak Ridge National Laboratory) were used as the starting materials. An x-ray fluorescence examination of the technetium powder showed no metal impurities within the limits of detection (i.e., less than 0.001%). To remove all volatile impurities, small elemental beads were prepared from these metal powders by repeated melting in an electron beam furnace at 10^{-6} Torr. The $Mo_{0.4}Tc_{0.6}$ alloy was prepared by arc melting weighed amounts of the beads in a zirconium-gettered argon atmosphere. The sample was arc melted four times with the button being turned over between each melting. An x-ray diffraction pattern for the sample showed the resulting material was single phase with a sharply defined A-15 structure.

The unit used for the quick-quench experiment consists of a small arc furnace modified to contain a copper hammer attached to the piston of an air cylinder and positioned so the hammer is directly above the water-cooled copper hearth.[1] The cooling rate is estimated at 10^5°C/sec. The sample is formed as a small disk between 25 and 50 μm thick. The x-ray diffraction trace of this material shows that complete conversion of the A-15 structure to the bcc structure had occurred. The diffraction lines for this phase are relatively broad due to thermal strains induced in the sample by the quick-quench operation. Since the bcc structure at this composition is not a stable phase, the sample could not be annealed. The lattice parameters determined for the starting materials and the two alloy preparations are given in Table I.

B. Calorimeter design

The apparatus used for these measurements is a recent-design low-addenda small sample calorimeter suitable for thin film and splat-cooled samples weighing only a few milligrams, as well as for larger (several hundred milligram) samples. This design was developed in 1975 by Räde[4] in Jülich, West Germany and has lower addenda than

TABLE I. Lattice parameters.

Sample	Crystal structure	a_0 (Å)	c_0 (Å)
Mo	A-2 (bcc)	3.1470 ± 0.0004	···
Tc	A-3 (hcp)	2.740 ± 0.001	4.399 ± 0.002
$Mo_{0.4}Tc_{0.6}$	A-15	4.9345 ± 0.0003	···
$Mo_{0.4}Tc_{0.6}$	A-2 (bcc)	3.098 ± 0.002	···

the bolometer design of Stanford.[5] In the calorimeter used for these experiments, a Scientific Instruments[6] model M2 exposed element Ge thermometer weighing 0.7 mg was bonded with EPO-TEK H62 epoxy[7] to the rough ground surface of a 1.27-cm disk of 0.2-mm-thick single-crystal sapphire. A Cr-Ti heater is evaporated on the sapphire disk. Samples are thermally bonded to the sapphire using Wakefield thermal grease.[8] Four 0.0762-mm-Au–7-at.%-alloy wires[9] make electrical contact via EPO-TEK H31 LV electrically conducting epoxy to the heater and two of the 0.0254-mm Au wires bonded to the M2 thermometer by the manufacturer. In addition to electrical and thermal contact, these four wires provide mechanical support.

Temperature standards for this calorimeter are a Cryocal encapsulated Ge thermometer, factory calibrated from 1.5–40 K and a Scientific Instruments encapsulated Ge thermometer, factory calibrated from 20–40 K. Agreement of the two calibrations is better than 0.040 K in the region of overlap, and the liquid-He point is better than 0.003 K. Data using the decay constant method[5] is taken by a Tracor Northern NS-570 signal averager, sampling the off-null signal of a Wheatstone bridge driven and detected by a PAR HR-8 lock-in amplifier at 1400 Hz. One arm of the Wheatstone bridge is the M2 thermometer, the other a decade resistance box. Other details of measurement may be found in the literature.[4,5]

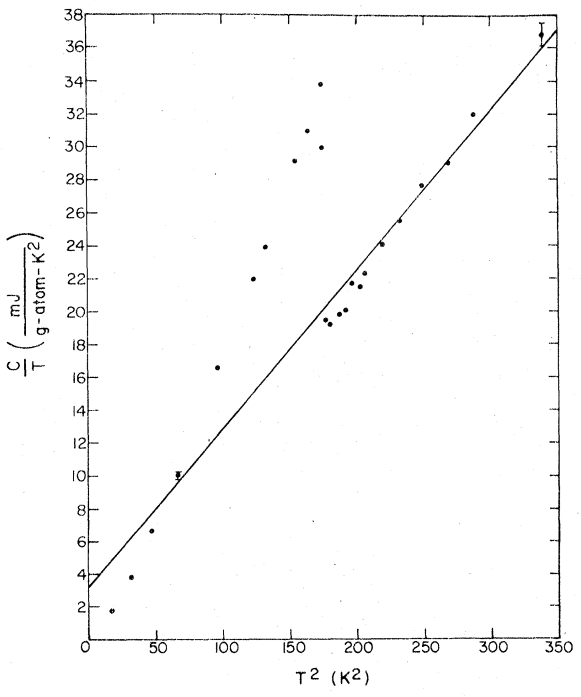

FIG. 1. Specific heat of A-15 $Mo_{0.4}Tc_{0.6}$. The line is derived from Eq. (3).

The specific heat of a 27-mg piece of high-purity Ge was measured as a standard from 4 to 24 K using the decay constant method, with an absolute accuracy of ±5%. Ge was used for the calibration since it is available in high purity, with a well-known specific heat,[10] and has a low specific heat with no electronic contribution. This latter property makes Ge a good test for the accuracy of the calorimeter's addenda since the sample constituted less than 50% of the total specific heat in the calibration measurement. The specific heat of the EPO-TEK H62 and H31 LV epoxies was measured and has been reported.[11]

For the A-15 $Mo_{0.4}Tc_{0.6}$, the addenda were less than 45% of the total specific heat at 4 K and less than 25% at 20 K. The bcc sample of $Mo_{0.4}Tc_{0.6}$ weighed only 11 mg and the addenda were approximately 60% of the total specific heat over the temperature range of measurement. Since the addenda error is the main source of error in the measurement of the net sample specific heat, this results in the larger error bars of Fig. 3 versus those of Fig. 1 for the A-15 data. Self-heating by the radioactive Tc was not a problem, contributing at most a 0.02-K temperature error.

III. RESULTS AND DISCUSSION

A-15 $Mo_{0.4}Tc_{0.6}$

The data for the A-15 $Mo_{0.4}Tc_{0.6}$ are given in Fig. 1. The size and sharpness of the superconducting transition are immediately apparent. The width, from onset to completion, of the transition is less than 0.15 K. T_c is taken as the midpoint of the transition. The size of the discontinuity is typically represented by $C_{es}(T_c)/\gamma T_c$, where $C_{es}(T_c)$ is the superconducting electronic specific heat at T_c,

$$C_{\text{supercond.}} \equiv C_s \equiv C_{\text{latt.}} + C_{es} . \tag{1}$$

γ is the coefficient of the linearly temperature-dependent term of the normal-state specific heat,

$$C_n = \gamma T + \beta T^3 , \tag{2}$$

and T_c is the transition temperature. Another commonly used measure of the discontinuity, $\Delta C/\gamma T_c$, where $\Delta C = C_s - C_n$, is merely $C_{es}(T_c)/\gamma T_c - 1$ since $C_{en}(T_c) = \gamma T_c$.

From Fig. 1 the intercept γ is 3.3 mJ/(g-atom K²). The slope of the curve is β, from which the Debye temperature Θ_D is derived to be 271 K [$\Theta_D^3 = 1944/\beta$, where β is in J/(g-atom K⁴)]. A thermodynamic stricture further aids determination of γ and β, in that, for second-order transitions,

$$\int_0^{T_c} \frac{C_n}{T}\, dT = S_n(T_c) = S_s(T_c) = \int_0^{T_c} \frac{C_s}{T}\, dT\,, \qquad (3)$$

where $S_n(T_c)$ is the normal-state entropy at temperature T_c and $S_s(T_c)$ is the superconducting-state entropy at T_c. Since the parameters of Eq. (2) are dependent on how the normal-state data is extrapolated to $T=0$, Eq. (3) enables the knowledge of the measured superconducting specific heat to improve the accuracy of the extrapolated normal-state data. Frequently high-T_c A-15 superconductors do not obey Eq. (2) above T_c, instead exhibiting an additional, T^5, term. Since Eq. (2) has two independent parameters, this makes extrapolation of Eq. (2) to $T=0$ more difficult. Fortunately, the data of Fig. 1 obey Eq. (2), the Debye law, well. Thus, the line drawn in Fig. 1 to satisfy Eq. (3) falls near all the data points.

Using this value of 3.3 mJ/(g-atom K^2) for γ, $C_{es}(T_c)/\gamma T_c$ for A-15 $Mo_{0.4}Tc_{0.6}$ is found to be 5.4, a remarkably high number. This number depends critically on γ, (see error discussion below) but although this γ is relatively low for A-15 compounds (see Table II), it is of the same size as the γ for Mo_3Ir. The next highest normalized superconducting electronic specific heat, $C_{es}(T_c)/\gamma T_c$, is 3.9 for $NbN_{0.91}$.[12]

Related to this extremely high discontinuity in the specific heat of A-15 $Mo_{0.4}Tc_{0.6}$ is an equally unusual energy gap. According to the BCS theory, the superconducting electronic specific heat C_{es} has an exponential temperature dependence

$$C_{es}/\gamma T_c = ae^{-\Delta(T)/kT}\,. \qquad (4)$$

From Fig. 2, this $\Delta(T)$ near T_c is approximately $3.26kT_c$. Normalized as $2\Delta/kT_c$, this 6.5 result for A-15 $Mo_{0.4}Tc_{0.6}$ is far greater than the previous highest known values of 4.8 for Nb_3Sn,[13] 4.6 for Hg,[14] 3.95 for Pb,[14] and 3.8 for V_3Si.[12] The value of 6.5 is a finite-temperature result, and not the higher $\Delta(T=0)$ extrapolation from tunneling experiments as is true for Hg and Pb.

The thermodynamic critical magnetic field $H_c(T=0)$ may be calculated via

$$\int_0^{T_c} (C_s - C_n)\, dT = \frac{VH_c^2(0)}{8\pi}\,, \qquad (5)$$

where V is the volume of a gram atom, calculated from the lattice parameter to be 9.043 cm^3. The value for $H_c(0)$ obtained from Eq. (4) is 2845 G. This is not particularly unusual; Table II shows $H_c(0)$ for Nb_3Sn is 5350 G. Coupled with Eq. (5) is the following equation:

$$\left.\frac{dH_c}{dT}\right|_{T=T_c} = \left(\frac{4\pi(C_s - C_n)}{T_c}\right)^{1/2}. \qquad (6)$$

The two equations are commonly referred to as the

TABLE II. Parameters for the $Mo_{0.4}Tc_{0.6}$ system with data for other superconductors for comparison.

Compound	Reference	T_c (K)	γ (mJ/g-atom K^2)	Θ_D (K)	$C_{es}(T_c)/\gamma T_c$	$2\Delta/kT_c$	λ	$H_c(0)$ (G)	$\left.\frac{-dH_c}{dT}\right\|_{T=T_c}\left(\frac{G}{K}\right)$	$V\left(\frac{cm^3}{g\text{-atom}}\right)$
A – 15 $Mo_{0.4}Tc_{0.6}$		13.2	3.3 ± 0.2	271 ± 3	5.37 ± 0.4	6.5 ± 0.2	1.1 ± 0.1	2845 ± 40	450 ± 15	9.043
bcc $Mo_{0.4}Tc_{0.6}$		12.7	6.8 ± 0.3	258 ± 3	3.14 ± 0.2	6.2 ± 0.2	1.1 ± 0.1	2430 ± 40	450 ± 15	8.953
bcc $Mo_{0.5}Tc_{0.5}$	25	12.6	4.60	300						
Nb_3Sn	13	18.1	13.1	228	3.4	4.8	1.67	5350	590	11.11
Mo_3Ir	25	8.5	3.34	325						
Pb	14	7.2	3.36	105	3.64	3.95	1.55	803	216	18.25
Hg	14	4.15	1.86	72	3.18	4.6	1.6			
BCS					2.43	3.52				

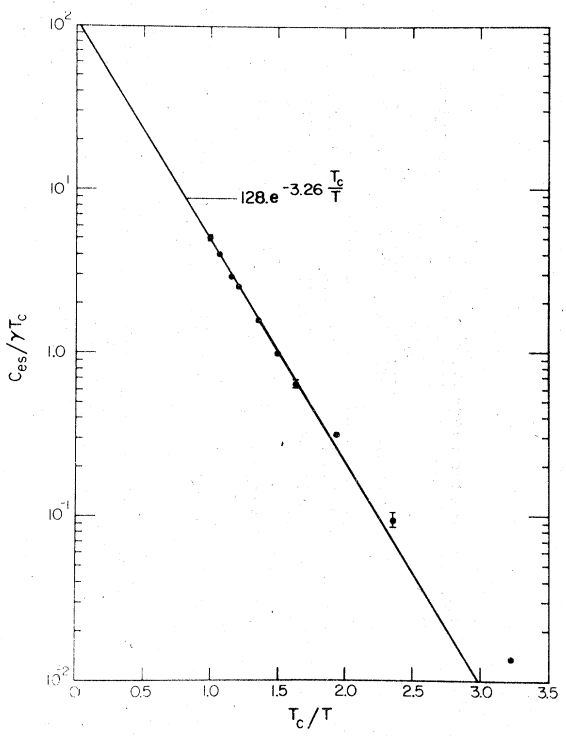

FIG. 2. Superconducting electronic specific heat of A-15 $Mo_{0.4}Tc_{0.6}$.

Rutgers relations. Since there is no division by the relatively small γ on the right-hand side of Eq. (6) this parameter (450 G/K) too is not unusually large, even though it involves ΔC.

Clearly the $2\Delta/kT_c$ and $C_{es}(T_c)/\gamma T_c$ parameters (see Table II) for this A-15 are novel. The BCS predictions are also shown in Table II along with typical values found for other superconductors. The two parameters are self-consistent, however, since they both imply strong coupling. Also, by scaling the values for $2\Delta/kT_c$ for Pb and Hg given in Table II, i.e.,

$$\frac{2\Delta}{kT_c} \text{ (scaled for } A\text{-15 } Mo_{0.4}Tc_{0.6}) = \frac{C_{es}(T_c)/\gamma T_c \text{ (for } A\text{-15 } Mo_{0.4}Tc_{0.6})}{C_{es}(T_c)/\gamma T_c \text{ (for Pb or Hg)}} \times 2\Delta/kT_c \text{ (for Pb or Hg)}$$

one finds that the scaled values for $2\Delta/kT_c$ bracket that for $Mo_{0.4}Tc_{0.6}$. Another way of estimating Δ is via the BCS relation

$$\frac{2\Delta(0)}{kT_c} = \frac{H_c(0)}{T_c}\left(\frac{2\pi V}{3\gamma}\right)^{1/2}. \quad (7)$$

This gives 5.14, another indication that this sample does not behave as predicted by BCS.

Another parameter used in determining the strength of the electron-phonon coupling is λ, given by the empirical relation of McMillan[15] as

$$\lambda = \frac{1.04 + \mu^* \ln(\Theta_D/1.45T_c)}{(1 - 0.62\mu^*)\ln(\Theta_D/1.45T_c) - 1.04}, \quad (8)$$

where Θ_D is the Debye temperature and μ^*, the Coulomb coupling constant, is taken as 0.13. This gives a λ, the electron phonon coupling parameter, of 0.99.

Recently, a simplified equation involving λ and measured parameters has been put forth,[16]

$$T_c = \tfrac{1}{20}\Theta_D(\lambda - 0.25). \quad (9)$$

Using $T_c = 13.22$ K and $\Theta_D = 271$ K, this gives a value for λ of 1.23. A resolution of the discrepancy between the two empirical formulas depends on measuring λ independently by tunneling experiments, measuring T_c, and measuring the specific heat to determine Θ_D. The strong coupling system suggested[16] for this purpose was the Tl-Pb-Bi system of alloys, with λ's measured by tunneling ranging from 0.78 to 2.13.[17] Some work[18] on this system had been done, and recently Hermans *et al.*[19] have begun an exhaustive study of the specific heat of this system. These results, not yet spanning the entire system, indicate that Eq. (9) is by far more accurate for λ's of 1.5 and larger, as might be expected since McMillan claimed accuracy only below $\lambda = 1.0$. By extrapolating the results of Hermans *et al.*[19] to $Pb_{0.6}Tl_{0.4}$, with a λ of 1.38,[17] Eqs. (8) and (9) are seen to bracket the correct answer—Eq. (9) giving too high a result by about 10% and Eq. (8) too low by 15%. This approximate treatment of the validity of the two equations for λ in this region suggests a λ for A-15 $Mo_{0.4}Tc_{0.6}$ of about 1.1. This value, as may be seen from Table II, is appreciably smaller than the strong coupling elements Pb and Hg, although still relatively large. Since λ involves an integral over the entire phonon spectrum, it is possible that structure in the phonon spectrum, as seen in[20] Mo-Re by neutron diffraction, could account for the anomalously large values of the energy gap and specific-heat discontinuity. Certainly a closer look at the electronic and phonon properties via tunneling is strongly suggested.

The possible errors in the values obtained for $C_{es}(T_c)/\gamma T_c$ and $2\Delta/kT_c$ are given in Table II. For the discontinuity, the largest error is introduced by the value used for γ, since the error in T_c is ≤ 0.030 K and error in $C_{es}(T_c)$ comes from error in $C_s(T_c)$ ($=\pm 3\%$) and in the lattice contribution that is subtracted to obtain C_{es}. This lattice contribution error is not greater than 5%, which could decrease C_{es} by about the same amount since for this material $C_{\text{latt.}}$ is approximately equal to C_{es} at T_c. The probable error in γ, however, is in a

direction to increase $C_{es}/\gamma T_c$ since, as can be seen in Fig. 1, the curve drawn to satisfy the entropy restraint, Eq. (3), gives a larger γ than would come from a simple straight line through the data extrapolated to $T=0$.

The error in $2\Delta/kT_c$ is strictly from the data C_s ($\pm 2\%$ in this range) and from the subtracted lattice contribution, $C_{\text{latt.}}$ [see Eq. (1)]. The error bars in Fig. 2 for C_{es} come mainly from the lattice contribution error. For the data near T_c, the errors are quite small ($\pm 3\%$) since C_s ($\pm 2\%$) is twice $C_{\text{latt.}}$ ($\pm 5\%$). At lower temperatures, $C_{\text{latt.}}$ becomes similar in size to C_s, causing a larger error ($\pm 11\%$) in the difference, C_{es}. Since the value of Δ in Fig. 2 is obtained from the data near T_c, the error is only 3%–4%. The deviation upward from the linear behavior of the lower temperature data in Fig. 2 may be due to a second gap or to anisotropy of the gap. This is discussed more fully below for the bcc sample.

bcc $Mo_{0.4}Tc_{0.6}$

The specific-heat data for this sample are given in Fig. 3. The discontinuity is noticeably smaller and the transition is broader (0.4 K) than for the A-15 and centered at 12.7 K. The electron density of states coefficient γ is appreciably larger [6.8 mJ/(g-atom K^2)] than in the A-15 allotrope and the lattice is noticeably softer in the bcc (Θ_D = 258 K). This softer lattice, coupled with the larger γ, causes the gram-atom normal-state specific heat to be larger ($\beta \propto 1/\Theta_D^3$) for the bcc material. The curve in Fig. 3 drawn to fit the data satisfies the entropy constraint of Eq. (3) very well (better than 5%).

An important comparison of the two allotropes can be made using a result of the BCS theory of superconductivity,

$$kT_c = 1.14\hbar w_q \exp[-1/N(0)V] , \qquad (10)$$

where $N(0)$ is the density of states at the Fermi level, V is the pairing potential arising from the electron-phonon interaction [$\lambda \approx N(0)V$], and $\hbar w_q \approx k\Theta_D$. As stated in the Introduction, this study was partly to understand why the bcc allotrope of $Mo_{0.4}Tc_{0.6}$ has essentially the same T_c as the A-15 allotrope. Equation (10) along with the γ and Θ_D parameters suggest one qualitative answer. It is seen from these parameters [$\gamma \propto N(0)$ and $\Theta_D \propto w_q$] that, given a fixed V, the bcc allotrope would have a much higher T_c due to the doubled density of states appearing in the exponential far outweighing the 5% smaller Θ_D of the bcc appearing in the pre-exponential factor in Eq. (10).

It is this larger density of states that may cause the similarity in T_c of the two allotropes. The λ calculated for the bcc $Mo_{0.4}Tc_{0.6}$ is given in Table II and is the same as that for the A-15. This, coupled with the γ's, implies a large pairing potential V for the A-15 material with its linear chains of atoms.

The discontinuity for the bcc $Mo_{0.4}Tc_{0.6}$, as measured by $C_{es}(T_c)/\gamma T_c$ at T_c is 3.1. This would probably be only slightly higher if it were possible to prepare the bcc $Mo_{0.4}Tc_{0.6}$ with a sharp transition. By extrapolating C_s to the transition temperature of the A-15, 13.2 K, the $C_{es}(T_c)/\gamma T_c$ for a sharp transition sample is 3.28, due to the larger linear term coefficient γ in the denominator. However, Fig. 4 shows that the energy gap for the bcc sample is approximately as large as that for the A-15. The error discussion for these two parameters [$C_{es}(T_c)/\gamma T_c$ and $2\Delta/kT_c$] is similar to that above for the A-15 material, except that the error contributed by the uncertainty in γ is less since the entropy constraint [Eq. (2)] agrees with the line through the data somewhat better than for the A-15. Thus, the percentage error in γ and the ratio $C_{es}(T_c)/\gamma T_c$ is improved.

The upturn of $C_{es}/\gamma T_c$ away from the linear extrapolation of the higher-temperature data is a prominent feature of Fig. 4. There are three possible explanations for this upturn: (i) presence of

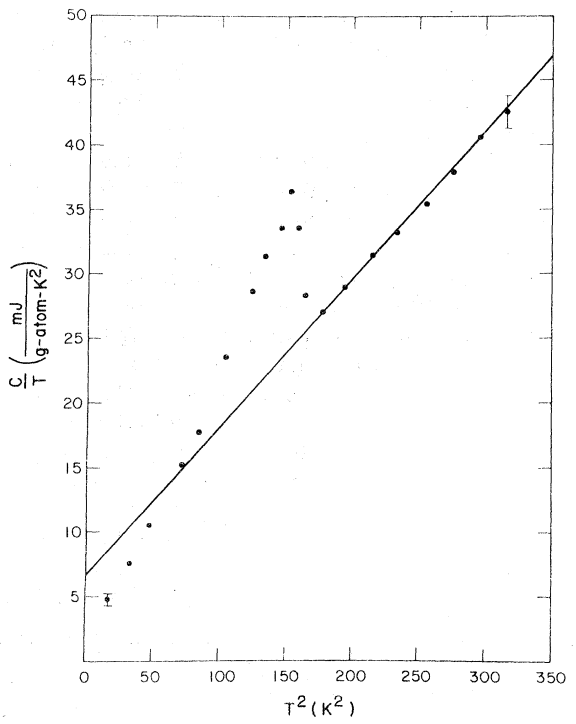

FIG. 3. Specific heat of bcc $Mo_{0.4}Tc_{0.6}$. The line is derived from Eq. (3).

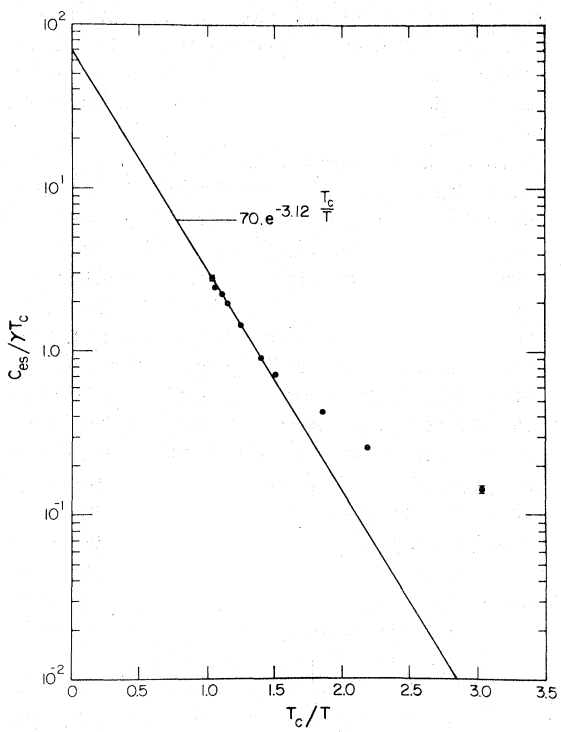

FIG. 4. Superconducting electronic specific heat of bcc $Mo_{0.4}Tc_{0.6}$.

normal material with a contribution to C_s of $\gamma_s T$; (ii) an anisotropic energy gap—at lower temperatures the electron excitations are preferentially across the narrower portions of the gap; and (iii) the existence of two gaps[21] caused by overlapping s and d bands at the Fermi surface.

The upturn could be explained by the presence of normal material with a γ_s of 2.7 mJ/(g-atom K^2). This would imply one of several things: 40% of the bcc $Mo_{0.4}Tc_{0.6}$ is normal at 4 K; local regions of Mo exist, but pure Mo has a γ of only[22] 2.1 mJ/(g-atom K^2) and thus this explanation is unphysical; local regions of Tc exist, amounting to again an unphysical amount since the γ for Tc is[23] 4.3 mJ/(g-atom K^2) and only 0.6 g-atom of Tc is present, leading to all of the Tc being normal. The first possibility, that of 40% of the $Mo_{0.4}Tc_{0.6}$ being normal, must also be rejected, as no second phase is found to be present in the x-ray study.

Secondly, this upturn may be due to an anisotropic gap, as seen[24] in pure V at much lower relative temperatures, below T_c/T of 5. Both this and the following explanation are similar, both require very pure material. As stated above, the purity of these $Mo_{0.4}Tc_{0.6}$ samples is about 99.99%, similar to that of the V work.[24] One must question, however, if the dissimilar, randomly placed Mo and Tc atoms in the lattice sites constitute sufficient scattering probability to serve as "impurities." Although the two are adjacent elements in the periodic table, this cannot be ruled out. Although anisotropy may seem an odd explanation to invoke for a bcc structure, V is also bcc.

The third possible explanation is that of an incipient second gap that, at low temperatures, would also follow straight-line behavior as predicted by Eq. (4), *albeit* with lower slope. This seems a likely explanation because of the larger $N(0)$ for the bcc material compared with the A-15. This larger density of states could very well be explained by an s band overlapping the d band in the bcc material and not in the A-15, thus causing the second gap behavior of Fig. 4.

IV. CONCLUSIONS

The specific heat of the allotropic pair of bcc and A-15 $Mo_{0.4}Tc_{0.6}$ has been measured. Extremely large energy gaps exist in both materials. This is accompanied by a large discontinuity $C_{es}/\gamma T_c$ in the A-15 material. The possibility of a two-gap model being applicable to the bcc $Mo_{0.4}Tc_{0.6}$ is given credence by the larger density of states observed for the bcc over the A-15 material. This larger density of states is also at least one reason behind the almost equally high T_c for the bcc structure $Mo_{0.4}Tc_{0.6}$ as for the A-15, contrary to the large decrease observed for other bcc–A-15 allotropic pairs. Tunneling experiments are being pursued to further investigate the unusual properties of these two superconductors.

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