Investigation of the second transition in $U_{1-x}Th_xBe_{13}$

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We have prepared extremely high-purity samples of $U_{0.97}Th_{0.03}Be_{13}$ and $U_{0.962}Th_{0.038}Be_{13}$ and conducted a study of the specific heat in zero field on the as-prepared and annealed samples. In addition we have measured the low-temperature specific heat of the 3 at. % Th sample in 0.25, 0.5, 0.75, 1.0, 1.25, 2, and 3 T magnetic fields in order to accurately determine H'_{c2} for both transitions. The high-purity unannealed 3.8 at. % Th samples show larger and sharper transitions than previously reported; annealing further improves the transitions such that $\Delta C(T_{c2})$ for the 3 at. % Th sample is more than twice as large as previously reported, with T_{c2} being approximately the same. A γ value of 2300 mJ/mol K^2 for $U_{0.97}Th_{0.03}Be_{13}$ is derived from entropy considerations and the 0-field and 3-T specific-heat data. The separation between the two transitions is significantly improved with annealing—$\Delta C(T_{c1})$, which is essentially only a shoulder on $\Delta C(T_{c2})$ for unannealed 3.8 at. % Th samples, becomes a clearly distinct transition upon annealing. The results of the field measurements are that the $H_{c2}(T)$ data for the two transitions are, within our (rather good) precision, parallel; this includes the data as $H\rightarrow 0$, i.e., $H'_{c2}(T_c)$ for the two transitions is the same, $\approx -45\pm 3$ T/K. This result is consistent with the H'_{c2} value reported for pure UBe_{13} suppresses. These field results contradict the two previous specific-heat-in-field studies (on unannealed lower-purity samples with significantly more scatter in the H_{c2} data) of the two transitions in $U_{1-x}Th_xBe_{13}$. The equality of $H'_{c2}(T_{c1})$, $H'_{c2}(T_{c2})$, and H'_{c2} for pure UBe_{13} demonstrated here is difficult to explain on the basis of current theories that consider the lower transition as a magnetic superconducting state.

I. INTRODUCTION

Since the discovery[1] of a second transition in the specific heat of $U_{1-x}Th_xBe_{13}$, $0.01<x<0.06$, a great deal of controversy has surrounded the identification of this transition at temperature T_{c2} below the superconducting transition T_{c1}. Important further experiments include (1) the finding[2] of a large λ-shaped peak in the ultrasound attenuation at T_{c2}, considered evidence for a magnetic description of the transition; (2) the discovery[3] that the lower critical field, H_{c1}, climbs more sharply with decreasing temperature below T_{c2}, considered as evidence for a superconducting description of the transition; (3) the report,[4] most recently with improved precision,[5] that muon spin resonance indicates an effective electronic magnetic moment of 10^{-3}–$10^{-2}\mu_B$/U atom is formed below T_{c2} in this Th composition range in $U_{1-x}Th_xB_{13}$. Recently, it has been argued[5–7] that a possible resolution of these conflicting arguments might be a magnetic superconducting state, for which several theoretical descriptions[6,7] have been proposed.

To date, no study of the influence of annealing on the behavior of the second transition (e.g., transition temperature, transition width, size of the specific heat discontinuity ΔC, separation of the two transitions) has been made. Only two studies of the bulk behavior (i.e., specific heat) of the second transition in $U_{1-x}Th_xBe_{13}$ as a function of magnetic field have been carried out.[8,9] These studies [where $x=3.3\%$ (Ref. 8) and 3% (Ref. 9)] had a lowest nonzero field of 1 T and a rather broad (0.07 K) bulk transition at T_{c2} in zero field, with some scatter present in the H_{c2} versus T data. Since theories (e.g., Ref. 7) of the second transition have advanced to the point of using ΔC and T_{c2} values in their calculations, and since any comparison of the superconducting state at T_{c1} and that at T_{c2} would benefit from a precise knowledge of the respective H_{c2} values, particularly due to the superconductivity-magnetism duality of the second transition, we have undertaken the present study.

II. EXPERIMENTAL

Samples of $U_{0.97}Th_{0.03}Be_{13}$ and $U_{0.962}Th_{0.038}Be_{13}$ were prepared in a Zr-gettered, high starting purity (99.9999) Ar atmosphere in an arc melter using a water-cooled Ta hearth. The use of a Ta hearth was to avoid the small uptake ($\sim$20 ppm) of Cu by the Be on repeated melting on a Cu hearth that has been reported.[10] As is known,[11] 0.4 at. % Cu in UBe_{13} suppresses superconductivity below 0.015 K; although 20 ppm would, by linear interpolation, only cause a 5-mK depression in T_c, we tried as far as possible to use improved conditions. The U used is the highest purity commercially available of which we are aware, i.e., electro-transport-refined U rod from Ames Laboratory. A typical purity analysis is 99.985% (with [Cr], [Cu], [Mn], [Fe]<2 ppm, [Ni]~ 7 ppm, and [Si]~ 100 ppm). What is interesting to note from a gross properties point of view is that the purity of the U rod is such that when it is cut (using ceramic-jaw cutters), the metal is ductile, in contrast with normal purity (99.9%) U from Los Alamos. The Be used in 99.9999% single crystal "scrap" from Atomergic. We have also tried (once) preparing pure UBe_{13} Ames U and zone-refined, extremely high-purity Be and found essentially the same T_c (0.972 K) as when the Atomergic Be is used. The Th

used was Th "crystal bar" prepared using the van Arkel process and having a purity of 99.99%. Sufficient extra Be was added to insure proper stoichiometry after three successive homogenization melts. The actual stoichiometries achieved were $U_{0.9705}Th_{0.0295}Be_{13.186}$ and $U_{0.9616}Th_{0.0384}Be_{12.931}$. It is important to note that UBe_{13} (which is the only compound present in the U-Be phase diagram) forms quite readily with the first melting, as can be seen, e.g., from x rays or specific heat at T_c. Thus, three melts is more than sufficient to insure homogeneity. However, to improve homogeneity of the U and Th, these two elements were melted together first, and flipped between each melt, for a total of three times. (Due to the lack of solubility of Th in U, this is at best only a *pro forma* attempt to provide better homogeneity.)

X-ray measurements of the samples were then taken, indicating single phase material with lattice parameters in agreement with Ref. 1. Some pieces of the unannealed samples were kept for specific-heat measurements and other possible further characterization. For each sample, a large (~500 mg) piece was placed above the opening of a small BeO crucible in the bottom of which was a 10–15 mg piece of Atomergic Be (melting point 1284 °C) which served as a source of Be vapor during annealing. (The sample and the Be were not in direct contact.) This assembly (samples perched atop a small BeO crucible containing pure Be) was placed inside a separate, large BeO crucible, which was then covered with a BeO lid. This was then sealed in a $\frac{3}{4}$-in.-diam Ta "bomb" which was simply a Ta tube with a bottom and top lid arc melted on while clamped in a Cu heat sink thermally attached to a water-cooled Cu hearth in a Zr-gettered, high-purity Ar gas arc melter. All BeO parts are previously outgassed at ~1700 °C in an induction furnace at 10^{-6} mm vacuum. The BeO crucible is always kept upright after placing in the Ta bomb to insure that the BeO lid remains in place to (mostly) contain the Be vapor. The samples, each sealed in their separate Ta bomb, were first annealed for 500 h at 1400 °C, then checked for weight loss and a small piece removed for specific heat, then resealed for an additional 720 h of annealing at 1400 °C. The Ta bombs were kept under dynamic vacuum (50 μm) in an alumina process tube in a computer controlled Lindberg furnace.

The Be vapor sources did not totally evaporate, even after the full 1220 h. The total weight loss, expressed as a fraction, was 0.004 for the 3 at. % Th sample and 0.002 for the 3.8 at. %Th sample. Assuming the loss was entirely Be, the resultant stoichiometries were $U_{0.9705}Th_{0.0295}Be_{13.026}$ and $U_{0.9616}Th_{0.0384}Be_{12.849}$. Without the crucible lid and, to a lesser extent, the Be vapor source, the weight losses would have been unacceptably high.

The specific heats were measured in a small sample calorimeter described elsewhere,[12] in a ^{3}He apparatus down to 0.3 K in a CCL magnet. Temperature in field was determined using a speer 220 W carbon resistor, following the method of Ref. 13. All specific-heat measurements were taken using the same platform, so that the internal precision of the data is better than 1%. The absolute accuracy of the specific heat is about ±5% below 1 K.

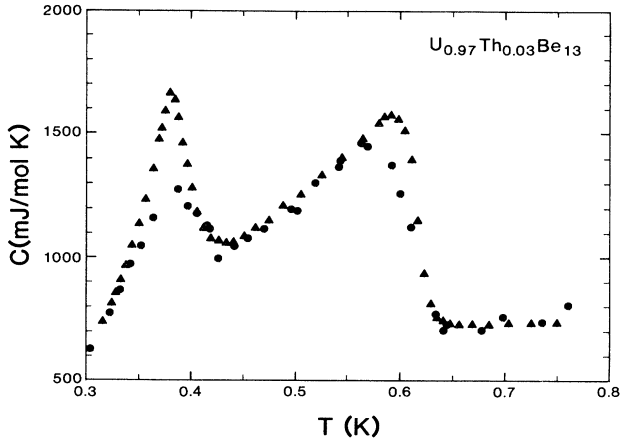

FIG. 1. Specific heat vs temperature for high-purity $U_{0.97}Th_{0.03}Be_{13}$, annealed for 1220 h at 1400 °C (triangles) and for $U_{0.9692}Th_{0.0308}Be_{13}$ from Ref. 1 (circles).

III. RESULTS AND DISCUSSION

Figures 1 and 2 show the zero-field specific heats of the 1220-h, 1400 °C annealed 3 at. % and 3.8 at. % Th samples, respectively, at low temperatures along with data on unannealed 3.08 at. % and 3.78 at. % Th samples from Ref. 1. (These latter data are typical for previously reported[8,9] samples.)

Specific-heat data from the present work's unannealed 3 at. % Th sample (not shown) show a peak, at essentially the same T_{c2}, intermediate in size between the two shown in Fig. 1, while at the peak at T_{c1} of our unannealed 3 at. % Th sample essentially overlays that shown in Fig. 1 from Ref. 1. In the case of the present work's unannealed 3.8 at. % sample, the specific-heat data (not shown) display slight (~10%) enhancement in the size of both peaks over those shown in Fig. 2 from Ref. 1,with the

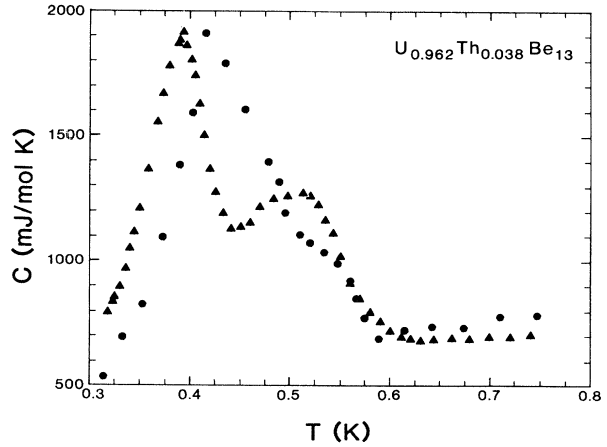

FIG. 2. Specific heat vs temperature for high-purity $U_{0.962}Th_{0.038}Be_{13}$, annealed for 1220 h at 1400 °C (triangles) and for $U_{0.9622}Th_{0.0378}Be_{13}$ from Ref. 1 (circles).

$\Delta C(T_{c1})$ anomaly still just a shoulder on the lower, $\Delta C(T_{c2})$ anomaly. (Specific-heat data from the present work's 500-h annealed samples showed essentially an intermediate behavior.)

Thus, based on this discussion and the data presented in Figs. 1 and 2, both the higher purity of the starting materials and the annealing evidently lead to increases in ΔC, especially $\Delta C(T_{c2})$ for 3 at. % Th, and to improved separation of the two transitions, especially for 3.8 at. % Th. The specific-heat parameters derived from Figs. 1 and 2 [$\Delta C(T_{c1})$, $\Delta C(T_{c2})$, and $T_{\text{peak 1}}$, $T_{\text{peak 2}}$, ΔT_1, ΔT_2] are collected in or calculable from Table I. To better compare the specific-heat data for the two annealed samples, these data are presented together in Fig. 3. [It is interesting to note how ΔC and T_c vary with annealing in *pure* UBe_{13}, made from the same high-purity starting material, where homogenization of a small amount of Th on the U sublattice is not necessary. With 3.5 weeks at 1200 °C, ΔC increases by ~15%, T_c (bulk) onset remains approximately constant, and the width, ΔT_c, decreases from 0.06 to 0.03 K—i.e., essentially the same behavior seen with the more thorough annealing in the upper transition at T_{c1} for 3 at. % Th doping, Fig. 1 and Table I.]

It is clear that previous work has underestimated $\Delta C(T_{c2})$ for 3 at. % Th by at least (additional annealing might give further increase) a factor of two. This has significance for understanding the nature of this second transition (e.g., coupling strength of the superconducting pairing) and affects parameters theoretically derived (see, e.g., Ref. 7) from equations involving $\Delta C(T_{c2})$. Moreover, this observed increase in $\Delta C(T_{c2})$ implies a concomitant increase in the normal state γ ($\equiv C/T$ as $T\rightarrow 0$) for this annealed 3 at. % Th sample, since the superconducting state entropy, S_s ($\equiv \int_0^{T_{c1}^{\text{onset}}} C^{\text{superconducing}}/T\,dT$), for a second-order phase transition must be equal to S_{normal}, which then may be used to infer C^{normal}/T for $T<T_{c1}$. Thus, we can use the measured $C_{\text{superconducting}}$ data below T_{c1} (=0.63 K) for the 3 at. % Th sample (Fig. 1), the measured C_{normal} ($H=3$ T) data in the same sample (Fig. 4) down to 0.42 K, and the required equality of the entropies $S_s(T_{c1})=S_n(T_{c1})$ to infer C^{normal}/T as

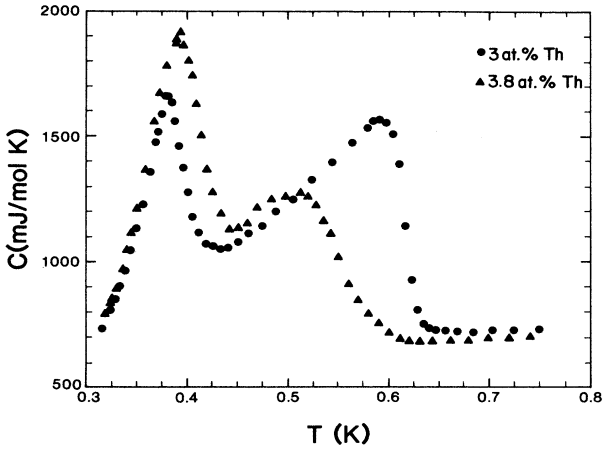

FIG. 3. Specific heat vs temperature of annealed $U_{0.97}Th_{0.03}Be_{13}$ (circles) and $U_{0.962}Th_{0.038}Be_{13}$. Some of the qualitative details of the phase diagrams to date (see, e.g., Refs. 5 and 7) are altered by the present results. As deduced from the values in Table I, we see here that T_{c1} changes significantly as a function of Th content for $0.03\leq x\leq 0.038$; based on T_{c1} (peak), the difference in T_{c1} for our annealed samples between 3 and 3.8 at. % Th is about twice that seen on unannealed samples (Ref. 1 and Figs. 1 and 2). Also, these data show T_{c2} (3.8 at. % Th) > T_{c2} (3 at. % Th) for our samples.

$T\rightarrow 0$, i.e., γ, for $U_{0.97}Th_{0.03}Be_{13}$. In pure UBe_{13}, where C/T at $T_c^+=0.96$ K is ~800 mJ/mol K^2, a similar procedure gives γ ($\equiv C/T$ as $T\rightarrow 0$) = 1020 mJ/mol K^2. As one can see in Fig. 4, C^{normal}/T at 0.42 K is already ~1400 mJ/mol K^2. To match the *observed*[14] superconducting state entropy, this C/T has to rise to 2300±200 mJ/mol K^2 at $T=0$. (The error bar is conservative.[14]) This is a very large γ result. What is further interesting to note is that this large normal state γ value (needed to match the superconducting state entropy of $U_{0.97}Th_{0.03}Be_{13}$) essentially exactly makes up for the smaller entropy observed above 1 K in the Th-doped sample (see Fig. 5). That is to say, the almost complete lack in the data of $U_{0.97}Be_{0.03}Be_{13}$ of the broad anomaly

TABLE I. Specific-heat parameters derived from Figs. 1 and 2.

	Lower transition		Upper transition	
	$C/T_{\text{peak}}/C/T$ at T_{onset} (J/mol K^2)	$T_{\text{peak}}/T_{\text{onset}}$ (K)	$C/T_{\text{peak}}/C/T$ at T_{onset} (J/mol K^2)	$T_{\text{peak}}/T_{\text{onset}}$ (K)
Ref. 1, 3.08 at. % Th	3.3/2.35	0.395/0.43	2.55/1.0	0.56/0.62
This work, 3 at. % Th annealed 1220 h	4.4/2.35	0.38/0.42	2.625/1.1	0.59/0.63
Ref. 1, 3.78 at. % Th	4.55/~2	0.41/0.52	C/T(Shoulder)[a] ~2/1.1	0.52(Shoulder)/0.58
This work, 3.8 at. % Th annealed 1220 h	4.87/2.55	0.39/0.44	C/T=2.44/1.1	0.513/0.60

[a]Our unannealed sample also has a poorly defined T_{c1}.

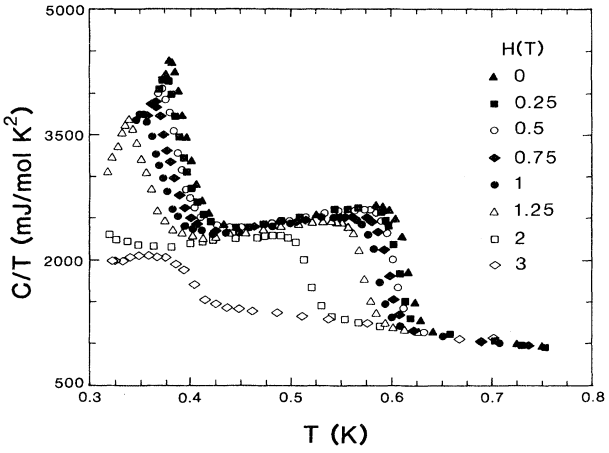

FIG. 4. Specific heat divided by temperature vs temperature for high-purity annealed $U_{0.97}Th_{0.03}Be_{13}$ as a function of field. The data below T_{c2} are not shown for $H < 1.25$ T for the sake of clarity.

observed in the specific-heat of UBe_{13} at 2 K, Fig. 5, turns out to be balanced by the greater entropy observed below 1 K in $U_{0.97}Th_{0.03}Be_{13}$ versus UBe_{13}. Thus S (6 K) for UBe_{13} = 3110 mJ/mol K [with S (1 K) = 920], while S (6 K) = 3030 mJ/mol K for $U_{0.97}Th_{0.03}Be_{13}$ [with S (1 K) = 1380]. There exists a fixed amount of entropy associated with the f electrons in heavy fermion systems, which can go variously into magnetic order, anomalies in C (e.g., at 2 K in UBe_{13} and 4 K in $CeCu_2Si_2$), or large γ as $T \to 0$. Obviously, the contribution of these limited f degrees of freedom to the anomaly in C at 2 K for UBe_{13} (Fig. 5) leads to a factor of 2 lower γ than seen in $U_{0.97}Th_{0.03}Be_{13}$ where, essentially, there is no such anomaly.

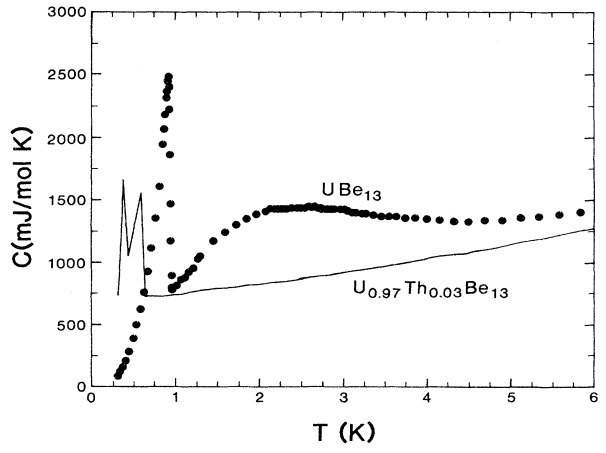

FIG. 5. Specific heat vs temperature for pure (annealed at 1200°C for 3.5 weeks, see text), UBe_{13} (circles) and for $U_{0.97}Th_{0.03}Be_{13}$ (solid line). Note the lack of any significant anomaly in C above T_c for the Th-doped sample, and the coming together of the two sets of data near 6 K.

In terms of the phase diagrams derived for $U_{1-x}Th_xBe_{13}$, it is an open question where and how T_{c1} and T_{c2} merge (see Fig. 3) with increasing Th concentration. At present, based on unannealed, lower-purity samples, it is believed[5] that the two transitions merge at 4.3% at. % Th. Since instead of the shoulder at T_{c1} on $\Delta C(T_{c2})$ observed in an unannealed sample, annealing gives two distinct transitions (Fig. 3) for a 3.8 at. % Th sample, presumably the phase boundary (i.e., where T_{c1} and T_{c2} merge) occurs in well-annealed, high-purity samples at $x > 0.043$.

More important for understanding the second phase transition may be its magnetic behavior. Previous specific-heat data[8] in field (where the lowest field applied was 1 T) were used to suggest that the slope of the critical field ($\equiv H'_{c2}$) at T_{c2} was larger than that (−44 T/K, Ref. 15) for pure UBe_{13}, and that H_{c2} versus T for the lower transition has a higher slope than the H_{c2} versus T data for the upper transition. Our field specific-heat data on annealed $U_{0.97}Th_{0.03}Be_{13}$ are shown in Fig. 4; the midpoint temperatures of the two transitions versus field are shown in Fig. 6. In order to more clearly compare the two H_{c2} curves, the lower curve is plotted in Fig. 7 shifted upwards in temperature by a constant amount such that the two 0.25 T points coincide. Thus, the impression that one gets from Fig. 6, i.e., that the two H_{c2} curves for our high-purity, annealed $U_{0.97}Th_{0.03}Be_{13}$ samples are parallel is, to within a very small deviation, born out in Fig. 7. A simple fit ($H_{c2} = H_{c2}(0)[1-(T/T_c)^2]$) to the three lowest field H_{c2} data points ($H \leq 0.75$ T) gives $H'_{c2} = -42.6$ T/K for the lower transition and −46.9 T/K for the upper transition. (Details of the number of points used in the fit do not affect the essential equality of the two H'_{c2} values.) Thus, we find that, within our error, these values are almost identical, and consistent[16] with

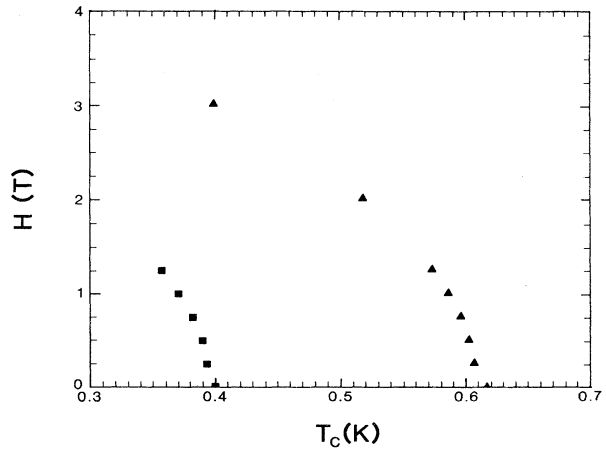

FIG. 6. From the specific-heat data in Fig. 4, the midpoints of the transitions (i.e., T_c) are plotted vs the field applied for both transitions. The zero field T_c for both transitions is shifted to slightly higher temperatures than would be expected from an extrapolation of the low field points. This is also seen in Ref. 8 for unannealed $U_{0.967}Th_{0.033}Be_{13}$, as well as in UPt_3.

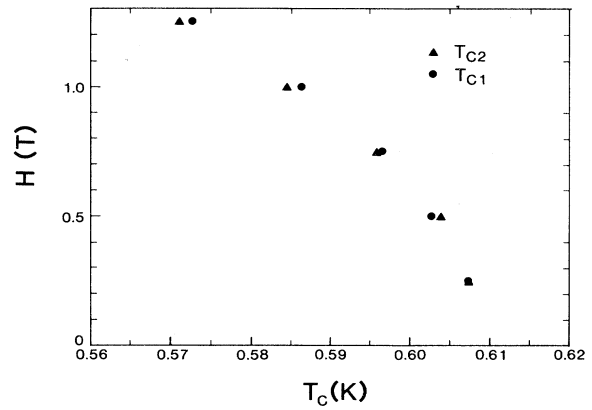

FIG. 7. The data from Fig. 6 are replotted, with all the data from the lower transition (triangles) shifted upwards in temperature by +0.213 K so that the two points at $H=0.25$ T coincide. The slight disagreement between the sets of data is within our error limits.

the value[15] for pure UBe_{13} (−44 T/K). This is in contradiction to the results of Ref. 8.

What do these values mean for understanding the lower transition? It certainly seems coincidental that a superconducting state of different type, with magnetic attributes, has an identical critical field slope at T_c with pure UBe_{13} and, within a small discrepancy, with the upper, "normal" transition of the doped $U_{0.97}Th_{0.03}Be_{13}$. The explanation previously put forward[3] that the two transitions are both normal superconducting transitions from separate, poorly connected parts of the Fermi surface seems to fit the observed equality in H'_{c2} better. If this explanation were indeed correct, then it would follow that in order to calculate $\Delta C/\gamma T_c$ for the two transitions, one would use only a part of the total γ_T of 2300 mJ/mol K^2 for each transition, i.e., $\Delta C_1/x\gamma_T T_{c1}$ and $\Delta C_2/(1-x)\gamma_T T_{c2}$. Using the values from Table I to calculate ΔC for the annealed 3 at. % Th sample (extrapolation of the C/T data—see Fig. 4—to make an idealized, sharp transition changes these ΔC values less than 4%), and assuming $x=0.5$, i.e., $\gamma_1=\gamma_2=1150$ mJ/mol K^2, one gets $\Delta C_1/\gamma_1 T_{c1}=1.33$ and $\Delta C_2/\gamma_2 T_{c2}=1.78$. For the annealed 3.8 at. % Th sample, using $\gamma_1=\gamma_2$ and the values from Table I, $\Delta C_1/\gamma_1 T_{c1}=1.17$ and $\Delta C_2/\gamma_2 T_{c2}=2.02$. Other reasonable assumptions (e.g., that $\Delta C_1/\gamma_1 T_{c1}=\Delta C_2/\gamma_2 T_{c2}$ to find the proportion of γ assignable to each transition) give $\Delta C/\gamma T_c$ values around 1.6 that also are quite normal appearing in comparison with either BCS ($\Delta C/\gamma T_c=1.43$ for weak-coupled superconductivity), with common elemental superconductors (e.g., $\Delta C/\gamma T_c=2.65$ for strong-coupled Pb and 1.60 for Sn), or with pure UBe_{13} itself. ($\Delta C/\gamma T_c=1.66$ for our annealed, high-purity starting material UBe_{13} sample, Fig. 5, using an extrapolated γ of 1100 mJ/mol K^2.) (This value for UBe_{13} is essentially the same as previously seen.[17])

In fact, using the converse explanation, i.e., that the whole Fermi surface, with a $\gamma=2300$ mJ/mol K^2, goes superconducting at T_{c1} in thorium-doped UBe_{13}, gives unrealistically low values of $\Delta C_1/\gamma T_{c1}$ of 0.67/0.59 for 3 at. % Th/3.8 at. % Th. Since at least in the case of our annealed 3 at. % Th sample not much further increase in ΔC due to sharpening of ΔT_c can be expected, such unrealistically low values of $\Delta C_1/\gamma T_{c1}$ must be seen as supporting the idea[3] of the Fermi surface in $U_{1-x}Th_xBe_{13}$ going superconducting in two distinct parts.

As a pure speculation, it is interesting to pursue the implications of this (one piece of the Fermi surface after the other going superconducting) model one step further. It is generally agreed that heavy fermion systems have local moment character of their f electrons at higher temperatures which is somehow (e.g., via Kondo screening) compensated at lower temperatures. The herein observed γ of 2300 mJ/mol K^2, and the therefrom calculated electron effective mass, m^*, for $U_{0.97}Th_{0.03}Be_{13}$, is more than twice that for UBe_{13}, $CeCu_2Si_2$, and UPt_3—the other heavy fermion superconductors. Could the cause of the magnetic moment observed[4,5] at T_{c2} be simply a failure of the compensation mechanism when a gap opens up in the electronic energy spectrum of a very large m^* material? In other words, is the low-frequency part of the f-electron energy spectrum critical for effective f-moment shielding when m^* exceeds a certain limit? If this is the case, then the μSR work[18] that shows no local moment behavior at the huge ΔC observed in $U(Be_{1-x}B_x)_{13}$ done for $x=0.0023$ (where $\Delta C/\gamma T_c=2.8$ and $\gamma=1350$ mJ/mol K^2) needs to be extended to higher x where, although $\Delta C/\gamma T_c$ falls, γ continues to rise[19] up to 2300 mJ/mol K^2.

In summary, annealed high-purity samples of UBe_{13} doped with 3 and 3.8 at. % Th show larger, more separated transitions than observed previously. The critical field behavior of both transitions in the 3 at. % Th sample agrees with that of pure UBe_{13}, while the γ for $U_{0.97}Th_{0.03}Be_{13}$ is a factor of two larger than that for pure UBe_{13}. This increased γ stems from increased degreees of freedom/entropy below 1 K. Thus, the degrees of freedom/entropy lost in pure UBe_{13} at the broad specific-heat anomaly at 2 K appear as the second transition in $U_{1-x}Th_xBe_{13}$.

ACKNOWLEDGMENTS

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