

Crystallographic imperfections and exotic superconductivity of UBe_{13}

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The symmetry of the superconducting gap is related to the symmetry of the crystal structure. In unconventional superconductors, big changes in the critical temperature, critical field, or even the gap structure can happen even for small perturbations of the lattice. In this paper, we use microstructuring to study aluminum-free micro crystals of UBe_{13} which are expected to be closer to a “perfect” material than previously studied aluminum-grown single crystals. We compare the effect of minuscule imperfections on the value of the critical temperature and critical field, which, in the case of UBe_{13} , has drastic effects, supporting its unconventional nature. We conjecture that this likely arises from the oxidation state of uranium, which is sensitive to its crystallographic environment. Our findings suggest that uranium-based materials provide not only an excellent reservoir of unconventional phenomena, but also a way to identify the gap symmetry of unconventional superconductors.

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I. INTRODUCTION

Unconventional superconductivity, i.e., superconductivity with strongly momentum-dependent pairing often driven by a nonphonon mechanism, is observed in many material classes – from high-temperature cuprates and pnictides to low-temperature heavy-fermion materials [1,2]. The response to disorder/imperfections can provide a lot of information on the underlying superconducting order parameter; in particular, this is a common indication of whether or not a given superconductor is unconventional. Unconventional superconductors include several uranium-based compounds. In fact, among nearly two dozen of known uranium-based superconductors, nearly half are currently classified as unconventional, with several spin-triplet candidates [3–8].

Typically, unconventional superconductors are thought to be exceptionally sensitive to crystallographic imperfections

and disorder [9–12]. For many uranium-based systems, this is also the case-minute changes on the atomic level make a large difference in their ground states, which can likely be linked to the oxidation state of uranium, which is very sensitive to its immediate crystallographic environment [13]. Crystallographic defects are tiny and diverse: Stacking faults in UPt_3 [14–16], spatial inhomogeneities of URu_2Si_2 [17–21], as well as uranium deficiencies coupled with the local breaking of translational symmetry in UTe_2 [13,22–27]. UBe_{13} single crystals, grown from aluminum flux, were conveniently available just when the interest in this material first arose [28,29]. However, it took considerable effort to realize and prove that these crystals are in fact never UBe_{13} , but rather $\text{UBe}_{13-x}\text{Al}_x$ [30,31], i.e., that the aluminum from the flux enters the crystal structure at the beryllium position (see the orange region of “unintentional substitution” in Fig. 1). With subsequent temperature treatment, the aluminum atoms start to leave the structure and, with longer annealing, vacancies – as a result of aluminum removal – appear, producing UBe_{13-x} . The respective amounts are small, on the order of 0.1 at. %, but superconductivity of this material changes not only in the value of T_c and the upper critical field curve [34], but also sometimes revealing two superconducting anomalies [31], making UBe_{13} somewhat similar to UTe_2 (although the imperfections in the latter are even more subtle [13]). In $\text{UBe}_{13-x}\text{Al}_x$, the partial substitution of beryllium by aluminum is also accompanied by an increase in lattice

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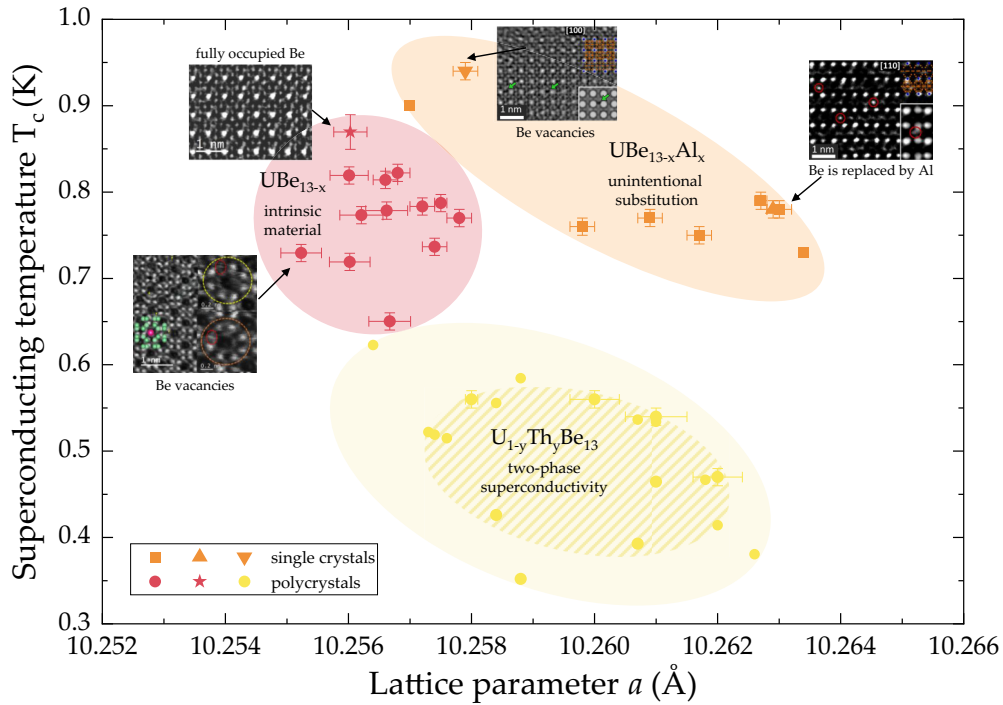


FIG. 1. Interplay of chemistry and physics in $U_{1-y}Th_yBe_{13-x}Al_x$: superconductivity as a function of structural imperfections, as reflected by the value of T_c (vertical axis) vs lattice parameter a (horizontal axis). Three distinct regions exist, representing three different materials – UBe_{13-x} (red, present study), $UBe_{13-x}Al_x$ (orange [31]), and $U_{1-y}Th_yBe_{13}$ (yellow [32,33]) – all showing superconductivity, albeit of different character. In some cases, imperfections on the atomic scale can only be seen by means of transmission electron microscopy (insets).

volume ($\sim 0.05\%$, see Fig. 1), which makes it somewhat easier to track microscopic imperfections quantitatively. Furthermore, the change in volume, induced by unintentional substitution of aluminum from flux, is of similar magnitude to the case when uranium is substituted by thorium [35–37] (see the yellow region in Fig. 1). Remarkably, both $UBe_{13-x}Al_x$ (orange region) and in $U_{1-y}Th_yBe_{13}$ (yellow region) show double superconducting transitions. So, in principle, this could be related to crystallographic strain or disorder. Gallium-grown single crystals show similar behavior to the crystals grown from aluminum flux – likely a result of gallium substitution into the lattice [38].

In this sense, especially considering that currently there is a lack of other established ways to grow single crystals of UBe_{13} , the only way to prepare pure, i.e., impurity-free UBe_{13} , is by crystallizing the binary melt by which one obtains polycrystalline products. This work combines a careful synthesis of UBe_{13} with the microscale isolation of single crystallites, giving access to the nominally cleanest possible UBe_{13} so far, both in terms of structural imperfections and single-phase character.

As demonstrated in this work, even in single-phase UBe_{13} some beryllium vacancies can exist, as a result of the synthetic route. Hence, we now refer to this material as UBe_{13-x} . While from the chemical point of view the amount of this imperfection is nontrivial to quantify, the effect on the superconductivity is dramatic. For a lattice change on the order of 0.006% , the corresponding change in T_c is 28% . In comparison, a similar change in T_c for $YBa_2Cu_3O_{7-\delta}$ is only possible when the unit cell volume is changed by 0.5% [39]. In another uranium-based heavy-fermion superconductor UTe_2 , the vol-

ume change of 0.09% splits one transition into two, while an $\sim 0.5\%$ volume change destroys superconductivity completely [13]. Even when high-energy electron irradiation is employed to gently perturb the lattice of unconventional superconductors, a stronger disorder is typically induced [40–45]. These examples show how large the effects of minute changes in lattice on the critical temperature are in UBe_{13} , placing it in a separate paradigm, even among unconventional superconductors.

II. RESULTS AND DISCUSSION

A. Structural characterization

Polycrystalline samples of UBe_{13-x} obtained by arc-melting contain U or Be as a secondary phase, depending on the starting material composition. Physical properties of polycrystalline UBe_{13-x} are affected by the grain boundaries, produced by both types of minority phases (see Fig. 2). The grains of UBe_{13-x} are in fact fairly large, at least $50\ \mu m$ in size, as evident from Fig. 2(a). By using a focused ion beam patterning, we create single-grain devices from these samples. A careful device design [46,47] can circumvent U or Be inclusions [see Figs. 2(b)–2(e)] giving access to the intrinsic resistivity of UBe_{13-x} . Contrary to previous studies, starting compositions far from 1:13 have been considered in this work; the nominal elemental ratios start with $UBe_{7.56}$ (UBe_{13-x} with U as the secondary phase) and go to $UBe_{23.76}$ (UBe_{13-x} with Be as the secondary phase). An unexpected result of this exploration was the large variation in the value of critical temperature T_c for UBe_{13-x} grown from either U or Be excess. By significantly changing

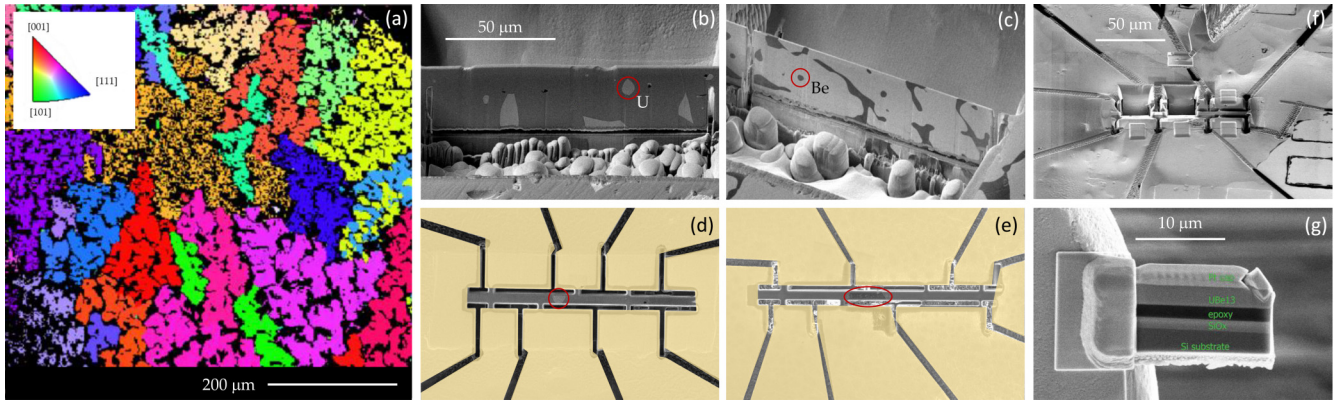


FIG. 2. Extraction of intrinsic electrical resistivity of UBe_{13-x} : (a) Electron backscatter diffraction is used to estimate the grain size. (b) and (c) Inclusions of U (light gray) or Be (dark gray) secondary phases is easily recognizable in the lamellae, extracted from polycrystalline material. (d) and (e) Microscale devices, fabricated from polycrystalline samples. The current flow is left to right, which means that several voltage pairs avoid inclusions of U or Be; this allows to capture the intrinsic resistivity of UBe_{13-x} . (f) and (g) A piece from a microscale device is then used to pinpoint defects on the atomic scale by means of transmission electron microscopy.

the starting U to Be ratio, the value of T_c is changed by nearly 30%, namely, from 0.65 to 0.88 K (see Fig. 3). The corresponding change in the lattice parameter a for the whole range is 0.04%, while the difference for the samples with the highest (green) and lowest (red) T_c is only 0.006%. Overall,

the unit cell parameter a is staying virtually the same (see the red region of Fig. 1). This region is in stark contrast to the orange and yellow regions of the same figure. What is revealed by the transmission electron microscopy [Figs. 3(e) and 3(f)] on pieces extracted from the microscale devices [Figs. 2(f) and

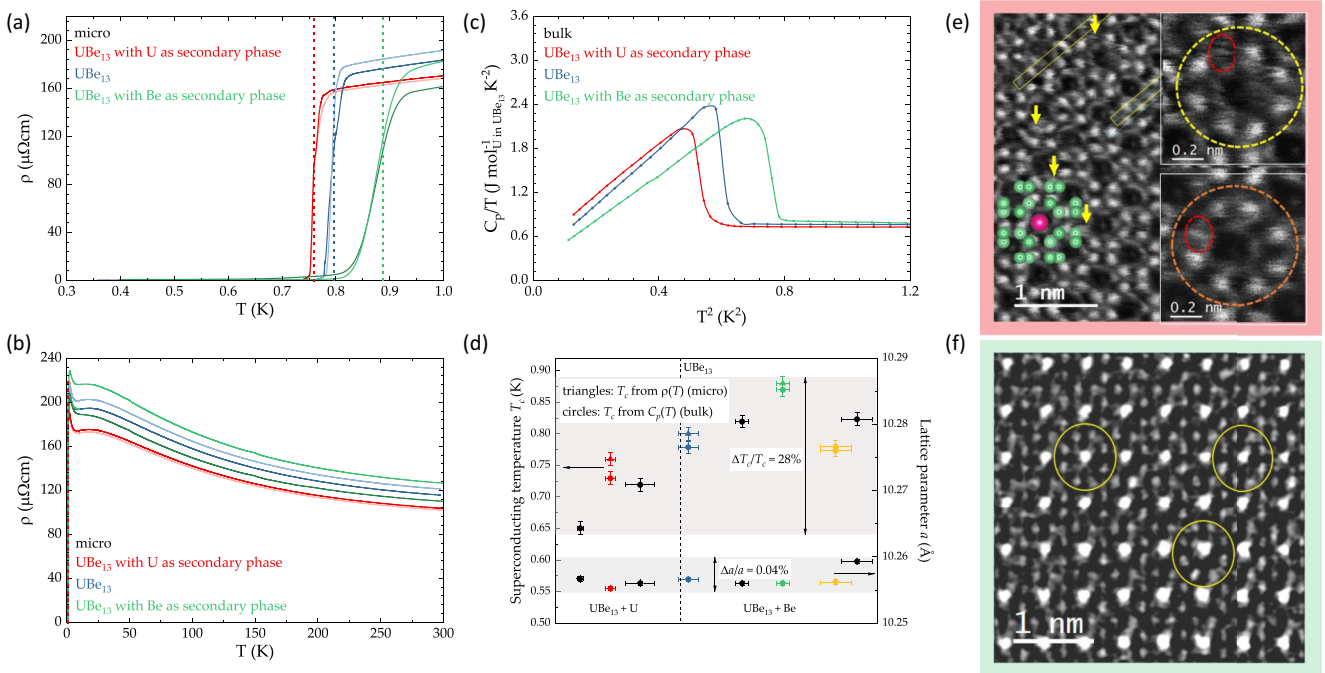


FIG. 3. Intrinsic superconductivity of UBe_{13-x} : (a) and (b) Microscale resistivity reveals that the superconducting transition temperature of UBe_{13-x} is changing by almost 30%, depending on whether polycrystalline material is prepared with U (red) or Be (green) excess. For all samples, only one superconducting anomaly is observed, with its position being different for different starting U:Be ratios. (c) The specific heat data follow the same trend. (d) All of the UBe_{13-x} grains within a given sample have the same composition, given the agreement between the value of T_c extracted from the microscale (triangles, resistivity) and bulk (circles, specific heat) data. The corresponding change in the lattice parameter a is negligible (right axis). (e) and (f) The only difference is seen in the transmission electron microscopy, revealing imperfect UBe_{13-x} (red) and perfect UBe_{13} (green) material. Panels (e) and (f) show the samples with red and green markers in (d), respectively. Yellow arrows mark beryllium vacancy positions – the exact amount of this beryllium deficiency is estimated to be below 0.1%.

2(g)] is that the amount of Be in the UBe_{13-x} structure varies slightly. Quantification of Be by means of traditional chemical analysis is not always trivial [48–52] and for this study, a single-crystal analysis was not possible since a single-phase specimen of a suitable size ($20 \times 20 \times 20 \mu\text{m}^3$) could not be isolated. Nonetheless, the transmission electron microscopy, coupled with lattice parameter refinements based on powder data, give an insight into the structural variations in UBe_{13-x} . It is important to note that while the importance of Be in UBe_{13} was suspected, it has not been investigated much up until now [31,36,53,54]. It is of course crucial to understand why the removal of less than one out of 24 Be atoms (average observed by transmission electron microscopy) within the environment of U in UBe_{13-x} makes such a big difference. Again, quantitatively speaking, it is hard to put a number on the abundance of this defect. Comparing with UTe_2 , where the lattice parameter change on the order of 0.09% arises from a defect on the order of 0.01 [13], it is reasonable to suppose that the amount of beryllium deficiency in UBe_{13-x} is even less. In principle, with the U-Be distances ($d_{\text{U-Be}} = 3.012 \text{ \AA}$) being much smaller than the U-U distances ($d_{\text{U-U}} = 5.128 \text{ \AA}$), the structure of UBe_{13-x} can be represented by U-Be polyhedra with a coordination number of 24. In this regard, it is instructive to examine what happens in conventional superconductors with cagelike structures.

A strong sensitivity to small changes in the lattice has been observed in several cagelike superconductors, including YB_6 [55–59], Lu/YB_{12} [60–62], $\text{Th}_4\text{Pt}_{16}\text{Be}_{33}$ [63–66], $\text{LaRu}_4\text{As}_{12}$ [67], $\text{Ba}_8\text{Ge}_{43}$ [68,69], $\text{Ba}_6\text{Ge}_{25}$ [70–72], and $\text{Ba}_8\text{Si}_{46}$ [73–76]. Here, small changes in the occupancy of the cage drive changes in the atomic environment at the center of the cage, strongly influencing the bonding within the structure. In principle, for phonon-mediated superconductivity, which is driven by (i) density of states at the Fermi level and (ii) electron-phonon coupling, a structural change will directly affect phonon frequencies. In fact, it has been shown previously that “rattling” can enhance both superconductivity [77–81] as well as effective electron mass [82,83]: in general, the larger the cage, the stronger is the “rattling” [84]. In this sense, both superconductivity and the enhanced effective electron mass of UBe_{13-x} could, in principle, originate from “rattling.” However, a crystallographic analysis of UBe_{13-x} [85], as well as other related compounds with the NaZn_{13} -type cubic structure [86], did not point to rattling, consistent with the superconductivity of UBe_{13-x} not being phonon-driven. Another parameter is likely at play, namely the oxidation state of uranium. This has also been stipulated to be the reason for the T_c suppression in UTe_2 [13,87]. This scenario is supported by the magnetic susceptibility data, shown in Fig. S2 [88] – both the effective magnetic moment and Weiss temperature, extracted from the Curie-Weiss fits, change for different UBe_{13-x} samples, showing that the local environment has a significant effect on the f -electron state. It is important to note that the previously reported anomaly in magnetic susceptibility data around $T = 160 \text{ K}$ (single crystals of $\text{UBe}_{13-x}\text{Al}_x$ [89]) can likely arise due to the presence of Be-doped U (see Fig. S2 [88]). Nonetheless, the intrinsic magnetism of UBe_{13-x} does appear to change: the Curie-Weiss fitting in the 200–300 K temperature range reveals that the effective magnetic moment (μ_{eff}) and magnetic correlation

strength (as reflected by the Weiss temperature Θ_W) are actually the smallest for the sample with full Be occupancy and highest T_c (green symbols). It is also likely that the valence electron concentration in the Be framework of UBe_{13-x} is able to accommodate these valence fluctuations of U. This is contrasted by the case of UB_{12} [90], where no superconductivity is observed, even though structurally these two materials are quite similar. Another possible effect of Be variation in UBe_{13-x} is a very small (beyond detectable) change in the cage volume, i.e., application of minuscule pressure. This can, in principle, have rather significant effects on the band structure and effective masses that could affect the superconducting state. Though we argue below that the superconductivity of UBe_{13-x} does not appear to be extremely sensitive to pressure tuning.

B. Strain effects

Since UBe_{13-x} microcrystals are always surrounded by a secondary phase of either Be or U, which contracts upon cooling differently from UBe_{13-x} , it is necessary to examine the possible effect of strain. It is known that T_c of unconventional superconductors can be very sensitive to strain, e.g., in ruthenate [91] or iron-based superconductors [92]. For Co-doped CaFe_2As_2 , it was demonstrated that the superconducting properties can even be effectively manipulated through the strain imposed by flux inclusions [93,94]. Using previous reports of the uniaxial pressure sensitivity, derived from thermal expansion, $dT_c/dp_a \approx 4 \text{ mK/kbar}$ and a bulk modulus of 108 GPa [95], we estimate the sensitivity of T_c to strain, $\varepsilon_a = (\Delta a/a)$, to be $dT_c/d\varepsilon_a \approx 4 \text{ K}$. Thus, in order to explain a change of 30% in the value of T_c , a tensile strain of about 2.5% would be needed. However, the differential thermal expansion between UBe_{13} and elemental Be (see Supplemental Material [88]) is vanishingly small and results in tensile strains on the order of 0.01% only. Even the differential thermal expansion between UBe_{13} and elemental U induces a maximum strain of about -0.15% at low temperatures. Moreover, microscale (possible strain due to a substrate [96]) and bulk (possible strain due to U/Be inclusions) trends in the evolution of T_c are identical [triangles versus circles in Fig. 3(d)]. We therefore conclude that strain effects created by the U/Be inclusions cannot fully account for the shift of T_c , observed in this study.

C. Thermodynamic measurements

Overall, it appears that superconductivity in UBe_{13-x} is very sensitive to the exact ratio of U and Be with a large variation of T_c . It is important to distinguish UBe_{13-x} vs $\text{UBe}_{13-x}\text{Al}_x$ vs $\text{U}_{1-y}\text{Th}_y\text{Be}_{13}$ – as summarized in Fig. 1. In previous studies, a number of experimental results on aluminum-flux-grown single crystals and arc-melted polycrystalline samples have been reported, and their conflicts regarding superconducting symmetries make it difficult to reach a coherent understanding: while a specific heat analysis [37,97], Josephson tunneling [98], and penetration depth measurements [99] have been performed on a polycrystalline material (i.e., UBe_{13-x}), a number of studies on single crystals (i.e., $\text{UBe}_{13-x}\text{Al}_x$) [100–103] should be considered separately.

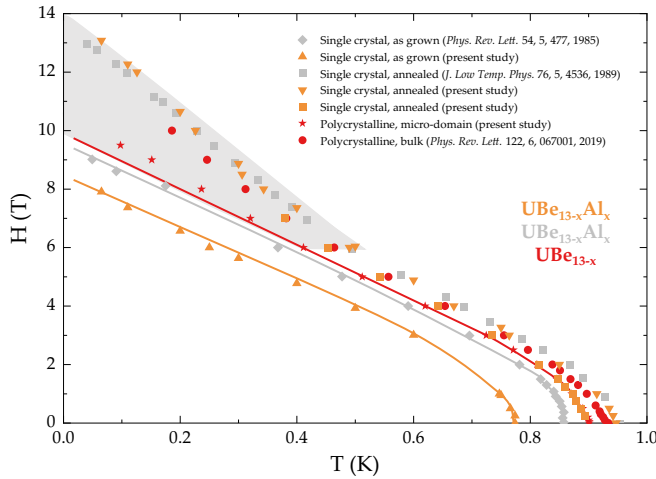


FIG. 4. Comparison of critical fields of $\text{UBe}_{13-x}\text{Al}_x$ and UBe_{13-x} from previous studies on single crystals (gray symbols [29,111]), together with the current work on single crystals (orange), polycrystalline microscale (red stars), and bulk (red circles) samples.

This raises the question of what kind of superconducting symmetry is realized in UBe_{13-x} and if it is different from the one in $\text{UBe}_{13-x}\text{Al}_x$.

The upper critical field of $\text{UBe}_{13-x}\text{Al}_x$ is anomalous (see Fig. 4), far exceeding the BCS Pauli limit ($\mu_0 H_P \simeq 1.6$ T), which supports an occurrence of a spin-triplet superconductivity [97,104–106]. In the O_h cubic symmetry fully gapped spin-triplet representations exist [107]: The fully gapped A_{1u} state or a mixed state of A_{1u} and the (nematic) E_u state, consistent with the nodeless behavior in low-temperature specific heat [108]. The pronounced curvature of $H_{c2}(T)$ near T_c indicates strong paramagnetic effects and an enhanced H_P , which is also compatible with the A_{1u} state [109,110]. Furthermore, samples with a high T_c exhibit an upturn of H_{c2} at roughly $T/T_c = 1/2$, whereas lower- T_c samples do not [34]. The origin of this upturn has been controversial [34,111–114] and can in principle have various reasons such as strong-coupling or multiband effects, or field-enhanced pairing interactions. A successful description combines a field-induced weak E_u component mixed with the dominant A_{1u} state. Our measurements of H_{c2} on two single crystals, grown from aluminum flux, as well as those obtained on microscale devices made out of arc-melted polycrystals, shown in Fig. 4, confirm this trend without a noticeable difference between UBe_{13-x} and $\text{UBe}_{13-x}\text{Al}_x$. Although the dependence of the superconducting transition temperature T_c on the lattice parameter a differs systematically between UBe_{13-x} and $\text{UBe}_{13-x}\text{Al}_x$ (Fig. 1), the impacts of beryllium vacancies versus aluminum inclusions on the critical field curve in UBe_{13} seem to be similar.

III. SUMMARY AND OUTLOOK

It is clear that UBe_{13} is an unconventional superconductor, which makes its ground state highly susceptible to atomic-scale imperfections on both U and Be sites. This material is also one of the oldest spin-triplet candidates, which makes a retrospective analysis of lattice quality vs the observed phe-

nomena much needed. In this paper, we establish an extreme sensitivity of UBe_{13} to beryllium defects: the removal of less than 0.1% of beryllium does not change the unit cell volume, but reduces T_c by nearly 30%. It is important to note that in the UBe_{13} sample with the highest T_c , we do not find any defects (beryllium or other kinds), despite implementing the same meticulous analysis on both macro- and atomic scales: The numerous transmission electron microscopy images, obtained as part of this study, do not show any indications of imperfections. While it is inherently difficult to prove crystallographic perfection, the samples of UBe_{13} with the highest T_c are different from both polycrystalline and single-crystalline samples of UBe_{13} for which we conclusively show defects. Even for an unconventional superconductor, such a delicate structure-property relationship is unusual.

The similar trend between T_c and the upper critical field curve of UBe_{13-x} and $\text{UBe}_{13-x}\text{Al}_x$ might be explained with a similarity of the samples with highest T_c . Since annealing of Al-grown single crystals removes Al from the structure and leaves Be vacancies [31], it seems that high- T_c samples from both growth methods share a small amount of Be vacancies. In the picture of admixed A_{1u} and E_u triplet superconducting states in UBe_{13} [104,105], variations in their relative weight explain the observed trends: Since the A_{1u} state is a fully gapped superconducting state with a well-defined phase, it is sensitive to imperfections, but likely more robust than the lower symmetry order parameter such as E_u . Thus, for higher- T_c samples, A_{1u} prevails, and a weaker E_u leads to the anomalous upward curvature in H_{c2} for $T_c/2$. For samples with lower T_c , imperfections lead to a suppression of both components with a stronger reduction of the E_u state, so that the upturn disappears. The temperature dependence of the critical field of the microscale UBe_{13} (stars in Fig. 4) is in between those extreme cases and shows a small upturn. Hence, the imperfections leading to this relation between the upturn and T_c seem to be similar in aluminum-free polycrystals with beryllium vacancies and single crystals with aluminum inclusions.

The UBe_{13} material investigated in this work exemplifies a known challenge in condensed matter physics: sample-to-sample variations that can lead to unreliable and irreproducible results [115]. We overcome the synthetic challenge by using microcrystallites to establish the intrinsic properties of UBe_{13} . This work provides a methodological example of how reducing the sample size can enable the reliable determination of the intrinsic properties of even relatively complex solid-state materials. This is particularly crucial for compounds whose properties cannot be reliably predicted from theoretical considerations alone.

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DATA AVAILABILITY

The data that support the findings of this article are not publicly available upon publication because it is not technically feasible and/or the cost of preparing, depositing, and hosting the data would be prohibitive within the terms of this research project. The data are available from the authors upon reasonable request.

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