Unusual sensitivity of superconductivity to strain in iron-based 122 superconductors

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Co-doped $BaFe_2As_2$ has been previously shown to have an unusually significant improvement of T_c (up to 2 K, or almost 10%) with annealing 1–2 weeks at 700 °C or 800 °C, where such annealing conditions are insufficient to allow significant atomic diffusion. While confirming similar behavior in optimally Co-doped $SrFe_2As_2$ samples, the influence on T_c of strain induced by grinding to ∼50 μm sized particles, followed by pressing the powder into a pellet using 10 kbar pressure, was found to increase the annealed transition width of 1.5 K by approximately a factor of ten. Also, the bulk discontinuity in the specific heat at T_c, ΔC, on the same pellet sample was completely suppressed by grinding. This evidence for a strong sensitivity of superconductivity to strain was used to optimize single-crystal growth of Co-doped $BaFe_2As_2$. This strong dependence (both positive via annealing and negative via grinding) of superconductivity on strain in these two iron based 122 structure superconductors is compared to the unconventional heavy Fermion superconductor UPt_3, where grinding is known to completely suppress superconductivity, and to recent reports of strong sensitivity of T_c to damage induced by electron-irradiation-induced point defects in other 122 structure iron-based superconductors, $Ba(Fe_{0.76}Ru_{0.24})_2As_2$ and $Ba_{1-x}K_xFe_2As_2$. Both the electron irradiation and the introduction of strain by grinding are believed to only introduce nonmagnetic defects, and argue for unconventional superconducting pairing.

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I. INTRODUCTION

Since the discovery [1] of iron-based superconductivity (for reviews, see Refs. [2–5]), a number of unusual properties in these fascinating materials have been discovered. Sefat *et al.* discovered [6] superconductivity in $Ba(Fe_{1-x}Co_x)_2As_2$, with the peak of the superconducting transition temperature T_c, dome versus composition at $x = 0.1$ and the maximum T_c equal to 22 K in as-prepared single crystals.

One of the properties of these materials which aroused interest was the substantial increase in T_c in $Ba(Fe_{1-x}Co_x)_2As_2$ with annealing. Gofryk *et al.* [7], in the first annealing experiments, reported that crystals of $Ba(Fe_{1-x}Co_x)_2As_2$ attained $T_c = 25$ K and a decrease in the transition width ΔT_c of 25% after 2 weeks at 800 °C. Kim *et al.* [8] further investigated T_c versus annealing in $Ba(Fe_{1-x}Co_x)_2As_2$. They found in their optimized self-flux grown samples that as-prepared crystals with $T_c = 25$ K reached T_c values as high as 26.6 K via annealing for 1 week at 700 °C. With further optimization using finer gradations (in all, 17 different compositions between $x = 0.05$ and 0.30) in Co concentration, Tam *et al.* [9] found as-prepared $Ba(Fe_{1-x}Co_x)_2As_2$ crystals with $T_c = 25.5$ K, with comparable annealing [10] resulting in $T_c^{\mathrm{onset}} = 27.2$ K.

Such annealing, for 1 week at less [11] than 60% of T_{melt} (i.e., without appreciable atomic diffusion) of pure $BaFe_2As_2$, resulting in such rapid increases in T_c (∼1.7 K or ∼7% of T_c), seemed more effective than in other superconductors. [For example, annealing elements like Cd or Zn to narrow ΔT_c is done [12] at 95% of T_{melt}. The 14.4-K T_c superconductor YNi_2B_2C, prepared by melting together the constituents, when annealed at 1200 °C for 5 days (72% of T_{melt} [13]), shows [14] no change either in T_c or ΔT_c.] The possibility that this unusual response of T_c with annealing is a clue to the unusual superconductivity in iron-based superconductors motivated us in the present work to further investigate annealing in a second 122 structure system, $Sr(Fe_{1-x}Co_x)_2As_2$.

The results of this investigation as detailed below suggest a possible answer to the puzzle first posed by Gofryk *et al.* of why superconductivity in $Ba(Fe_{1-x}Co_x)_2As_2$ is improved so rapidly with relatively short annealing at only ≈60% of T_{melt}.

II. EXPERIMENTAL

Single-crystal samples of nominal composition $Sr(Fe_{0.86}Co_{0.14})_2As_2$ (near optimal doping) were prepared using self-flux growth techniques as in Refs. [8,9]. The crystals nucleate out of self-flux (FeAs) during a slow cool (3 °C/hr) between 1200 and 900 °C, followed by a more rapid cooling (75 °C/hr) to room temperature. Crystals are then separated from the self-flux mechanically. A single crystal of mass 18.3 mg was chosen for a series of measurements on the same sample: measurement of magnetic susceptibility χ and specific heat C on the unannealed crystal, annealing (700 °C for 2 weeks) of this crystal in an outgassed alumina crucible sealed via arc melting into a niobium cylinder containing an As vapor source [8–9], then measurement of χ and C on the annealed crystal.

Following the comparison of χ and C for the same single crystal, unannealed and annealed, the annealed crystal was ground in an inert atmosphere glove box in an agate mortar for 2–3 minutes for x-ray characterization. Before being x-rayed, the powder was pressed into a pellet [at 150 000 psi (10 kbar) to avoid poor thermal contact between the grains] and the susceptibility of the pressed powder pellet was measured. When these data showed severe degradation of the superconductivity, the specific heat on the pressed powder pellet was also measured. All of these measurements were on the same 18.3-mg single crystal, or the 12.9-mg pressed pellet from the powder therefrom.

The size of the powder making up the pellet (and of the powder made from a second crystal discussed below) was roughly determined by breaking it up as gently as possible with the blunt end of a wooden Q-tip and passing the powder through successively-sized sieves. Approximately half of the powder passed through a 270 mesh sieve (hole size 53 μm) and none of the powder passed through a 325 mesh sieve (hole size 45 μm).

In order to obtain an x-ray pattern on annealed powder to compare linewidths and therefore strain between ground and annealed samples, a separate [15] crystal of mass 43 mg was ground in the inert atmosphere glove box. One part of the powder was x-rayed and measured via magnetic susceptibility and a second part annealed (without an As vapor source) for 2 weeks at 700 °C, and then measured by x-ray diffraction as well as by magnetic susceptibility. In addition, x-ray diffraction was measured on the unannealed single crystal to obtain the linewidths of 00L reflections as discussed below.

III. RESULTS

It was expected from the previous annealing work [8–9] on $Ba(Fe_{1-x}Co_x)_2As_2$ crystals that T_c^{onset} and the bulk transition width of the specific heat of the single crystal of $Sr(Fe_{0.86}Co_{0.14})_2As_2$ would increase and narrow respectively upon annealing for 2 weeks at 700 °C. The susceptibility (Fig. 1) and specific heat (Fig. 2) data of the unannealed and annealed 18.3-mg single crystal of $Sr(Fe_{0.86}Co_{0.14})_2As_2$ confirm this expectation. T_c^{onset} increases with annealing as measured by the susceptibility/bulk specific heat by ∼1.5 K/0.9 K and the transition in the specific heat at T_c, ΔC, sharpens considerably. These results are indeed comparable to those [7–9] in optimally doped $Ba(Fe_{1-x}Co_x)_2As_2$.

What was not known previously [7–9] is the very strong influence of grinding, followed by pressing into a pellet, on the superconductivity. As shown in Fig. 1, grinding [16] the annealed 18.3-mg crystal to a grain size of no smaller than 45 μm, followed by pressing into a pellet, results in a large increase (from ∼1.5 K to over 14 K) in the transition width of the superconducting transition as measured by the magnetic susceptibility. Further, as shown in Fig. 3, the specific heat of the pressed pellet of this same ground powder shows that the specific heat discontinuity at T_c, ΔC, present in both the annealed and unannealed crystal (Fig. 2) is totally smeared out in the ground material.

In order to quantify the amount of strain introduced by the grinding, a separate 43-mg Co-doped $SrFe_2As_2$ crystal was ground [15] in the same fashion, and some of the (homogenized) powder was annealed for 2 weeks at 700 °C. We then measured x-ray diffraction and susceptibility on a portion [15] of the unannealed powder from this second crystal as well as on an annealed portion of the powder. The susceptibility of the unannealed starting crystal and on the ground powder are consistent with the results in Fig. 1, while the susceptibility data of the annealed powder are consistent both in T_c^{onset} and transition width with the annealed single crystal shown in Fig. 1, i.e., the annealed powder T_c and ΔT_c are improved vis-à-vis the unannealed single crystal. Analysis [17] (Fig. 4) of the x-ray linewidths of various (*hkl*) reflections of the annealed and unannealed powders between 55° and 110° 2Θ results in a strain ε for the annealed powder of 0.0008 ± 0.0001 and for the unannealed powder of 0.0011 ± 0.0001, a small but—as evident from Fig. 4—easily measurable [18] difference. Thus, although the annealing does cause a change in the amount of strain in the material, the small amount of the difference indicates a high sensitivity of the superconductivity to strain. Consistent with the susceptibility result (not shown) just discussed [that T_c (unannealed crystal) $<$ T_c (annealed

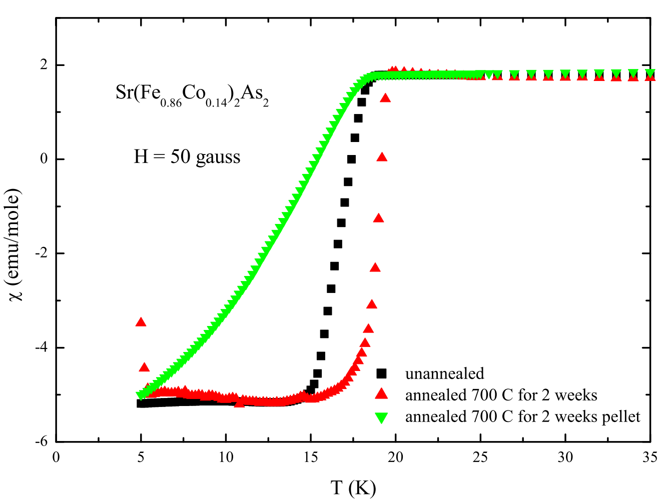

FIG. 1. (Color online) Magnetic susceptibility χ vs temperature of an 18.3-mg single crystal of $Sr(Fe_{0.86}Co_{0.14})_2As_2$ in three conditions: unannealed crystal (black squares), crystal after annealing at 700 °C for 2 weeks (red triangles), and ground powder pressed into a pellet (green inverted triangles), >45 μm diameter, made from the annealed single crystal. T_c^{onset} increases ∼1.5 K with annealing, while the transition width ΔT_c decreases from 2.4 to 1.5 K.

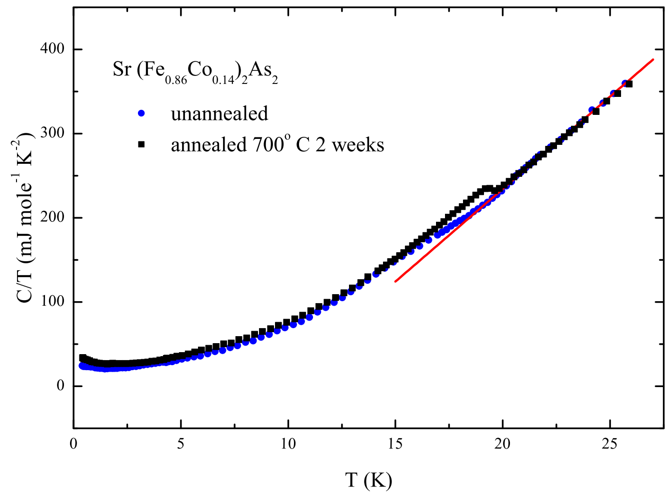

FIG. 2. (Color online) Specific heat divided by temperature *C/T* vs temperature of an 18.3-mg single crystal of $Sr(Fe_{0.86}Co_{0.14})_2As_2$, unannealed (solid blue squares) and annealed at 700 °C for 2 weeks (solid black circles.) T_c^{onset} as measured by the bulk specific heat improves by ∼0.9 K with annealing, comparable to work [8] on $Ba(Fe_{1-x}Co_x)_2As_2$. The red line is an extrapolation of the normal state data to below T_c. The finite intercept of C/T (T → 0), defined as $\gamma_{residual}$, in these samples is ∼20 mJ/molK^2, which is larger than that found [8] in Co-doped $BaFe_2As_2$ and could indicate the presence of some normal material in the crystal. However, the measured discontinuity in C at $T_c = 19.5$ K, $\Delta C/T_c = 19$ mJ/molK^2 in the annealed sample is within 25% of that [8] for Co-doped $BaFe_2As_2$.

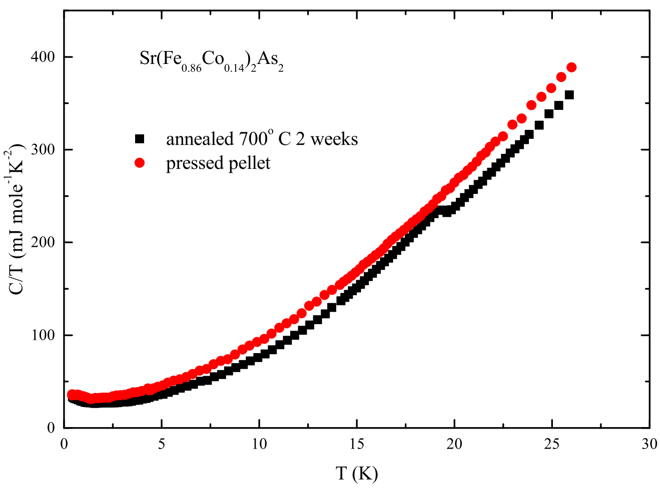

FIG. 3. (Color online) Specific heat divided by temperature *C/T* versus temperature for annealed single crystal, 18.3 mg (solid black squares) and a pressed pellet of the ground powder, 12.9 mg (solid red circles), >45 μm diameter, from the same annealed crystal of $Sr(Fe_{0.86}Co_{0.14})_2As_2$. The absolute error bar for the pressed pellet data above 20 K is ±5% vs ±3% for the single crystal due to the larger addenda contribution (38% vs 17%). However, the relative precision (±1%–2%) between the two measurements is sufficient to state that the larger *C/T* for the pressed pellet sample is qualitatively correct.

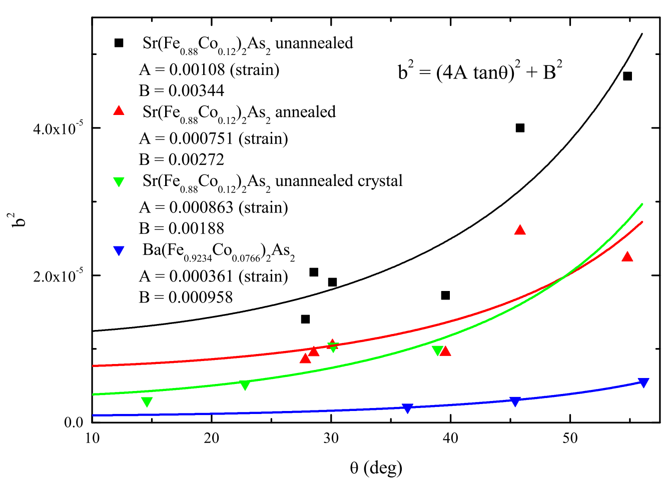

FIG. 4. (Color online) Analysis of the linewidth, *b* (radians), vs angle of the x-ray reflections (*hkl*) to determine strain of unannealed (black squares) and annealed (red triangles) powder, as well as—using (00L) reflections—of an as-grown single crystal (green inverted triangles) of $Sr(Fe_{0.88}Co_{0.12})_2As_2$ and of a cooled-to-600 °C crystal (i.e., quasiannealed) of $Ba(Fe_{0.9234}Co_{0.0766})_2As_2$, blue inverted triangles. For example, for the (0010) line the full width (when plotted vs Θ) at half maximum in units of 10^{-3} radians for the four different samples is 4.15, 3.09, 3.15, and 1.45 respectively. "B" is the instrumentally caused line broadening. Errors bars for strain are ±0.00005 except for the Co-doped Ba 122 sample, where the error bar is only ±0.00002. (Note that a term $(\{0.9\lambda/D\cos\Theta\}^2)$ in the equation for the linewidth that involves the particle size, *D*, is omitted since, with $D \sim 50\,\mu$m, the term is negligible.)

powder)], the analyzed strain in Fig. 4 in the unannealed single crystal of $Sr(Fe_{0.88}Co_{0.12})_2As_2$ is indeed slightly larger [19] than that of the annealed powder, 0.000863 versus 0.000751.

These results make it evident that the superconductivity in Co-doped $SrFe_2As_2$ is (1) very sensitive to strain and (2) the strain present in ground powder, with its very broadened ΔT_c and $\Delta C \to 0$, vs that in annealed material differs by a relatively small amount (~30%). Thus the question arises: what is the minimal amount of annealing necessary to improve the superconductivity in unannealed single crystals? Phrased in another way, what minimal further heat treatment on the as-grown self-flux crystals [with the slow (3 °C/hr) cooling halted at 900 °C] is necessary to remove the strain that the present work implies is introduced by cooling from 900 °C to room temperature at 75 °C/hr? Reference [8] states that annealing at 600 °C has essentially no effect on T_c^{onset} or ΔT_c in Co-doped $BaFe_2As_2$, therefore presumably removing this small amount of residual strain in the as-grown single crystals cooled slowly to 900 °C requires thermal treatment above 600 °C.

In order to make a first attempt at answering this question, and in a different 122 structure iron based superconductor in order to broaden the applicability of these results, we undertook the following. To verify indeed that the strain involved is produced by cooling at 75 °C/hr the as-grown crystals from 900 °C to somewhere above 600 °C (based on the Ref. [8] result), we have reproduced/altered the growth procedure in our previous thorough study of annealing in Co-doped $BaFe_2As_2$ (Refs. [8,9]) for $x = 0.0766$ (a composition slightly below that of optimal doping) as follows. Two batches of $Ba(Fe_{0.9234}Co_{0.0766})_2As_2$ crystals were grown in self flux, one heated to 1200 °C, cooled at 3 °C/hr until 900 °C, followed by cooling at 75 °C/hr to room temperature (the original procedure, followed also herein for Co-doped $SrFe_2As_2$). The second batch was identical in every respect except it was cooled from 1200 °C down to 600 °C at 3 °C/hr, and then at 75 °C/hr to room temperature. This extra temperature region of slow cooling did not result in larger crystals, since the crystals have already formed [20] by 900 °C, but it adds slow cooling (roughly equivalent to annealing for the same length of time at a fixed intermediate temperature) over a period of about 3 days from 900 down to 700 °C. The susceptibility of single crystals from both batches is shown in Fig. 5. Clearly, the strain removed by annealing at 700 °C for 2 weeks in the present work, or at 700 °C for 1 week as in Refs. [8,9], can also be removed by merely cooling at 3 °C/hr further down in temperature, past the previous 900 °C changeover-in-cooling rate point, to 600 °C. In fact, as shown in the strain analysis graph, Fig. 4, the cooled-to-600 °C crystal of $Ba(Fe_{0.9234}Co_{0.0766})_2As_2$ shows a strain only half of that of the annealed for 2 weeks at 700 °C powdered sample—arguing for the effectiveness of the slow cooling procedure.

IV. DISCUSSION AND CONCLUSIONS

The first conclusion that can be reached is the solution to the puzzle raised by the previous annealing work [7–9] on Co-doped $BaFe_2As_2$: why does annealing 1–2 weeks at 700 °C, only 60% of T_{melt} have such an important effect on T_c^{onset} and the bulk specific heat transition width, ΔT_c? Clearly, the superconductivity in both the Co-doped $SrFe_2As_2$ and

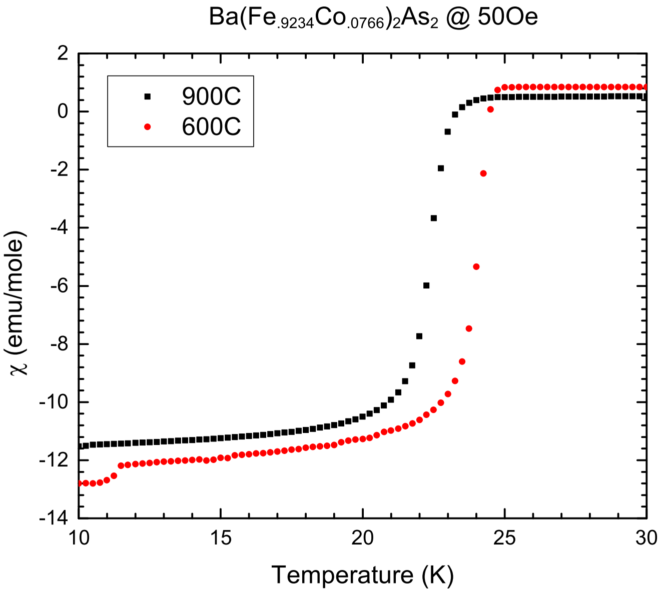

FIG. 5. (Color online) Magnetic susceptibility χ vs temperature for single crystals with the nominal composition of $Ba(Fe_{0.9234}Co_{0.0766})_2As_2$ prepared either by cooling at 3 °C/hr from 1200 °C to 900 °C, followed by cooling at 75 °C/hr to room temperature (black points) or by cooling at 3 °C/hr down to 600 °C, followed by cooling at 75 °C/hr to room temperature (red points). The difference in T_c (either midpoint or onset) is approximately 1.7 K, the same as found [9] after 1 week at 700 °C annealing as-grown crystals slow cooled down to 900 °C. Note the somewhat sharper onset to superconductivity upon cooling in the red points, whereas the black data is somewhat more rounded.

$BaFe_2As_2$ 122 iron based superconductors—and presumably other 122's and 111's as well (although see the discussion below of $CaFe_2As_2$)—is extremely sensitive to strain as shown by the results presented above.

Before we discuss why T_c and ΔT_c are in Co-doped $SrFe_2As_2$ and $BaFe_2As_2$ are so sensitive to strain, we now make a digression in order to discuss whether the current results will apply to Co-doped $CaFe_2As_2$. First, it is important to note that results exist detailing how annealing affects the normal state properties in all three 122's. Annealing (at either 350 or 700 °C between 1 and 30 days) has been shown [21] to have a relatively small (1–6 K) effect on the magnetic and structural transitions for $BaFe_2As_2$ and $SrFe_2As_2$ (at 135 and 200 K, respectively), and to change the *c*-axis lattice parameters at room temperature by less than 0.01 Å. This is in stark contrast to $CaFe_2As_2$ (quenched from 960 °C in order to decant the crystals from the FeAs self-flux), where annealing at 400 °C for 1 week changes [22] the low temperature structure from a noncollapsed tetragonal phase below 100 K to an orthorhombic, antiferromagnetic state below 170 K, and increases [21] the *c*-axis lattice parameter by 0.152 Å. As an explanation for the "extreme case" of $CaFe_2As_2$, Ref. [22] explains that the effect of annealing is to remove very fine, ∼10 nm, precipitates whose average strain field mimics the effect of 0.4 GPa pressure, and to allow the formation of the necessary-for-superconductivity orthorhombic antiferromagnetic state. Since (1) Refs. [8,9] found that annealing at temperature *above* 600 °C is necessary to improve T_c in $Ba(Fe_{1-x}Co_x)_2As_2$ (where the current work tells us that the annealing is causing the removal of strain harmful for superconductivity), and since (2) Ref. [23] finds that annealing at 600 °C and above in Co-doped $CaFe_2As_2$ forms the noncollapsed tetragonal, non-magnetic, inimical-to-superconductivity phase, the removal of strain from grinding ⇒ improved T_c results presented here in $Sr(Fe_{1-x}Co_x)_2As_2$ likely cannot be used to optimize superconductivity ($T_c \sim$ 16 K) in Co-doped $CaFe_2As_2$. It would be interesting to measure the amount of strain present in the rather low-T_c Co-doped $CaFe_2As_2$ samples—is the strain larger than seen in the higher-T_c $Sr(Fe_{1-x}Co_x)_2As_2$ and $Ba(Fe_{1-x}Co_x)_2As_2$, or is the explanation the difference [23] in the order of the magnetic behavior in $CaFe_2As_2$ (strong first order) and the lack [23] of coexistence between magnetism and superconductivity anywhere in Co-doped $CaFe_2As_2$?

Returning now to our main question: why are T_c and ΔT_c in Co-doped $SrFe_2As_2$ and $BaFe_2As_2$ so sensitive to strain? Such a strong dependence of superconductivity on strain in Co-doped Ba and Sr 122 is indicative of an unconventional superconducting mechanism. One well known example [24] of a very strain sensitive superconductor is UPt_3, where grinding [24–25] totally destroys superconductivity (from a T_c of ∼0.5 K to below 0.05 K as measured by susceptibility). The f-wave pairing symmetry in UPt_3 is expected to be very sensitive to damage and defects [26].

Theory [5] suggests that the pairing mechanism, the so called $s\pm$ scheme, where the order parameter changes sign between different sheets of the Fermi surface, favored for the iron-based superconductors, is also extremely sensitive to defects, not just magnetic defects as are known to degrade conventional superconductors but also including nonmagnetic [27] defects introduced by grinding. Thus the original intent of the present work—to see if understanding the unusually rapid improvement of $T_c^{\rm onset}$ with annealing at only 60% of $T_{\rm melt}$ for just 1 week in $BaFe_{2-x}Co_xAs_2$ could shed light on the superconductivity in iron based superconductors—has produced evidence for extreme sensitivity of the superconductivity to defects introduced via grinding. This is reminiscent of the behavior of the known unconventional superconductor UPt_3.

Another way to introduce nonmagnetic defects in the lattice is via electron irradiation, which has been performed on 122 iron superconductors, UPt_3, as well as in the unconventional cuprate high-temperature superconductors—thus allowing a quantitative intercomparison among all three. Electron irradiation, using 2.5 MeV electrons, by approximately 1.1 10^{19} e/cm^2 gives T_c reductions from the unirradiated T_{c0} (T_c/T_{c0}) of 0.84 for [28] UPt_3, 0.80 for [29] $Ba(Fe_{0.76}Ru_{0.24})_2As_2$, 0.87/0.66 for [30] $Ba_{1-x}K_xFe_2As_2$ ($x =$ 0.19/0.34), and 0.92 for [31] $YBa_2Cu_3O_7$. These results are consistent with the extreme sensitivity of the superconductivity in Co-doped $SrFe_2As_2$ to grinding and the induced strain therefrom found in the present work. Moreover, the relief of a small amount of strain by replacing a 75 °C/hr cooling from 900 to 600 °C with 3 °C/hr cooling and the concomitant increase of $T_c^{\rm onset}$ by ∼1.7 K in Co-doped $BaFe_2As_2$ is further consistent with the electron irradiation evidence [29–30] for the extreme sensitivity of iron based superconductivity, with presumed [29–30] $s\pm$ pairing symmetry, to nonmagnetic defects.

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[16] Since the 122 iron based superconductors such as Co-doped $BaFe_2As_2$ and Co-doped $SrFe_2As_2$ are quite brittle, such grinding involves qualitatively (this will be quantified via x-ray linewidth analysis in later discussion) only a small application of force.

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[19] These two rather close values of strain each have error bars that make them almost equivalent. Unfortunately, the single-crystal (00L) reflections, except for (008) are relatively faint and require long counting times for precision.

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[25] As a further comparison of the current results with those for UPt_3, annealing at 1200 °C (71% of T_{melt}) for 12 hours unannealed single crystals of UPt_3 grown from Bi flux sharpens the susceptibility transition width (from 50 to 30 mK, or from 10% to 6% of the $T_c = 0.5$ K, comparable to the 12 to 8% of $T_c = 19.8$ K narrowing of ΔT_c shown in Fig. 1 here), increases T_c^{onset} by 6% (vs ~7% for annealing at 60% of T_{melt} for 2 weeks in the current work), and sharpens the anomaly ΔC in the specific heat (Ref. [24]).

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[27] Neither low-temperature susceptibility in a 50 G applied field nor zero-field specific heat measurements on the ground $Sr(Fe_{0.86}Co_{0.14})_2As_2$ sample in the present work gave any indication of additional magnetic character, such as increased magnetic susceptibility above T_c or additional upturn in *C/T* below 1 K, upon grinding. However, both the annealed ($\Delta T_c \sim 2$ K) and unannealed ($\Delta T_c \sim 10$ K) powders whose strains are compared in Fig. 4 do show (similar) saturation behavior in their magnetization with increasing field, which is consistent with increased magnetic behavior upon grinding. However, such behavior does not appear to be affecting T_c or ΔT_c.

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