Fluctuations in the vicinity of the quantum critical point of (Sr,Ca)$_3$Ir$_4$Sn$_{13}$ revealed by high-energy X-ray diffraction study

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(Dated: November 20, 2019)

We explore the evolution of the structural phase transition of (Sr,Ca)$_3$Ir$_4$Sn$_{13}$, a model system to study the interplay between structural quantum criticality and superconductivity, by means of high-energy x-ray diffraction measurements at high pressures and low temperatures. Our results confirm a rapid suppression of the superlattice transition temperature $T^*$ against pressure, which extrapolates to zero at a critical pressure of $\approx 1.79(4)$ GPa. The temperature evolution of the superlattice Bragg peak in Ca$_3$Ir$_4$Sn$_{13}$ reveals a drastic decrease of the intensity and an increase of the linewidth when $T\to 0$ K and $p\to p_c$. Such anomaly is likely associated to the emergence of quantum fluctuations that disrupt the formation of long-range superlattice modulation. The revisited temperature-pressure phase diagram of (Sr,Ca)$_3$Ir$_4$Sn$_{13}$ thus highlights the intertwined nature of the distinct order parameters present in this system and demonstrates some similarities between this family and the unconventional superconductors.

I. INTRODUCTION

Compounds displaying the interplay between superconductivity (SC) and electronic instabilities have been extensively studied during the past years due to their rich phase diagrams as a function of doping, pressure or magnetic fields. In most cases, superconductivity is found in the vicinity of electronic instabilities of magnetic origin, where the pairing mechanism is mediated by spin fluctuations and the superconductivity is unconventional. The proximity of superconductivity to nonmagnetic structural instabilities, on the other hand, is rare and searches for a quantum critical point (QCP) resulting from a tunable structural phase transition has provoked great interest due to its promise role of stabilizing or even enhancing the pairing mechanism. Thus, accessing superconducting materials where a detailed study of structural quantum criticality and its impact on superconductivity can be explored is highly desirable.

In this context, the ternary intermetallic stannides such as R$_2$Ir$_4$Sn$_{13}$, where R = Sr, Ca and T=Ir, Rh, have attracted special attention due to the existence of a second-order structural phase transition below $T^*$, its putative coexistence with a superconducting state below $T_C$ and its suppression upon applying pressure or chemical substitution. The resulting phase diagram is very suggestive of strong interplay between different order parameters and remarkably resembles the phase diagrams of the heavily studied unconventional superconductors. However, the role of these order parameters and whether they are coexisting, competing or cooperating between each other are still a matter of debate.

Sr$_3$Ir$_4$Sn$_{13}$ and Ca$_3$Ir$_4$Sn$_{13}$ exhibit an anomaly in the temperature-dependent electrical resistivity and magnetic susceptibility measurements below $T^* \sim 147$ K and $T^* \sim 33$ K, followed by a superconducting transition at $T_C = 5$ K and $T_C = 7$ K, respectively. Single-crystal x-ray diffraction and neutron-scattering measurements revealed that such anomaly is produced by a second-order structural phase transition from a simple cubic parent structure (space group $Pm\bar{3}n$ at 300 K), to a superlattice variant ($I\overline{3}d$), where the lattice parameter is twice that of the room temperature phase. The complete substitution of Sr by Ca, which corresponds to a positive pressure of $\sim 5$ GPa, reduces $T^*$ and this behavior continues for the (Ca$_{1-x}$Sr$_x$)$_3$Ir$_4$Sn$_{13}$ series under external pressure. Full suppression of $T^*$ is predicted at a structural quantum critical point $\approx 1.8$ GPa for Ca$_3$Ir$_4$Sn$_{13}$. Several experimental probes suggest that such structural instability is associated with a charge density wave (CDW) transition involving the conduction electrons. Such idea is supported by a decrease in the carrier density and a formation of a partial energy gap at the Fermi surface through the onset of the structural phase transition. Moreover, muon spin relaxation measurements revealed a strong enhancement of the superfluid density and a dramatic increase of the pairing strength above $\approx 1.6$ GPa, giving evidence of the presence of a QCP. Although several investigations have been realized on this system, in none of these experiments has the CDW been microscopically probed inside the superconducting phase and the question remains whether it survives in this low temperature phase. Since the feature associated with the structural transition is weakened when approaching the QCP, a range of experimental probes have so far failed to identify it. In this sense,
whether the CDW and superconducting states coexist and the exact pressure at which the CDW disappears are yet to be determined or confirmed.

With the improvement of x-ray diffraction techniques to an extended pressure range it is now possible to explore the evolution of superlattice structures and their instabilities when approaching a pressure-driven QCP. Here, using high-energy x-ray diffractions measurements we have performed a detailed study of the evolution of the superlattice structure of (Sr, Ca)$_3$Ir$_4$Sn$_{13}$ against pressure and temperature. We find that the superlattice transition temperature $T^*$ is rapidly suppressed with increasing pressure and extrapolates to zero at a critical pressure of $P_s \approx 0.1$ GPa. Such anomaly is also manifested by a large decrease of the static coherence length ($\xi$) when the temperature approaches to zero. Since information about fluctuations can also be obtained from the Bragg diffraction peaks coming from a static order parameter, our results suggest that quantum fluctuation effects is likely the mechanism behind the destruction of the long-range CDW modulation in (Sr, Ca)$_3$Ir$_4$Sn$_{13}$. The presence of strong quantum fluctuations competing with CDW modulation and possibly with superconductivity makes the phase diagram of (Sr, Ca)$_3$Ir$_4$Sn$_{13}$ reminiscent of unconventional superconductors.

II. SAMPLES AND METHODS

Single crystals of (Sr, Ca)$_3$Ir$_4$Sn$_{13}$ were grown by flux method as described elsewhere. The crystal structure and phase purity were determined by XRD on powdered crystals (not shown). Ambient pressure synchrotron XRD data ($E = 8.33$ keV) were collected on single crystals ($\sim 2 \times 1 \times 1$ mm$^3$) at beamline P09 at PETRA III, DESY. The high quality of the crystal was verified by a mosaic spread of $0.01^\circ$ determined at the (0, 4, 1) Bragg reflection in the room temperature $Pm\overline{3}n$ phase.

High pressure single crystal XRD experiment on (Sr, Ca)$_3$Ir$_4$Sn$_{13}$ was performed at P07 beamline of PETRA III, DESY. A single crystal of (Sr, Ca)$_3$Ir$_4$Sn$_{13}$ was inserted in a 10 T cryomagnet installed on top of the triple-axis diffractometer. Pressure calibration was achieved by measuring the pressure-dependence of the orthorhombic splitting of $(2, 0, 0)/(0, 2, 0)$ Bragg peaks on La$_{1.975}$Ba$_{0.025}$CuO$_4$ as described in Refs 29 and 30. Further details of the pressure calibration can be found in the Supplemental Material. The bulk properties of the superlattice modulation as well as the crystal structure were studied by transmission geometry, vertical scattering, with c axis along the beam direction.

III. RESULTS

A. High-pressure XRD on (Sr, Ca)$_3$Ir$_4$Sn$_{13}$

At ambient pressure, single crystal XRD measurements on (Sr, Ca)$_3$Ir$_4$Sn$_{13}$ reveal a series of satellite peaks below $T^* = 38.2(1)$ K at $q_{SL} = \tau + q_{SL}$, where $\tau$ is the wave vector of the room temperature phase and $q_{SL} = (0.5, 0.5, 0)$ the propagation vector of the superlattice structure (see Fig. S1 in Supplemental Material). No reflections associated with $q_{SL} = (0.5, 0.5, 0)$ or $(0, 0, 0.5)$ modulations were found, in agreement with previous studies. The temperature and pressure dependence of the superlattice peak $Q_{SL} = (3.1, 5, 0.5)$ are summarized in Figure 1(a-f). The measurements consisted of rocking (Fig. 1(a)) and 2$\theta$ scans collected at several pressures and temperatures. At $p = 0.09$ GPa, the integrated intensity of the superlattice peak grows gradually on cooling below $\sim 32$ K (see Figure 1(b)). A saturation of the peak intensity seems to take place below the superconducting temperature $T_c \sim 7$ K, as also observed at ambient pressure. Interestingly, for pressures higher than $p \sim 0.17$ GPa, the superlattice peak intensity is partially suppressed below $T \sim 15$ K. This suppression is enhanced upon pressure increase up to $p = 0.62$ GPa, above which total suppression of the superlattice
peak intensity is observed. The temperature dependence of the superlattice peak intensity for different pressures (Fig. 1(b)) were fitted by a power law $\propto (1 - T/T^*)^{23}$ and the best fit to the data near $T^*$ corresponds to the critical temperatures displayed in Figure 1(c).

Further insight into the partial suppression of the superlattice peak intensity is given by the temperature dependence of the pseudo-Voigt linewidths. The linewidths were extracted from the 29 scans at the lattice and superlattice Bragg peaks (4, 2, 0) and (3, 1.5, 0.5), respectively (Figs. 1(d) and 1(e), respectively). At ambient pressure, the superlattice peak is resolution limited, while it develops a small but finite width at 0.09 GPa (correlation length $\xi \approx 340 \text{ Å}$) indicating the CDW is long-range ordered (see Supplemental Material). At low pressures ($p \lesssim 0.17$ GPa), the linewidth of the superlattice modulation (Fig. 1(e)) is comparable to that of the Bragg reflection (Fig. 1(d)) and is mostly temperature-independent at low temperatures. Upon further pressure increase, the linewidth increases 30% from $p = 0.09$ GPa to $p = 0.57$ GPa at $T = 5$ K. Surprisingly, the widths at low temperatures ($T \approx 5$ K, $p = 0.54 - 0.62$ GPa) are comparable to the values observed in proximity to the structural phase transition ($T^* \approx 20 - 32$ K, $p = 0.09 - 0.02$ GPa), indicating that a competing order of similar energy scales is likely to be developing at low temperatures and high pressures. This same feature is highlighted in Fig. 1(f), where the temperature dependence of the correlation length of the superlattice peak (3, 1.5, 0.5) for two different pressures are plotted: a correlation length of $\xi \approx 153$ Å can be observed in either $T = 5$ K and $p = 0.57$ GPa or $p = 0.09$ GPa and $T \approx 32$ K curves.

We have also explored the crystal structure of Ca$_3$Ir$_4$Sn$_{13}$ under pressure. The evolution of the cubic lattice parameter $a$ at $T = 5$ K was obtained through the analysis of selected structural Bragg peaks ((0, 0, 4) and (4, 2, 0)). No discontinuities in lattice parameters or signatures of a structural phase transition were found in the entire pressure range measured (Fig. 2(a)). Such lattice constant is well-characterized by a single-parameter Birch equation of state (EoS) with bulk modulus of $B = 72(13)$ GPa and $a$-axis compression rate of $\Delta \alpha/a_0 = -0.4(1)$ %/GPa, where $a_0$ is the lattice parameter at $p = 0.09$ GPa.

In order to verify whether the partial suppression of the superlattice intensity is due to a competition between CDW and superconductivity, we have probed the effect of application of magnetic field on the superlattice modulation. A maximum field of 9 T was applied along the $\mathbf{Q}_{\text{SL}} = (3, 1.5, 0.5)$ direction. The field dependence of

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**FIG. 1.** (Color online) (a) Evolution of the (3, 1.5, 0.5) superlattice integrated peak intensity at $p = 0.09$ GPa. (b) Temperature dependence of the (3, 1.5, 0.5) superlattice peak intensity at several pressures. The pressure dependence of $T^*$ for Ca$_3$Ir$_4$Sn$_{13}$ is shown in (c). (d, e) Evolution of the pseudo-Voigt linewidth against temperature at selected pressures for (4, 2, 0) Bragg and (3, 1.5, 0.5) superlattice peaks, respectively, extracted from 29 scans. The inset in (e) highlights the linewidths at low temperatures. (f) Temperature dependence of the static correlation length, $\xi$, of the (3, 1.5, 0.5) superlattice reflection at $p = 0.09$ GPa and $p = 0.57$ GPa. The grey line indicates the similar correlation lengths observed at $T = 5$ K and $p = 0.57$ GPa and at $T \approx 32$ K and $p = 0.09$ GPa. Details of the calculation of the correlation length can be found in the Supplemental Material. The estimated pressure error bar is $\pm 0.1$ GPa.
FIG. 2. (Color online) (a) Pressure dependence of the lattice parameter $a$ extracted from lattice Bragg reflections in the low-temperature $Ih 3d$ space group and its fit to a single parameter Birch equation of state (EoS). (b) Magnetic field dependence of the superlattice modulation peak intensity at $(3, 1.5, 0.5)$ for $T = 5$ K and $p = 0.62$ GPa. The dataset was collected with a magnetic field applied along $Q_{SL} = (3, 1.5, 0.5)$ direction.

the intensity of the superlattice reflection $(3, 1.5, 0.5)$ at $T = 5$ K is shown in Fig. 2(b). Application of a magnetic field has no significant effect on the superlattice peak intensity. We note that previous studies report that a magnetic field of $\mu_0H = 9$ T is enough to suppress the superconductivity in this material at $p = 0.66$ GPa, where the normal state is disclosed down to 2 K.

**B. High-pressure XRD on Sr$_3$Ir$_4$Sn$_{13}$**

A detailed single crystal XRD study under pressure was conducted on Sr$_3$Ir$_4$Sn$_{13}$ compound. Sr$_3$Ir$_4$Sn$_{13}$ displays a series of satellite peaks below $T^* = 151.2(1)$ K at the same reciprocal space positions observed for Ca$_3$Ir$_4$Sn$_{13}$ (Fig. S1 in Supplemental Material). Figure 3(a) displays the temperature dependence of the intensity at several superlattice peaks under pressure. As expected from the high-pressure electrical resistivity measurements, a drastic suppression of the superlattice transition temperature is observed. The temperature dependence of the superlattice peak intensity were also fitted by the power law $\propto (1 - T/T^*)^{5/3}$ and the best fit to the data near $T^*$ corresponds to the critical temperatures displayed in the phase diagram of Fig. 4.

Fig. 3(b) shows the evolution of the intensity of the superlattice peak $(3, 2.5, 0.5)$ normalized by the Bragg reflection $(3, 2, 0)$ against pressure. Due to temperature constraints ($T_{min} \sim 40$ K), the superlattice peak intensity was probed up to the highest pressure of $\sim 4.6$ GPa.
The total superlattice peak suppression is expected to take place at \( \sim 7.7 \) GPa based on the linear extrapolation of the pressure evolution of \( T^* \), as it will be discussed below.

**IV. DISCUSSION**

It has been shown that the combination of physical and chemical pressure has strong influence on the superlattice phase of the 3-4-13 series of compounds, such as the \((Ca_{x}Sr_{1-x})_{3}Ir_{4}Sn_{13}\), where bulk measurements reveal a suppression of the second-order structural phase transition at \( T^* \)\(^{14-16}\). Our XRD measurements on \((Sr, Ca)_{3}Ir_{4}Sn_{13}\) constitute a detailed structural study of this class of material under pressure, contributing to the advancement of the temperature-pressure phase diagram, which up to now are based solely on electrical resistivity, magnetic susceptibility and muon spin relaxation measurements\(^{16}\).

Among the \((Ca_{x}Sr_{1-x})_{3}Ir_{4}Sn_{13}\) series at ambient pressure, \(Ca_{3}Ir_{4}Sn_{13}\) displays the smallest difference between its order parameters \( (T^* \sim 38 \) K and \( T_C \sim 7 \) K at ambient pressure), thus more prone to external stimuli, such as applied physical pressure. Indeed, our high-energy, high-pressure XRD measurements on \(Ca_{3}Ir_{4}Sn_{13}\) reveal that \( T^* \) is rapidly suppressed by pressure at a rate of \( dT^*/dp \approx -19.3 \pm 0.3 \) K/GPa, with the superlattice modulation intensity vanishing completely above \( \sim 0.62 \) GPa. Such result corroborates with our resistivity measurements, where the resistivity anomaly associated with \( T^* \) was last seen at \( T^* \approx 21(1) \) K and \( p = 0.55(5) \) GPa. Combined with the high-pressure XRD data on \(Sr_{3}Ir_{4}Sn_{13}\), \( T^* \) extrapolates to zero yields at a critical pressure of \( p_c \approx 1.79(4) \) GPa (black filled half-circle in Fig. 4), in striking agreement with values found in literature\(^{15,16}\).

Interesting to note is the pressure-induced partial suppression of the superlattice peak intensity for temperatures below \( 15 \) K (Fig. 1(b)). Such anomaly is also manifested in the pseudo-Voigt linewidth as a function of temperature for selected pressures (Fig. 1(e)). Closer to ambient pressure, the linewidth has little to no dependence with temperature. For \( p > 0.09 \) GPa and \( T < 15 \) K, the CDW modulation becomes less long-range ordered, with \( Q_{SL}\)-width increasing when temperature is lowered to \( T \sim 5 \) K. Fig. 1(f) shows the correlation length of the CDW modulation, which decreases from \( \xi \approx 341 \) Å at \( p = 0.09 \) GPa to 153 Å at \( p = 0.62 \) GPa at \( T \sim 5 \) K, a reduction of 55% (going from \( \sim 17.6 \) to \( \sim 8 \) CDW wavelengths\(^{38}\)). Our results strongly suggest that a competing order of similar energy scales is developing at low temperatures and high pressures. The possible nature of such order parameter and its implications will be discussed below.

In order to determine the mechanism behind the partial suppression of the superlattice modulation, we have investigated the evolution of the lattice parameter against pressure. No sign of a structural phase transition could be observed in the entire pressure range measured, suggesting that the crystal structure remains within the superlattice variant unit cell \( I\overline{F}3d\). This was further supported by the pseudo-Voigt linewidths extracted from the lattice Bragg peaks: a 5% peak broadening was found from \( p = 0.09 \) to 0.57 GPa, but such value is likely attributed to the natural peak broadening related to application of pressure rather than to the onset of a structural phase transition. Evidence for competition between CDW and superconductivity was also investigated through measurements of the superlattice peak under applied magnetic field. From the field-dependent data shown in Fig. 2(b), we found that an applied magnetic field of 9 T along \( Q_{SL} = (3, 1.5, 0.5) \), which should be enough to suppress the superconducting phase in \(Ca_{3}Ir_{4}Sn_{13}\)\(^{37}\), has no effect on the superlattice modulation intensity. This is in contrast with observations in cuprate materials, such as \(YBa_2Cu_3O_{6.65}\)\(^{31}\), where application of magnetic field suppresses superconductivity and enhances CDW ordering.

It should be noted that fluctuation effects\(^{6,39,40}\) might be playing a crucial role in the decrease in coherence length of the CDW modulation at \( T < 15 \) K and \( p > 0.09 \) GPa. Analysis of the linewidth of the 2\( \theta \) scans indicate that at \( p = 0.09 \) GPa the profile shape is mostly Gaussian and evolves towards Lorentzian when approaching the superlattice phase transition at \( T^* \) (see Fig. S4 in
Supplemental Material). For higher pressures, on the other hand, the profile shape has a significant contribution from the Lorentzian line shape, indicating that the superlattice phase correlation is exponentially decaying in real space already at low temperatures\(^{39,40}\). This result could be consistent with a disorder pinning scenario\(^{41-43}\), where the CDW phase distortion is distributed over a spatial range across the pinning site\(^{42}\). However the short correlation length (\(T < \text{perlattice peak intensity for first derivative of the temperature dependence of the superlattice phase correlation is exponentially distribution from the Lorentzian line shape, indicating that other hand, the profile shape has a significant contribution}) observed at \(p = 0.62\) GPa is unlikely to accommodate several disorder sites within a coherent volume to pin the CDW domain\(^{40}\). Thus, we believe that the mechanism for destroying the long-range CDW modulation in Ca\(_3\)Ir\(_4\)Sn\(_4\) is probably the increasing quantum fluctuations when \(T \to 0\) K and \(p \to p_c\). Indeed, muon spin relaxation measurements\(^{46}\) have pointed out the importance of fluctuations when approaching the quantum critical point, which might be the origin of the enhancement of the superconducting phase above \(p_c\). Quantum fluctuations has also been highlighted in LuPt\(_{2-x}\)Pd\(_x\)In system, where \(T_C\) presents a dome-shaped doping dependence with highest value exactly where the CDW transition disappears\(^{46}\).

Finally, we have constructed the temperature-pressure phase diagram of Fig. 4. Results from XRD measurements on (Sr, Ca)\(_3\)Ir\(_4\)Sn\(_3\), electrical resistivity experiments on Ca\(_3\)Ir\(_4\)Sn\(_3\) as well as other studies found in literature are reported. The phase diagram also depicts the apparent short-range order phase related to the partial suppression of the superlattice peak intensity in Ca\(_3\)Ir\(_4\)Sn\(_3\) and possibly to the quantum fluctuations in this material. To better follow the evolution of such phase, we have extracted the temperature \(T'\) from the first derivative of the temperature dependence of the superlattice peak intensity for \(T < 15\) K (see Supplemental Material\(^{34}\) for more details). As mentioned before, our results suggests an enhancement of the quantum fluctuations when \(T \to 0\) K and \(p \to p_c\), which is likely the mechanism behind the partial/total suppression of the superlattice modulation at low temperatures. Although not probed in our experiment, we believe that a reentrant CDW modulation at higher pressures are unlikely to happen due to the lack of observation of such feature in bulk measurements. The presence of quantum fluctuations competing with CDW modulation and possibly with superconductivity makes the phase diagram of (Sr, Ca)\(_3\)Ir\(_4\)Sn\(_3\) reminiscent of unconventional superconductors. Particularly for Ca\(_3\)Ir\(_4\)Sn\(_3\), these different types of orders occur on comparable temperature scales and can compete/cooperate on an almost equal footing revealing their intertwined nature. Further experimental efforts will help to determine more accurately the nature of the quantum fluctuations in the low-temperature, high-pressure phase. For instance, measurements of the diffuse elastic line width in the anomalous phase could determine the critical exponent that controls the correlation length of the fluctuations.

V. CONCLUSION

Here we have performed a detailed study of the evolution of the superlattice structure of (Sr, Ca)\(_3\)Ir\(_4\)Sn\(_3\) against pressure by means of high-energy XRD measurements. We found that the superlattice transition temperature \(T^*\) is rapidly suppressed with increasing pressure and extrapolates to zero at a critical pressure of \(p_c \sim 1.79(4)\) GPa, in agreement with values found in literature for the same family of compounds\(^{15,16}\). Our XRD measurements on Ca\(_3\)Ir\(_4\)Sn\(_3\) revealed an anomaly related to a partial suppression of the superlattice peak intensity, which takes place at low temperatures (\(T < 15\) K) and high pressures (\(p > 0.09\) GPa). Such anomaly is also manifested by an increase of the pseudo-Voigt linewidth of the 2\(\theta\) scans when the temperature approaches to zero. With no apparent origin on a structural phase transition or a competition with the superconducting phase that emerges at \(T_C \sim 7\) K and reaches its maximum at \(p \sim 4\) GPa, our results suggest that quantum fluctuation effects is possibly the mechanism behind the destruction of the long-range CDW modulation. The revisited temperature-pressure phase diagram of (Sr, Ca)\(_3\)Ir\(_4\)Sn\(_3\) highlights the intertwined nature of the distinct order parameters and demonstrates some similarities of this family of supposedly conventional BCS superconductors\(^{15,16,44-46}\) and the unconventional superconductors.

ACKNOWLEDGMENTS

We thank O. Gutowski for his assistance at P07 beamline of Petra III. We also thank M. Eleotero, J. Fonseca and N. M. Souza-Neto for the help with high pressure diffraction measurements at XDS beam line at LNLS (proposal No. 20160202). Part of this research was carried out at PETRA III at DESY, a member of Helmholtz Association (HGF). LSI Veiga is supported by the UK Engineering and Physical Sciences Research Council (Grants No. EP/N027671/1 and No. EP/N034694/1). EM Bittar is supported by the Conselho Nacional de Desenvolvimento Científico e Tecnológico (CNPq) (Grant No. 400633/2016-7).

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