Local structure and superconductivity of the $Ce_{1-x}La_xRu_2$ Laves phase system

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We have studied local structure of the Laves phase $Ce_{1-x}La_xRu_2$ superconductor by Ru K-edge extended x-ray absorption fine-structure measurements focusing on the small La concentration regime where the transition temperature T_c passes through a local maximum. We find that correlated Debye-Waller factor of the Ru-Ru bonds follows T_c with the varying La concentration in the system. Although, this remarkable T_c correlation on the local atomic structure suggests important role of the electron-lattice interactions, the band-structure effects seem more likely the reason to drive the anomalous superconducting behavior and the T_c maximum in this $4f$ system.

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I. INTRODUCTION

Recent discoveries of high T_c superconductivity in the MgB_2 binary system[1] and coexistence of superconductivity and ferromagnetism in alloys such as UGe_2 (Ref. 2) and $ZrZn_2$ (Ref. 3) have raised further interest in other simple binary structures with fascinating transport properties.[4] Among others, the $CeRu_2$ is a good example[5,6] of such binary systems exhibiting superconductivity at a T_c of $\sim$6.2 K and having cubic-Laves structure (C15) as the $ZrZn_2$. This is the highest T_c among the Ce intermetallics ($4f$ systems) and, in addition, the system possesses a wide range of interesting properties such as mixed valence and coexisting superconductivity and magnetism with small moment.[6] Several experiments are consistent with the picture of a strong-coupling superconductivity in the system.[7] It is now known that the $CeRu_2$ superconductivity is not solely derived by the Ru $4d$ electrons but appears to have significant influence from the Ce $4f$ states.[8–10] In fact, the $CeRu_2$ is an interesting example where the f electrons represent both localized and itinerant character with appreciably wide $4f$ band.[9,10]

Chemical substitution at the Ce site has been one of the approaches used, to explore the rich physics involved with the interplay of localized and itinerant character of the Ce $4f$ electrons, and to get further insight on the superconductivity of this system. The $Ce_{1-x}La_xRu_2$ provides a model case with the end member $LaRu_2$ to be a formally $4f^0$ system, showing superconductivity at 4.4 K.[11–16] T_c shows an anomalous change from $\sim$6.2 K, 0.3 K, and 4.4 K, respectively, at $x=0$, 0.5, and 1 (Refs. 11 and 12) while a continuous expansion of the unit cell with increasing of the La concentration occurs. In addition, at low concentration, $0<x<0.15$, an anomalous T_c behavior has been observed with an increase of T_c with a maximum of 6.7 K in the range of $x \sim 0.06-0.09$ before the decrease with further increasing of the La concentration.[11,12] Similar behavior was observed also in other Laves phase such as the $Th_{1-x}(La,Nd)_xRu_2$ system and attributed to alloying effect in these materials.[17] It was also indicated that the phonons could be important for the anomalous T_c dependence. Incidentally a similar behavior of T_c has been observed in the superconducting binary systems (e.g., $Re_{1-x}Os_x$, $Re_{1-x}Ru_x$) (Ref. 18) and $Au_{1-x}(Pd)_xGa_2$-type ternary alloys.[19] In addition, recent experiments have revealed a pressure induced new structural phases in the $CeRu_2$.[16] T_c shows an increase up to $\sim$7.5 K followed by a faster decrease down to $\sim$4 K with increasing pressure.[15,16] This abnormal change in T_c with external pressure indicates an interesting behavior of the electronic correlations in the $Ce_{1-x}La_xRu_2$ system. The anomalous T_c dependence on the pressure appears similar to the one found in the $Au_{1-x}(Pd)_xGa_2$ alloys, which has been assigned to a Fermi-surface transition.[19] Also, the new structural phases appearing under external pressure in the $CeRu_2$,[16] and their possible relation with the superconductivity motivates further studies on this $4f$ superconductor. Here, we address the anomalous T_c dependence and investigate the local structure of the $Ce_{1-x}La_xRu_2$ system, focusing on the low-concentration regime.

We have used Ru K-edge x-ray absorption fine-structure (EXAFS) spectroscopy, a fast ($\sim 10^{-15}$ s) and local ($\sim$5–6 Å) probe,[20] to enlighten a possible role of local displacements in the anomalous superconductivity of the $Ce_{1-x}La_xRu_2$ system. The local probe has been used also due to the fact that diffraction does not reveal any evident structural anomaly as a function of La concentration at ambient pressure.[11] Moreover, the $Ce_{1-x}La_xRu_2$ is a short coherence length ($\sim$30–60 Å) superconductor and hence local interactions are expected to be important for its superconductivity. We have measured distribution of the local Ru-Ru displacements (dynamic and static), given by the correlated Debye-Waller factor (DWF) of the Ru-Ru bonds. The DWF shows an anomalous change at the low La substitution regime where T_c appears with anomalous increase, indicating un-

usual electron-lattice interaction in this short coherence length strong-coupling superconductor.

II. EXPERIMENTAL DETAILS

A. Sample preparation and characterization

Samples of $Ce_{1-x}La_xRu_2$ ($x=0$, 0.03, 0.06, 0.09, 0.12, 0.15, 0.20, 0.75) were prepared using arc melting under argon atmosphere. The La, Ce (of Koch-Light Lab., with purity of 99.7% and 99.9%, respectively) and Ru (Johnson Matthey GmbH, 99.99%) metals, degassed at 800 °C under high vacuum, were used for the synthesis. The melting process was repeated three times for the homogeneity, followed by annealing of the melt buttons (wrapped in tantalum foil, placed in quartz tube with high vacuum) at 1100 K for 12 days.

The samples were characterized by x-ray diffraction (Cu K_α source with Si standard) for the structural properties.[21] The diffraction data show $MgCu_2$-type structure with inevitable impurities (estimated to be $<2-3\%$, perhaps originated from the small impurities of the starting metallic ingredients). The lattice parameters were measured to be 7.544 Å, 7.547 Å, 7.551 Å, 7.555 Å, 7.561 Å, 7.565 Å, 7.573 Å, 7.671 Å, respectively, for $x=0$, 0.03, 0.06, 0.09, 0.12, 0.15, 0.20, 0.75 samples. All the samples were characterized for their superconducting properties using a single coil induction method.[21] T_c of the samples were found to be 6.17, 6.45, 6.55, 6.57, 6.14, 5.35, 4.77, 1.42 K, respectively, with increasing La concentration.

B. X-ray absorption measurements

Ru K-edge absorption measurements were performed on powder samples of $Ce_{1-x}La_xRu_2$. The absorption measurements were made at the beamline BM29 of the European Synchrotron Radiation Facility (ESRF), Grenoble, where the synchrotron radiation emitted by a bending magnet source at the 6 GeV ESRF storage ring was monochromatized using a double crystal Si(311) monochromator. The Ru K_α fluorescence yield was collected using multielement Ge detector array covering a large solid angle of the fluorescence emission. The samples were mounted in a closed cycle two stage He refrigerator to perform the measurements at low temperature (30 K). The sample temperature was controlled and monitored within an accuracy of ±1 K. As our routine experimental approach, several absorption scans were collected to limit the noise level to the order of 10^{-4} ensuring the reproducibility of the spectra, in addition to the high signal-to-noise ratio. Standard procedure was used to extract the EXAFS signal from the absorption spectrum,[20] followed by the x-ray fluorescence self-absorption correction before the analysis.[22]

III. RESULTS AND DISCUSSION

Figure 1 shows Ru K-edge EXAFS oscillations on several $Ce_{1-x}La_xRu_2$ samples at low temperature (30 K). The EXAFS oscillations were extracted from the absorption spectra measured on the powder samples and corrected for the fluorescence self-absorption effect. The EXAFS oscillations are weighted by k^2 to highlight the higher k region and the oscillations with high signal-to-noise ratio could be seen up to $k=17$ Å^{-1}. There are evident differences between the EXAFS oscillations (see, e.g., the oscillation around $k=8-9$ Å^{-1} and in the k range above ~12 Å^{-1}), including a clear overall damping of the EXAFS oscillations with increasing La. The differences in the local structure could be better discriminated in the Fourier transforms of the EXAFS oscillations providing real-space information.

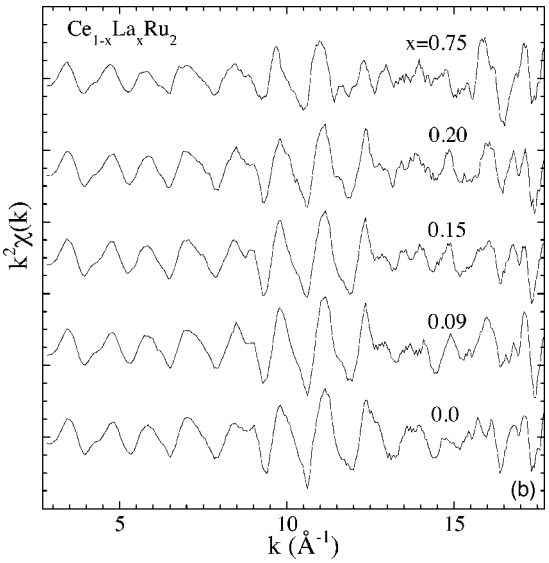

FIG. 1. EXAFS oscillations (multiplied by k^2) extracted from the Ru K-edge absorption spectra measured on $Ce_{1-x}La_xRu_2$ system for several La concentrations at low temperature (30 K).

Figure 2 shows magnitude of the Fourier transforms, $|\mathrm{FT}(k^2\chi(k))|$, of the EXAFS oscillations (weighted by k^2) measured on the representative samples. The Fourier transforms (FTs) were performed between $k_{min}=3$ Å^{-1} and $k_{max}=17$ Å^{-1} using a Gaussian window. The FTs are not cor-

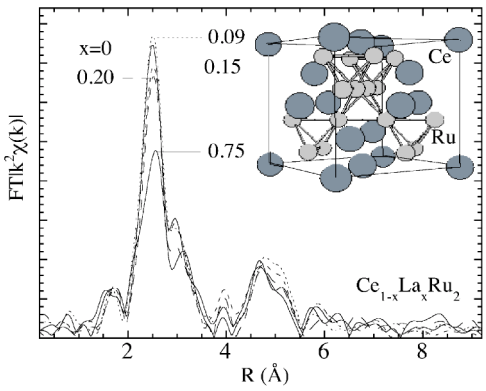

FIG. 2. Fourier transforms of the EXAFS oscillations showing global atomic distribution around the Ru in the $Ce_{1-x}La_xRu_2$ system. The Fourier transforms are performed between $k=3$ and 17 Å^{-1} and not corrected for the phase shifts. The main peak ~2.5 Å is due to Ru-Ru bonds while peak structure ~3 Å represents Ru-Ce(La) bonds. Crystal structure of the $CeRu_2$ is shown as an inset.

rected for the phase shifts due to the photoelectron backscattering and represent raw experimental data. Here it is worth recalling the $CeRu_2$ crystal structure (see inset of Fig. 2) belonging to the C15 family. The $CeRu_2$ lattice has Ce atoms forming a diamond-type lattice and the Ru atoms forming a network of corner sharing tetrahedra. The Ce has 16 nearest neighbors at ~3 Å (12 Ru atoms and 4 Ce atoms). On the other hand, the Ru has eight nearest-neighbor Ru atoms, at $R \sim 2.5$ Å, Following the geometry around the photoabsorbing Ru, we can assign the Fourier-transform peaks in Fig. 2, with the main peak at $R \sim 2.5$ Å, being due to the Ru-Ru bonds, while the peak at ~3 Å, corresponding to the Ru-La(Ce) bonds. At higher R value the peaks in the FTs are due to multiple scattering of the photoelectron exiting at the Ru site [see, e.g., the doublet structure at ~5 Å, due to multiple scattering of Ru-Ru-Ru and Ru-La(Ce)-Ru]. From the first look it is easy to distinguish evident local structural differences between different samples. The Ru-Ru peak is strongly influenced by the La substitution and gets damped in the higher La content sample. Also, the Ru-La(Ce) peak shows a La concentration dependence and seems to move towards higher R, indicating increased Ru-La(Ce) bond length with increasing La. There are evident differences at higher value of R, however, the peaks contain contributions of complex multiple scattering that are out of the present discussion and may be better discussed looking at the near edge (XANES) spectrum.

We will focus on the Ru-Ru shell that shows a large change with the La concentration. The Ru-Ru FT peak amplitude as a function of La content is plotted in Fig. 3, showing interesting behavior of the local structure. The peak amplitude shows a small increase before a gradual decrease, revealing a clear anomaly in the local structure with the La concentration. To ensure this behavior we have used k weighted FT in such a way to separate the Ru-Ru contribution from the Ru-La(Ce) peak, however, the result remains the same (see, e.g., middle panel of Fig. 3). Incidentally the FT amplitude anomaly is quite similar to the behavior of the superconducting transition temperature of the system, plotted in Fig. 3 (Ref. 21). Although the FT anomaly is quite small, the analogous T_c behavior merely suggests the existence of some correlation between the two quantities, i.e., the local structure of the Ru site and the superconductivity.

Here we continue our focus on the Ru-Ru and Ru-La(Ce) displacements (i.e., on first two shells around the Ru) and their evolution with the La concentration. The EXAFS signals due to the two shells are well separated from the multiple-scattering contributions and we can easily analyze them separately. We have used the "standard procedure" to analyze the EXAFS considering a Ru-Ru distance and a Ru-La(Ce) distance corresponding to the two coordination shells where the effective DWF include all distortion effects. It should be recalled that the number of parameters which may be determined by EXAFS is limited by the number of independent data points: $N_{ind} \sim (2\Delta k \Delta R)/\pi$, where Δk and ΔR are, respectively, the ranges in k and R space over which the data are analyzed.[18] In the present case $\Delta k = 14$ Å^{-1} and $\Delta R = 2$ Å, give $N_{ind} \sim 18$ for the two shell EXAFS. Except the radial distance R and the corresponding DWF, all other parameters were kept fixed in the least-squares paradigm to

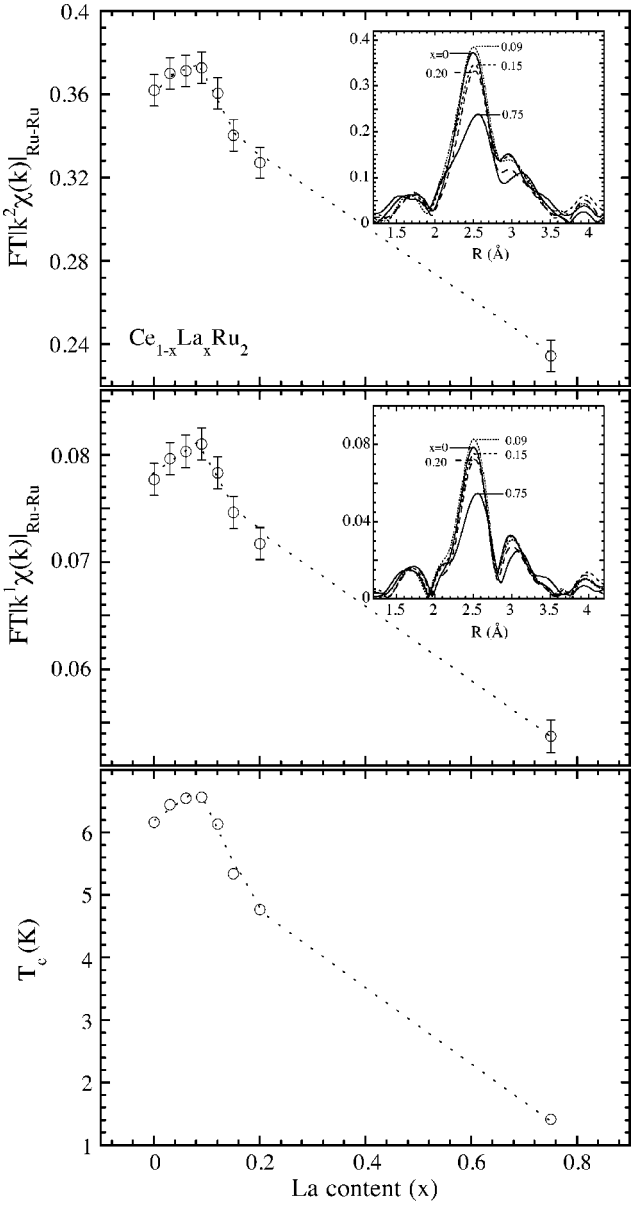

FIG. 3. Fourier-transform (FT) main peak amplitude (Ru-Ru) is plotted as a function of La concentration. The FT amplitudes have been plotted for the two cases of EXAFS oscillations multiplied by k^2 (upper) and k (middle). The corresponding FTs are shown as inset for several La concentrations. The error bars represent maximum deviation of the peak amplitude, estimated by taking peak intensity taken at several R points around the peak position. The superconducting transition temperature (T_C) as a function of La concentration is also shown (lower).

make a suitable comparison between the extracted parameters for variable La. Starting parameters were taken from the diffraction studies.[11,21] For simplicity Ru-Ce and Ru-La signals were modeled considering a single shell and a single DWF. We have also made an effort to analyze the Ru-Ce(La) shell, with the La occupancy being a free parameter to see if some kind of clustering appears with the La (similar approach as discussed by Shibata *et al.* for manganites[23]), however, unphysical parameter values were obtained. This seems reasonable since we are dealing with small La substitutions

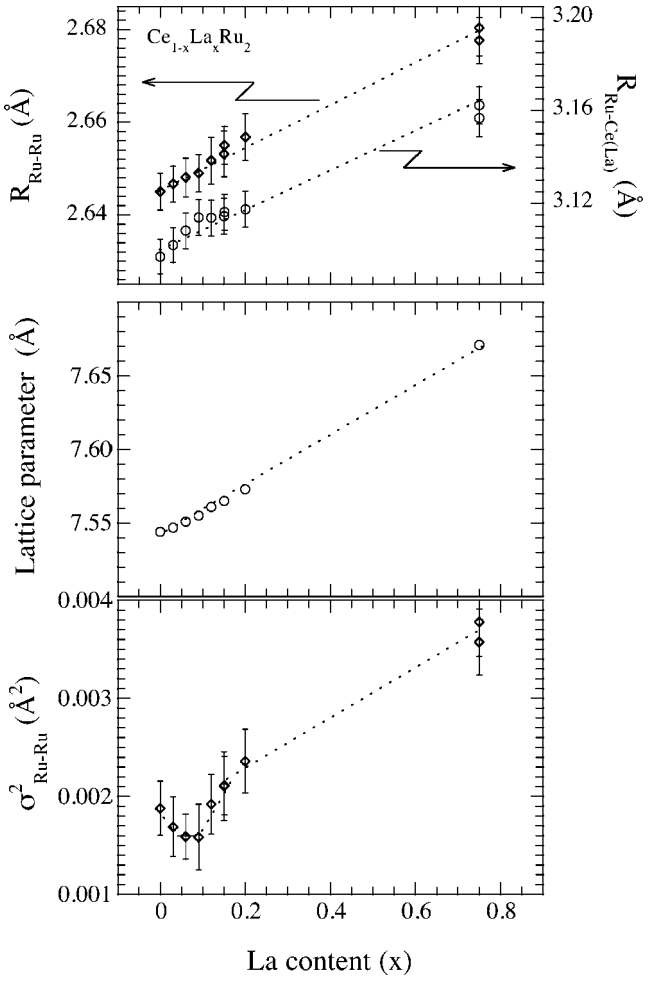

FIG. 4. Ru-Ru and Ru-Ce(La) distances for the $Ce_{1-x}La_xRu_2$ system as a function of La concentration showing an almost linear increase (upper), consistent with the variation of the lattice parameter (middle). The Debye-Waller factor of the Ru-Ru pairs is also plotted (lower), revealing a clear change with a small anomaly at low La concentration. The error bars represent the uncertainties estimated by creating correlation maps between different parameters.

and the correlation among the parameters is stronger than what has been found in the manganites.[23]

The average radial distances and the DWF are shown in Fig. 4 as a function of La concentration. There is a gradual increase of the average Ru-Ru and Ru-La(Ce) distances with increasing La concentration, consistent with the diffraction studies showing increase of lattice parameters due to larger size of the La (Fig. 4).[11,21] The results are also consistent with the recent EXAFS study on the $CeRu_2$ system.[24] Here the Ru-Ru distance shows an increase of about 0.04 Å, while the Ru-Ce(La) distance gets longer by ~0.08 Å, with variation of La concentration from $x=0$ to 0.75.

We can observe some evident changes for the DWF of Ru-Ru pairs (lower panel of Fig. 4) while, within the experimental uncertainties, we could not see any significant variations in the DWF of the Ru-Ce(La) pairs (0.0045 ± 0.0009), indicating smaller influence of the latter on the anomalous superconductivity. On the other hand, the DWF of Ru-Ru bond shows an overall increase with increasing La concentration with a small anomaly at the low La concentration. Although the low-concentration anomaly of the DWF is small, it appears intrinsic to the studied system.

It is useful to recall that the EXAFS DWF measures the mean-square relative displacements, i.e., represents the distance broadening between the pair of absorber (Ru) and the scatterer (Ru). Therefore, in contrast to the mean-square displacements, measured by diffraction Debye-Waller factors, the EXAFS DWF depends on fluctuation in the pair distances, providing measure of displacement-displacement correlation function. In addition, the EXAFS DWF is related to the local vibrational density of states, and hence provides information on the local vibrational structure.[20]

If static structural distortions are present, the EXAFS DWF σ^2 could be given as a superposition of a temperature independent (static σ_s^2) and temperature dependent (dynamic σ_d^2) terms, i.e., $\sigma^2 = \sigma_s^2 + \sigma_d^2$. The present data suggest that the DWF of the Ru-Ru pair is approximately inversely proportional to the superconducting transition temperature of the system, being smaller while T_c is higher. This provides an indication that the anomalous superconductivity at low doping should have some correlation with electron-lattice interaction, as the DWF is directly related to the local vibrational structure, involving the Ru-Ru bonds. We should mention that, although information on the complete vibrational structure of the $CeRu_2$ is unavailable, we believe that the main contribution to the electron-phonon coupling should come from the modes due to Ru atoms (analogous to other Laves phases for which the vibrational structure is well known[25]). This is due to the fact that a major contribution to the electronic density of states at the Fermi level in this system is provided by the Ru 4*d* orbitals.[26]

Let us assume that the DWF could be given by the simple correlated Einstein model, approximating the local vibrational spectrum with a single δ function (Ru-Ru phonon mode) centered at the effective vibrational frequency (ω_E). At low temperature, where the zero-point motion dominates, the Debye-Waller factor could be given as

$$\sigma_d^2 \approx \frac{\hbar}{2\omega_E m_r},$$

where m_r is the reduced mass and ω_E is the Einstein frequency of the Ru-Ru pair. Therefore, assuming that the same phonon mode, related with the Ru-Ru participates in the superconductivity of the $CeRu_2$ and the $Ce_{1-x}La_xRu_2$, the local decrease of the DWF could mean an increase of the phonon frequency $(\omega_E \approx \hbar/2m_r\sigma^2)$. Since we cannot distinguish static and dynamic contributions from the present measurements, we have assumed that the static part, σ_s^2 increases monotonically with the La concentration. Therefore, the local T_c anomaly at low La concentration appears to have some correlation with the energy of the local phonon modes related with the Ru-Ru.

In the strong-coupling theories of superconductivity, the electron-phonon coupling constant is generally given as[27,28]

$$\lambda_{ph} = \sum_i \frac{\eta_i}{M_i \omega_i^2},$$

where the sum runs over all the atoms i, with masses M_i, the phonon frequency ω_i, and the Hopfield parameter η_i describing the electronic contribution.[28,29] η_i is given by the density of states at the Fermi level, multiplied by the square of the electron-phonon matrix element averaged over the Fermi surface. Assuming that the electronic system remains unchanged, the increase of ω_i should result in a decrease in λ_{ph}, thereby lowering T_c. This is in contrast to the experimental observations showing increase of T_c on increasing the phonon frequency. Therefore, with the assumption of constant η_i, the electron-phonon coupling is unable to explain the local maxima in T_c at small La concentration. However, local effects could appreciably change η_i, by changing the electron-phonon matrix element,[29] and hence it is not simple to rule out the electron-phonon interaction and more work is required.

Recently it has been experimentally observed that local atomic disorder somehow favors the superconductivity in the heavy fermion $CeCu_{2+x}Si_2$ superconductor.[30] Indeed, while the local structure is intrinsically disordered for the superconducting system, there is a long-range atomic order for the case of the nonsuperconducting system. A spin-Peierls-like ground state in the periodic Anderson model, proposed for the heavy fermion superconductors,[31] has been argued to be consistent with these experiments revealing local inhomogeneities in the $CeCu_{2+x}Si_2$.[30] Evidently, our results appear inconsistent with the above model[31] because T_c is higher while the DWF is smaller, i.e., smaller local disorder (see, e.g., Fig. 4). However, since the $CeRu_2$ is a strong-coupling superconductor with coexisting ferromagnetism, the contradicting behavior leads us to think for another possibility, such as a competing role of the spin fluctuations and phonons in the superconductivity of $CeRu_2$, as discussed for the isostructural $ZrZn_2$ system.[28,32]

Here we wish to recall that in the binary systems (e.g., $Re_{1-x}Os_x$, $Re_{1-x}Ru_x$),[18] T_c shows a local increase at low concentration, which has been explained to be due to band smearing and change of Fermi-surface topology, derived by impurity induced modification of the electron-scattering rate, leading to an increased density of states at the Fermi level. Making an analogy with these binary alloys, we cannot rule out the similar effect, considering the experimental fact that the high-pressure studies on the $CeRu_2$ reveal unusual behavior. Here it is worth mentioning that the Sc substitution in the $CeRu_2$ does not show the anomalous T_c dependence in the low-concentration regime[33,34] due to the fact that the Sc-Sc distance is less than the Ce-Ce distance, i.e., no chemical pressure on the Ce-Ce. On the other hand, the new (unrecognized) structural phases induced by pressure in the $CeRu_2$ appears to be correlated to the T_c anomaly.[15,16] In the $Ce_{1-x}La_xRu_2$ system, a band-structure smearing could also occur due to change in the chemical potential introduced by local change in the Ce valence states[8–10] resulting in increased density of states at the Fermi level and hence T_c.

In summary, we have measured local structure of Laves phase $Ce_{1-x}La_xRu_2$ superconductor by high-resolution Ru K-edge EXAFS as a function of La concentration in the host $CeRu_2$ lattice. We find that the local displacements show anomalous change in the low La concentration regime, similar to T_c of the system, revealed by the Ru-Ru Debye-Waller factor as well as the corresponding Fourier-transform amplitude. The observation of a minimum in the Debye-Waller behavior of the Ru-Ru bonds at small La concentration appears to indicate the important role of the Ru channels within this Laves phase. We have discussed that a competing role of spin fluctuations and phonon could be important, along with the band-structure smearing and change in the Fermi-surface topology.

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