Electronic Inhomogeneities in the superconducting phase of CaFe$_{1.96}$Ni$_{0.04}$As$_2$ single crystals

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Abstract. Superconductivity in Iron-Arsenic based pnictides emerges in close proximity to an antiferromagnetic (AFM) ordered parent state and the AFM phase overlaps with superconducting (SC) phase in some pnictides for certain range of doping. CaFe$_{2-x}$Ni$_x$As$_2$ belongs to this category, where both the phases overlap. Here we use scanning tunneling microscopy and spectroscopy to investigate the local electronic properties of underdoped CaFe$_{1.96}$Ni$_{0.04}$As$_2$ single crystals in the vicinity of the boundary of the two phases. Both resistivity and magnetic measurements show that a tiny portion ($\sim 1.2\%$) of this compound becomes superconductor below the SC onset temperature $T_C \sim 15$ K. Topographic images show reasonably flat surface with signatures of atomic resolution. High temperature spectra are spatially homogeneous and show signatures of spin density wave (SDW) gap with a finite density of states near the Fermi energy. Below $T_C$, spectra show significant spatial inhomogeneity with a SDW gap everywhere but at some locations we also see an asymmetric or symmetric depression in $\sim \pm 5$ meV energy range together with the SDW gap. Inhomogeneity reduces significantly as the temperature goes above $T_C$ and disappears completely far above $T_C$. These observations are discussed in terms of an inhomogeneous electronic phase that may exist due to the vicinity of this composition to the SC dome boundary on the underdoped side of the phase diagram.

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1. Introduction

The discovery\cite{1} of Iron-Arsenic (Fe-As) based pnictide superconductors with high critical temperature ($T_C$) provide us a system with rich physics. As in the cuprates, heavy fermions and organic superconductors, superconductivity in Iron-Arsenic based pnictides also emerges in close proximity to an antiferromagnetic (AFM) ordered parent state and $T_C$ has dome-shaped dependence on doping or pressure\cite{2,3,4,5,6,7,8,9,10,11,12,13}. If the AFM and superconducting (SC) phases overlap in the phase diagram, the maximum $T_C$ is found close to the extrapolated end point of the AFM transition. Conventional electron-phonon pairing mechanism cannot explain such high-$T_C$ superconductors. It is widely believed that magnetic fluctuations have an important role in the origin of high-$T_C$ superconductivity. Quantum fluctuations associated with the quantum critical point (QCP)\cite{14,15} may also have a crucial role in superconductivity. Muon spin relaxation ($\mu$SR) and Mössbauer spectroscopy on LaFeAsO$_{1-x}$F$_x$ show a discontinuous first-order-like transition from SDW to SC state without any coexistence of SDW and SC phases\cite{2}. Neutron scattering in CeFeAsO$_{1-x}$F$_x$ reveals a continuous second order transition, but the SDW and SC phases touch only at T = 0 and it could be a quantum critical point\cite{3}. On the contrary, in other cases such as scanning tunneling microscopy and spectroscopy on NaFe$_{1-x}$Co$_x$As\cite{4}, $\mu$SR\cite{5,6,7} and neutron diffraction\cite{8} study on Ba$_{1-x}$K$_x$Fe$_2$As$_2$, $\mu$SR study on Ba(Fe$_{1-x}$Co$_x$)$_2$As$_2$\cite{9,10}, $\mu$SR study on SmFeAsO$_{1-x}$F$_x$\cite{11}, $\mu$SR and nuclear quadropole resonance study on SmFe$_{1-x}$Ru$_x$AsO$_{0.85}F_{0.15}$\cite{12} and $^{75}$As NMR study on Ba(Fe$_{1-x}$Ru$_x$)$_2$As$_2$\cite{13} show coexistence of the two phases. These two phases may coexist microscopically or in a phase separated way. SmFe$_{1-x}$Ru$_x$AsO$_{0.85}F_{0.15}$\cite{12} and Ba(Fe$_{1-x}$Ru$_x$)$_2$As$_2$\cite{13} display phase separation while NaFe$_{1-x}$Co$_x$As\cite{4}, Ba(Fe$_{1-x}$Co$_x$)$_2$As$_2$\cite{9,10} show microscopic coexistence. In some pnictides for example Ba$_{1-x}$K$_x$Fe$_2$As$_2$ and SmFeAsO$_{1-x}$F$_x$, the issue of whether they have microscopic or phase separated AFM and SC coexistence is not completely clear yet\cite{5,6,8,7,11}. Therefore, pnictides with compositions close to the SC dome boundary on the under doped side are very attractive to study the evolution between magnetism and superconductivity.

With its atomic scale structural and spectroscopic imaging capabilities, scanning tunneling microscopy and spectroscopy (STM/S) is an ideal probe to investigate the local electronic properties of these systems. In our variable temperature STM/S we also investigate the temperature evolution of the electronic properties to see how they correlate with various phases at different temperatures.

CaFe$_{2-x}$Ni$_x$As$_2$ has a very rich phase diagram. The parent compound CaFe$_2$As$_2$ undergoes a spin density wave (SDW) transition near $T_{SDW} = 170$ K\cite{17,18}. Around $T_{SDW}$ a structural transition is also observed, where the symmetry changes from tetragonal ($I4/mmm$) to orthorhombic ($Fmmm$)\cite{17,18}. Electron doping by partially replacing Fe by Ni suppresses the SDW and the structural transitions, leading to superconductivity in CaFe$_2$As$_2$\cite{19}. With increase of Ni concentration, $T_{SDW}$ start to decrease. Furthermore a drop in resistivity occurs at $\sim 15$ K for $x = 0.027$ which at
higher doping develops into a pure superconducting transition \cite{19}. SDW phase vanishes completely at $x = 0.06$ \cite{19}.

Here, we report temperature dependent STM/S studies of underdoped in situ cleaved CaFe$_{1.96}$Ni$_{0.04}$As$_2$ single crystals in 5.4 K - 292 K temperature range. We observed atomically flat terraces with homogeneous tunneling DOS between 292 K and 78 K and these spectra correlate well with the SDW gap. However the spatially resolved spectra below superconducting onset temperature $T_C$ show inhomogeneous local tunneling DOS with two kinds of spectra: spectra with only SDW gap and spectra with some signature of asymmetric or symmetric suppression in DOS near ± 5 mV bias range together with the SDW gap. In the later case, some spectra show one peak near +5 mv and some spectra show a sharp decrease in DOS at negative bias without any peak. Although these spectra are unlike those described by Bardeen-Cooper-Schrieffer (BCS) theory that show two sharp coherence peaks, but they correlate well with the SC phase, as this asymmetric or symmetric suppression near ± 5 mV disappears above $T_C$. We also observed atomically resolved surfaces at the lowest studied temperature (5.4 K). A preliminary version of this work was also reported by us in a conference \cite{20}.

2. Experimental details

Single crystals of CaFe$_{1.96}$Ni$_{0.04}$As$_2$ were grown \cite{19} by the high temperature solution growth using Sn-flux under identical condition as mentioned in Ref.\cite{21}. Electrical resistivity was measured using a standard four-probe method using a closed cycle refrigerator. DC magnetization was measured in a superconducting quantum interference device magnetometer. STM/S studies were done in cryogenic vacuum using a homemade variable temperature STM with fresh-cut Pt$_{0.8}$Ir$_{0.2}$ tips. The STM head is based on a design published earlier \cite{22} and we use RHK electronics and software. Standard ac-modulation technique was used for STS measurements with a modulation amplitude between 1 and 10 mV and frequency 2731 Hz.

A tunnel spectrum (dI/dV) differs from the actual DOS due to thermal smearing and the voltage dependence of the tunneling matrix element. The temperature smears out the spectral features of width less than a few $k_B T$. For small bias the tunneling matrix element is almost independent of bias voltage and the measured dI/dV displays thermally smeared local electron density of states. But for large bias we cannot ignore the voltage dependence of the tunneling matrix element. By plotting d(lnI)/d(lnV), one can normalize away the effect of the tunnel matrix element on the spectra \cite{23}. So for small bias (20 mV) we plot dI/dV directly to show the DOS, while d(lnI)/d(lnV) is plotted for large bias (250 mV), which sharpens the spectral features.

STM/S measurements were performed on in situ cleaved single crystals. The in situ cleaving was done at room temperature and at $4 \times 10^{-6}$ mbar pressure before transferring the sample to the STM head at low temperature (78 K). STM/S measurements were performed in two different temperature ranges: 78 K to 292 K and 5.4 K to 19.7 K. To obtain the temperature below 78 K, we used liquid helium. We took
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**Figure 1.** Temperature dependence of the in plane electrical resistivity for as-grown $\text{CaFe}_{1.96}\text{Ni}_{0.04}\text{As}_2$ single crystal. The inset shows an expended view of the region near the superconducting transition.

$\frac{dI}{dV}$ Vs V spectra at different positions of the crystal surface over an area of around $2 \times 2 \mu\text{m}^2$ for all studied temperatures. The tunneling spectra in the temperature range 78 K to 292 K were taken with 100 pA tunnel current and 250 mV bias. Whereas, the tunneling spectra in temperature range 5.4 K to 19.7 K were taken with 100 pA tunnel current and 20-250 mV bias.

3. Results and Analysis

Fig. 1 shows the in-plane electrical resistivity of as-grown $\text{CaFe}_{1.96}\text{Ni}_{0.04}\text{As}_2$ single crystal. The in-plane resistivity decreases slowly with decreasing temperature below room temperature down to 121.5 K. Then there is a relatively broad upturn starting at $T_{SDW} = 121.5\text{K}$ due to SDW transition. This rise in resistivity is attributed to the opening of a gap in parts of the Fermi surface giving rise to a loss in DOS at $E_F$ upon entering the SDW state. However, resistivity starts decreasing rapidly as the temperature is reduced further indicating that the Fermi surface is only partially gapped in the SDW state. As the temperature decreases further, a sharp drop in the resistivity occurs at $\sim$ 15 K due to the onset of superconducting order. But the resistivity does not go to zero down to 1.3 K. Full superconducting transition develops only at slightly higher Ni doping (0.053 and 0.06)[19] with an onset $T_C$ of 15 K. The drop in the resistivity at $\sim$ 4 K is due to the presence of traces of Sn on the surface of single crystals.

Fig. 2 shows the temperature dependence of magnetic susceptibility $\chi$ measured under zero field cool (ZFC) condition at 50 Oe. Field was applied perpendicular to the c-axis of the crystals. The susceptibility remains unchanged (nearly zero) with decreasing temperature down to 15 K. $\chi$ starts decreasing for temperature below $\sim$ 15 K and becomes negative. But $4\pi\chi$ does not saturates to -100 % down to 4 K. This
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Figure 2. Temperature dependence of the zero field cooling (ZFC) magnetic susceptibility for $\text{CaFe}_{1.96}\text{Ni}_{0.04}\text{As}_2$ single crystal in 50 Oe.

Figure 3. (Color online) Simultaneously acquired (a) topographic and (b) conductance images (area: $140 \times 140$ nm$^2$) of $\text{CaFe}_{1.96}\text{Ni}_{0.04}\text{As}_2$ single crystal at 292 K taken with a junction bias of 500 mV, a tunnel current of 60 pA and an ac-modulation voltage of 10 mV. (c) Topographic profile along the lines marked in (a). (d) $dI/dV$ variation along the marked line in (b), which is less than 9%.

indicates that the system is not fully shielded. Assuming demagnetization factor to be zero for $H \parallel a - b$ plane, measured shielding fraction is only $\sim 1.2\%$ at 5 K. Thus only a small portion of the system is superconducting at 4 K.

Simultaneously acquired STM topographic and conductance images of in situ cleaved $\text{CaFe}_{1.96}\text{Ni}_{0.04}\text{As}_2$ single crystal at 292 K are shown in Fig. 3. Topographic image in Fig. 3 (a) shows a nice step-terrace morphology. The surface is atomically flat over the terraces, with an rms roughness over any terrace as $\sim 0.11$ nm. At all studied temperatures, the STM topographic images show a similar kind of step-terrace
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Figure 4. (Color online) (a) and (b) are the STM topographic images at 5.4 K of $69.22 \times 69.22$ nm$^2$ and $13.17 \times 13.17$ nm$^2$ respectively. Image in (a) was taken with a junction bias of 100 mV and a tunnel current of 100 pA while that in (b) was taken with constant height mode.

The line profile in Fig. 3 (c) along the line marked in the topographic image in Fig. 3 (a) shows that the terraces are separated by twice the atomic steps of height $\sim 1.2$ (±0.11) nm [19]. The topographic image in Fig. 4 at the lowest studied temperature (5.4 K) shows signatures of atomic resolution. The conductance has very little variation over the flat terraces except for few isolated bright (yellow) spots. Magnified conductance image near these spots does not show same contrast indicating these spots are arise due to noise. The variation of conductance along a line marked in the conductance image in Fig. 3 (b) is shown in the Fig. 3 (d) showing a homogeneous nature. There is a little jump in the conductance at the steps resulting from feedback instability at the steps.

The temperature dependent tunneling spectra, $dI/dV$ versus $V$ between temperatures 78 K and 292 K are shown in Fig. 5 (a). Each plotted spectrum at a particular temperature is a spatial average of about one hundred spectra. We plotted spatial average because spatially resolved spectra in this temperature range (78 - 292 K) show very little inhomogeneity as seen from the conductance map discussed earlier. Spectra at 78 K at different locations of the sample are shown in Fig. 6 (a). The conductance near zero bias for the low temperature spectra shows a dip indicating the presence of a partial gap, while that at higher temperatures only has a noticeable curvature. $d(lnI)/d(lnV) - V$ is plotted in Fig. 5 (b) which eliminates the effect of the voltage dependence of the tunneling matrix element and sharpens the gap feature, as discussed earlier. Above $T_{SDW}$ (121.5 K), there is a broad depression in the $d(ln I)/d(ln V)$-V spectra which becomes more pronounced as the temperature goes below $T_{SDW}$. We attribute this to the opening of a partial gap at the $E_F$ upon entering the SDW state. Similar gap was seen in EuFe$_2$As$_2$ [24]. The spectra at 147 K (above $T_{SDW}$) has a weak signature of gap. The gap signature above the SDW transition may arise from short-range SDW fluctuations [23].

The spatially resolved $dI/dV$ - V spectra are shown in Fig. 6 (b) at 5.4 K. These spectra show significant spatial inhomogeneity as compared to that at 78 K. However, all the spatially resolved $dI/dV$ - V spectra show SDW gap. But the spectra do not show
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Figure 5. Temperature dependent (a) $dI/dV$ and (b) $d(\ln I)/d(\ln V)$ spectra of CaFe$_{1.96}$Ni$_{0.04}$As$_2$ single crystals between 292 K and 78 K. Spectra were taken with a junction bias of 250 mV and a tunnel current of 100 pA. Consecutive spectra have been shifted uniformly upwards for clarity.

any signature of superconducting gap as expected from the resistivity behavior. These spectra were taken using 10 mV ac modulation voltage. We reduced ac modulation voltage to 1 mV as well as the bias range to see the low energy features more carefully.

Spatially resolved $dI/dV$ spectra along a line of a topographic image taken with 1 mV ac modulation are shown in Fig. 7 at 5.4 K. These $dI/dV$ spectra are plotted in Fig. 7(b) to (e). Each spectrum at a particular location is an average of 10 - 20 spectra and the distance between two consecutive locations is 2.4 nm. We clearly see that the nature of the spectra changes over nm length scale. Fig. 8 shows a spectrum over a large energy range indicating the coexistence of SDW and SC phases at the same location.

We also took $dI/dV$ - $V$ spectra at a number of different locations on the surface of the crystal. These $dI/dV$ - $V$ spectra at 5.4 K at different locations are plotted in Fig. 9(a) - (d) showing significant spatial variations similar to those obtained along a line as discussed earlier. None of the spectra show true superconducting gap with two coherence peaks but there is some signature of asymmetric suppression in DOS in ±5 mV bias range in some of the spectra. In some spectra, one peak is observed near +5 mV in the $dI/dV$ spectra. In some cases a sharp decrease in DOS occurs at negative bias without any peak. Few spectra show a symmetric depression over ±5 mV bias. In some locations the spectra show a rising DOS with a minima near zero bias but without any low energy features, i.e. these spectra are similar to the ones above 20 K.

We measured tunnel spectra at different locations across $T_C$ at four more temperatures (8.6, 11.8, 15.8 and 19.7 K) to see how the inhomogeneity evolves with temperature. Fig. 9(e) - (h) show $dI/dV$ spectra at 19.7 K at different locations. Below
Figure 6. $dI/dV$ spectra of CaFe$_{1.96}$Ni$_{0.04}$As$_2$ single crystals taken at different locations of the sample at (a) 78 K and (b) 5.4 K. These spectra were taken with a junction bias of 250 mV and a tunnel current of 100 pA. Consecutive spectra have been shifted uniformly upwards for clarity. Insets show the spatially averaged $dI/dV$ spectra.

Figure 7. (Color online) (a) STM topographic image at 5.4 K. (b)-(e) Spatially resolved $dI/dV$ spectra along the marked line of the topographic image in (a). Separation between two consecutive spectra is 2.4 nm. Spectra were taken with a junction bias of 20 mV bias and a tunnel current of 100 pA.
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**Figure 8.** $dI/dV$ spectra of CaFe$_{1.96}$Ni$_{0.04}$As$_2$ single crystals at 5.4 K. Spectra were taken with a junction bias of 30 mV and a tunnel current of 100 pA. Downward arrow indicates the SC gap.

**Figure 9.** (a)-(d) $dI/dV$ spectra at different locations taken with a junction bias of 20 mV and a tunnel current of 100 pA at 5.4 K. They have been grouped in different panels based on similarity between spectra. (e)-(h) $dI/dV$ spectra at different locations taken with same junction parameters but at 19.7 K.
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Figure 10. (a) Spatially averaged dI/dV spectra with a sharp change in DOS at -ve bias at different temperatures and (b) Spatially averaged dI/dV spectra with a peak at +ve bias at different temperatures.

$T_C$, the dI/dV spectra show qualitatively similar inhomogeneities as observed at 5.4 K with some reduction in sharpness in features. Above $T_C$, the spatially resolved dI/dV spectra do not show any peaks and the spatial variation is markedly reduced. We would like to state that we cannot track the same area as a function of temperature in our STM as the relative xy-shift between tip and sample with temperature is significant.

Based on the observed spectra we divided them into three categories: spectra with a sharp change in DOS at -ve bias, spectra with a peak at +ve bias, and spectra with no sharp change. We took the spatial average of the spectra showing peak at +ve bias and of those showing sharp change in DOS at -ve bias. We plot the temperature dependence of these two types of spatially averaged spectra in Fig. 10(a) and (b), which clearly demonstrate the evolution of the DOS across the $T_C$. Below $T_C$, there is some depression in the DOS near the Fermi energy (at zero bias) and this depression becomes more pronounced as the temperature goes down.

Our observed tunnel spectra at low temperatures are unlike those of typical superconductors, which show a BCS gap with two coherence peaks in DOS. However, the observed asymmetric or symmetric depression in DOS correlates well with the bulk superconductivity below $T_C$. Above $T_C$, there is no peak or depression in the tunnel spectra but as the temperature crosses the superconducting transition temperature, peak or depression in the tunnel spectra starts to appear. The depression in DOS at low energies is most pronounced at the lowest studied temperature. The temperature variation of the tunnel spectra across $T_C$ clearly indicates that this peak disappears above superconducting transition. Moreover, from the quality of the images with some
signatures of atomic resolution, we believe that we are probing an intrinsic surface. If we take a spatial average of these spectra we do get a gap-like structure with symmetric depression in DOS and weak peak-like features corresponding to BCS coherence peaks. Asymmetric spectra with gap have been seen routinely in high-$T_C$ cuprates [25, 26] with an energy gap and two coherence peaks. So the spectra that we observed are somewhat peculiar but correlate with the SC transition.

4. Discussions

All of the tunnel spectra below $T_C$ show a SDW gap and some of them have both SDW and SC gap. This suggests that only a fraction of the system becomes SC below $T_C$. Emergence of inhomogeneous DOS only below the SC onset temperature is somewhat puzzling. It is possible that some small inhomogeneities do exist above $T_C$ and they evolve into more clearly visible features below $T_C$. These features mainly involve an asymmetric depression in DOS near Fermi energy in 5 mV energy range. This energy scale is consistent with the typical superconducting gap reported in some of the pnictides [4, 27]. Susceptibility data also show that a tiny fraction ($\sim 1.2\%$) of the system is in the SC state below $T_C$. Thus below $T_C$, a major portion of the system is in non-SC state and a tiny portion is in SC state. Below $T_{SDW}$, a portion of the Fermi surface is disappearing due to the SDW transition. Thus the Cooper pairing occurs when a portion of the Fermi surface is already gapped by the magnetic order.

In our STM/S we have observed the inhomogeneities on a very small length-scale ($\sim 1$ nm). A very homogeneous Ni distribution at this doping (0.04) will give $\sim 1.4$ nm average separation between Ni atoms in a-b plane. So we cannot reconcile our results with clustering of Ni atoms as that would lead to an inhomogeneity over a much larger length scale. We have also seen the same inhomogeneous spectra in 3-4 different surfaces of these crystals, which makes it very unlikely that Ni is segregating over length scales larger than what is accessible to our STM scanner. This is further ruled out from smooth susceptibility data without any sharp jumps. Signatures of atomic resolution and atomic steps with flat terraces make the surface contamination a very unlikely possibility.

In a typical pnictide phase diagram (see Fig. 11), which is similar to cuprate superconductors, there is the famous superconducting dome and a phase boundary extending to much higher temperatures that separates the SDW phase from the paramagnetic phase. Thus the phase boundaries touch the $T = 0$ axis at three points ($x_1$, $x_2$ and $x_3$) and it is not fully clear if all the three points are QCPs. The temperature dependent resistivity, penetration depth and spin-lattice relaxation measurements have strongly suggested that $x_2$ is a QCP in pnictides [14, 15]. In cuprates field doping near $x_1$ point has also revealed a QCP due to crossover between SC phase and an insulator phase[28]. Larger inhomogeneities have been observed in underdoped cuprates than the overdoped ones [29]. Superconductor to insulator transition has also been reported in heavily disordered conventional superconductors although it is not clear if this disorder also acts as a dopant and directly affects the SC order [30]. In the latter
case inhomogeneities have also been systematically studied using STM/S [31].

The inhomogeneities reported here have similarities to some of those seen in disordered conventional superconducting films and also under-doped cuprates close to and below $T_C$. The composition of our crystals is close to the superconducting dome boundary on the under-doped side. Proximity to this boundary makes the non-SC phases easily accessible and presence of disorder will further help in nucleating such phases. We believe that in proximity to this crossover point, which may be a QCP, the system will be extremely susceptible to disorder and eventually the disorder might influence this crossover more than doping. Our results are suggestive of the presence of a QCP near $x_1$ in pnictides. The composition of studied sample is very close to $x_1$ and this proximity to QCP will amplify the effect of disorder arising due to Ni dopants.

5. Conclusions

In conclusion, we presented a detailed temperature dependent STM/S investigation of underdoped $\text{CaFe}_{1.96}\text{Ni}_{0.04}\text{As}_2$ single crystals. Temperature dependent resistivity measurement shows that $\text{CaFe}_{1.96}\text{Ni}_{0.04}\text{As}_2$ single crystals undergo both SDW at $\sim 121.5$ K and SC transition at $\sim 15$ K. Magnetic measurement confirm a tiny portion ($\sim 1.2\%$) of this compound being superconductor below the SC onset temperature $T_C \sim 15$ K. Our STM measurements show atomically flat terraces separated by monatomic steps with signatures of atomic resolutions at lowest temperature indicate good surface quality. Above $T_C$, the tunnel spectra show homogeneous local DOS with a SDW gap. However, the tunnel spectra show inhomogeneity in local DOS in superconducting state with SDW gap. But low energy scale spectra at low temperatures ($< T_C$) show an asymmetric or a symmetric dip in DOS at $\pm 5$ meV energy in some of the spectra. In some locations the spectra show a rising DOS with a minima near zero bias but without any low energy features, i.e. these spectra show only SDW gap. We attribute this asymmetric or symmetric depression to the SC transition as it correlates well with $T_C$. Thus
whenever SC phase arises below $T_C$, it coexists with SDW phase at the same location. Which also indicates that Cooper pairs can be formed even when the Fermi surface is partially gaped due to SDW transition. However, as the temperature goes above the superconducting transition temperature, the inhomogeneity disappears. Inhomogeneity could be attributed to the chemical inhomogeneity, in particular distribution of Ni substitutions but the three different types of qualitatively different spectra do not fit this scenario. Observed inhomogeneity over such a small length-scale ($\sim 1$ nm) and smooth susceptibility data also exclude the possibility of segregation of Ni. We believe the inhomogeneities in this underdoped compound below $T_C$ are intrinsic due to proximity to the non-sc phase.

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