

Temperature-Dependent Transformation of the Magnetic Excitation Spectrum on Approaching Superconductivity in $\text{Fe}_{1+y-x}(\text{Ni}/\text{Cu})_x\text{Te}_{0.5}\text{Se}_{0.5}$

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(Received 20 January 2012; revised manuscript received 9 October 2012; published 28 November 2012)

Spin excitations are one of the top candidates for mediating electron pairing in unconventional superconductors. Their coupling to superconductivity is evident in a large number of systems, by the observation of an abrupt redistribution of magnetic spectral weight at the superconducting transition temperature, T_c , for energies comparable to the superconducting gap. Here we report inelastic neutron scattering measurements on Fe-based superconductors, $\text{Fe}_{1-x}(\text{Ni}/\text{Cu})_x\text{Te}_{0.5}\text{Se}_{0.5}$, that emphasize an additional signature. The overall shape of the low energy magnetic dispersion changes from two incommensurate vertical columns at $T \gg T_c$ to a distinctly different U -shaped dispersion at low temperature. Importantly, this spectral reconstruction is apparent for temperatures up to $\sim 3T_c$. If the magnetic excitations are involved in the pairing mechanism, their surprising modification on the approach to T_c demonstrates that strong interactions are involved.

DOI: [10.1103/PhysRevLett.109.227002](https://doi.org/10.1103/PhysRevLett.109.227002)

PACS numbers: 74.70.Xa, 61.05.fg, 75.25.-j, 75.30.Fv

In weak-coupling models of magnetically mediated superconductivity, magnons essentially replace phonons as the pairing bosons [1]. By assumption, the interaction between the electrons and bosons is not strong enough to significantly modify the bosonic excitation spectrum. In conventional systems, superconductivity does modify the self-energy of the phonons, causing changes in the energy-dependent line shape, but there is no significant change in the phonon dispersion [2,3]. In many unconventional superconductors, including high- T_c cuprates [4–7], heavy Fermion superconductors [8,9], and the recently discovered Fe-based superconductors [10–12], one observes, on cooling below T_c , the gapping of low-energy spin fluctuations and a shift of spectral weight to a “resonance” peak. Empirically, the magnetic spectrum found above and below T_c tends to be qualitatively the same.

Here we study the low-energy spin fluctuations in single-crystal samples of the superconductor $\text{Fe}_{1+y}\text{Te}_{0.5}\text{Se}_{0.5}$ (the “1:1” system, $T_c = 14$ K) as we perturb the system by making partial substitutions for Fe. Substituting 2% and 4% of Ni reduces T_c to 12 and 8 K, respectively, while 10% of Cu results in an absence of superconductivity, as shown in Fig. 1(a). Our inelastic neutron scattering measurements show that low energy ($\hbar\omega \lesssim 12$ meV) magnetic excitations transform from having two peaks clearly (about a quarter of Brillouin zone) away from the M point in reciprocal space [(0.5, 0.5, 0) using the two-Fe unit cell] at high temperature in the normal state, to having a broad maximum at the M point at low temperature in the

superconducting phase. This drastic change on the magnetic dispersion between the superconducting and nonsuperconducting phases suggests that strong correlations between electrons have to be taken into account when the magnetic and electronic properties of the “1:1” system are considered.

Single crystals of $\text{Fe}_{1+y-x}(\text{Ni}/\text{Cu})_x\text{Te}_{0.5}\text{Se}_{0.5}$ were grown by a unidirectional solidification method [13] at Brookhaven National Laboratory. The lattice constants are $a = b = 3.81$ Å, and $c = 6.02$ Å, using the two-Fe unit cell. For convenience, we label these samples as Ni02, Ni04, and Cu10, according to the amount of Ni/Cu doping on the Fe site. To minimize Fe interstitials, a nominal composition of $y = -0.02$ was used for all three samples. The neutron scattering experiments on the two Ni02 and Ni04 samples were carried out on the BT7 triple-axis spectrometer at the NIST Center for Neutron Research, using beam collimations of open-50'-S-50'-240', a fixed final energy of 14.7 meV, and two pyrolytic graphite filters after the sample. The Cu10 sample was measured on the HB1 triple-axis spectrometer at the High Flux Isotope Reactor, Oak Ridge National Laboratory, with beam collimations of 48'-40'-S-60'-240', fixed final energy of 13.5 meV, and two pyrolytic graphite filters after the sample. No static order around (0.5, 0, 0.5) was found in any of the three samples. The inelastic scattering experiments were all performed in the (HK0) zone, so that the scattering plane is defined by the [100] and [010] wave vectors. All data have been normalized into absolute units of $\mu_B^2 eV^{-1}/\text{Fe}$ by

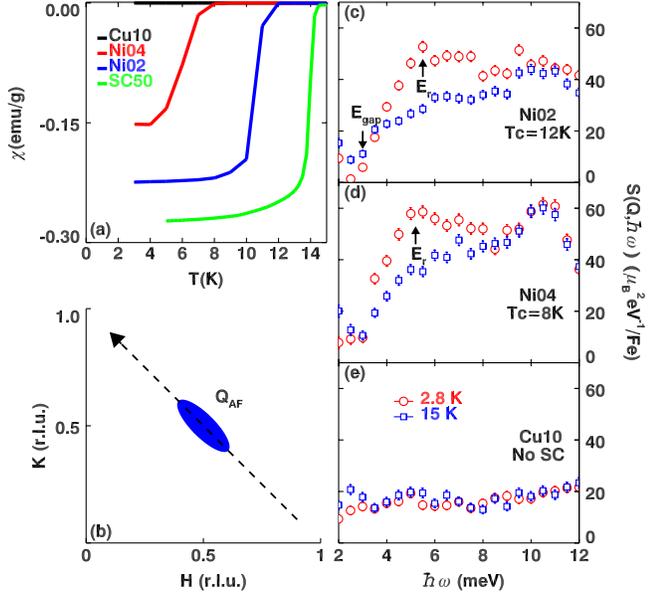


FIG. 1 (color online). Magnetic susceptibility and inelastic neutron scattering measurements performed on the NiO2, NiO4, Cu10, and SC50 ($\text{FeTe}_{0.5}\text{Se}_{0.5}$) samples. (a) Magnetic susceptibility measurements, and (c), (d), (e) magnetic neutron scattering intensity measured at \mathbf{Q}_{AF} with $T = 2.8$ K (red circles) and 15 K (blue squares). The error bars represent the square root of the number of counts. The fitted background obtained from constant-energy scans has been subtracted from all data sets. The (HK0) scattering plane is plotted in (b) while the dashed line denotes the direction for the Q scans shown in Fig. 2.

incoherent elastic scattering intensities from the samples. X-ray diffraction measurements of lattice parameters were performed at beam line X22B of the National Synchrotron Light Source, Brookhaven National Laboratory.

We are interested in the magnetic excitations near the M point, which we define as $\mathbf{Q}_{\text{AF}} = (0.5, 0.5, 0)$. Note that this is different than the ordering wave vector $(0.5, 0, 0.5)$ of the parent compound FeTe, but it is the same in-plane wave vector characteristic of magnetic scattering in other Fe-based superconductors. Figures 1(c)–1(e) show the measured inelastic neutron scattering intensity as a function of energy obtained at $T = 2.8$ K and 15 K for all three samples. It has been established in previous studies [14–17] that the unperturbed superconductor has a magnetic resonance peak at $E_r \sim 7$ meV. Here we see that E_r decreases to ~ 6 and 5 meV in the NiO2 and NiO4 samples, respectively, while there is no observable resonance in the nonsuperconducting Cu10. One can also see a spin gap of about 3 meV in NiO2, but the gap is more difficult to resolve for NiO4.

Things get more interesting when we look at the wave vector (\mathbf{q}) dependence of the magnetic scattering. It has been established in previous studies [14,15,18] of superconducting $\text{FeTe}_{1-x}\text{Se}_x$ that the magnetic excitations disperse from \mathbf{Q}_{AF} only in the transverse direction, along $[1, -1, 0]$. Figure 2 shows scans along this direction for the NiO4 sample at a series of energies, illustrating the

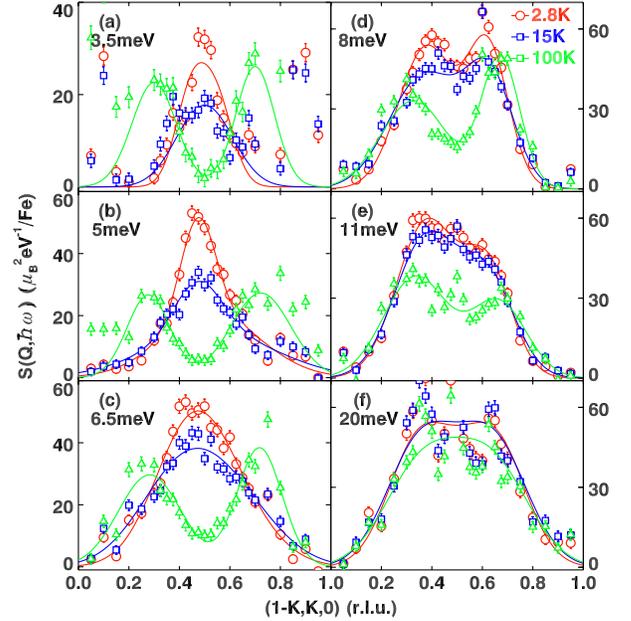


FIG. 2 (color online). Wave vector dependence of the magnetic scattering intensity along the transverse direction through \mathbf{Q}_{AF} [see Fig. 1(b)] for the NiO4 sample at $T = 2.8$ K (red circles), 15 K (blue squares), and 100 K (green triangles), obtained at excitation energies (a) 3.5 meV, (b) 5 meV, (c) 6.5 meV, (d) 8 meV, (e) 11 meV, and (f) 20 meV [which was measured in a higher zone, near $\mathbf{Q} = (1.5, 0.5, 0)$]. Solid lines are guides to the eye. [The spurious peaks near $K = 0.1$ and 0.9 in (a) have the temperature dependence of phonons, and are only significant at the lowest energies.]

variation of the \mathbf{q} dependence as the temperature changes from 2.8 K ($\ll T_c$) to 15 K ($\approx T_c$) and then up to 100 K ($T \gg T_c$). The variations are minor at the higher energies, as in Figs. 2(e) and 2(f), but become dramatic for $E \sim E_r \approx 5$ meV, as in Figs. 2(a)–2(c). The change from $T \ll T_c$ to $T \approx T_c$ is simply the standard resonance behavior. The feature that we wish to emphasize is the change from a single commensurate peak at $T \approx T_c$ to a pair of well-resolved incommensurate peaks at $T \gg T_c$. This change cannot be confused with a temperature-dependent change in peak width.

The same data are presented again, slightly cleaned up and in a different format, in Figs. 3(a)–3(c). The lower-temperature data exhibit a U -shaped dispersion, with the bottom of the U at $\sim E_r$. Except for the change in the resonant peak, the basic shape of the dispersion does not really change on crossing T_c . In contrast, the dispersion at 100 K is qualitatively different: it looks like the legs of a pair of trousers. It also looks very similar to the low-temperature dispersion of the nonsuperconducting Cu10 sample shown in Fig. 3(d). (Note that limited measurements on a nonsuperconducting Ni10 sample are consistent with the Cu10 results.)

There is clearly a major change in the low-energy portion of the dispersion between 15 and 100 K, but how does it change between those temperatures? This is illustrated in

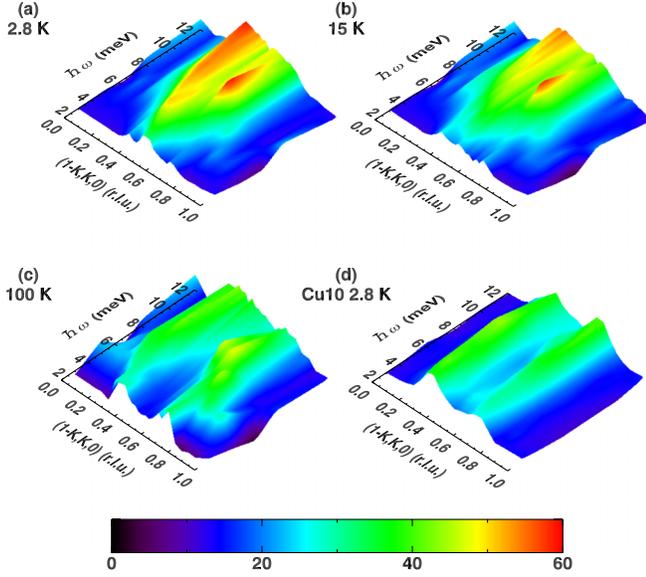


FIG. 3 (color online). Magnetic scattering intensity plotted for the Ni04 sample in energy-momentum space at (a) 2.8 K, (b) 15 K, and (c) 100 K. Results for the Cu10 sample measured at 2.8 K are plotted in (d). The data have been smoothed, and nonmagnetic sharp spurious signals [see Fig. 2(a)] have been removed for better visual effects.

Fig. 4. Focusing in particular on the results for the Ni04 sample, in Fig. 4(e) we see that the crossover is continuous in temperature, but with a reasonably defined midpoint at 30 ± 10 K. For Ni02, the midpoint may be closer to 40 K. In both cases, the crossover occurs at temperatures of order $3T_c$. We previously observed [17] hints of this temperature dependent modification of the dispersion in superconducting

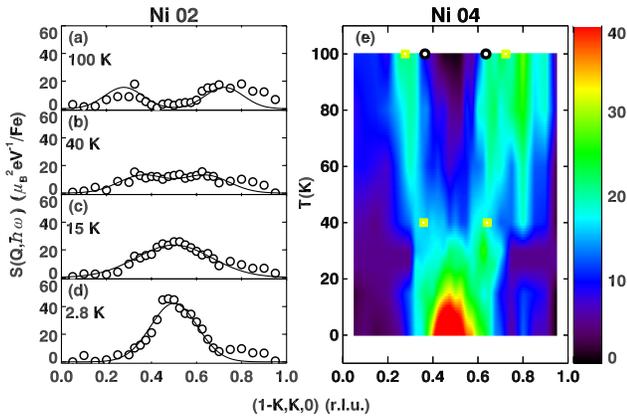


FIG. 4 (color online). Thermal evolution of the magnetic scattering at $\hbar\omega = 5$ meV. The data are measured through \mathbf{Q}_{AF} along the transverse direction for the Ni02 sample at (a) 100 K, (b) 40 K, (c) 15 K, (d) 2.8 K, and (e) for the Ni04 sample plotted as an intensity contour map in temperature-wavevector space. The data have been smoothed. The yellow and black symbols in (e) denote the corresponding peak positions for the Ni02 sample (yellow squares) and for a superconducting $\text{Fe}_{1+\delta}\text{Te}_{0.35}\text{Se}_{0.65}$ sample (black circles) [17].

$\text{FeTe}_{0.35}\text{Se}_{0.65}$; however, the high-temperature incommensurability was not as large nor as well resolved as for the Ni- and Cu-doped samples [see Fig. 4(e)].

It is possible to see the incommensurate columns of magnetic scattering even at low temperature when the superconductivity is suppressed, as shown for the Cu10 sample in Fig. 3(d). A similar low-temperature spectrum has been observed previously in non-bulk-superconducting “1:1” samples such as $\text{Fe}_{1.04}\text{Te}_{0.73}\text{Se}_{0.27}$ [18] and $\text{Fe}_{1.10}\text{Te}_{0.75}\text{Se}_{0.25}$ [19]. Thus, whether one destroys the superconductivity with excess Fe or by sufficient substitution of Cu (or Ni), the impact on the magnetic excitations is qualitatively similar.

There is an evident pattern that superconducting 1:1 samples have commensurate or almost commensurate magnetic excitations at the resonance energy, while non-superconducting samples have incommensurate excitations. Our results for the Ni-doped samples show that it is possible for a sample to transform from the incommensurate phase at high temperature to the low-energy-commensurate phase on cooling. The commensurability appears at the energy scale of the resonance energy at a temperature of $\sim 3T_c$, which is coincidentally also comparable to the maximum pressure-induced T_c in the $\text{Fe}_{1+y}\text{Te}_{1-x}\text{Se}_x$ system [20,21].

The temperature dependence of the magnetic spectrum has motivated us to check for related changes in other properties. We note that an x-ray scattering study of $\text{Fe}_{1.03}\text{Te}_{0.43}\text{Se}_{0.57}$ detected a transition to an orthorhombic phase on cooling below 40 K. Although such a transition has not been detected in our Ni04 sample, x-ray diffraction measurements indicate an anomalous in-plane expansion for $T \leq 60$ K. Similar behavior was observed in neutron diffraction measurements of $\text{Fe}_{1+y}\text{Te}_{1-x}\text{Se}_x$ for $0.1 \leq x \leq 0.2$ (with $x = 0.2$ being the maximum Se concentration examined in that work) [22]; at smaller x , the transition to the monoclinic phase was observed.

In the iron-based superconductors, it has been proposed that there are competing electronic instabilities similar to those in the cuprates [23,24]. The existence of a nematic phase that is directly related to orbital order has been proposed and discussed in detail [25]. In addition to antiferromagnetism and superconductivity, the material also has a propensity toward xz/yz orbital ordering, which has been observed directly by angle-resolved photoemission spectroscopy in the case of $\text{Ba}(\text{Fe}_{1-x}\text{Co}_x)_2\text{As}_2$ [26]. Such ferro-orbital ordering has been shown theoretically to couple strongly to the commensurate magnetic correlation in both the strong coupling [27] and weak coupling [24] picture.

With Se doping, disorder due to the mixture of Se and Te [28], as well as our partial substitutions for Fe, will tend to frustrate long-range ordering. The abnormal behavior of the in-plane lattice parameter reported in Ref. [22] and in our Fig. 5, is likely related to local structural changes similar to the structural phase transition in the parent

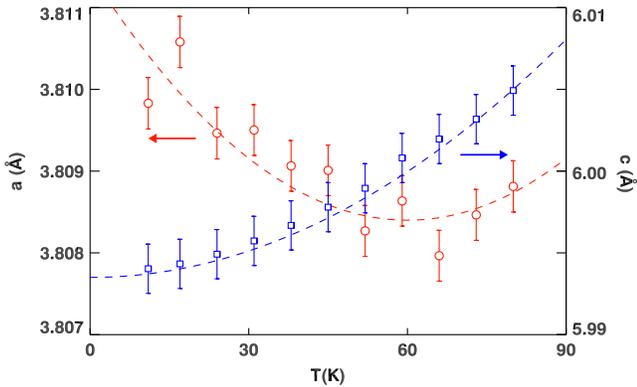


FIG. 5 (color online). Lattice parameters a (red circles) and c (blue squares) measured on the Ni04 sample.

compounds, but occurring on a much smaller length scale. With the suppression of long-range order of structural modulations, long-range orbital ordering will also diminish. However, point-contact measurements on both the 1:2:2 and 1:1 compounds have shown that electronic nematicity arising from orbital fluctuations exists even above the structural transition temperature [29,30]. This indicates that even without long-range order, orbital correlations can still play an important role. The abnormal temperature dependence of the in-plane lattice parameters could be related to a freezing of local orbital correlations. We suggest that the crossover we observe at $\sim 3T_c$ reflects such an orbital freezing “transition” in the presence of disorder.

On the other hand, Fermi surface topology is also believed to affect the magnetic response in the Fe-based superconductors. Incommensurate magnetic response has been observed in a number of Fe-pnictide systems, and in some cases has been attributed to nesting between electron and hole Fermi pockets [31,32]. In the 1:1 compounds, the low Fermi energies [33] measured at both the electron ($\epsilon_F \approx 10 \pm 1$ meV) and hole pockets ($\epsilon_F \approx 4 \pm 2.5$ meV) mean that nesting effects should be quite sensitive to temperature. It is notable that our observed changes in magnetic dispersion of the Ni04 sample occur at $T \sim \epsilon_F/k_B$ for the hole pockets measured for a Ni-free sample [33]. Indeed, there are other signatures showing a change of electronic correlations in the 1:1 systems in the same temperature scale around $3T_c$. We find that Pallecchi *et al.* [34] observed a systematic sign change in the thermoelectric power for $0 \leq x \leq 0.45$ (with 0.45 being the maximum x studied) at temperatures comparable to that of the incommensurate to commensurate transition observed in our measurements. The changes in the thermopower provide direct evidence for modifications of the electronic density of states close to the Fermi level. This is consistent with changes in the optical conductivity of a sample with $x = 0.45$ by Homes *et al.* [35,36]. Between room temperature and 100 K, there is strong, frequency-independent damping of the conductivity. By 18 K, a few degrees above T_c , the damping is reduced for energies below 20 meV.

The degree of temperature-dependent transformation of the magnetic spectrum is unusual among unconventional superconductors. For example, in superconducting $\text{YBa}_2\text{Cu}_3\text{O}_{6+x}$ systems [4–6], the spin resonance develops at commensurate wave vectors below T_c , while above T_c the spectrum of magnetic excitations broadens in \mathbf{Q} but does not show any dramatic change in structure [37]. In superconducting $\text{La}_{2-x}\text{Sr}_x\text{CuO}_4$ the spin resonance occurs at lower energies where the spin fluctuations are incommensurate [38,39], both in the normal and superconducting phases. Returning to the analogy with electron-phonon coupling, strong interactions can lead to a modification of the spectrum through a structural phase transition, as occurs [40] in Nb_3Sn at a temperature above the superconducting T_c . In the present case, strong interactions appear necessary to cause the transformation from incommensurate to commensurate magnetic excitations.

Strong spin correlations near \mathbf{Q}_{AF} are needed for most electronic mechanisms of pairing [23,41–44]. In such a scenario, the momentum of the repulsive spin excitations couples the nearly nested hole and electron pockets, and in turn allows a superconducting gap to develop on both sets of pockets, though with opposite phases. Obviously, an incommensurate spin correlation of a very different momentum (about a quarter of the Brillouin zone away) would seriously impair the development of superconductivity in this kind of weak coupling scenario. More generally speaking, such a large change in the momentum reflects a dramatic change of the *short-range* spin and orbital correlation that hosts the superconductivity. It is thus not surprising that superconductivity can be entirely absent within such a different correlation. On cooling, do the electronic and magnetic correlations adjust themselves to enable the spin-fluctuation mechanism? If so, what are the energetic tradeoffs associated with this transformation? And can interactions strong enough to achieve this transformation lead to effectively the same pairing mechanism as the one identified from a weak-coupling approach? We hope that these questions will be addressed by future investigations.

We thank Igor Zaliznyak for useful discussions. Work at BNL is supported by the Office of Basic Energy Sciences, U.S. Department of Energy under Contract No. DE-AC02-98CH10886. Work at Berkeley is supported by the same office through Contract No. DE-AC02-05CH11231. The research at ORNL was sponsored by the Scientific User Facilities Division, Office of Basic Energy Sciences, U.S. DOE. Z. J. X. and J. S. W. contributed equally to this work.

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