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Evidence for superconducting fluctuations at 180 K: influence of Zn, Fe and Co doping on the phonon renormalization of $YBa_2Cu_3O_{7-\delta}$

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Abstract. Detailed studies on the mid-infrared-active phonon spectra of $YBa_2(Cu_{1-x}M_x)_3O_{7-\delta}$; M = Co, Fe and Zn for $0 \leqslant x \leqslant 10\%$, have been carried out from 30 K to room temperature. The phonon mode near 580 cm^{-1} softens at the superconducting transition temperature (T_c), as reported earlier. The *new* observation is a marked difference in the temperature dependence of the phonon in Co- and Fe-doped compounds as opposed to Zn-doped and undoped material. We observe that the phonon begins to soften at temperatures *well above* T_c—in some cases $\sim 2T_c$ ($\approx$ 180 K)—in the Co- and Fe-doped $YBa_2Cu_3O_{7-\delta}$, whilst no such behaviour is detected in Zn-doped and undoped samples. We argue that this precursor phonon softening above T_c in Co- and Fe-substituted samples is due to superconducting fluctuations. Our proposal is that the transition-metal-ion substitution affects the *dimensionality* of the system and the ramifications of this are crucial for explaining the observed differences between Zn/undoped $YBa_2Cu_3O_{7-\delta}$ and Co/Fe-doped compounds. Our observations cast doubt on the idea that 2D superconductors like $YBa_2Cu_3O_{7-\delta}$ show a Kosterlitz–Thouless behaviour. Instead, it appears that $T_c \to 0$ K and fluctuations are reduced for the fully decoupled layers.

1. Introduction

An interesting and unusual feature of the high-temperature copper-oxide superconductors is the behaviour of many of their normal-state properties in the neighbourhood of the superconducting transition. For example, with decreasing temperature the resistance in undoped or Zn-doped $YBa_2Cu_3O_{7-\delta}$ exhibits a sharp drop at T_c from its linear temperature dependence in the normal-state [1], whereas in Co- or Fe-doped samples, the resistance begins to decrease gradually *well above* T_c before dropping abruptly at the critical temperature itself. It is found empirically (e.g. [2]) that the resistance begins to decrease at temperatures as high as $2T_c(\approx 180$ K) for Co/Fe content $x \approx 4\%$: (at.%, i.e. x =0.04 in $YBa_2(Cu_{1-x}M_x)_3O_{7-\delta}$; in this paper M = Zn, Fe or Co) and the resistance starts to fall at temperatures closer to T_c for lower Co/Fe doping levels.

Precursor effects in transport quantities are often attributed to 'superconducting fluctuations', but evidence to support this claim is not readily accessible through bulk measurements (i.e. transport phenomena, rather than features in thermodynamic equilibrium). Somewhat more direct evidence emerges from specific heat

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(C_P) measurements [3], although in this case a clear separation between the effect of superconductivity and lattice anharmonicity is not possible. On the other hand, the temperature dependence of phonon modes [4–10] provides information on a *local* length scale. This length scale (determined by the phonon absorption process and depending on the normal coordinates of individual phonon modes) is typically well below 30 Å [11, 12]. Local order is therefore indistinguishable from long-range order, provided that the correlation length of the order parameter exceeds this inherent length scale of the experiment. The coupling of phonon-mode frequencies to the superconducting order parameter has been demonstrated previously in Co-doped and undoped $YBa_2Cu_3O_{7-\delta}$ [4, 5] where it is shown that the anomalous decrease in frequency observed at T_c for the phonon mode at 580 cm^{-1} correlates directly with the order parameter in the superconducting state. These investigations are now extended to encompass Fe- and Zn-doped $YBa_2Cu_3O_{7-\delta}$ samples.

In this paper we report that superconducting fluctuations increase with Fe and Co doping but *not* with Zn content in $YBa_2Cu_3O_{7-\delta}$. We also find for the Fe- and Co-doped materials that the temperature where the superconducting fluctuations become apparent is approximately the same magnitude as observed in other bulk properties, i.e. between T_c and $2T_c$. The crucial point here is that we are measuring the thermodynamic order parameter of the normal-superconductor transition on a *local* length scale, leading us to conclude that the normal-state behaviour above T_c is due to fluctuations of the superconducting order parameter. This implies the existence of superconducting clusters above T_c in the Fe- and Co-doped $YBa_2Cu_3O_{7-\delta}$ but not, or to a much lesser extent, in the undoped or Zn-doped compound.

This interpretation echoes previous detailed investigations on Bi, Ga and Pb superconducting thin films [13]. The DC electrical conductance was measured for many films of different thicknesses convincingly demonstrating the existence of a temperature-dependent superconductivity-related contribution to the electrical conductance above T_c. In favourable cases this was observed at temperatures as high as $2T_c$. Furthermore, theories based on the idea of superconducting carriers above T_c as a consequence of thermal fluctuations gave a satisfactory account of the empirical observations.

At first glance it is difficult to imagine why structurally similar compounds like Co/Fe-doped $YBa_2Cu_3O_{7-\delta}$ on one hand and Zn-doped/undoped $YBa_2Cu_3O_{7-\delta}$ on the other hand should exhibit such fundamentally different behaviour. One possible answer proposed in this paper is that the dimensionality of the phase transition is modified by doping. In geometrical terms, it is assumed, as is the current thinking, that the CuO_2 conducting planes are essential to the occurence of superconductivity in the copper oxides. Indeed, the electrical properties in the normal state do seem to indicate a predominantly 2D character of the wavefunctions. In the superconducting state, however, coupling between the layers becomes essential and the superconducting properties are 3D in character. It is crucial for the understanding of the high-temperature superconducting properties that the phase transition *increases* the dimensionality (2D→3D). Reduction of the 3D state in $YBa_2Cu_3O_{7-\delta}/PrBa_2Cu_3O_{7-\delta}$ layered films has shown that T_c decreases [14–16], although the 2D limit is ill-defined due to the penetration of the wavefunction into the surrounding matrix.

An alternative way to reduce the dimensionality is to dope the material with atoms which substitute on the Cu(1)† CuO-chain site only. This leads to an increas-

† Notation as used in Jorgensen *et al* [17].

ing decoupling between CuO_2 planes with increasing doping level. The mechanism involved in such a decoupling can be considered in the spirit of the apical-oxygen model [18, 19]. In this scheme the Emery Hamiltonian is coupled to the displacement of the O(4) atom in an anharmonic potential. It is reasonable to expect that substituting 'impurities' onto Cu(1) sites would change the local form of the anharmonic potential around the O(4) atom and thus alter the coupling between adjacent CuO_2 planes.

It is known (from neutron diffraction, EXAFS, Mössbauer spectroscopic studies etc) that Fe and Co substitute for copper ions preferentially on Cu(1) whereas divalent Zn tends to occupy both Cu(2) and Cu(1) sites ([9, 20–26] and references therein). The Fe- and Co-doped compounds undergo an orthorhombic to tetragonal structural phase transition at $x \approx 2.5\%$—in a similar fashion to the removal of oxygen at $\delta \approx 0.5$ in the undoped compound—whilst the Zn compounds do not. Also, Zn dramatically reduces T_c even in low concentrations, in contrast to Co and Fe which do not depress T_c until about $x \approx 2.5\%$.

Following the idea that Fe and Co substitutions onto Cu(1) sites gradually decouple the CuO_2 planes, we would expect that fluctuations above T_c will be created and enhanced with increasing doping level. Conversely, in general terms, doping with Zn on Cu(1) and Cu(2) sites will not have this effect and no increase of the fluctuations is expected.

In this paper we present experimental evidence showing that local superconducting states exist for $T \gg T_c$ in Co- and Fe-doped $YBa_2Cu_3O_{7-\delta}$ but *not* in Zn-doped or undoped material.

2. Experimental procedure

$YBa_2Cu_3O_{7-\delta}$ and $YBa_2(Cu_{1-x}M_x)_3O_{7-\delta}$ samples; M = Co, x =1.5%, 2.8%, 4.0%, 7.0%, M = Fe, x = 0.5%, 1.0%, 2.0%, 2.5%, 4.0%, 7.0%, 10.0%; M = Zn, x =1.0%, 3.0%, 5.0%, 7.0% were prepared using the powdering/sintering method. Details of the procedure and subsequent characterization methods used, including x-ray analysis, resistance and magnetic measurements are described elsewhere [1, 2, 20, 27].

A check on the microstructure of the Zn-doped samples was carried out on a Jeol JEM-2000ex transmission electron microscope (TEM). Extensive TEM studies on Fe- and Co-doped $YBa_2Cu_3O_{7-\delta}$ have already been reported by other authors [20, 28].

After being ground in Spex electrical ball mill, the powdered superconductors—ca 0.1 μm diameter grains—were mixed with dry CsI and pressed (at 10 ton cm^{-2}) into disc-shaped pellets of 13 mm diameter at room temperature. Each pellet contained 2 mg of superconductor, and identical pellets of CsI diluent were used as references. All pellets were stored under dry, cool conditions (dessicator, silica gel) and measured within 12 h of creation.

A Bruker 113v FTIR spectrometer set at 2 cm^{-1} resolution utilizing a liquid-nitrogen cooled MCT detector was used for the absorption measurements. Spectra were recorded in the mid-infrared ($\gtrsim$ 450 cm^{-1}) under vacuum with the pellet in a Leybold He-cryostat equipped with KRS-5 windows, thus allowing a range from 30 K to room temperature to be achieved. An Aspect 3000 computer was used for the data collection and processing. This involved a least-squares band-profile analysis of the spectra using Voigt profiles, enabling the absolute frequency of each phonon band to be directly estimated from the spectrum with a random error in measurement of ± 0.5 cm^{-1}.

3. Results

The infrared absorption spectra of Fe-, Zn- substituted $YBa_2(Cu_{x-1}M_x)_3O_{7-\delta}$ and undoped $YBa_2Cu_3O_{7-\delta}$ are presented in figure 1. For the spectra of the Co-doped compound see [4]. In a few samples—e.g. 0.5% Fe, figure 1(*a*)—tiny traces of $BaCO_3$ impurity phase, at $\sim 690\ cm^{-1}$ and $\sim 857\ cm^{-1}$, were identified. As phonon lines in all spectra are fairly sharp, (i.e. no inhomogeneous line broadening was found) it is evident that M atoms have been incorporated into the structure in a fairly homogeneous manner. No evidence is found for cluster formation or segregation of M on twin boundaries or other lattice imperfections.

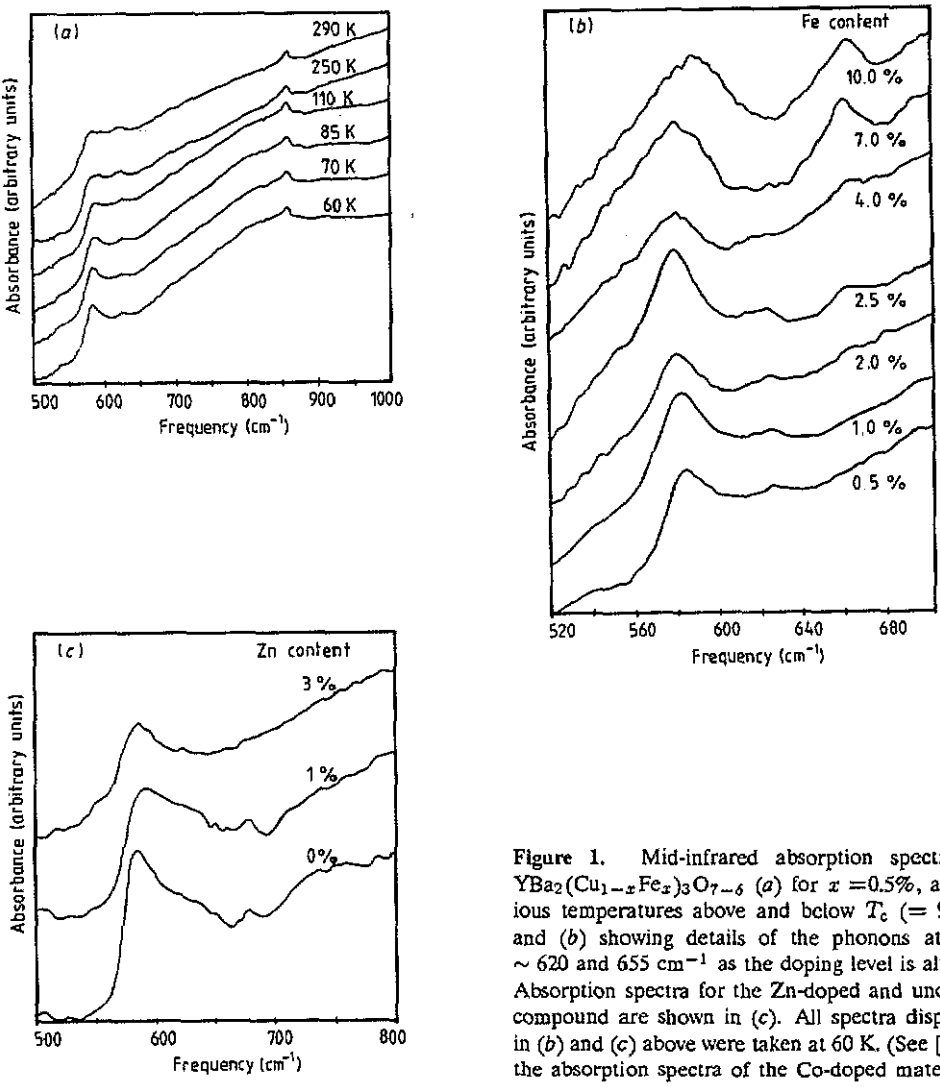

Figure 1. Mid-infrared absorption spectra of $YBa_2(Cu_{1-x}Fe_x)_3O_{7-\delta}$ (*a*) for $x = 0.5\%$, at various temperatures above and below T_c (= 91 K) and (*b*) showing details of the phonons at 580, ~ 620 and 655 cm^{-1} as the doping level is altered. Absorption spectra for the Zn-doped and undoped compound are shown in (*c*). All spectra displayed in (*b*) and (*c*) above were taken at 60 K. (See [4] for the absorption spectra of the Co-doped material).

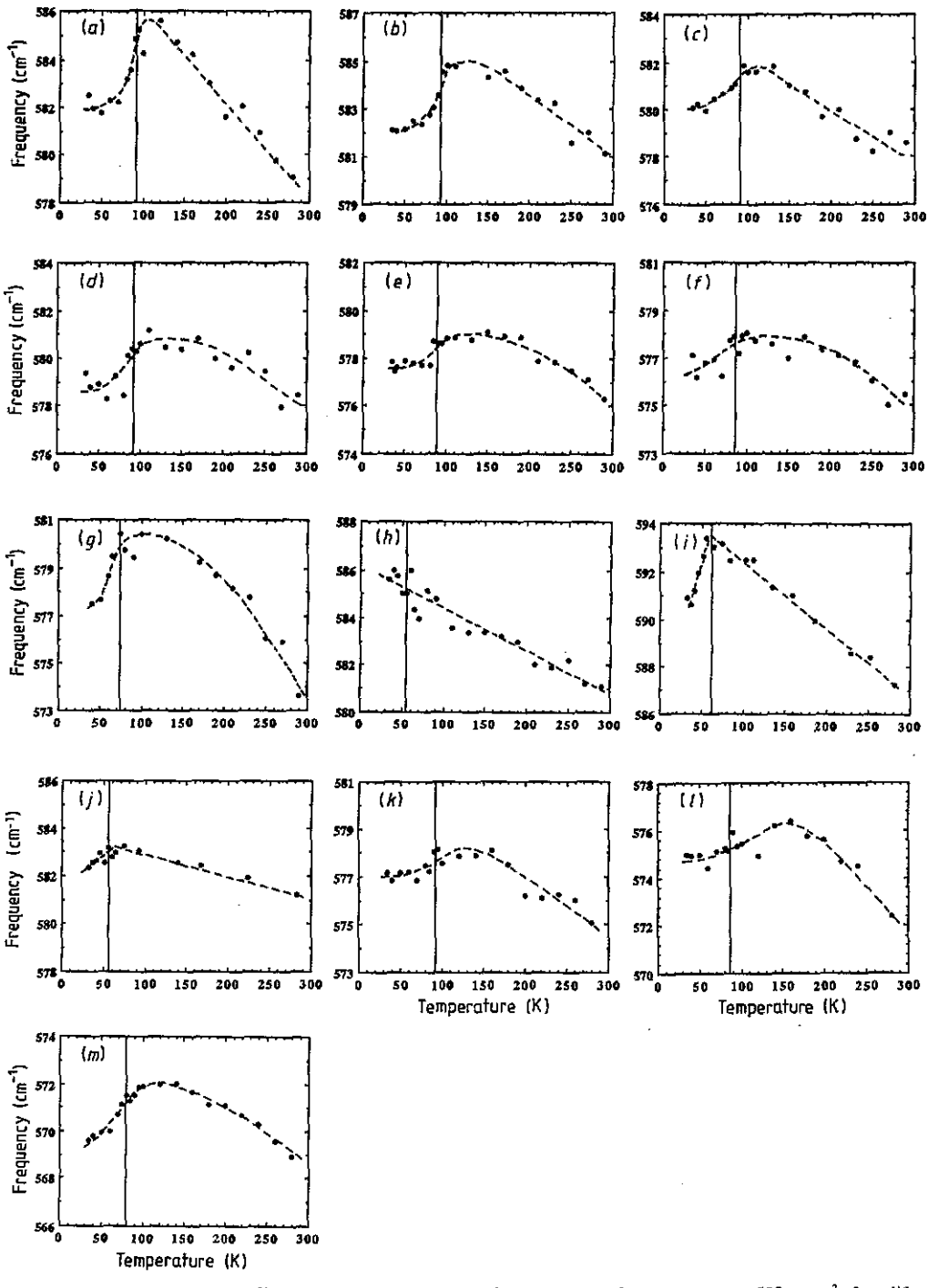

Figure 2. Temperature dependence of the phonon frequency near 580 cm^{-1} for different concentrations of Fe, Zn and Co—dashed lines are guides for the eye. Doping concentrations are as follows. Undoped (*a*); Fe doped: (*b*) 0.5%, (*c*) 1.0%, (*d*) 2.0%, (*e*) 2.5%, (*f*) 4.0%, (*g*) 7.0%, (*h*) 10.0%; Zn doped: (*i*) 1.0%, (*j*) 3.0%; and Co doped: (*k*) 1.5%, (*l*) 2.8% and (*m*) 4.0%. The vertical lines indicate T_c as obtained from resistance and magnetic susceptibility measurements (see text for details).

The temperature dependence of the mode frequency for the phonon near 580 cm^{-1} (figure 2) displays the anomalous behaviour reported previously [4–10] and can be characterized by three main features. Firstly, at temperatures far above the onset of superconductivity, one observes a hardening of the mode with $d\bar{\nu}/dT \gtrsim -3.9 \times 10^{-2}$ cm^{-1} K^{-1}. This hardening at $T \gg T_c$ is attributed to the volume–strain behaviour of the weakly anharmonic crystal and can be formally described by the Grüneisen relationship (see [4]),

$$\gamma_i = -\partial(\ln\omega_i)/\partial(\ln V)$$

for the ith mode, with V the molar volume. We find that the value of the mode Grüneisen parameter γ_i is comparable for all samples ($\sim$ 2.0) indicating that all M-substituted structures exhibit similar bulk volume–strain behaviour at temperatures well above T_c.

Secondly, at temperatures below T_c a strong phonon anomaly is observed which is not correlated with an equivalent anomaly in the cell volume and thus cannot be associated with the weakly anharmonic lattice. It has been demonstrated that this phonon softening is *directly correlated* with the order parameter in the superconducting state [4, 5, 29]. The mode softening below T_c is unobserved in $YBa_2(Cu_{1-x}M_x)_3O_{7-\delta}$ samples with $x \gtrsim 10.0\%$ Fe, $x \gtrsim 7.0\%$ Co, and $x \gtrsim 5.0\%$ Zn, as the critical temperature for these compounds is outside the low-temperature limit of our apparatus. The change in phonon frequency with decreasing temperature is a maximum at ~ 5 cm^{-1} in undoped $YBa_2Cu_3O_{7-\delta}$ and is just resolvable at ~ 1 cm^{-1} in the 3.0% Zn-doped compound.

The third feature is a phonon softening that begins at temperatures well above T_c in the Fe- and Co-doped compounds but is *not seen* in the undoped and Zn-doped compounds. This is most apparent in the 2.8% Co and 2.5% Fe samples where the phonon mode starts to soften at $\sim 2T_c$ (figure 2). In contrast, the Zn-doped and undoped samples show sharp frequency changes with temperature at T_c.

The temperature dependence of the intensity of this phonon mode (see figure 1(*a*)) has been analysed in detail by Güttler *et al* [5].

TEM analysis showed that the crystal microstructure of the Zn-doped material consists of a lamellar twin configuration with average spacing of $\sim$ 40 nm. The twin domain walls are oriented along the form {110} and generally the microstructure is very similar to that of undoped $YBa_2Cu_3O_{7-\delta}$ [20, 28]. As expected 'tweed' microtwinning was not evident since its occurrence is linked with the orthorhombic–tetragonal structural phase transition in Co and Fe doped $YBa_2Cu_3O_{7-\delta}$.

4. Discussion

A consideration of polarized infrared reflection measurements or oriented thin films [30, 31], single crystals of the tetragonal $\delta = 1.0$ phase [32] and polycrystalline infrared measurements [4, 33] leads to the following phonon assignment. In the orthorhombic phase the mode near 580 cm^{-1} is attributed to a Cu(1)–O(4) vibration of B_{1u} symmetry with electric dipole moment oriented along the c direction. In the tetragonal phase a corresponding $E//c$ A_{2u} symmetry Cu(1)–O(4) mode becomes active in the 655 cm^{-1} region. This is not seen in the Zn-doped compounds since they do not exhibit an orthorhombic–tetragonal structural phase transition with increasing x. In many samples a small peak at $\sim$ 620 cm^{-1} is just discernible. This

we attribute to Cu(2)–O(2),O(3) B_{2u}, B_{3u} symmetry vibrations. These modes are not intense probably as a result of the anisotropy in $YBa_2Cu_3O_{7-\delta}$ which has the effect of screening out all modes bar the $E//c$ B_{1u} species [30].

As explained in the above introduction, phonon absorption of infrared radiation measures the thermodynamic order parameter associated with the superconductivity on a spatially local scale. The precursor mode softening indicates that fluctuating droplets or clusters of superconducting material can exist above the critical temperature at which the macroscopic order parameter disappears, i.e. T_c. We have found that no such fluctuations exist in pure $YBa_2Cu_3O_{7-\delta}$ and the Zn-doped compound; for these materials the phase transition follows essentially mean-field behaviour. Conversely, our experiments show that precursor effects *do* occur in Co- and Fe-doped $YBa_2Cu_3O_{7-\delta}$. The relevant temperature interval for fluctuations at $T > T_c$ is first estimated using thermodynamic arguments. For this purpose we consider the amplitude function of the order parameter Q and it is sufficient to concentrate on the Hamiltonian;

$$\mathcal{H} = \tfrac{1}{2}A(T - T_c)Q^2 + \tfrac{1}{4}BQ^4 + \tfrac{1}{2}g(\nabla Q)^2. \tag{1}$$

Quantum saturations at $T < T_c$ are ignored at this point [34]. The partition function is given by

$$Z = \int \exp\left(-\beta \int \mathcal{H}\, \mathrm{d}^d r\right) D(Q) \tag{2}$$

where d is the dimension of the space. We can rescale Q and r via

$$q = Q\left(2A(T-T_c)/B\right)^{-1} \qquad u = r\left(g/A(T-T_c)\right)^{-1/2}$$

and obtain the rescaled partition function

$$Z = \int \exp\left\{-\mathrm{g} \int \mathrm{d}u\, \left[(\nabla_u q)^2 + q^2 + q^4\right]\right\} D(Q)$$

with the Ginzburg number [35] defined by

$$\mathrm{g} \equiv (4\beta/B)\left[\tfrac{1}{2}A(T - T_c)\right]^{2-d/2}\left(\tfrac{1}{2}g\right)^{d/2}.$$

Fluctuations do not occur for $\mathrm{g} \to \infty$ in the thermodynamic limit. At $T \approx T_c$ we find the classical upper critical dimension $d_c = 4$. This dimension is obviously changed if we change the Hamiltonian in (1). A typical example of such a change is to a tricritical Hamiltonian with a $\frac{1}{6}CQ^6$ term replacing the $\frac{1}{4}BQ^4$. Using the same line of argument we find $d_c = 3$, indicating that fluctuations are non-critical in this case (ignoring logarithmic corrections). The same result $d_c = 3$ applies if long-range elastic forces are coupled with the order parameter [36]. Salje [29] used the argument that such coupling with the phonon bath might be the physical reason for the apparent mean-field behaviour of $YBa_2Cu_3O_{7-\delta}$.

We now ask what happens when T_c is lowered due to doping. The Ginzburg number now has to be calculated in the quantum limit ($T_c \to 0$ K, [37]). The critical dimension is then reduced because the consideration of the kinetic energy leads an additional integration over time in the partition function (2). As a result we find

that $d_c^{T_c \to 0} = d_c - 1$ for the spherical model [35]. This means that the fluctuations decrease rapidly with decreasing critical temperature.

Let us now consider the role of dopants. Our model Hamiltonian is certainly inappropriate for any quantitative estimates but we can arrive at some conjecture if we concentrate on two aspects of doping. Firstly, Co and Fe replace Cu(1) in the interlayer sites [9, 20–26]. Structural data show that the most sensitive reaction of the crystal structure to doping is related to the apical oxygen, O(4). The importance of this oxygen for the occurrence of superconductivity, in particular its anharmonic vibrations, was emphasised by Müller [19] and is the crucial part of the apex-oxygen model by Frick *et al* [18]. In this model the standard Emery Hamiltonian [38] is coupled to the displacement of O(4) in an anharmonic potential. The new entity can then be analysed in a manner similar to a 'structural polaron' [39] in which the structural relaxation forms an essential part of the condensation process. Doping changes the local O(4) potential and hence changes the transition temperature i.e. T_c decreases with increasing doping. The second effect of doping is to change the character of the coupling between the CuO_2 layers. In the case of Co and Fe doping, it would be reasonable to expect a decoupling effect and thus, an effective reduction in the overall dimensionality of the phase transition. In the case of Zn doping, T_c reduces dramatically although the 3D character of the phase transition appears to remain unchanged.

We now focus our attention on the fluctuation effects observed above T_c. No fluctuations occur in the Zn-doped and undoped samples and in these cases a classical Landau–Ginzburg analysis is appropriate. If we follow the idea that the role of Fe or Co doping serves to decouple the CuO_2 planes, it is expected that increasing the level of doping will lower T_c and extend the Ginzburg interval. As T_c hardly changes for $x \lesssim 2.5\%$ (the 'plateau' region in the T_c versus x curve) we anticipate that the temperature interval for fluctuations will be largest near the edge of the plateau i.e. as $x \to \approx 2.5\%$. At higher doping levels, $T_c \to 0$ K with increasing x in an approximately linear fashion and consequently $d_c^{T_c \to 0} \to d_c - 1$. Fluctuations are thus suppressed and the phase transition follows Landau–Ginzburg (mean-field) behaviour once again. The corresponding 'phase-diagram' is sketched in figure 3(*a*). The shaded area indicates the fluctuations—the formation of superconducting clusters at temperatures well above T_c.

This analysis is in contrast to the finite-scaling argument, in which the results of an x–y model are analysed in $3 - \epsilon$ dimensions. Critical fluctuations are always present in this model and increase as $\epsilon \to 1$. At $\epsilon = 1$, a Kosterlitz–Thouless (KT) transition [40] occurs with strong short-range order at $T > T_c$. Note that the KT transition temperature (T_{KT}) is finite whereas doping experiments show that $T_c \to 0$ K as x is increased in $YBa_2(Cu_{1-x}M_x)_3O_{7-\delta}$. The 'phase diagram' in this case is shown in figure 3(*b*).

An examination of figure 2 for the larger doping levels shows that our experiments support a diagram of the type 3(*a*), *not* of the type 3(*b*). This result obviously hinges on the idea that doping does indeed cause inter-layer decoupling. On the other hand, investigations of the properties of layered $YBa_2Cu_3O_{7-\delta}/PrBa_2Cu_3O_{7-\delta}$ super-lattice structures [14–16], seem to support the idea of a finite T_{KT} for a single $YBa_2Cu_3O_{7-\delta}$ layer. However, there may be some doubts about this conclusion, because the fact that only one double CuO_2 layer exists in a $PrBa_2Cu_3O_{7-\delta}$ matrix does not mean that the wavefunction is strictly two-dimensional. Only small extensions in the third dimension are enough to immediately increase T_c to a finite value. Unfortunately,

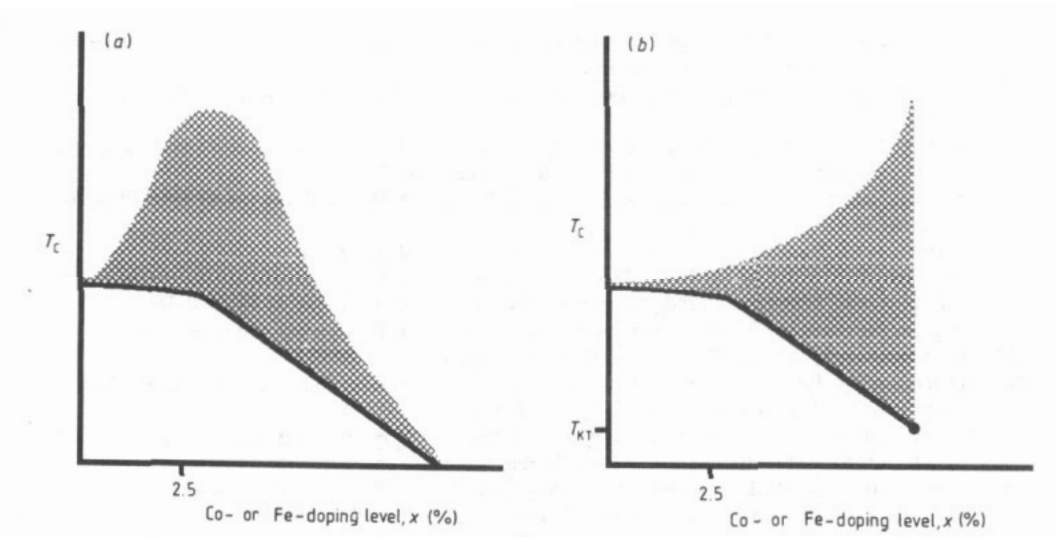

Figure 3. 'Phase diagram' of Co- or Fe-doped $YBa_2Cu_3O_{7-\delta}$. The cross-hatched area represents the region in which superconducting fluctuations are effective.

the only information on the superconducting fluctuations at $T > T_c$ in these layered structures comes from the width of the resistive transition observed in transport measurements (e.g. [15]).

Further work on high-T_c materials is under way in order that more understanding be gained on the nature of the superconducting fluctuations above T_c.

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