Complete elastic moduli of La$_{2-x}$Sr$_x$CuO$_4$ ($x =0.00$ and 0.14) near the tetragonal-orthorhombic structural phase transition

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We present resonant ultrasound spectroscopy measurements of the complete elastic moduli of La$_{2-x}$Sr$_x$CuO$_4$ ($x=0.00$ and 0.14). The x dependence of both the absolute values of the moduli in the tetragonal phase and the temperature dependences of the moduli near the structural phase transition is weak. In both materials c_{44} and $\frac{1}{2}(c_{11}-c_{12})$ are unaffected by the transition, c_{33} shows a steplike drop broadened by fluctuations, and c_{11} and c_{66} decrease as $1/(T-T_{SPT})$ for $T>T_{SPT}$ (where T_{SPT} is the structural phase transition temperature). We also present measurements of the anisotropic thermal expansion coefficients in La$_{1.86}$Sr$_{0.14}$CuO$_4$ using a high-resolution capacitance dilatometer. These data when taken together raise serious questions about the standard interpretation of the structural phase transition in La$_{2-x}$Sr$_x$CuO$_4$.

La$_{2-x}$M$_x$CuO$_4$ ($M =$Sr, Ba, or Ca) has attracted much attention in recent years. The system's phase diagram is extremely rich.[1,2] The stoichiometric compound is an insulating antiferromagnet, but when doped appropriately, the material becomes superconducting with a maximum transition temperature near 40 K. Of the doped compounds La$_{2-x}$Sr$_x$CuO$_4$ has the simplest structure, undergoing only a single structural phase transition (SPT) from tetragonal to orthorhombic crystal symmetry at a temperature (T_{SPT}) which depends strongly on the amount of Sr present.[3]

Above the SPT La$_{2-x}$Sr$_x$CuO$_4$ has the body-centered tetragonal K$_2$NiF$_4$ structure.[4] The tetragonal-orthorhombic transition has been interpreted qualitatively as being driven by a zone-edge soft-phonon mode corresponding to the tilting of the structure's oxygen octahedra in the [1,1,0] or [1, −1,0] directions.[5] The transition occurs near 525 K for the stoichiometric compound and decreases rapidly with increasing Sr concentration ($dT_{SPT}/dx \cong -2500$ K).[6]

We present here resonant ultrasound spectroscopy (RUS) measurements of the complete elastic moduli of La$_{2-x}$Sr$_x$CuO$_4$ ($x =0.00$ and $x \cong 0.14$) as a function of temperature, the only such determinations for single-crystal samples. RUS (Ref. 7) is a technique for determining elastic moduli, particularly appropriate for small rectangular-parallelepiped single crystals. Rather than measuring the transit time of traveling waves (as in pulse-echo techniques), we measure a body's mechanical

resonances (standing waves). Because a body's density, dimensions, and elastic moduli determine its mechanical resonances,[8] the measurement of a sufficient number of its resonance frequencies permits, through an inverse calculation, the determination of its elastic moduli. This technique has the advantage of determining all elastic moduli simultaneously with high accuracy, using low-measurement frequencies (0.5–3 MHz), and requiring maximum sample dimensions of only 1 mm or less. The details of both the measurement and data analysis procedures are discussed at length in Ref. 7.

For this study we have focused on two carefully prepared and characterized rectangular-parallelepiped samples: a nearly optimally doped sample (i.e., one giving the highest superconducting transition temperature) and a stoichiometric one. We have also measured the resonance frequencies as a function of temperature of several unoriented fragments of La$_{2-x}$Sr$_x$CuO$_4$ ($x =0.10$ and 0.18) in an attempt to establish the universality as a function of Sr concentration of the temperature-dependent effects we have observed.[9]

The La$_{1.86}$Sr$_{0.14}$CuO$_4$ crystal was grown by the traveling solvent floating-zone technique.[10] Magnetization measurements on this sample indicate bulk superconductivity. The crystal displayed full shielding diamagnetism at 4 K in a 10-G field and an onset temperature for superconductivity of 36 K in a field of 20 G, with a Meissner fraction of 6% for the c axis parallel to the field.[11] When combined with the observed sharpness of the SPT, mea-

TABLE I. Tetragonal elastic moduli of $La_{1.86}Sr_{0.14}CuO_4$ and La_2CuO_4. Elastic moduli are in units of 10^{12} dynes/cm^2.

T (K)	Compound	c_{11}	c_{33}	c_{23}	c_{12}	c_{44}	c_{66}
300	$La_{1.86}Sr_{0.14}CuO_4$	2.67	2.57	0.99	0.65	0.68	0.59
580	La_2CuO_4	2.45	2.48	0.95	0.61	0.63	0.52

sured ultrasonically, these data also indicate good sample homogeneity. The Sr concentration was determined by measuring the structural phase-transition temperature with RUS and deducing the Sr concentration from the known phase diagram. Thus, modifications of the phase diagram would lead to adjustments in the estimation of Sr concentration.

The La_2CuO_4 crystal was pulled from a CuO flux using the Kyropolous method and a Pt seed.[12] This particular growth technique was developed specifically for this study in an attempt to produce crystals which are untwinned in the orthorhombic phase. Magnetic susceptibility and electrical resistance measurements on smaller pieces of the same crystal have been made to verify its quality.[12]

The elastic moduli determined for each crystal in its tetragonal phase are shown in Table I. The La_2CuO_4 values are about 10% lower than those of $La_{1.86}Sr_{0.14}CuO_4$ because of the higher temperature at which they were obtained (a softening of 3% for every 100 K of temperature increase is quite typical). These data provide the first indication that Sr is a rather weak dopant in terms of ultrasonic properties. This result is not too surprising: the ionic radius of Sr is also sufficiently close to that of La that only one SPT is observed in this system as opposed to the two SPT's that are observed when La_2CuO_4 is doped with barium, a much larger atom.[13]

At 300 K, La_2CuO_4 is orthorhombic and, due to the doubly degenerate soft phonon which is presumed to drive the SPT, usually twinned. The high Q's measured for our sample with RUS (where Q is the quality factor of a mechanical resonance, i.e., one of the normal modes of vibration of the sample, and is defined as the center frequency of the resonance divided by its full width at half maximum), polarized light examinations, and low-stress growth conditions all support the hypothesis that our crystal is untwinned at room temperature. The fitted elastic moduli for our untwinned crystal, as well as several previous measurements, are shown in Table II. Burma *et al.* measured twinned crystals and thus could
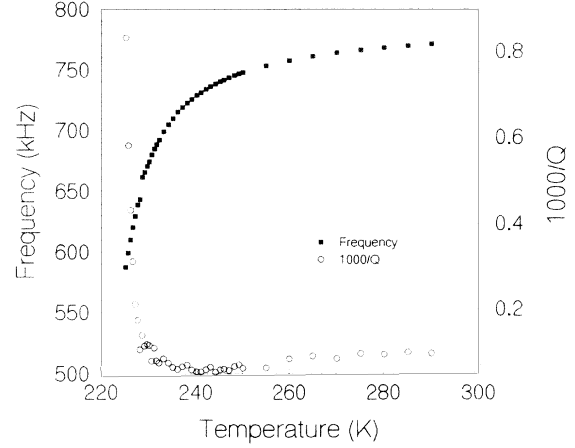

FIG. 1. Temperature dependence of the resonance frequency and the inverse of the quality factor (proportional to ultrasonic attenuation) for a vibrational mode in $La_{1.86}Sr_{0.14}CuO_4$ which depends mostly on c_{66}.

only determine effective tetragonal moduli.[14] They also had to introduce a monoclinic distortion to reconcile self-consistently all their measured sound velocities, which may explain the large disagreement for some moduli. The data of Migliori *et al.* are an earlier RUS determination of the moduli using a different crystal of La_2CuO_4.[15]

We have also measured the elastic moduli of both crystals as a function of temperature near their respective SPT's. The temperature dependences of the resonance frequencies for our $La_{1.86}Sr_{0.14}CuO_4$ crystal fall into three general classes. A representative resonance frequency from each of these classes is shown in Figs. 1–3. In all cases we observe a rise in ultrasonic attenuation within 5–10 K of the transition temperature ($T_{SPT} \cong 220$ K). The behavior of the frequencies, on the other hand, is quite different. Some modes show softening over the entire temperature range (Fig. 1), while others only soften very near the transition (with the decrease beginning at the same temperature that the attenuation begins to rise, Fig. 2) or are unaffected by the transition (Fig. 3). A large softening over a wide temperature range has also been observed by several other groups using either vibrating reed or pulse-echo techniques.[16,17] In previous RUS work it was established that this softening arises from changes in c_{66}, one of the in-plane shear moduli, and that this

TABLE II. Elastic moduli of orthorhombic La_2CuO_4 at room temperature: comparison between literature and this work. All values are in 10^{12} dynes/cm^2.

c_{11}	c_{22}	c_{33}	c_{23}	c_{13}	c_{12}	c_{44}	c_{55}	c_{66}	Ref.
1.72	1.71	2.00	0.73	0.73	0.90	0.65	0.66	0.97	15
	2.23[a]	2.72		0.96[a]	1.32		0.68[a]	1.05	14
1.75	1.73	2.65	0.98	0.90	0.90	0.66	0.67	0.99	This work

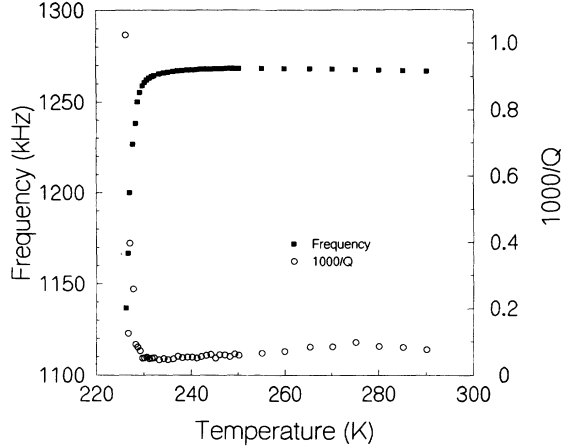[a]Measurement on twinned crystals. Only effective tetragonal moduli were found.

FIG. 2. Temperature dependence of the resonance frequency and the inverse of the quality factor for a vibrational mode in La$_{1.86}$Sr$_{0.14}$CuO$_4$ which depends mostly on c_{33}.

FIG. 4. Temperature dependence of the three principal shear moduli in La$_{1.86}$Sr$_{0.14}$CuO$_4$. The solid line is the fitted Curie-Weiss behavior: $c_{66}(T)=61.5-203/(T-218)$.

modulus has a Curie-Weiss temperature dependence [i.e., varies with temperature as $1/(T-T_{SPT})$ for $T > T_{SPT}$].[18] The elastic moduli calculated from our resonance frequencies are shown in Figs. 4 and 5. Consistent with the resonance frequency data, three characteristic behaviors are observed: $\frac{1}{2}(c_{11}-c_{12})$, a pure shear modulus, and c_{44} are essentially independent of temperature, c_{33} shows a steplike drop near T_{SPT} broadened by several degrees, and c_{11} and c_{66} exhibit a Curie-Weiss temperature dependence.

Unfortunately, the scatter in the fitted moduli and the consequent error bars become large near the phase transition. This is due to an unknown symmetry-breaking effect displayed by our crystal. The effect is shown in Fig. 6. The resonance frequencies of two distinct normal modes of vibration, one varying rapidly with temperature and one essentially constant, cross. Although these vibrational modes are in different symmetry classes and,
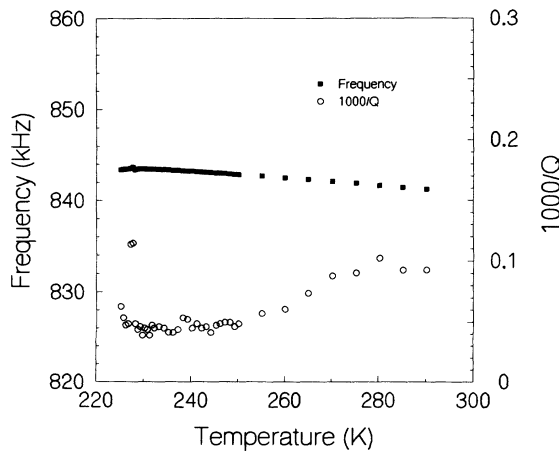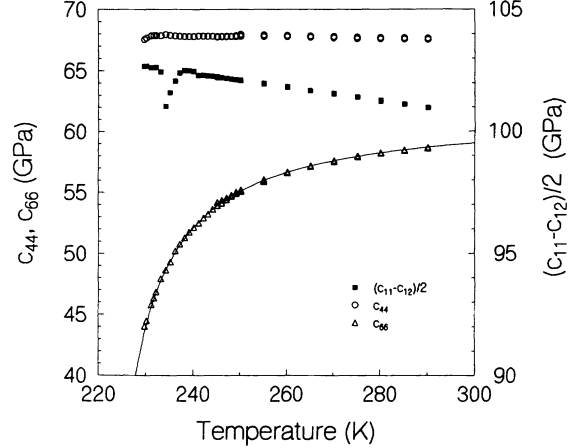
thus, should be orthogonal,[7] they repel each other, indicating that some symmetry (either microscopic or macroscopic) in the crystal is broken. Such avoided crossings are observed, with varying magnitude, for many modes in our crystal. The size of the effect is not amplitude dependent and thus cannot be attributed to a simple anharmonicity. While the avoided crossings may be caused by a dopant-induced symmetry breaking, our inability to reproduce it in other doped systems argues for an extrinsic effect. Whatever its source, the avoided crossings represent an effect beyond the scope of our mathematical model for a resonating solid, producing progressively larger scatter in the fitted moduli as the transition temperature is approached and the number of crossings increases.

The experimental situation is much cleaner for our stoichiometric La$_2$CuO$_4$ crystal. No avoided crossings are observed, and a sufficient number of resonance fre-
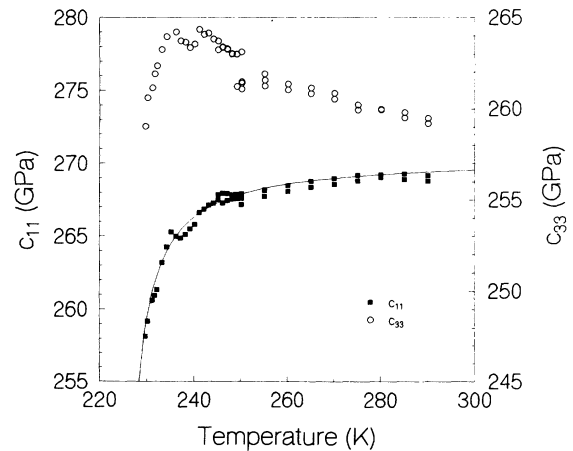

FIG. 3. Temperature dependence of the resonance frequency and the inverse of the quality factor for a vibrational mode in La$_{1.86}$Sr$_{0.14}$CuO$_4$ which depends mostly on c_{44}.

FIG. 5. Temperature dependence of the two principal compressional moduli in La$_{1.86}$Sr$_{0.14}$CuO$_4$. The solid line is the fitted Curie-Weiss behavior: $c_{11}(T)=270.4-65.5/(T-224)$.
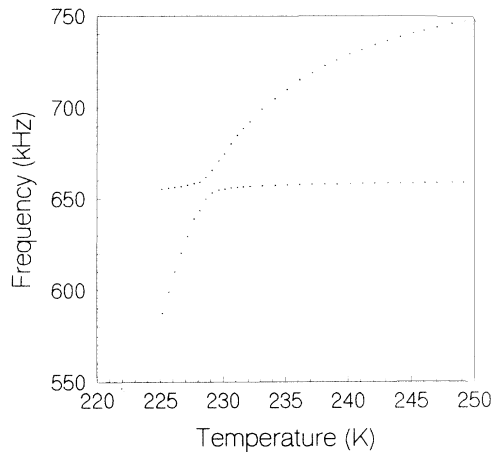
FIG. 6. Temperature dependence of two resonance frequencies near the structural phase transition in $La_{1.86}Sr_{0.14}CuO_4$. In our model, the vibrational modes are orthogonal. The interaction and resulting avoided crossing represent an unexplained symmetry breaking in our crystal.

FIG. 8. Temperature dependence of the two principal compressional moduli in La_2CuO_4. The solid line is the fitted Curie-Weiss behavior: $c_{11}(T) = 246 - 146/(T - 520)$.

quencies can be tracked to permit determination of the complete set of elastic moduli to within several degrees of the transition. These moduli are shown in Figs. 7 and 8. Similar to our observations in $La_{1.86}Sr_{0.14}CuO_4$, three characteristic behaviors are observed: $\frac{1}{2}(c_{11} - c_{12})$ and c_{44} are essentially independent of temperature, c_{33} shows a steplike drop, and c_{11} and c_{66} exhibit a Curie-Weiss temperature dependence. An increase in ultrasonic attenuation is also observed near the phase transition (the temperature dependence for a representative mode is shown in Fig. 9). The temperature range over which c_{33} falls and attenuation rises coincide and, when expressed as $\Delta T/T_{SPT}$, is the same (approximately 5%) as that observed for the Sr-doped sample. Thus, aside from strongly shifting the transition temperature, the presence of strontium does not significantly affect the ultrasonic properties of $La_{2-x}Sr_xCuO_4$. Measurements of resonance

frequency as a function of temperature for unoriented single-crystal fragments with other Sr concentrations confirm the universality of the observed large modulus-specific softening.[9]

The SPT in $La_{2-x}Sr_xCuO_4$ is generally understood as resulting from a zone-edge soft-photon mode corresponding to the rigid tilting of oxygen octahedra. Using a Ginsburg-Landau analysis with the octahedral tilt angles as the order parameters, Axe[19] has explained not only the tetragonal-to-orthorhombic transition in $La_{2-x}Sr_xCuO_4$ but also the subsequent orthorhombic-to-low-temperature-tetragonal transition in $La_{2-x}Ba_xCuO_4$. In order to study the effect of the SPT on elastic moduli, the Ginsburg-Landau free energy must be generalized to include the elastic energy for a tetragonal solid and the proper strain-order-parameter coupling. This expression
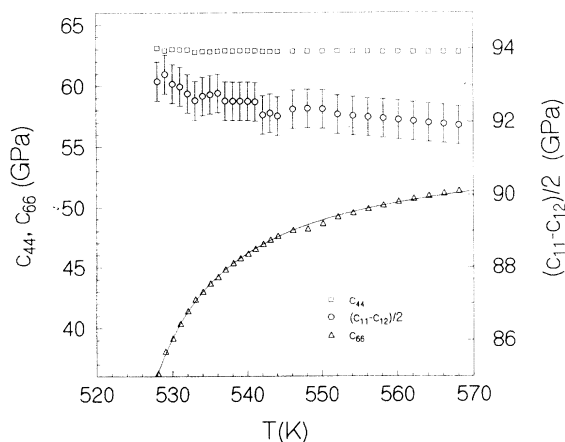

FIG. 7. Temperature dependence of the three principal shear moduli in La_2CuO_4. The solid line is the fitted Curie-Weiss behavior: $c_{66}(T) = 55.2 - 207/(T - 517)$.
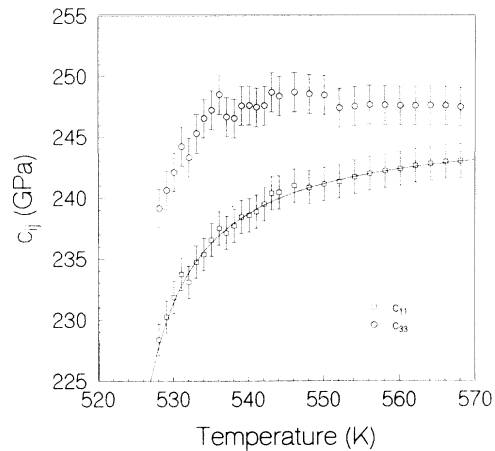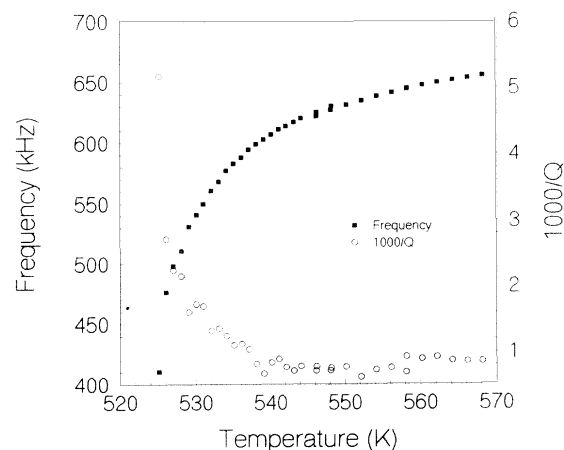

FIG. 9. Temperature dependence of the resonance frequency and the inverse of the quality factor for a representative vibrational mode in La_2CuO_4. The inverse of the quality factor is proportional to ultrasonic attenuation.

for the free energy can be written[20-23]

$$F = F_{GL} + F_e + F_{c'}$$

with

$$F_{GL} = a(\eta_1^2 + \eta_2^2) + u(\eta_1^2 + \eta_2^2)^2 + v(\eta_1^4 + \eta_2^4) ,$$

$$F_e = \tfrac{1}{2}c_{11}(\varepsilon_1^2 + \varepsilon_2^2) + c_{12}\varepsilon_1\varepsilon_2 + c_{13}(\varepsilon_1 + \varepsilon_2)\varepsilon_3$$
$$+ \tfrac{1}{2}c_{33}\varepsilon_3^2 + \tfrac{1}{2}c_{44}(\varepsilon_4^2 + \varepsilon_5^2) + \tfrac{1}{2}c_{66}\varepsilon_6^2 ,$$

and

$$F_c = [k_{xx}(\varepsilon_1 + \varepsilon_2) + k_{zz}\varepsilon_3](\eta_1^2 + \eta_2^2) + k_{xy}\varepsilon_6(\eta_1^2 - \eta_2^2) , \quad (1)$$

where F_{GL} is the usual Ginsburg-Landau expansion for a two-component order parameter where η_1 and η_2 correspond to octhadral tilts in the [1,1,0] and [1,−1,0] directions, respectively, F_e is the elastic energy for a tetragonal solid, and F_c is the coupling between elastic strains ε_i and order parameters η_i. This free energy predicts step-like changes in the elastic moduli at the SPT:[24]

$$\Delta c_{11} = -\frac{k_{xx}^2}{2(u+v)} ,$$

$$\Delta c_{12} = -\frac{k_{xx}^2}{2(u+v)} ,$$

$$\Delta c_{66} = -\frac{k_{xy}^2}{2(u+v)}$$

$$\Delta c_{33} = -\frac{k_{zz}^2}{2(u+v)} , \quad (2)$$

$$\Delta c_{13} = -\frac{k_{xx}k_{zz}}{2(u+v)} ,$$

$$\Delta c_{44} = 0 .$$

The predictions agree well with our data for c_{33}, c_{44}, and $\tfrac{1}{2}(c_{11} - c_{12})$. c_{44} and $\tfrac{1}{2}(c_{11} - c_{12})$ are essentially unchanged near the transition (i.e., $\Delta c_{44} = 0$ and $\Delta c_{11} = \Delta c_{12}$). Although the change in c_{33} is somewhat rounded rather than steplike, this rounding, which occurs over the same range in temperature as the rise in attenuation, is attributable to thermal fluctuations and is consistent with the transition widths observed in other perovskite materials.[24] Fluctuations of the order parameter, which corresponds physically to the tilting of oxygen octahedra, give rise to a nonzero mean-squared displacement of the octahedra which produce modulus softening and ultrasonic attenuation in the tetragonal phase. The temperature dependences of c_{11} and c_{66}, however, cannot be explained with this free energy.

Our previous attempts to explain the large Curie-Weiss softening of c_{66} in the Sr-doped crystal relied on either a large fluctuation region[18] or dopant-induced symmetry breaking.[24] In the two-dimensional Gaussian approximation[25] the elastic moduli are expected to show Curie-Weiss softening in the fluctuation regime. Thus, it was argued that the compound's layered structure gave rise to two-dimensional fluctuations over a very wide temperature range (of order 100 K). The source of the large tem-

perature range for c_{66} softening and the much smaller temperature range over which attenuation increased was not understood. The observation of mode repulsion in our Sr-doped crystal gave rise to an alternative interpretation. Mode repulsion is a signature of a possibly intrinsic symmetry breaking. This symmetry breaking, the result of a random-local-field dopant,[26] destroys short-range translational invariance and modifies the allowed strain-order parameter couplings at the zone edge. If this were coupled to the zone center, a linear coupling between strain and order parameter [i.e., $F(\eta,\varepsilon) \propto \eta\varepsilon$] in the Ginzburg-Landau free energy would result, which would naturally give rise to Curie-Weiss softening.[27] Our subsequent measurements on stoichiometric La$_2$CuO$_4$ do not support such an interpretation. In the absence of doping and avoided crossings, the Curie-Weiss softening persists, and the temperature range over which c_{33} softens and attenuation increases is a much more reasonable estimate of the fluctuation region. The temperature range over which fluctuations are expected to contribute can be estimated with the Ginzburg criterion.[25] Using a specific-heat jump at the SPT of 0.03 J/g K (Ref. 28) and taking the coherence length to be a few angstroms, the size of an octahedron, gives a fluctuation range of order 0.1 K, not the 100 K needed to account for c_{66} softening.

In order to further investigate the distinction between in-plane and out-of-plane elastic moduli, we have also measured the in-plane, $\alpha_{\|}$, and out-of-plane, $\alpha_{\perp}$, thermal expansion coefficients of our La$_{1.86}$Sr$_{0.14}$CuO$_4$ single crystal using a high-resolution capacitance dilatometer. These data are shown in Fig. 10 and are in good agreement with those of Braden et al.[29] Similar to the in-plane, c_{11}, and out-of-plane, c_{33}, compressional elastic moduli, $\alpha_{\|}$ varies over a wide temperature range while $\alpha_{\perp}$ is only affected near the SPT, emphasizing the distinction between in-plane and out-of-plane behavior in La$_{2-x}$Sr$_x$CuO$_4$.

To understand this distinction, we must examine the physical details of the structural phase transition more carefully. Consider a single plane of Cu-O octahedra in a
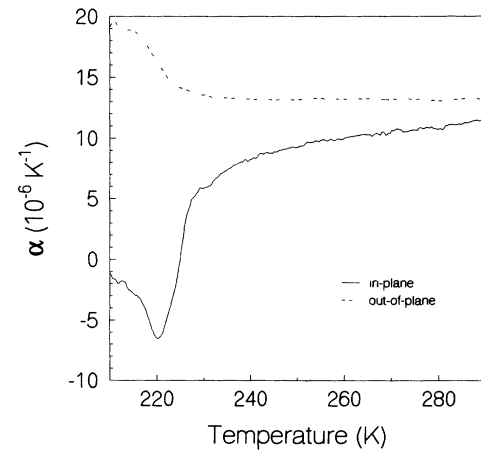

FIG. 10. Temperature dependence of the in-plane, $\alpha_{\|}$, and out-of-plane, $\alpha_{\perp}$, coefficients of thermal expansion for La$_{1.86}$Sr$_{0.14}$CuO$_4$.

single domain so that all the octhadral tilts will be in the same crystallographic direction (i.e., $\eta_1 \neq 0$, $\eta_2 = 0$). At the phase transition the octahedra tilt, producing a corrugated structure. If the octahedra were rigid, as we have assumed thus far, the crystal should contract in the [1,1,0] direction due to the coupled tilts. Experimentally, however, the crystal expands. Elastic neuron-diffraction experiments on powder samples[30] have shown that, in addition to rigidly tilting, the four in-plane oxygens scissor about the Cu, transforming a square in the plane into a rectangle while leaving the individual Cu-O bond lengths unchanged. The scissoring angle is approximately 10% of the tilt angle. This effect has been verified with single-crystal measurements.[31] While the tilting doubles the unit cell because neighboring octahedra are corner-sharing and tilt in opposite directions, the scissoring stretches each octahedra in the same way. Thus, the distortion of the crystal at the structural phase transition combines a zone-edge octahedral tilt with a much smaller zone-center deformation of the octahedra.[32,33] While it is not possible to formulate rigorously a model of the transition which incorporates these effects while still maintaining the full group symmetry of the crystal, we believe this zone-center distortion is central to the complete understanding of the elastic properties of $La_{2-x}Sr_xCuO_4$.

In summary, we have presented a determination of the complete elastic moduli of $La_{1.86}Sr_{0.14}CuO_4$ and La_2CuO_4 as a function of temperature near the tetragonal-orthorhombic structural phase transition. From these data we conclude that Sr acts as a weak dopant and does not produce significant changes in the ultrasonic properties of the solid. Both the magnitude of the elastic moduli and their temperature dependence are essentially unchanged by the presence of strontium. Three distinct behaviors are observed: c_{11} and c_{66} show a large temperature-dependent softening proportional to $1/(T - T_{SPT})$, c_{33} shows a steplike transition near T_{SPT} broadened by fluctuations, and c_{44} and $\frac{1}{2}(c_{11} - c_{12})$ are unchanged by the transition. A similar distinction between in-plane and out-of-plane behavior has also been observed in measurements of the anisotropic coefficients of thermal expansion. We have argued that this distinction may arise from the usually neglected in-plane distortion of the Cu-O octahedra at the SPT. Finally, we speculate that such a distortion may also be significant in the similar and previously not-understood elastic properties of other layered perovskite compounds.[21,22]

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