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Anomalies of attenuation and phase velocity of surface acoustic waves in $YBa_2Cu_3O_{7-\delta}$ thin films in the vicinity of T_c

M. Stenger[1], S. Baumhoff[1,a], J.-Y. Prieur[1,2], J. Joffrin[1], and J.-P. Contour[3]
[1] Laboratoire de Physique des Solides, Université de Paris-Sud, 91405 Orsay Cedex, France
[2] Laboratoire LMDH, Université Pierre et Marie Curie, 4 Place Jussieu, 75252 Paris Cedex 05, France
[3] Unité Mixte de Physique CNRS/THOMSON, Domaine de Corbeville, 91404 Orsay Cedex, France

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Abstract. We present measurements of the attenuation and phase velocity of surface acoustic waves in thin $YBa_2Cu_3O_{7-\delta}$ films as a function of temperature, in magnetic fields up to 3.6 T applied parallel to the *c*-axis of the films. We have observed anomalies in both, the attenuation and the phase velocity in the vicinity of the superconducting critical temperature which do not depend on the magnetic field. Possible origins of these anomalies, observed, to our knowledge, for the first time in $YBa_2Cu_3O_{7-\delta}$ thin films, are discussed and compared to bulk acoustic wave experiments. We present a kind of feedback technique for surface acoustic waves which improves the sensitivity of this type of measurement. The actual sensitivity limits are mentioned.

PACS. 62.40.+i Anelasticity, internal friction, stress relaxation, and mechanical resonances – 73.50.Rb Acoustoelectric and magnetoacoustic effects – 74.25.Ld Mechanical and acoustical properties, elasticity, and ultrasonic attenuation

1 Introduction

Ultrasonic investigations have provided important information about the classical superconductors in the vicinity of and below the superconducting critical temperature T_c. Some examples are the diminution of the acoustic attenuation due to the opening of a gap in the electronic density of states at the superconducting transition (see, *e.g.*, [1]), and the structural phase transition in the A15-type compounds like V_3Si and $NbSe_3$ (see, *e.g.*, [2,3]). For the high T_c-superconductors, most of the acoustic measurements have been performed over large temperature ranges without emphasis on the region around T_c. This is probably due to the difficulty of preparing bulk samples showing good crystalline quality, which are large enough to allow investigation by acoustic methods with sufficient experimental resolution.

A possibility to avoid this difficulty is the use of thin films and surface acoustic waves (SAW). Epitaxial thin films of $YBa_2Cu_3O_{7-\delta}$ are nowadays available with good crystalline quality. Surface acoustic waves permit the collection of information about the elastic properties of thin films. Unfortunately, the propagation of SAWs is determined by the elastic properties of the substrate as well. The properties of the film material influence the characteristics of surface acoustic waves with a weighting factor which depends on the ratio of the thickness h of the film and the wavelength λ of the SAWs. Thus, the experimental sensitivity to changes in the film material using SAW is diminished compared with the use of bulk acoustic waves in bulk samples.

We will present a kind of "feedback"-technique which makes it possible to compensate for this loss of sensitivity when performing SAW measurements. We will also present the first results of velocity and attenuation measurements of SAW's in the vicinity of T_c obtained by using this technique to investigate two thin films of $YBa_2Cu_3O_{7-\delta}$ deposited on $LiNbO_3$-substrates, of good crystalline quality and critical temperatures above 85 K.

2 Sample preparation

The most straightforward way to create surface acoustic waves involves the use of a piezoelectric crystal as a substrate. We have used $1 \times 15 \times 15$ mm^3 Y-cut $LiNbO_3$-crystals to deposit $YBa_2Cu_3O_{7-\delta}$ films by pulsed laser ablation (PLD). The $YBa_2Cu_3O_{7-\delta}$ films have been prepared *in situ* by PLD using a frequency tripled Nd:YAG laser with a pulse length of 5 ns at a repetition rate of 2.5 Hz. The power density used in the present study was 100 MW/cm^2 at a wavelength of 355 nm, the corresponding deposition rate being in the range of 2 Å/s. The deposition has been carried out using a three step process. The first step is the deposition of a $YBa_2Cu_3O_{7-\delta}$ buffer layer with a thickness of 150 Å at a low temperature of 650 °C to prevent diffusion of oxygen and lithium from

[a] e-mail: baumhoff@lps.u-psud.fr

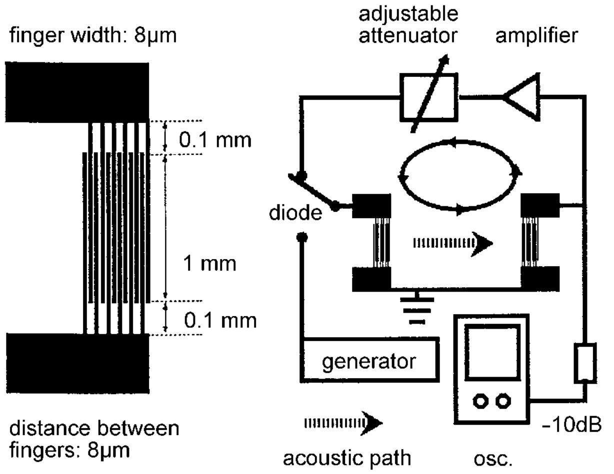

Fig. 1. Geometry of the interdigital transducers used for the generation of SAW's (left) and sketch of the reinjecting electro-acoustic circuit (right). Only a small part (−10 dB) of the electric signal recovered at the right transducer is detected (the detecting circuit is symbolized by the oscilloscope), the rest is amplified and send to the left transducer. The diode switch serves to direct the rf-pulses in the reinjecting circuit. The circulation sense of the signals is indicated in the middle of the sketch.

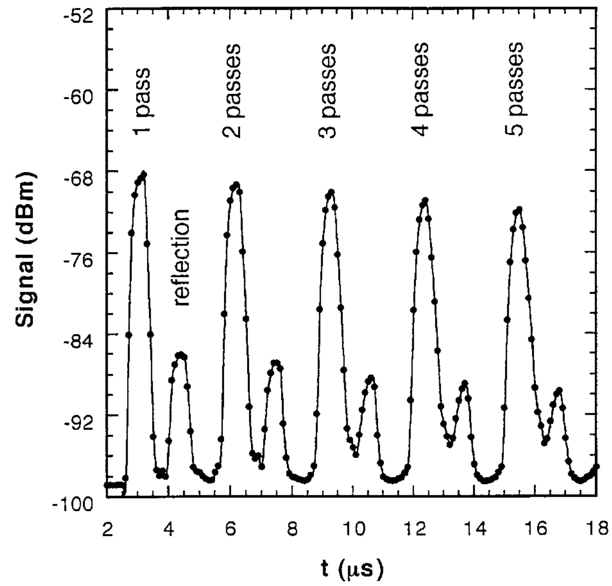

Fig. 2. Sequence of 5 echoes obtained with the setup sketched in Figure 1, shown for sample n°1. The parasitic small echoes between the main signals are −18 dB smaller than the main echoes, the spectrum being registered with a log-amplifier.

the substrate [4]. Then, the temperature is increased rapidly to 735 °C without stopping the deposition (within 2 min, corresponding to a thickness of the intermediate layer of 240 Å). The temperature, measured at the surface of the substrate holder, is finally maintained at 735 °C until the end of the growth. The total thickness of the films was 8000 Å and 950 Å, respectively. The deposition of the films has been carried out in 0.4 mbar of pure oxygen. At the end of the growth, the pressure was increased up to 400 mbar before the sample was cooled down to 100 °C in 30 min.

The resulting films show high critical temperatures of 87 K and 85.5 K. Investigation with X-rays proved that their c-axis is perpendicular to the film surface. The length of the c-axis is, for both samples, 11.70 Å. This elevated value is probably the result of the combination of a slight oxygen deficiency [5] (the critical temperatures below 90 K of our films support this assumption) and the elastic stress induced by the rather elevated lattice mismatch between the substrate and the film materials. Φ-scan analysis revealed, that the a/b-axes of our films are rotated by 45° compared to the Z/X-axes of the substrate, as was reported by Höhler *et al.* [6] for sputtered films. This orientation reduces the lattice mismatch to about 6%.

The films were lithographically patterned into a strip of 3×8 mm^2. By a lift-off process, thin gold finger structures (with a finger width and a distance between two fingers of 8 μm) were deposited by laser ablation at both ends of the $YBa_2Cu_3O_{7-\delta}$-strip, to serve as interdigital transducers. A sketch of these IDT's including the dimensions is given in Figure 1 to the left.

3 Experimental setup

Applying a pulse of an alternating voltage with frequency f to the two electrodes of the emitting transducer results, *via* the piezoelectric effect, in a surface acoustic wave propagating perpendicular to the fingers. This wave creates an alternating electric field at the opposite (receiving) IDT, which is then sent into a power divider to generate two signals of equal amplitude. One is processed through a log-amplifier, a detector and finally measured with a digital oscilloscope to monitor the power of the wave. The second signal is processed through a limiting amplifier and phase comparator to monitor the sine and cosine of the phase. The resonance frequency of the IDT's is fixed by the distance between two fingers of the same polarization. For our setup, this distance is 32 μm. It corresponds to frequencies of about 110 MHz.

To increase the resolution of surface acoustic wave experiments only a small part (−10 dB) of the electromagnetic signal obtained at the receiving transducer was fed into the detecting circuit. The largest part of the signal was, by means of an amplifier and an adjustable attenuator, compensated for the losses which occur during the transformations of the electromagnetic signal to an acoustic signal and back, reinjected into the emitting transducer, generating again a surface acoustic wave, propagating along the same path and thus experiencing the same attenuation and phase shift. The total gain of the reinjection chain of the circuit is of the order of 25 dB, thus giving the appearance to the main echoes of being cycled without losses (see Fig. 2). The electromagnetic signal created at the receiving transducer by this wave was treated in the same way and so forth. Thus, the distance the acoustic pulse travels in the sample is multiplied by a factor N which is equal to the number of injections of the "same"

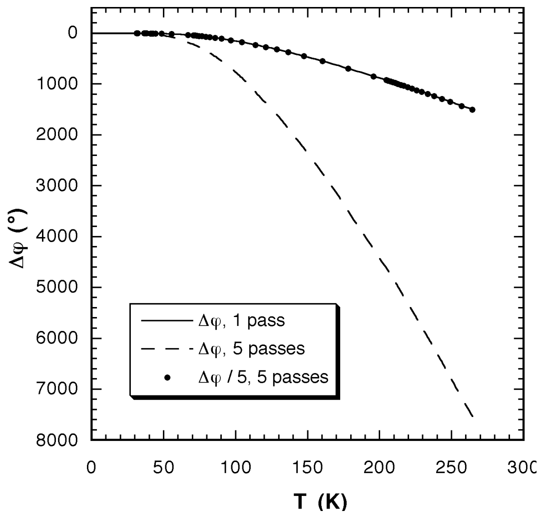

Fig. 3. Total phase variation for the acoustic signal issued from the first (full line) and fifth echo (dashed line) as a function of temperature. They are expressed in degrees. The points on the full line correspond to the phase variation of the fifth echo divided by five. They coincide exactly to the phase variation measured with the first echo.

electromagnetic pulse into the emitting transducer. The coherence of the signal being preserved, this increases the experimental resolution by a factor of N, for the attenuation measurements as well as for the determination of the phase velocity. Since the phase is determined by processing through a phase comparator working at 30 MHz intermediate frequency with further homodyne detection, the noise eventually added by the high frequency amplifier is rejected. Thus the noise observed on the signal is independent of the number of reinjections and only due to the last amplifier of the chain working at video frequency. Therefore the gain of sensitivity is really increased by the factor N. The same sensitivity would have been gained for a pulse travelling N times in the crystal, provided it would have existed and travelled without being attenuated. In Figure 2, for sample n°1, is shown a sequence of 5 echoes as they appear at the output of the log-amplifier. The parasitic small echoes between the main signals are reflections at the edges of the substrate. Their amplitude is 18 dB below the main signals. In the absence of reinjection, the parasitic signal appearing at the same time as the second main signal is 40 dB below that signal, consequently the perturbation it induces on measurements is negligible. In Figure 3, the temperature dependence of the relative variation of the phase of the signal as determined for the 1st and for the 5th echo are shown (solid and dashed lines). The dependence of this variation as determined for the 5th echo has been divided by 5, and is also shown in Figure 3 (solid circles). There is a very good coincidence with the dependence as determined directly for the first echo, which proves that our reinjecting method works very well indeed.

In principle, in this way it should be possible to measure echoes which have run through the acoustic path ten or even a hundred times. Unfortunately, due to the geometry of our setup, there is a limit. The distance between the pair of transducers is 10 mm. They are placed at a distance of 2.5 mm from opposite edges of the substrate. This means, that 2 times the distance from the transducer to the edge and back exactly equals the distance between the transducers. Thus, there will be acoustic pulses, induced by the Nth electromagnetic pulse, which have been reflected at the edge of the substrate (the existence of these reflections is well established by the parasitic echoes visible in Fig. 2) and which will arrive at the receiving transducer at the same time as the acoustic pulse induced by the $(N+1)$th electromagnetic pulse, which has not been reflected at any edge. After several reinjections, one will not be able to observe echoes whose origin is unambiguous, but which are made up of echoes having run through different acoustic trajectories. It would be impossible to extract any physically meaningful information from these signals. This becomes visible in a distortion of the echos.

To diminish the influence of the signals reflected at the edges of the substrate, we have eroded these edges. This diminished the amplitude of the reflected pulses by 15 dB. After this treatment, echoes, which had run 5 times through the acoustic path for sample n°1, and echos having passed 3 times the acoustic path in the case of sample n°2, did not show any sign of distortion and could thus be used for our experiments.

As mentioned above, this increase of the experimental resolution is even more welcome in SAW-experiments on thin films than in bulk acoustic measurements because there is a inherent decrease of sensitivity to the method: attenuation and velocity of a SAW are determined both by the elastic properties of the substrate and by the elastic properties of the film. To obtain relative changes in the elastic properties of the film's material from SAW-measurements, one has to take into account a weighting factor which depends not only on the ratio of the film thickness h and the wavelength λ of the SAW, but also on the elastic properties of both materials. We have followed the approach introduced by Snider *et al.* [7] to calculate numerically this weighting factor for our experimental situation. We obtain $\alpha_{\rm SAW} = 0.08\alpha_{\rm bulk}$ and $\frac{\Delta v}{v}_{\rm SAW} = 0.08\frac{\Delta v}{v}_{\rm bulk}$, where $\frac{\Delta v}{v}_{\rm bulk}$ is taken to be a variation of the velocities of the transverse and the longitudinal modes in the bulk.

4 Experimental results

To observe possible changes in the elastic properties of $YBa_2Cu_3O_{7-\delta}$ thin films near the superconducting critical temperature, we have measured the amplitude and velocity variation of SAW's as a function of temperature around T_c taking care not to vary the temperature too rapidly (standard variation rates were between 0.05 K/min and 0.1 K/min). For sample n°2 we have performed these measurements also in three different magnetic fields of 1.2 T, 2.4 T and 3.6 T applied perpendicular to the sample surface, *i.e.*, parallel to the c-axis of the $YBa_2Cu_3O_{7-\delta}$ film.

We have observed, for the two good films investigated, anomalies in the amplitude and the relative variation of the velocity of surface acoustic waves in the vicinity of the critical temperature.

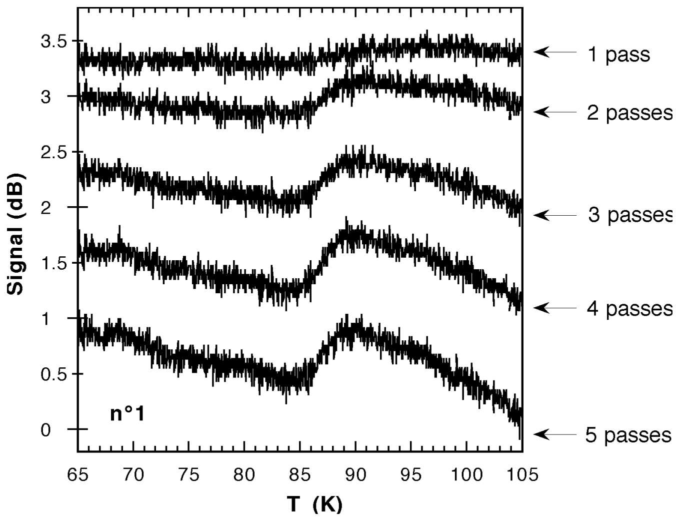

Fig. 4. Amplitude of the detected part of the electromagnetic signal obtained at the receiving transducer for a pulse having passed 1,2,3,4 and 5 times the acoustic path. One sees a step-like anomaly in the signals which have run several times through the same acoustic path. On the other hand, this anomaly is nearly invisible for the pulse having performed only one acoustic pass. This illustrates the usefullness of our experimental method sketched in figure 1.

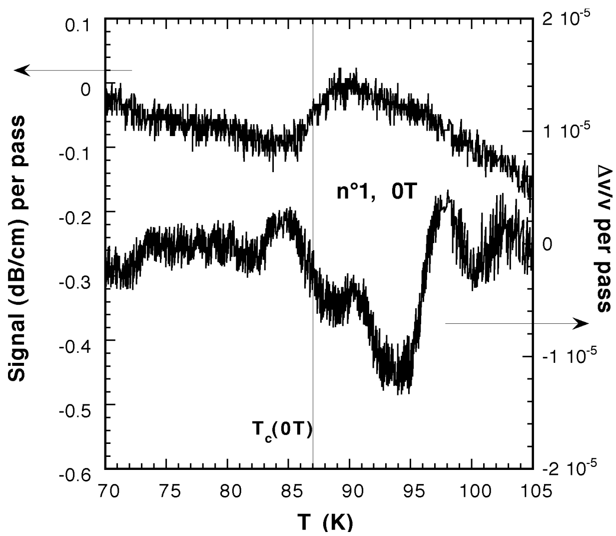

Fig. 5. Anomalies observed in the amplitude (upper part) and in the relative velocity of surface acoustic waves for sample n° 1, obtained for pulses having passed the same acoustic path 5 times, plotted per pass. The critical temperature indicated is the temperature, where the resistance has dropped to half of its normal value. Shown are the thin film measurements. To obtain their numerical values for bulk material, one has to take into account the proportionality factor given at the end of section 3.

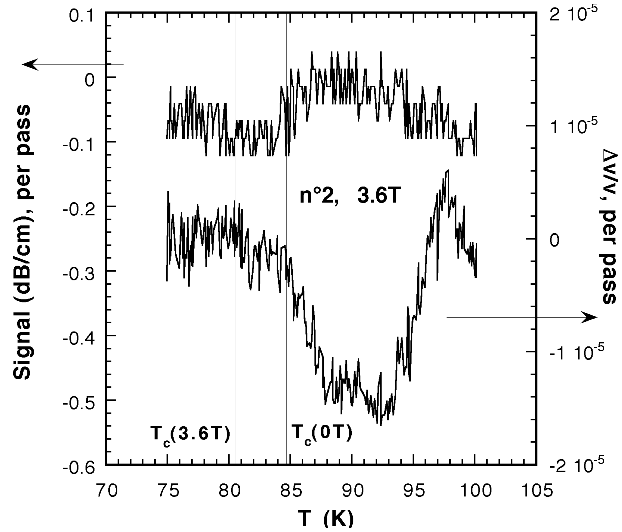

Fig. 6. Anomalies observed in the amplitude (upper part) and in the relative velocity of surface acoustic waves for sample n°2, obtained for pulses having passed the acoustic path 3 times with a magnetic field of 3.6 T applied perpendicular to the film surface and plotted per pass. The critical temperatures indicated are the temperatures, where the resistance has dropped to half of its normal value. Again, thin film measurements are shown. To obtain their numerical values for bulk material, one has to take into account the proportionality factor given at the end of section 3.

In Figure 4, the amplitude of the detected part of the electromagnetic signal obtained at the receiving transducer is shown for a pulse after 1, 2, 3, 4 and 5 acoustic passages (sample n°1). The step-like anomaly in the amplitude is easy to see for the pulse, which has run through the acoustic path several times. On the other hand, it is nearly invisible for the pulse having performed only one acoustic passage. This illustrates the usefullness of our reinjecting method. The size of the step is about 0.1 dB/cm per trajectory (Fig. 4). Its extension in temperature is about 5 K around T_c. Figure 5 shows simultaneously this step for the fifth echo and the corresponding change in velocity. Figure 6 shows this step as measured for sample n°2, in a magnetic field of 3.6 T. The size of the step is the same for both samples and does not vary under application of a magnetic field. Taking the weighting factor into account as described in Section 3, this corresponds to a step in the attenuation in $YBa_2Cu_3O_{7-\delta}$ of 1.3 dB/cm.

The observed variation of velocity with temperature has two origins: a small contribution around T_c from the film is superimposed on a large background contribution due to the smooth and monotonic change of the elastic constant of the substrate. To determine this background around T_c, we interpolated a parabolic function fitted to the variation measured outside the temperature interval 78 K and 97 K. Then, we have subtracted the fit values from the experimentally obtained points. We observe, for both samples, a decrease of the experimental values compared to the parabolic background of about $\frac{\Delta v}{v} = 1.5 \times 10^{-5}$ per acoustic passage, extended about 12 K above the critical temperature. This is shown for sample n°1 in Figure 5 together with the step in the amplitude already shown in Figure 4. This corresponds to a variation of the sound velocities in bulk-$YBa_2Cu_3O_{7-\delta}$ of 2×10^{-4}. The magnitude of that change around T_c is scarcely dependent of the procedure adopted to parameterize the background change of the velocity (extension of the interval of temperature, parabolic or higher order function), by virtue of physics it extends in a relatively narrow range of temperature giving a good confidence to its amplitude but not to its precise shape. In Figure 6, the same results are shown for sample n°2 in a magnetic field of 3.6 T.

Once again, the application of a magnetic field up to 3.6 T did not have any effect on this elastic anomaly. It is noteworthy, that the resistivity measurements might be more sensitive to any grain boundary effects than acoustic measurements, which probe the bulk of the material. In any case, the magnetic fields we applied are rather small compared to the large critical fields of $YBa_2Cu_3O_{7-\delta}$. The shift of the resistive transition caused by the application of the field is smaller than the extension in temperature of the elastic effects observed and is probably related to the intergrain properties while the acoustic wave probes the volume of each grain.

5 Discussion

Propagation of acoustic waves has been used as a standard probe for superconductivity. A decrease in the acoustic attenuation below T_c was explained long ago by the disappearance of the contribution of free electrons caused by the opening of an energy gap (see, for example [1]).

For the high T_c-superconductors, this effect is expected to be much smaller than for the classical superconductors. The smaller mean free path for the electrons and the smaller density of carriers in these materials lead to a very small acoustic absorption. This absorption can be calculated as: $\alpha_{el} = \frac{4}{15}\left(\frac{Nmv_F}{\rho v}\right)k^2 l$, where N denotes the charge density, m the carrier mass, v_F the Fermi velocity, ρ the mass density, v the ultrasonic velocity, k the acoustic wave vector, and l the electron mean free path. For $YBa_2Cu_3O_{7-\delta}$, one can estimate $\alpha_{el} \approx 5 \times 10^{-4}$ dB/cm at 100 MHz (taking the following values: $N = 10^{22}$ cm^{-1}, $m = 9.11 \times 10^{-28}$ g, $v_F = 10^6$ cm/s, $v = 4000$ m/s, $\rho = 6.4$ g/cm^3, $l = 10$ nm and $k = 1500$ cm^{-1}). This is much too small to be seen in our experiments.

The attenuation of SAW's by $YBa_2Cu_3O_{7-\delta}$ thin films has also been interpreted in terms of the acousto-electric effect[8]. The electric field which accompanies a SAW in a piezoelectric substrate couples this wave to the sheet resistance of a thin film deposited on the substrate. This sheet resistance induces an absorption of energy, which depends on the dielectric and the piezoelectric coupling constants of the substrate, the sheet resistance of the film, the wavelength and the velocity of the SAW. Following Lee *et al.* [8], we can estimate this contribution to the attenuation of the SAW's to be $\alpha_{ae} \approx 6\times10^{-3}$dB/cm. This is also too small an effect to be seen with our experimental setup.

In any case, both effects should diminish the acoustic attenuation below the critical temperature. We observe an increase of the attenuation, thus our measurements can not be accounted for by the two processes presented.

The indifference to magnetic fields of the anomalies observed makes unlikely an interaction between vortices and the SAW's as a reason for these effects. The effects we observe are also situated rather above than below the critical temperature.

The discontinuity of the specific heat ΔC at the superconducting critical temperature T_c is related to the relative variation of the velocity of compressional bulk waves by the Ehrenfest relation [9,10]: $\frac{\Delta v}{v} = -\frac{1}{2}B\frac{\Delta C}{T_c}\left(\frac{dT_c}{dP}\right)^2$, where $B = \rho v^2$ is the bulk modulus. Using the results of Junod *et al.* $\Delta C = 50$ mJ/kg K^2 [11] and of Besson $dT_c/dP = 1.5$ K/GPa [12], one can estimate the resulting change in the relative variation of the sound velocity to be: $\frac{\Delta v}{v} \approx 4 \times 10^{-5}$. This is by a factor of 5 smaller than the effect we observe in the velocity. Furthermore, the discontinuity in the specific heat should occur at the critical temperature itself. The diminution of the relative variation of the SAW velocity we observe is extended to temperatures about 10 K above the critical temperature. Therefore, it seems unlikely that the discontinuity in the specific heat is the reason for the SAW velocity anomaly we observe.

A spin gap or pseudo gap has frequently been invoked to explain NMR-experiments which yield a susceptibility varying over a large temperature range [13]. The effects we have observed are localized in temperature. So, our measurements do neither support nor rule out the existence of such a magnetic gap.

Thus, there are difficulties to explain our experimental results in the framework of well-established theories. A clue which could perhaps lead to a qualitative interpretation of our observations can be obtained by closely regarding of the properties of another class of superconductors, the so-called A15 compounds (*e.g.*, V_3Ge, V_3Si). The structural phase transition just above T_c, which occurs in these superconductors [14], has been successfully explained in the framework of the model of Labbé and Friedel [15]: depending on the temperature, it can be energetically favorable for a material to raise energy to change the crystalline structure, if the amount of energy necessary is more than compensated by an energy gain of the electronic system. In particular, the ingredients necessary for this type of theory to apply are a singularity in the density of states, a band degeneracy of the density of states, a Fermi-level in the vicinity of this singularity (which is the case for the oxide superconductors [16]), and a coupling of the electron states to the deformation potential (see, *e.g.* [17]). All these prerequisite conditions are fullfilled for $YBa_2Cu_3O_{7-\delta}$. It is interesting to note that in a test experiment with another $YBa_2Cu_3O_{7-\delta}$ film whose superconducting transition was rather broad (10 K) we have not observed any elastic anomaly. This is consistent with the prediction of [18,19]: a 2d singularity in the density of states would be more affected by inhomogeneities than in the 1d case (A 15-compounds). The phase velocity decrease and the attenuation increase which we observe above T_c are similar to the effects of a structural phase transition. If the signal does not recover its high temperature value the existence of domains may well prevent it recovering for $T < T_c$. Moreover, any small step in the velocity through T_c will be washed out by the procedure adopted to extract the rapidly varying part. However, to see if the general idea of Labbé and Friedel applies to high-temperature superconductors and, notably, can explain our results, more theoretical work is necessary to permit a quantitative analysis.

Our measurements are, to our knowledge, the first acoustic experiments which show elastic anomalies just above T_c in thin $YBa_2Cu_3O_{7-\delta}$ films. Other thin film SAW-experiments on $YBa_2Cu_3O_{7-\delta}$ were focused on effects occurring over the whole temperature range between 4 K and room temperature [8,20]. On the other hand, elastic anomalies have been reported in longitudinal acoustic waves in bulk-$YBa_2Cu_3O_{7-\delta}$, in sintered samples [21] and in monocrystals [22,23]. The velocity anomalies detected in the sintered samples and in the longitudinal modes c11 and c33 in the monocrystals are of the same order of magnitude as our result and situated at the critical temperature, not above. They are interpreted in terms of the jump in the specific heat, even though the experimentally measured values are larger than predicted by theory. Kim *et al.* [23] found a cusp-like increase of the attenuation at 8 MHz of the c11-mode at T_c, which is of about the same size as the step-like increase observed by us, supposing a quadratic frequency-dependence of the attenuation.

It can not be excluded that the reason for the elastic anomalies found in our measurements is the same as for those observed by other groups, even if the temperature at which they appear is not the same. Particularly, if a kind of Labbé-Friedel-mechanism is a good explanation. The temperature, at which the anomaly takes place would depend strongly on the electronic structure of the samples, which could be slightly different for different kinds of samples. For example, it may be due to varying degrees of oxygenation. The filling of the band near a so-called Van Hove-singularity may lead to a density of states near the Fermi level which differs strongly even for small changes of δ. On the other hand, the dependence of T_c on δ is not very strong for small δ.

Finally, we should mention a quite different type of experiment on $YBa_2Cu_3O_{7-\delta}$ single crystals which supports our results. By ion-channeling, a dip in the Rutherford backscattering yield along the 001-axis has been observed around 100 K. This dip is pronounced for oxygen atoms, but also present for Copper atoms [24]. The authors claim, that these results suggest a lattice softening, even if the corresponding mode is not yet identified.

6 Conclusions

In summary, we have presented a procedure which allows an increase in the experimental sensitivity of SAW's. We have used this procedure to measure the temperature dependence of the attenuation and relative variation of phase velocity of surface acoustic waves propagating in 2 samples of thin $YBa_2Cu_3O_{7-\delta}$ films deposited onto $LiNbO_3$-substrates. We observe a step-like increase of the attenuation at about the critical temperature and a diminution of the relative variation of the SAW-velocity ranging from T_c to about 10 K above T_c. These experimental results can not be explained by the condensation of the carriers, nor by the electro-acoustic effect, nor by the discontinuity in the specific heat. Such effects have, to our knowledge, not yet been reported in thin film measurements. Our results are consistent which what has been reported for bulk acoustic measurements in $YBa_2Cu_3O_{7-\delta}$. We suggest, that a mechanism like that in the A15-compounds could perhaps lead to an explanation of the phenomena observed. A quantitative explanation of the effects in this framework would require further theoretical work.

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