Low-temperature elastic moduli and dilational and shear internal frictions of superconducting ceramic $GdBa_2Cu_3O_{7-\delta}$

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Longitudinal and transverse wave velocities, six kinds of elastic parameters, and dilational and shear internal frictions have been simultaneously measured as functions of temperature between room temperature and 77 K in the high-T_c superconductor $GdBa_2Cu_3O_{7-\delta}$. An abrupt increase from 117 K in bulk modulus and Lamè parameter suggests that the order parameter has a much stronger coupling with volume-nonpreserving distortions than with volume-preserving distortions. The 227 and 120 K peaks in dilational friction might be related to structural change and second-order phase transition due to variation of potential energy, respectively. For shear friction, the 214 and 106 K peaks could be attributed to barium atom relaxation with an apparent activation energy of 0.62 eV, and lattice instability, respectively.

I. INTRODUCTION

Since the discovery of cuprate oxide superconductors, much work has been carried out with ultrasonic techniques.[1–8] However, as concerns the experimental results and theoretical explanations of the phenomena observed there are different opinions. This discrepancy has generally been explained by very strong sensitivity of acoustic parameters to the quality of the sample preparation.[9,10] Three other causes may be added: first, low-temperature deterioration[11] of piezoelectricity in transducer materials such as PZT [$Pb(Zr_xTi_{1-x})O$] and $LiNbO_3$, which are epoxied onto the samples; second, failure to make different frequency measurements simultaneously; third, usage of relative elastic moduli in place of low-temperature ones. In the conventional method, these transducers are exposed to a cold temperature during measuring, and longitudinal and transverse waves with different frequencies are measured on separate runs. Because the ultrasonic attenuation of high porosity materials is strongly affected by the frequency,[12] we must pay attention to use the optimum wave with the same frequency. In addition, the acoustic wave velocity anisotropy factor $A=\sqrt{3}V_t/V_l$ is dependent on the kind of solid and temperature,[13,14] where V_l and V_t are longitudinal and transverse wave velocities, respectively; the relative elastic moduli are not appropriate parameters as a true bulk probe of superconductivity. Thus it is necessary to reinvestigate all the ultrasonic elastic data reported to this day with respect to these points of view.

On the other hand, internal friction is also often measured for investigation of strain relief, softening and lattice relaxation in superconductors,[1,3–8] but no one has drawn a physical line between longitudinal and transverse wave frictions. In particular, since cold deterioration in piezoelectricity arrests the occurrence of friction peaks in low-temperature region, the conventional method is not necessarily suitable.

Our interest lies in simultaneously determining all the elastic moduli and both dilational and shear internal frictions of a high-T_c superconductor through its superconducting transition. In this study, we report temperature dependence of the longitudinal and transverse wave velocities, their anisotropy factor, elastic (Young's, shear and bulk) moduli, Lamè parameter,[15] Poisson's ratio, dilational and shear internal frictions for high-T_c superconductor $GdBa_2Cu_3O_{7-\delta}$ (hereafter referred as Gd oxide) in view of the electron-phonon coupling for lattice hardening of superconducting crystals. As far as we know, no research work has been carried out previously on low-temperature, simultaneous measurement of all the parameters for the cuprate oxide superconductor, using both longitudinal and transverse waves with the same frequency. Since the longitudinal wave interacts with electrons and the transverse one lacks such interactions in superconducting state,[16] simultaneous measurement provides such useful information about structural changes, lattice instabilities, superconducting transition and electronic contributions for superconductors. Furthermore, simultaneous measurement can enable us to evaluate the effects of all elastic parameters for superconductivity. In particular, the temperature dependence of the shear and bulk moduli or Lamè parameter can explain the coupling of the superconducting order parameter to volume-preserving distortions or volume-nonpreserving distortions.

II. EXPERIMENTAL

Longitudinal and transverse wave velocities, all elastic moduli and internal friction values were accurately measured by means of the ultrasonic pulse sing-around method[17] with zero cross time detection and multiple delay circuits, since "main" and "trailing" signals always overlap in the propagation of spurious echoes. The experimental procedure is described in the previous paper.[14] Design of short-specimen buffer-rod (waveguide) system is schematically shown in Fig. 1. The specimen

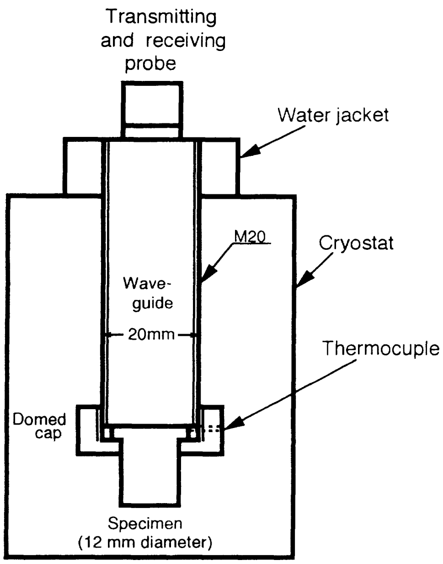

FIG. 1. Design of short-specimen buffer-rod system. The 20-mm-long specimen is mechanically coupled to 50-mm-long buffer rod. Cupler is of stainless steel SUS 304 (U.S. designation; SAE 30304).

was in the form of short rod fastened with the waveguide of Ti-6Al-4V alloy with threads of pitch 1.5 mm, to eliminate the generation of spurious signals by mode conversion at sides,[18] using domed cap nut of stainless steel. The transducers and the specimen were jointed to both sides of the waveguide by water-free glycerin grease (Sonicoat). The experiments were performed at 3.6 MHz, where optimum resolution was achieved. An apparent activation energy for a relaxation peak was determined using the frequencies of 1.7 and 2.3 MHz. The water jacket at the top of the rod warmed it so that the longitudinal wave generation PZT transducer at the top end was kept room temperature, to avoid the low-temperature deterioration. The specimen, which is vertically suspended in convenient cryostat, was measured from room temperature down to the boiling point of liquid nitrogen at a cooling rate of approximately 2 K/min. Because trailing pulses are generated when a longitudinal sound wave is propagating into a rod, both accurate longitudinal and transverse velocities are simultaneously determined in one run,[14,18] even if there are temperature gradient and the resulting velocity one through the waveguide between the room temperature and the cooled sides. No thermal shrinkage correlation was done in the calculation of velocity since it was expected to be small in comparison to the change of sound velocity. Increasing and decreasing rates in the internal friction at low temperature were calculated from echo amplitude of the ultrasonic wave at room temperature.[14] The specimen, which was sintered at 1113 K for 36 ks in air, showed zero resistivity at 89 K and abrupt decrease of the ac magnetic susceptibility at 93 K.[19] X-ray diffraction measurements were performed to ensure that the samples were single phased. From the specimen's mass and size, we estimated a macroscopic mass density of 4.768 Mg/m^3, implying void content of 31.1%. A unit cell volume at 77 K was calculated using data of $YBa_2Cu_3O_{7-x}$ superconductor (hereafter referred as Y oxide) reported by Tarascon *et al.*[20]

III. RESULTS

A. Temperature dependence of acoustic wave velocity

The longitudinal and transverse wave velocities as a function of temperature are shown in Figs. 2 and 3, respectively. As the temperature decreases from room temperature down to 117 K above T_c (=89 K), both velocities increase monotonically as they do for conventional solids. However the former begins to increase abruptly from 117 K, and then saturates (0.4% increase between 117 and 77 K) below T_c. This pronounced stiffening suggests that atomic bonding strength of the superconducting phase is larger than that of the normal conducting one. The latter shows a small decrease by 0.14% between 117 and 89 K, but no change below T_c. Almond *et al.*[21] have measured the ultrasonic wave velocity of Gd oxide ceramic in the temperature range between 42 and 290 K, and they have observed distinct increase below T_c in longitudinal and transverse curves.

In discussion of properties of solids, it is useful to use the acoustic wave velocity anisotropy factor. The temperature-dependence velocity anisotropy factor is shown in Fig. 4. The factor is almost the same from room temperature down to 117 K, but decreases from 117 K down to 89 K and then saturates below T_c. Since the solid with the anisotropy factor $A=1$ is elastically isotropic, the normal phase is apparently an isotropic body at room temperature in comparison with the degree of anisotropy of certain cubic crystals: iron 2.4, copper 3.2, lead 4.0, NaCl 0.7, KCl 0.36, LiF 1.6 (Ref. 13) and α-alumina 1.0, yttria-stabilized tetragonal zirconia crystal (Y)TZP 0.9, β'-sialon $(Si,Al)_3(N,O)_4$ 0.9 (Ref. 14) and α-SiC 1.1.[22] The decimal fraction indicates inactivity of the dilational mode or activation of the shear mode in elastic solids. It would be the former, assumed from the relative decrease of the transverse wave velocity to the longitudinal wave one at low temperature.

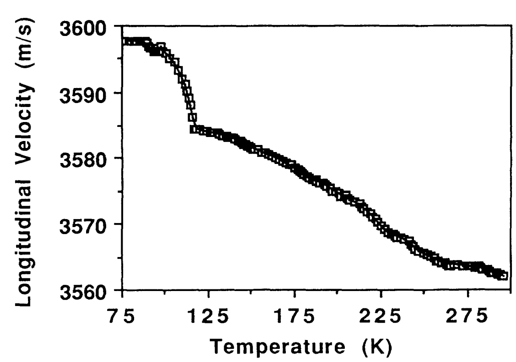

FIG. 2. Longitudinal wave velocity vs temperature at high-T_c superconductor $GdBa_2Cu_3O_{7-\delta}$.

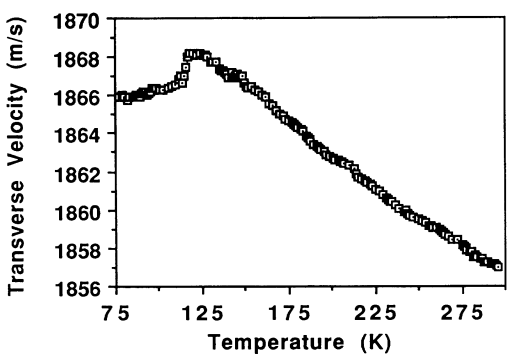

FIG. 3. Transverse wave velocity vs temperature at high-T_c superconductor $GdBa_2Cu_3O_{7-\delta}$.

B. Temperature dependence of elastic moduli

Since the only structural defects affecting the sound velocity are the voids,[10] the correct values of the elastic parameters of the bulk material must be estimated using the volume fraction of voids. However, since behavior in elastic parameters of the void-free materials is similar to that of the matrix material with voids, the elastic parameters of the matrix material serve conveniently as measures in place of those of the voids-free material. According to a previous paper,[14] Young's, shear and bulk moduli, Lamè parameter and Poisson's ratio were calculated by use of longitudinal and transverse wave velocities. In particular, Lamè parameter is more sensitive to structural change, lattice relaxation, grain boundary sliding in comparison with shear modulus,[14,23] because the former is a function of both longitudinal and transverse wave velocities, but the latter depends on the transverse one alone. These elastic results are shown in Figs. 5–9, respectively.

Young's and shear moduli increase down to 117 K as the temperature decreases, and then the former saturates but the latter slightly decreases. Since temperature dependence of Young's and shear moduli of the Gd oxide superconductors has not been observed by ultrasonic method, we compare these data with the elastic moduli of the Y oxide superconductors. Lee and Salama[24] have reported a monotonous increase below T_c in Young's and

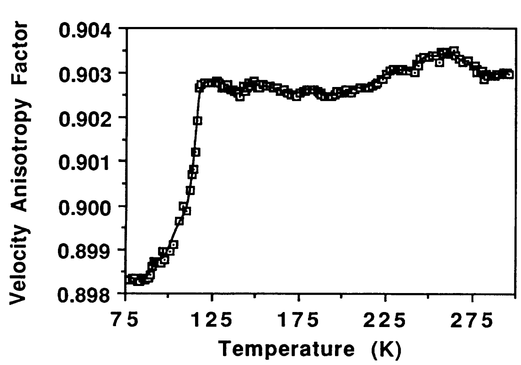

FIG. 4. Acoustic wave velocity anisotropy factor vs temperature in high-T_c superconductor $GdBa_2Cu_3O_{7-\delta}$.

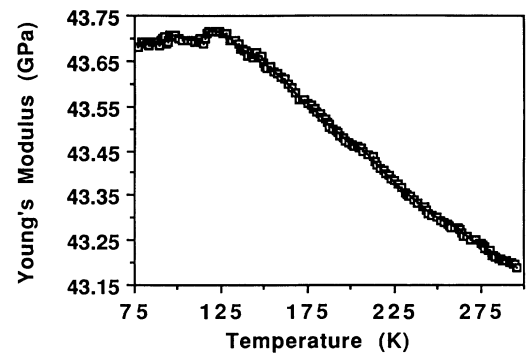

FIG. 5. Young's modulus of high-T_c superconductor $GdBa_2Cu_3O_{7-\delta}$ as a function of temperature.

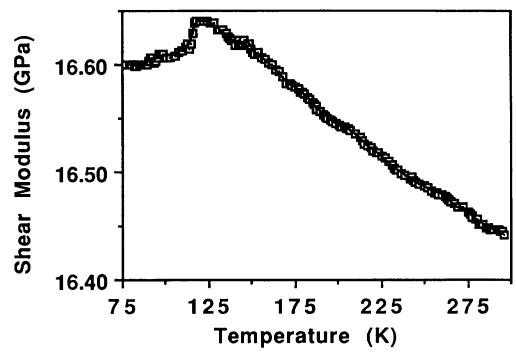

FIG. 6. Shear modulus of high-T_c superconductor $GdBa_2Cu_3O_{7-\delta}$ as a function of temperature.

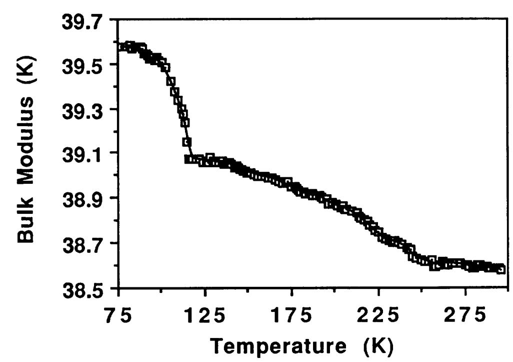

FIG. 7. Bulk modulus of high-T_c superconductor $GdBa_2Cu_3O_{7-\delta}$ as a function of temperature.

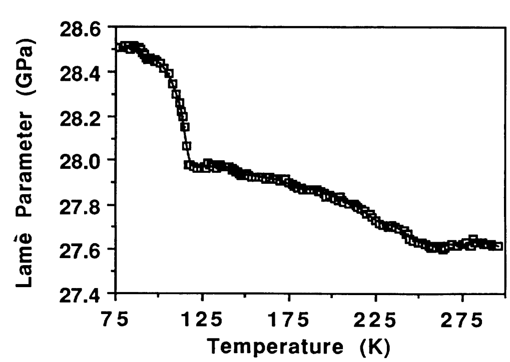

FIG. 8. Lamè parameter of high-T_c superconductor $GdBa_2Cu_3O_{7-\delta}$ as a function of temperature.

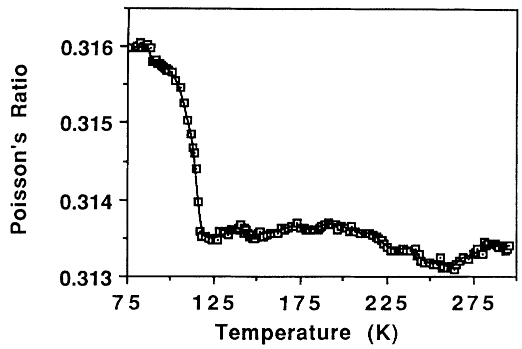

FIG. 9. Poisson's ratio of high-T_c superconductor $GdBa_2Cu_3O_{7-\delta}$ as a function of temperature.

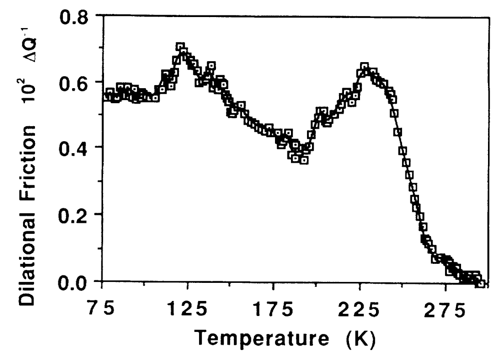

FIG. 10. Temperature dependence of dilational internal friction for high-T_c superconductor $GdBa_2Cu_3O_{7-\delta}$.

shear moduli of Y oxide, and Ledbetter *et al.*[16] have found hardening of shear modulus below T_c. Temperature dependences of bulk modulus and Lamè parameter resemble that of the longitudinal wave curve in Fig. 2, indicating enhancement of crystal rigidity below 117 K. Existing thermodynamic theory for a second-order phase transition predicts that shear and bulk moduli decrease during cooling from disordered phase to ordered one.[25] Therefore we cannot elucidate these behaviors by the standard thermodynamics of the BCS model.[26] The least hardening of the bulk modulus cannot be due to the carrier condensation alone. Poisson's ratio does not change distinctly between room temperature and 117 K, but it increases somewhat from 117 K. Since increase in Poisson's ratio means softening of crystal lattice, this increase from 117 K is reversed in contrast with increase in the bulk modulus and Lamè parameter. This could perhaps be due to apparent enhancement, as well as apparent peaks in isostatical hot pressed β'-sialon[14] and hot-pressed silicon nitride.[22] However, we cannot make any assignment at present time. Compared with the normal conducting phase, thus, the superconducting phase is more tough for C_{12} shear in the elastic constant matrix but more deformable for simple (uniaxial) shear. Together, these results suggest that an intermediate phase transition precedes the superconducting transition at 117 K, as reported by Bhattacharya *et al.*[5]

C. Temperature dependence of internal friction

Internal friction curves for longitudinal and transverse waves are shown in Figs. 10 and 11, as a function of temperature, respectively. The dilational friction shows two peaks at 227 and 120 K. In comparison with other data of the Gd oxide, Almond *et al.*[21] have observed two peaks at 200 and 70 K, but Brown, Migliori, and Fisk[27] have not observed any peaks in the temperature region from 112 to 78 K. In Y oxide ceramic,[10] a similar behavior was observed at 235 and 130 K. Both the 227 and 120 K peaks would be related to electron-phonon interaction or structural readjustment associated with variation of potential energy. The 227 K peak may be related to structural change due to the nonelastic accommodation of the second phase misfit domains which Cannelli *et al.*[28] have found in electrostatic excitation flexural vibration method. The 120 K peak, marked as T_s transition, would be due to a second-order phase transition, because the specific heat at T_c could be explained by the logarithmic potential divergence associated with strain relief of outer electrons.[29] In the literature,[30] T_s depends on the oxygen stoichiometry. The 120 K peak also corresponds to a kink temperature at 117 K in all the elastic moduli. Although Almond *et al.*[21,31] have concluded that the inflection in velocity for the Gd oxide is attributed to the velocity change accompanied by an elastic loss peak associated with a thermally activated process, this peak is not the thermal activated one, as described later. Furthermore, the decrease from 120 K in the vicinity of T_c is small in comparison to the abrupt decrease as expected for the BCS superconductor.[26,32]

On the other hand, the transverse curve reveals two peaks at 214 and 106 K during cooling. Almond *et al.*[21,31] have reported two peaks at 170 and 64 K for the Gd oxide. The striking peak at 214 K is similar to a transverse attenuation peak at 210 K in Y oxide single crystal.[4] Since the 214 K peak is shifted to a higher temperature with increasing frequency, the frequency dependency was investigated to clear up the microscopic nature of the thermally activated anelastic relaxation process. The frequency F varies with the peak temperature according to

$$F = F_0 \exp(E/kT) , \tag{1}$$

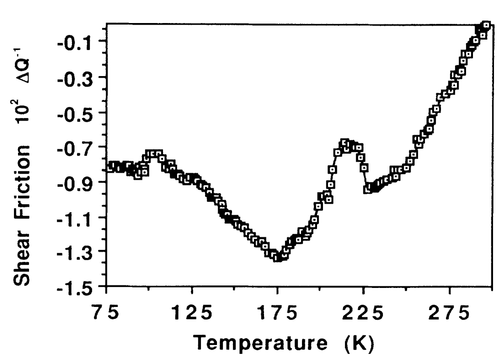

FIG. 11. Temperature dependence of shear internal friction for high-T_c superconductor $GdBa_2Cu_3O_{7-\delta}$.

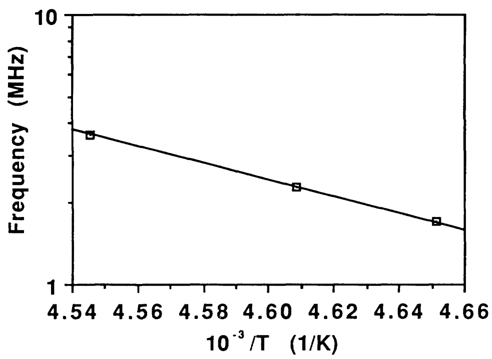

FIG. 12. Arrhenius plot of ultrasonic frequency for low-temperature peak in high-T_c superconductor $GdBa_2Cu_3O_{7-\delta}$.

so the apparent activation energy is found from the slope (Fig. 12) to be 9.96×10^{-20} J (=0.62 eV).

This value suggests change of the barium positions in the crystalline structure, because the activation energy of the smaller atoms such as Gd and O is smaller than 0.62 eV. In nonsuperconducting $YBa_2Cu_3O_6$, Cannelli, Cantelli, and Cordero[33] have found an anelastic relaxation peak with activation energy of 0.11 eV, which is caused by the stress-induced hopping of the residual oxygen atoms in the basal Cu-O planes. This barium relaxation model is also supported by a reduction of the separation of the barium atoms from the Cu(1) planes between 250 and 150 K.[34] Analogous relaxation results were shown for the Y-Ba-Cu-O (Refs. 3 and 7) and the La-Sr-Cu-O systems.[23,35,36]

As regards the origin of another small peak at 106 K, there are some possibilities, e.g., mechanical damping by lattice instability, phase transition, alterations of the twin structure and migration of oxygen atoms etc. According to physical meaning of the shear friction, the 106 K peak might be due to lattice instability. This structural instability is likely to relate to decrease in shear modulus from 117 K, which reflects a soft phonon mode. The lattice instability seems to be a common feature of all the layered superconducting perovskite ceramics.[5] For this reason, Bèal-Mond *et al.*[37] have reported that the lattice instability is associated with some intrinsic ordering of the oxygen vacancies.

IV. DISCUSSION

Acoustic wave velocity which is mainly phonon velocity, varies with temperature and pressure due to nonharmonity of potential between atoms. In the ultrasonic method, the pressure effect is negligible. On the other hand, flexural vibration and resonance frequency methods have a possibility of deterioration due to high frequency fatigue.[38] In the cuprate oxide superconductors, indeed, the complicated behaviors were observed in measurement by flexural vibrating[28,39] and vibrating-reed techniques,[40] compared with the ultrasonic one. Since enhancement of T_c in superconductivity is strongly affected by material conditions such as grain boundaries, heterogeneous phases, vacancy defects and metastable or nonequilibrium phases,[41] it would not be desirable for investigation of superconductors to use the vibrating methods with fatigue effect.

Since a ratio of a (=6 mm), the radius of the specimen, to λ (=1.0 mm), the wavelength, a/λ (=6.0 at room temperature), is greater than 2.5, the measured pulse wave velocity approaches the bulk velocity in an infinite elastic medium.[42] This high ratio can enable us to distinguish a physical meaning between pure mode III shear defined as shear modulus and mixed mode II-mode III shear designated as Lamè parameter.[43] In the ultrasonic pulse method, shear modulus is a function of the transverse wave velocity only, while Lamè parameter is a function of both longitudinal and transverse waves.

The decrease from 117 K in the anisotropy factor of Fig. 4 and increase in bulk modulus, Lamè parameter of Figs. 7 and 8 show a transition driven by a change in the mechanical properties of the system. By contrast to no change in Young's modulus in Fig. 5 and small decrease in shear modulus of Fig. 6, we can conclude that this hardening from 117 K is due to volume-nonpreserving distortion, rather than volume-preserving (uniaxial shear) distortions. This leads to a large coupling between the superconducting order parameter and the volume-nonpreserving distortion. Thus the hardening can also be interpreted in terms of a reduction of the screening of lattice distortions due to the depletion of carrier density, which couples strongly to longitudinal-acoustic phonons. To the best of our knowledge, however, no one has ever reported any theoretical model that anticipates or predicts such a hardening from T_s. Furthermore, this hardening endorses a superconducting model proposed by Fukuhara *et al.*,[41,44–45] in which superconductivity arises from singlet pairs in filled atomic shells lying below the conduction band. This model is based on a common feature of all kinds of superconductors, that is, the forced compression of the paired electrons toward a nucleus in the superconducting atom. T_c increases with the increase of the compressive pressure against the spherical electron clouds due to the pintch effect which is induced by excess oxygen in cuprate oxide superconductors and by the outermost electrons in the superconducting elements. In cuprate oxide superconductor, the superconducting atoms are forced to squeeze into the lattice skeleton modified by the strong bonds associated with the affinity of the outer s and p electrons in II-III group elements for oxygen.

The temperature dependences of the wave velocities can be further clarified by consideration of conversion anharmonic effects explained by the T_c-Θ_D relationship.[46] From the mean Debye velocity

$$V_m=\frac{1}{3}\left[\frac{1}{V_L^3}+\frac{1}{V_t^3}\right], \tag{2}$$

we can calculate the elastic Debye temperature

$$\Theta_D=\left[\frac{h}{k}\right]\left[\frac{3N}{4\pi}\right]^{\frac{1}{3}}V_m . \tag{3}$$

Here, h and k take their usual meanings, N denotes number of mass point in an average atomic volume V. To correct the measurements to the void free state, we used a model given by Lee and Salama,[24] taking into the effects of porosity ($n = 0.311$) consideration. Using the corrected longitudinal and transverse wave velocities of a 100% dense material along with $V = 175.2 \times 10^{-30}$ (at 296 K), 174.8×10^{-30} m^3 (at 77 K) and $N = 13$, Θ_D of the Gd oxide at 296 and 77 K are calculated to be 309 and 310 K, respectively. These values are fairly consistent with those (313 and 329 K) of Y oxide reported by Kim *et al.*[47] and Ledbetter *et al.*,[48] respectively, but are smaller than other reported values [426 K (Refs. 21 and 23), 500 K (Ref. 10)] of the Y oxides. Both values obtained in this study are almost the same between 296 and 77 K, indicating the temperature independency of the velocities, i.e., the elastic constants. In other words, the Debye temperature, i.e., phonon effect, might not be directly related to occurrence of superconductivity in the Gd oxide, although it is reported that many ordinary superconductors obey McMillan's T_c-Θ_D relationship[49] for conventional BCS materials.

As can be seen in abrupt increase from 117 K of bulk modulus and Lamè parameter during cooling in Figs. 7 and 8, slight decrease from 120 K in dilational friction of Fig. 10 and deviation from the T_c-Θ_D relationship, thus, these experimental results have put severe constraints on the possibility of application of the high-T_c superconductors to the BCS theory.

As reported in previous studies of ceramics[14,21] and metals,[35,50] physical properties of the dilational friction are different from those of the shear one; the former corresponds to superplasticity and recrystallization accompanied by relief of strain, and the latter is caused by phase transformation, solute solubility, softening of precipitate or glassy phases at grain boundaries and diffusion of ions. A common point of the former is motion of dislocation along grain boundaries or variation of potential energy between atoms, while that of the latter is atomic rearrangement in crystalline structure. Since the elastic strain field of the longitudinal wave changes the local electronic concentration of atoms, the phonon damping occurs when the longitudinal phonon comes across movement of dislocation. Here it should be noted that interatomic forces between two atoms are increasingly repulsive when they draw near each other, and thereby atomic force microscopy is based on sensing the forces between a sharp stylus and the surface of interest.[51] The interatomic interactions in the contact mode can be understood only by using molecular dynamic simulations with realistic potential.[50] Thus, it is clear that the dilational friction is a sensitive probe for electron-phonon interaction in superconductor. In ordinary superconducting studies, indeed, physical meanings of both frictions have been confused.

From the above mentioned results and discussions, putting four internal frictions (227, 214, 120, and 106 K), kink point (117 K) of elastic parameters and T_c ($=89$ K) in temperature order, it is clear that origins of physical phenomena observed in the Gd oxide change from causes in lattice size scale to contribution of electrons, especially outer electrons such as *s* and *p* ones, with decreasing temperature.

V. CONCLUSION

Characteristic elastic and damping behavior have been simultaneously measured as functions of temperature between room temperature and 77 K in the high-T_c superconductor $GdBa_2Cu_3O_{7-\delta}$. From 117 K in the vicinity of T_c, Young's and shear moduli saturate and slightly decrease, respectively, but the bulk modulus and Lamè parameter reveal abrupt increases. These results suggest that the order parameter has a much stronger coupling with volume-nonpreserving distortions than with volume-preserving distortions. The 227 and 120 K peaks in dilational friction are probably related to structural change and second-order phase transition due to variation of potential energy, respectively. For shear friction, the 214 and 106 K peaks might be interpreted by barium atom relaxation with an apparent activation energy of 0.62 eV, and lattice instability, respectively.

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