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# Resonance-type internal friction of a 1:1:1:2 Bi–Sr–Ca–Cu–O superconductor at very low frequencies

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**Abstract.** The internal-friction (IF) technique is applied to study high-$T_c$ superconducting Bi–Sr–Ca–Cu–O (BSCCO) system cuprates. IF measurements at five different frequencies from $10^{-2}$ to 5 Hz in a temperature range from room temperature to 330°C are carried out. Resonance-type behaviour of IF at a very low frequency ($10^{-2}$ Hz) appears once the sample is slightly deformed ($\epsilon \simeq 10^{-4}$). The IF characteristic of the high-frequency side of the IF-frequency resonance peak is thus determined. It is suggested that the resonance originates from two-dimensional defect vibration, and most probably the domain wall motion caused by dislocation translation.

## 1. Introduction

The techniques of internal friction (IF) and ultrasonic attenuation have been applied to high-temperature superconductors and have become well established in these materials. The ultrasonic technique proves data on the sound velocity (i.e. modulus), and various anomalies in the elastic constants have been found. The technique of very-low-frequency (VLF) IF, on the other hand, avoids some limitations of the ultrasonic technique such as the fairly high temperature of relaxation peaks (and hence the interference of other phase transitions) and low relaxation strength. Anelastic relaxation studies of a single-phase Y–Ba–Cu–O specimen by VLF IF showed a fundamental connection between the migration of oxygen vacancies and the onset of superconductivity (Zhang *et al* 1989, 1990). In this work we apply the VLF IF technique to study a 1:1:1:2 B–Sr–Ca–Cu–O (BSCCO) high-$T_c$ superconductor (HTSC), and a resonance-type IF is confirmed which differs substantially from the relaxation phenomena.

The response behaviour of solid parts to external vibration has been studied extensively in theory and application (Nowick and Berry 1972). IF refers to the mechanical energy loss from an oscillating solid through internal causes. In forced vibration of a solid sample, a resonant peak can be observed when the frequency $\omega$ of an external alternating stress is equal to the characteristic relaxation time $\tau$ of the system. IF, defined as the phase lag of strain behind the stress which is equal to $1/2\pi$ of the fractional energy loss per cycle, can be proved to be just the reciprocal of the commonly used quality factor $Q = f_r/(f_1 - f_2)$, where $f_r$ is the resonance frequency and $f_1$ and $f_2$ are two half-power points of the peak. Hence, the IF is denoted as $Q^{-1}$. To obtain the resonance peak, we prefer to vary the relaxation time $\tau$ while keeping the frequency $f$ constant rather than varying $f$ and keeping $\tau$ constant, since frequency scanning is more difficult. Thermal activation is

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commonly adopted to vary $\tau$, and $Q^{-1}$ versus $T$ plots at a certain frequency are measured in the IF experiment. The anelastic relaxation peak at temperature $T_p$ will shift to higher $T$ as the frequency $f$ increases (in $Q^{-1}$ versus $T$ measurements, only a few $f$-values are needed). The temperature dependence of $\tau$ explains the appearance of $Q^{-1}$ versus $T$ peaks. Furthermore, there is another type of IF behaviour: damping resonance. A resonance IF peak occurs at a definite frequency but disappears when $f$ changes. The resonance absorption of external mechanical vibration owing to microscopic mechanisms has been investigated in only a limited way. Two phenomena were studied: the absorption of acoustic vibration in stress-induced martensitic phase transformation (Mercier *et al* 1979) and the resonance absorption IF caused by dislocation-string vibration (Stern and Granato 1962). They occur in acoustic frequency ($10^2$–$10^3$ Hz) and megahertz frequency ($10^6$–$10^7$ Hz) ranges and are manifest in the form of $Q^{-1}$ versus $f$ peaks. There is no report on resonance absorption IF at VLFs ($10^{-2}$–$10^{-3}$ Hz). In this work we report VLF resonance-type IF in 1:1:1:2 BSCCO superconductors. We suggest that it originates from two-dimensional defects.

## 2. Probable origin of VLF resonance IF

The vibrating-string model serves as the theoretical basis for studying the one-dimensional resonance absorption process (Koehler 1952, Granato and Lücke 1956). A line (e.g. a dislocation string) or an area element defect of length $l$ and effective mass $m$, pinned at its ends or corners, undergoes forced one-dimensional vibration with damping coefficient $B$ and restoring force $\gamma$. The equation of motion in the displacement $u(y, t)$ is

$$m\ddot{u} + B\dot{u} - \gamma\left(\partial^2 u/\partial y^2\right) = \sigma_0 \exp(\mathrm{i}\omega t) \tag{1}$$

with boundary conditions $u(0, t) = u(l, t) = 0$, where $\sigma_0 \exp(\mathrm{i}\omega t)$ is the driving force. The length $l$ of the line or area element is the 'free' length of the microvibration. It is readily obtained that the resonance frequency $\omega_0$ (similar to a resonant mechanical system) and the apparent relaxation time $\tau$ are

$$\omega_0^2 \propto \gamma/ml \qquad \tau \propto Bl/\gamma \tag{2}$$

and a numerical factor as the proportional constant appears on the right-hand side of each equation. If the damping constant is large such that $\tau > 1/\omega_0$ and $\tau$ follows the Arrhenius relationship, relaxation-type behaviour occurs. On the other hand, if $\tau \leqslant 1/\omega_0$, the system shows resonance-type behaviour and a large IF peak will appear at $\omega \simeq \omega_0$. Let us substitute the dislocation data into equation (2), i.e. $\gamma \simeq \frac{1}{2}Gb^2$, $m \simeq Db^2 l$ and $l \simeq 10^{-6}$ m where $G$, $b$ and $D$ are the shear modulus of the material (about $10^2$ GPa), the magnitude of the Burgers vector of the dislocation and the material density (taken as $5\times10^3$ kg m$^{-3}$), respectively. We obtain $\omega_0 > 10^9$ Hz. It shows that the resonance absorption of a one-dimensional defect occurs at a fairly high frequency.

The vibrating-membrane model as a generalization of the two-dimensional case, in which the Laplacian operator $\nabla^2$ instead of $\partial^2/\partial y^2$ is used, can result in a lower resonance frequency. Equation (2) is applicable with $l$ as the dimension of each side of an area element pinned at its corners. The restoring force $\gamma$ can be very small for two-dimensional defects and its effective mass fairly large. Suppose that the effective thickness of a defect interface is about $10^3$ Å and the edge length is still 1 $\mu$m. Then the mass is $10^7$ times that of the dislocation string. If the restoring force is much smaller than that in the one-dimensional case, e.g. only $10^{-7}$ of that of a dislocation string, the resonance frequency $\omega_0$ drops to $10^2$–$10^3$ Hz, but is still not as low as $10^{-1}$–$10^{-2}$ Hz.

Area defects with an unconstrained perimeter can reach a low resonance frequency which pinned two-dimensional defects cannot. Its resonance frequency can be expressed approximately as

$$\omega_0^2 \propto \gamma/m. \tag{3}$$

Then $\omega_0$ drops to $10^{-1}$ Hz under similar conditions. To reach an even lower resonance frequency $f = 0.01$ Hz ($\omega_0^2 \simeq 4 \times 10^{-3}$), it is necessary for $\gamma$ to be smaller (about $10^{-9}$–$10^{-10}$ times the restoring force of a dislocation string). $\gamma$ of the area-defect motion induced by perimeter vibration cannot have this magnitude. However, if the interface motion is caused by translational motion of perimeter, the restoring force could be extremely small. For example, domain wall (DW) motion is generated from the translational motion of dislocations, e.g. the perimeter of an antiphase DW. The restoring force results from interaction between the moving DW and other defects so that $\gamma \ll \frac{1}{2}Gb^2$. Even for a reasonable DW thickness, e.g. 10 Å, it is still possible for resonance absorption to occur at $10^{-2}$ Hz.

HTSC cuprates all possess a layer structure, in which metal–oxygen layers are stacked in a variety of sequences with metal atoms often in unusual coordinations. HTSC cuprates can hence support a wide variety of structural defects (Jorgensen 1991). In addition to various point defects, there exist numerous two-dimensional defects such as stacking faults and DWs (orientation DWs and antiphase DWs). Resonance absorption due to these two-dimensional defects might occur readily at VLFs. In fact, resonance-type IF peaks do arise at about $10^{-2}$ Hz in Bi-type HTSCs. Their appearance conditions and characteristics will be discussed in the following section.

## 3. Experimentation

$BiSrCaCu_2O_y$ powder was prepared using the solid state reaction method from a mixture of $Bi_2O_3$ (CP), $SrCO_3$ (AR), $CaCO_3$ (AR) and CuO (AR) in a pre-determined ratio (Bi:Sr:Ca:Cu atomic ratio, 1:1:1:2) after ball grinding for 12 h, pre-heating at 820 °C for 3 h and cooling inside the furnace. The powder was ground again and compressed under 150 MPa into rectangular pieces of size 1.5 mm × 6.0 mm × 60 mm. They were sintered at 850 °C for 60 h and then quenched in liquid nitrogen. Characterization of the specimens follows similar procedures to those mentioned previously (see, e.g., Zeng *et al* (1989)). The four-terminal technique for resistivity measurement was adopted to determine the $T_c$ of samples in the experiment. An x-ray diffractometer (Rigaku Max-II) was used for structure identification and a differential scanning calorimeter (Perkin–Elmer DSC-IIc) for thermal analysis with a heating rate of 5 K $min^{-1}$. A multifunction IF inverted torsion pendulum system (MFIFA-I, Institute of Solid State Physics, Academia Sinica) was used for the IF measurements; this is computer controlled and can carry out simultaneous measurements of IF $Q^{-1}$ and shear modulus $G$ versus $T$ for several frequencies (five different frequencies in the range $10^{-2}$–5 Hz in our experiment) in forced-vibration mode. The precision of $Q^{-1}$ is better than 1% and that of the relative values of shear modulus is better than 1% too.

The IF was measured in the temperature range from room temperature to 330 °C under a low pressure of 10 Torr. The measurements obtained during heating were repeated each time that the sample was cooled to room temperature. Resistivity measurements were carried out to monitor the conduction of the sample before and after these thermal cycles. X-ray diffraction was also used to check the structural changes of the sample.

## 4. Results

Figure 1 shows the electrical behaviour ($R$ versus $T$) of a Bi-type superconductor sample: curve a for the as-sintered sample and curve b after the eighth cycle. Before the thermal test, the onset temperature is 98 K and the resistance drops fairly slowly to the $T_c(R = 0)$ of 77.5 K with further temperature reduction. After 8 cycles between room temperature and 330 °C under a pressure of 10 Torr, the sample resistance increases in general. A gentle transition starts at a similar temperature and, after 80 K, the resistance drops precipitously to a seventh of the 100 K resistance at the liquid-nitrogen temperature but is still not zero. It shows that the superconductivity of the BSCCO sample deteriorates after thermal cycling.

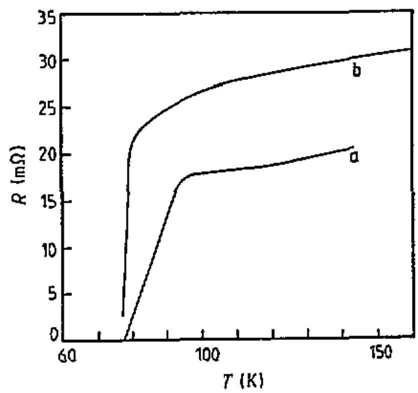


**Figure 1.** Resistance versus temperature curves of 1:1:1:2 BSCCO samples: curve a, as-sintered sample; curve b, the sample after the eighth cycle.

The x-ray diffraction patterns of the samples used for figure 1, curves a and b, are shown in figures 2($a$) and 2($b$), respectively. Although the sample illumination area and the instrument conditions are more or less similar, the diffraction intensity decreases in general, with only a few exceptions: intensified peaks (e.g. (008)) or markedly weakened peaks (e.g. (200) and (113)). Furthermore, the interplanar separation decreases and the crystal volume contracts. The least-squares fitting of the experimental data shows that the volume contraction ratios after 3 cycles and 8 cycles are 4.4% and 6.6%, respectively. This relates to the increase in oxygen deficiency owing to oxygen desorption under low pressures.

Figure 3 shows the temperature dependence of IF and $G$ of the sample, when heated in steps of 5 K in the first cycle, at five frequencies 0.010 Hz, 0.047 Hz, 0.224 Hz, 1.06 Hz and 5.00 Hz from top to bottom, respectively. It is similar to previous results (Zeng *et al* 1989). Below 150 °C, there is a spreading relaxation peak which shifts to higher temperatures with increasing frequency. The activation energy of this relaxation process is obtained from the Arrhenius graphs as 1.10±0.05 eV. The IF $Q^{-1}$ decreases with increasing frequency, which may suggest that the IF behaviour in the measuring temperature region is viscoelastic (Aklonis and Macknight 1983). However, the $Q^{-1}$ versus $T$ curve is independent of heating rate. Hence, the IF behaves more as the high-frequency side (HFS) of a $Q^{-1}$ versus $f$ resonance peak with $\omega_0 \leqslant 10^{-2}$ Hz. Repeated measurements in further thermal cycling would give similar results except that the low-temperature IF is slightly lowered and the high-temperature IF slightly increases (Zeng *et al* 1989).

However, once the sample is deformed slightly ($\epsilon$ of the order of $10^{-4}$) owing to bending or twisting, or even from unloading and refitting, its IF behaviour changes drastically. The sample tested in the first cycle was unloaded, deformed for about $10^{-4}$ and refitted; then the

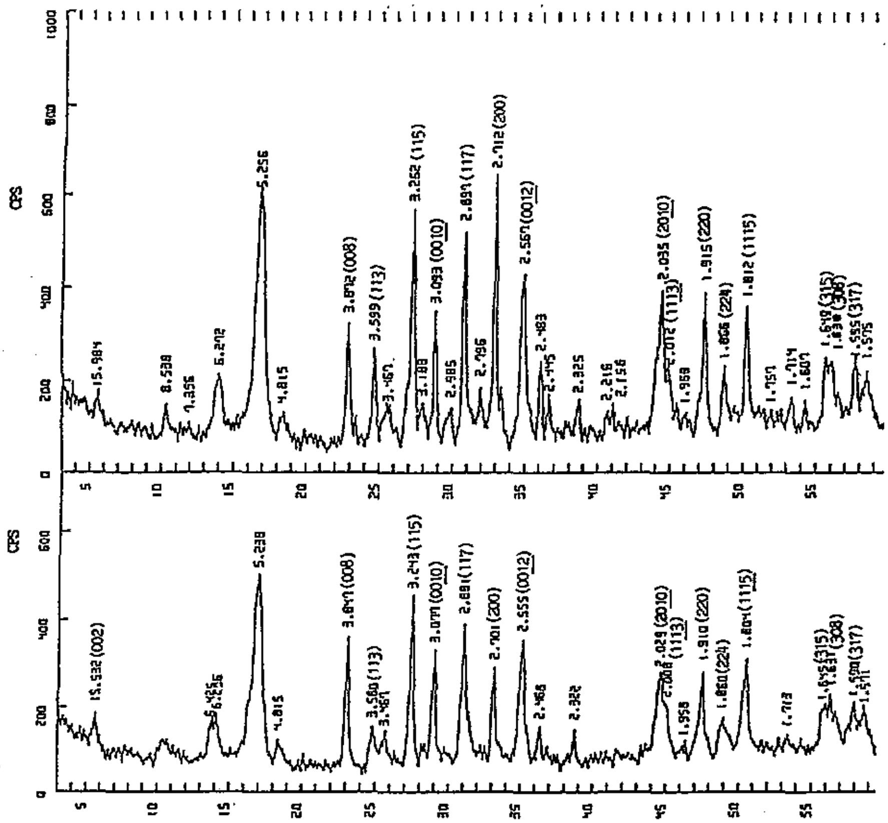

**Figure 2.** X-ray diffraction patterns of 1:1:1:2 BSCCO samples: (*a*) as sintered; (*b*) after the eighth cycle.

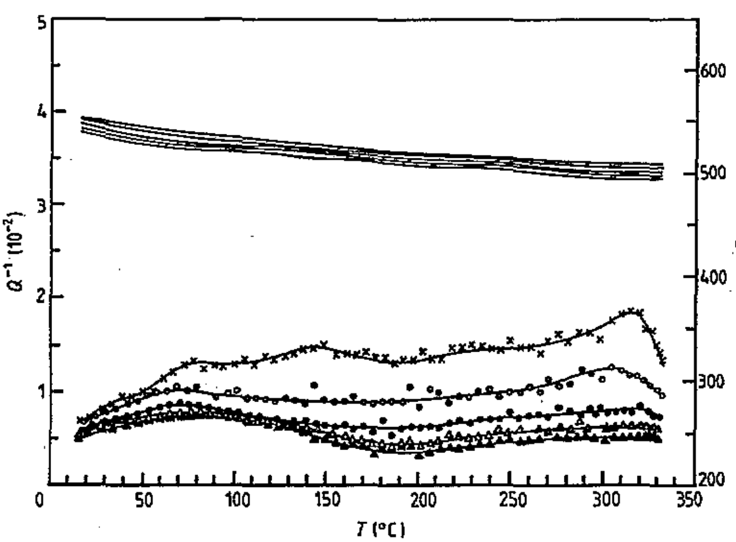

**Figure 3.** *IF and relative shear modulus versus temperature in the first heating:* ×, *0.010 Hz;* ○, 0.047 Hz; ●, 0.224 Hz; △, 1.06 Hz; ▲, 5.00 Hz.

IF was measured in further cycles. Figures 4(*a*), 4(*b*) and 4(*c*) shows the temperature dependences of the IF and $G$ in the second, fourth and fifth step-heating procedures, respectively. Figures 4(*a*) and 4(*b*) are the $Q^{-1}$ versus $T$ and $G$ versus $T$ curves at the same five frequencies as mentioned above (0.01 Hz or higher). Figure 4(*a*) shows that the broad relaxation IF peak below 150 °C still exists and the $Q^{-1}$ versus $T$ curve is lowered with increasing frequency. However, a unique high IF peak, labelled $A_1$, appears at $f_1$ with a height of about $2.5 \times 10^{-2}$ after background deduction. $A_1$ is reproduced well; in the subsequent third heating, it maintains its height and $T_p$ shifts only from 88 K to 85 K. Thus, to investigate any effects of the testing conditions on $A_1$, the sample was kept at 325 °C in vacuum in the torsion pendulum furnace for 1 h after the third cycle. When the sample had cooled to room temperature and measurements made in the fourth heating, $T_p$ had shifted to 78 °C and the intensity increased to about $3.5 \times 10^{-2}$ (figure 4(*b*)). The results of the fifth heating at $f_1 = 0.001$ Hz, $f_2 = 0.010$ Hz and $f_3 = 0.100$ Hz ($f_1 \leqslant 0.01$ Hz) are shown in figure 4(*c*). The peak temperature $T_p$ shifts back to 83 K and the peak broadens. It further shows the resonance characteristic of the peak $A_1$; it occurs only at $f = 0.010$ Hz in simultaneous three-frequency measurements.

The torsion pendulum system does have an intrinsic frequency but it is larger than 50 Hz at room temperature, far beyond the frequency region of the driving force. It probably excludes the system as the origin of resonance. To confirm the viewpoint that the resonance results from the sample itself, we further unloaded the sample, shortened the sling and refitted it. Figure 5(*a*) shows the results of the IF $Q^{-1}$ in the sixth heating at the same five frequencies from 0.010 to 5.00 Hz. Resonance still occurs at $f = 0.010$ Hz but now three IF peaks appear at 49 °C, 91 °C and 171 °C, labelled $A'_1$, $A'$ and $A_2$, respectively. The $Q^{-1}$ versus $T$ curves at other frequencies vary only slightly and the new tiny features are attributed to additional cold working during the refitting. Measurements in the seventh heating were carried out at lower frequencies (figure 5(*b*)): $f_1 = 5.0\times10^{-3}$ Hz, $f_2 = 0.010$ Hz and $f_3 = 0.020$ Hz. At $f_2$, $A'_1$ and $A_2$ shift to higher temperatures, 65 °C and 188 °C, respectively, and $A'$ disappears completely. $A'_1$ decreases a little in intensity but the intensity of $A_2$ does not vary. At $f_1 = \frac{1}{2}f_2$, $A'_1$ and $A_2$ are still present but with reduced intensity. The peak temperature of $A'_1$ is close to that of $A_1$ and its increment, about 16 °C, is comparable with that of $A_1$, which is about 10 °C. Both $A'_1$ and $A_1$ vary in intensity during thermal cycling. Therefore, $A'_1$ is identified as the previous $A_1$. $A'$ is temporary and $A_2$ is newly developed. At a higher frequency $f_3$ ($2f_2$), no peak is observed. This shows that the bandwidth of the resonance peak is less than 0.01 Hz. In the eighth thermal cycling, $A_1$ disappears and $A_2$ has a similar intensity. These results show that the resonance frequency 0.01 Hz is a characteristic of the deformed sample.

Resistive and x-ray measurements are performed when the thermal measurements are complete (figures 1(*b*) and 2(*b*)). Other measurements such as thermal expansion measurements (precision $10^{-3}$ cm) are also carried out to see whether they provide complementary information. The curve of linear expansion $\Delta l$ ($l = 60$ mm) versus $T$ (figure 6) shows three bends at 70, 100 and 130 °C, suggesting that there are some changes at these temperatures. The results of DSC measurements are not plotted here; there is no abnormal feature from room temperature to 350 °C and all heat versus $T$ curves of the sample before and after thermal cycles show a very broad endothermic peak at around 400 °C.

To investigate the effect of deformation relief, the sample was kept in a dessicator for 2 months and IF measurements were then carried out at the same five frequencies as before. Figure 7 shows the results. It can be seen that the resonance peak disappears and the covered relaxation peak emerges again. All $Q^{-1}$ versus $T$ curves shift to lower intensities when

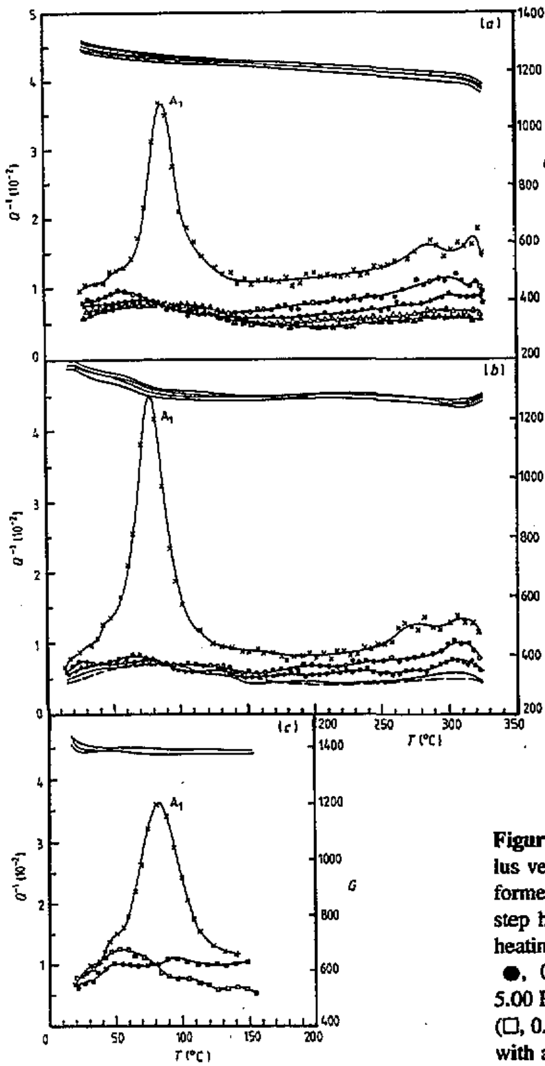


**Figure 4.** IF and relative shear modulus versus temperature for a slightly deformed sample: (*a*) during the second step heating; (*b*) during the fourth step heating (×, 0.010 Hz; ○, 0.047 Hz; ●, 0.224 Hz; ——, 1.06 Hz; - - - -, 5.00 Hz; (*c*) during the fifth step heating (□, 0.001 Hz; ×, 0.010 Hz; ■, 0.10 Hz) with a smaller temperature region.

the frequency increases, the same behaviour as previously. A feature appears in the range 140–210 °C, the temperature region of $A_2$. It shifts to a lower temperature with increasing frequency. It is associated with the sample history. We call it the resonance remnant.

## 5. Discussion

Experiments show that the 1:1:1:2 BSCCO HTSC can generate a resonance absorption type of IF peak at about $10^{-2}$ Hz when being deformed slightly. The IF intensity diminishes with increasing frequency when $f \geqslant 10^{-2}$ Hz (figures 3 and 4(*b*)). Hence, we can take the $Q^{-1}$ versus $T$ curves there as the HFS of a $Q^{-1}$ versus $f$ resonance peak. HTSCs at $T > T_c$ all possess this characteristic. The possibility of viscoelasticity is ruled out since the IF measured in HTSCs is independent of heating rate.

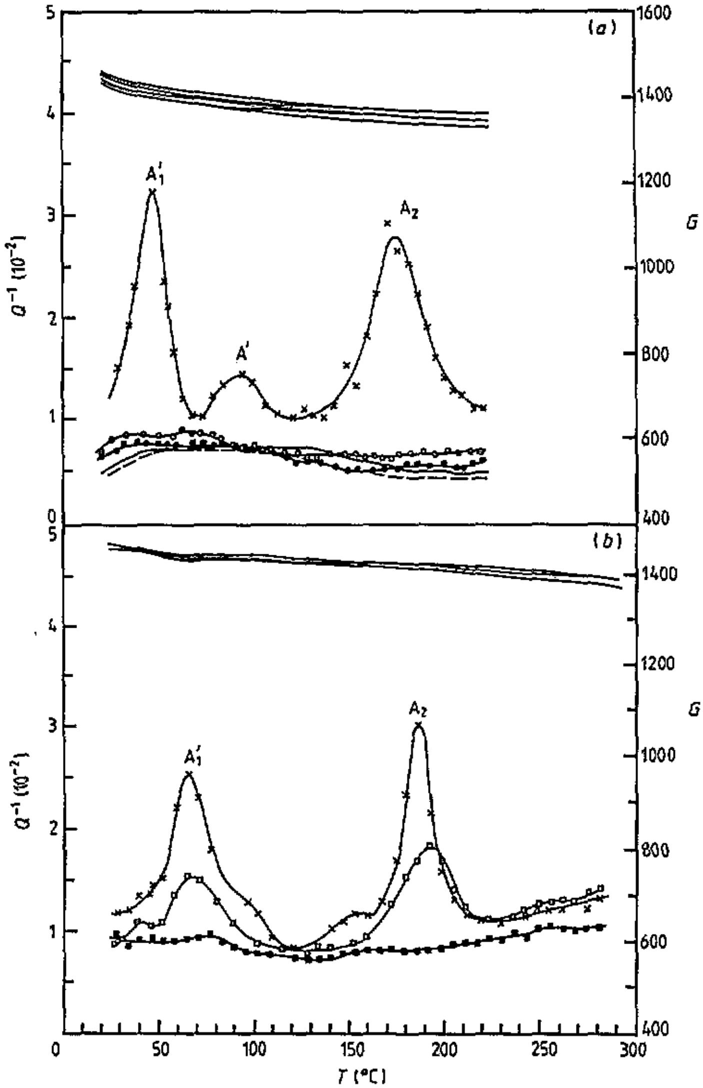


**Figure 5.** IF and relative shear modulus versus temperature for a shortened-and-refitted sample: (*a*) during the sixth step heating; (*b*) during the seventh heating with lower frequencies (□, 0.005 Hz; ×, 0.010 Hz; ■, 0.02 Hz; ○, 0.047 Hz; ●, 0.224 Hz; ——, 1.06 Hz, - - - -, 5.00 Hz).

Slight cold work is an essential condition for the generation of a resonance IF peak. It supports the inference that the resonance concerns DW motion caused by dislocation motion (see also Hazen *et al* (1988)). VLF resonance absorption of HTSCs may occur if 'there are flat DWs with loose constraints produced by essential and suitable deformation'.

All HTSCs possess the characteristic HFS of a resonance peak. Zhang *et al* (1992) show that on applying a magnetic field at $T > T_c$ (the normal state) the frequency dependence of the low-frequency IF and the HFS characteristic do not change but, when $T$ is lowered below

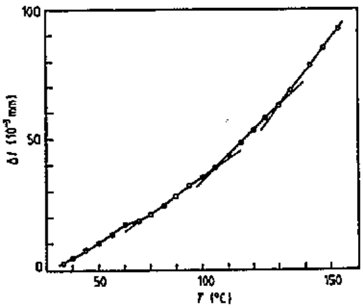


**Figure 6.** Linear expansion versus temperature curve of the sample after the eighth cycle.

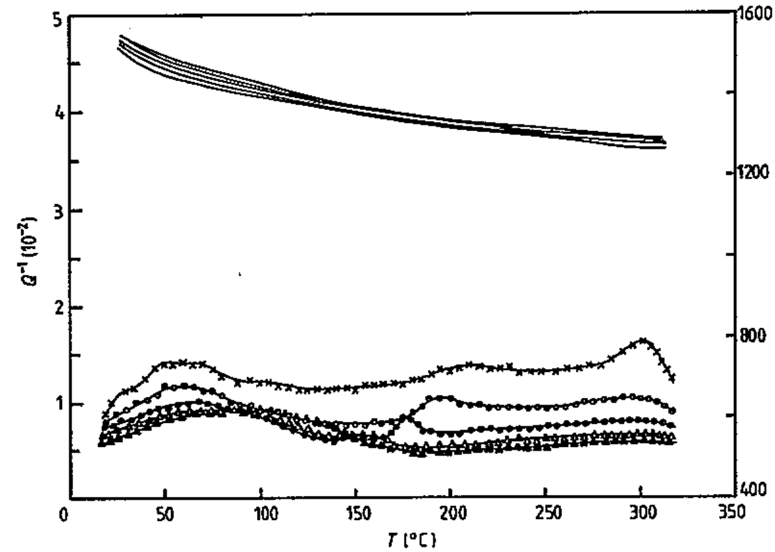


**Figure 7.** IF and relative shear modulus versus temperature for a sample with deformation relief (all symbols are the same as in figure 3).

$T_c$, the HFS characteristic reverts to the low-frequency side characteristic, i.e. $Q^{-1}$ increases with increasing frequency. If this change is related to a resonance peak, it implies that the resonance frequency $\omega_0$ increases suddenly in the superconducting transition, which reflects the sudden change in restoring force $\gamma$ of DWs. Then the sudden increase in restoring force of DW motion is associated with the appearance of superconducting carriers.

The resonance $Q^{-1}$ versus $T$ peak occurs as a result of the temperature dependence of the restoring force $\gamma$, which is related to $\omega_0$ since $\omega_0^2 \propto \gamma/m$. A definite applied frequency $\omega$ could only equal $\omega_0$ at a certain temperature $T_0$ but fails to match $\omega_0$ otherwise. A $Q^{-1}$ versus $T$ peak such as those in our results thus appears. Furthermore, if the bandwidth of the resonances $Q^{-1}$ versus $f$ peak becomes as wide as the resonance remnant, an 'abnormal' frequency dependence of the $Q^{-1}$ versus $T$ peak temperature $T_p$ occurs; $T_p$ decreases with increasing applied frequency $\omega$. This in fact shows that $\gamma$ decreases with increasing $T$.

The single peak $A_1$ splits into three peaks after further deformation but the resonance frequency, 0.01 Hz, does not vary. This implies presumably that there are newly freed DWs with different orientations and properties, and the DW density changes.

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