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To cite this article: G. D'Anna *et al* 1990 *EPL* **13** 465

EUROPHYSICS LETTERS 1 November 1990
Europhys. Lett., 13 (5), pp. 465-471 (1990)

Dynamic and Static Mechanical Measurements of Flux-Lattice Softening and Associated Hysteretic Depinning Dissipation Peak in $YBa_2Cu_3O_x$ Ceramic.

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(received 22 June 1990; accepted in final form 30 August 1990)

PACS. 74.60G – Flux pinning, flux motion, fluxon defect interactions.
PACS. 62.40 – Anelasticity, internal friction and mechanical resonances (inc. stress relaxation).

Abstract. – We have studied flux-lattice dissipation in $YBa_2Cu_3O_x$ ceramic using a forced inverted torsional pendulum. Reversible restoring force variations during temperature sweep suggest that flux lines experience a «hard-soft» transition between 50 K and T_c. Measurements of internal friction and modulus change as a function of the applied stress show a typical depinning behaviour. This dissipation peak during the temperature sweep is understood as a hysteretic depinning in which the critical force depends upon the penetration depth.

Introduction. – In «ideal» defect-free type-II superconductors, the flux line structure will begin to move whenever a force acts on it. The motion of vortex results in a dissipative effect and when the power dissipation exceeds a certain limit, a thermomagnetic instability builds up and superconductivity breaks down completely. In particular, since a transport current is equivalent to a force on the flux line system, ideal type-II superconductors are not able to carry transport currents without losses. The presence of defects, acting as pinning centres for the flux line, prevents this dissipative motion. However, a sufficient pin of the flux line structure can occur only after the formation of a flux-lattice, for which a collective pinning requires just a small number of defects. As pointed out by other authors [1-3], experiments in high-T_c materials as YBaCuO or BiSrCaCuO indicate a tendency of the flux line assembly to develop a fluidlike character below T_c instead of forming a regular lattice; in this condition the hope of obtaining a high critical current for technologic applications is obviously very small. Lately a considerable effort is made in order to understand which mechanisms are involved. Experiments using installations permitting the analysis of mechanical properties of flux-lattice go into the same direction.

In mechanical measurements on flux-lattice, via the interaction of the vortex with the material (pinning mechanism) the apparent modulus of the vortex lattice shows an important decrease and is accompanied by a dissipation peak. The temperature T_P at the peak can be reported in an H-T phase diagram; while the dependence of T_P on the external applied magnetic field has been intensively investigated [3-7], its dependence on other parameters as the frequency or the applied stress has not been explored. The dependence of the flux-lattice mechanical behaviour upon frequency and stress will, however, give important indications.

For the investigation of anelastic mechanical phenomena in solids, a great number of dynamic apparatuses have been utilized providing a frequency range which starts at about 10^{-5} Hz and which terminates at about 100 GHz (see for example [8]). In the last years many of these methods have been recuperated in order to investigate anelastic phenomena of flux-lattice in type-II superconductors; however only in a frequency range which starts at about 100 Hz (resonant pendulum-type [6]) and which terminates at about 10 kHz (high-Q oscillators [9, 3] and vibrating reed [5]). In the low-frequency range, typically from 10^{-5} Hz to 100 Hz, the subresonance method is used. In principle, such an experiment is very simple to carry out: a specimen is set into forced vibration at different set frequencies and the anelastic properties are found by noting the extent to which the strain lags behind the applied stress (phase angle ϕ) as well as their amplitude ratio (peak-stress on peak-strain σ_0/ε_0).

The results shown in this paper are obtained by the first instrument that permits to investigate anelastic mechanical properties of flux-lattice in type-II superconductors in the low-frequency (10^{-5} Hz to 10 Hz) and in the low-temperature (4.7 K to 500 K) range or as a function of stress [10].

Mechanical characteristics of the apparatus. – A schematic description of the apparatus is given in fig. 1. In practice the specimen under test is submitted to a harmonic torque in an inverted torsional pendulum. Torsional vibrations are obtained by the application of a low-frequency harmonic current to a pair of coils. In contrast to the free-vibration methods in which the strain is generally constant, the stress is strictly fixed by the current imposed in

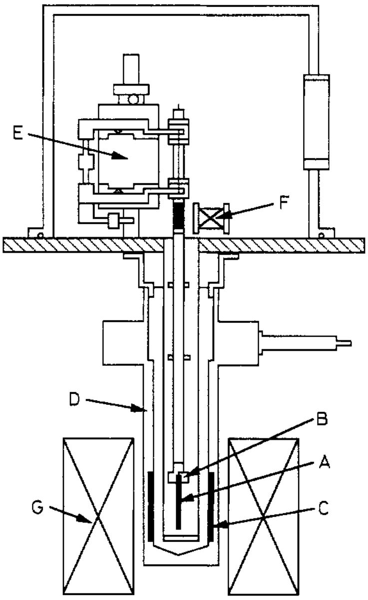

Fig. 1. – Sketch of forced inverted torsional pendulum apparatus. A: superconductor specimen; B: grip; C: furnace; D: cryostat; E: suspension system; F: excitation coils; G: external magnetic field.

the coils. The natural frequency of vibration of the complete system (typically 100 Hz) is set high enough to permit neglect of the inertial effect in the low-frequency range. The phase angle and the ratio of peak values between the current in the coils, proportional to the applied stress, and the specimen displacement, proportional to the specimen strain, is then measured as a function of either frequency, temperature, applied stress or time. The tangent of the phase angle gives directly the internal friction Q^{-1} and the peak values' ratio is proportional to the specimen elastic modulus change ΔE.

Superconductor specimens are $(1 \times 4 \times 40)\,\mathrm{mm}^3$ bars held only by the upper grip, the lower side of the specimen remains free. An external electromagnet permits to create a uniform magnetic field, perpendicular to the rotation axis, up to about 2000 Gauss in the sample region. The superconducting characteristics manifest themselves by a restoring force tending to align the sample to the static magnetic field. The superconducting samples can be regarded as completely rigid; thus the deformation of the flux-lattice is a flexion, rather than a torsion as in conventional pendulums for solids. The strain of flux-lattice can be varied from about $2.5 \cdot 10^{-4}$ to $8 \cdot 10^{-2}$, *i.e.* from relatively low amplitudes, avoiding nonlinear effects, to high ones.

In addition to dynamic quantities, *i.e.* internal friction and modulus change, a static measurement is possible with the same apparatus, by the detection of the «zero point». Zero point is the position, given by the displacement detector, in the absence of periodic stress. The specimen is free at its low end. Thus the small suspension tungsten wire (fig. 1) is used to measure the torque acting on the specimen: to this end, in the absence of magnetic field, an angle (typically of about 5 degrees) is imposed between the specimen and the direction of the applied field. In the flux-lattice mechanical experiments, the zero point is a measure of the restoring force tending to align the sample to the static external magnetic field; this quantity is a direct measure of the specimen magnetization and the sensitivity is high enough to compare our measurements with the results obtained by other methods (see for example [11]).

Results. – We next show the results in a sample of $YBa_2Cu_3O_x$ superconducting ceramic prepared by conventional processing from Y_2O_3, $BaCO_3$ and CuO high-purity raw materials. The raw materials were mixed by wet milling with agate media, calcined at 1173 K for 24 h

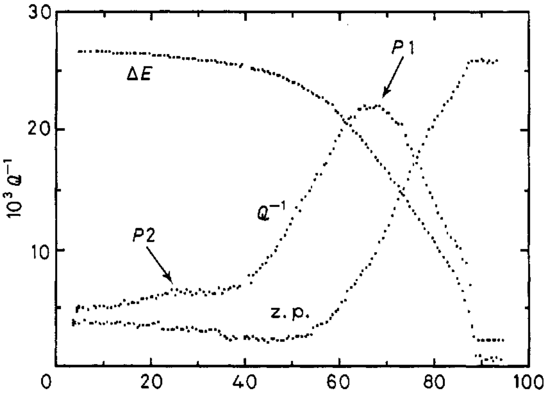

Fig. 2. – The graphs report typical internal friction, modulus change and zero point as a function of temperature during a 0.5 K/min heating at 1 Hz and 1800 G from 4.7 K to 100 K. (For modulus change and zero-point the units are not reported.) The critical temperature is $T_c = 92.5$ K measured by resistivity.

and remilled in acetone to avoid water reaction. Ceramic rectangular pieces were sintered at 1303 K for 24 h in oxygen and annealed at 673 K for 48 h. They are monophasic as seen by room-temperature X-ray measurements with a T_c value of 92.5 K. Density was 5.9 g/cm^3 with considerable open porosity. The result of a typical measurement at fixed magnetic field, frequency and effective stress, as a function of temperature, is shown in fig. 2. The internal-friction spectrum shows an abrupt increase 3 degrees below the transition temperature, followed by an important dissipation peak (P1); another small peak (P2) appears at about 30 K (the small P2 peak grows after a zero-field cooling and a high-temperature increase velocity). It is of interest to note the relatively high internal friction below the peak; this is an indication that even at low temperature the flux lattice shows some dissipative mechanisms and that it is not fully pinned [12]. Modulus change shows the expected decrease during temperature sweep. The static zero-point measurement indicates that at about 50 K, the restoring force tending to align the sample to the magnetic field starts to decrease; the restoring force change ΔF, defined as the difference between the measured «zero-point» above T_c and at low temperature (50 K), depends only on the applied field (ΔF depends linearly on the applied field between the experimentally accessible values from 300 to 2000 G) and is completely reversible between increasing and decreasing temperature. However, the restoring force begins to decrease at about 50 K for all accessible magnetic fields, in the limit of our sensitivity.

The results in fig. 3 show the internal friction and modulus change as a function of temperature for three different applied stresses. The temperature T_{P1} of the dissipation peak depends strongly on this parameter, as shown by its shift at higher temperatures with decreasing stress; at the same time the peak height increases. The softening occurs more and more abruptly as the stress decreases; this is shown by the modulus change spectra. From this kind of measurement, the H-T «phase diagram» can be traced by reporting the magnetic field as a function of the dissipation peak temperature T_{P1}; fig. 4 shows these diagrams for five different applied stresses, with the linear extrapolation at zero field. It is obvious that these «transition» lines depend strongly upon the stress [4]; in particular the slope seems to increase at low stress. Figure 5 shows the T_{P1} as a function of the applied stress, for three different magnetic fields, with an exponential interpolation.

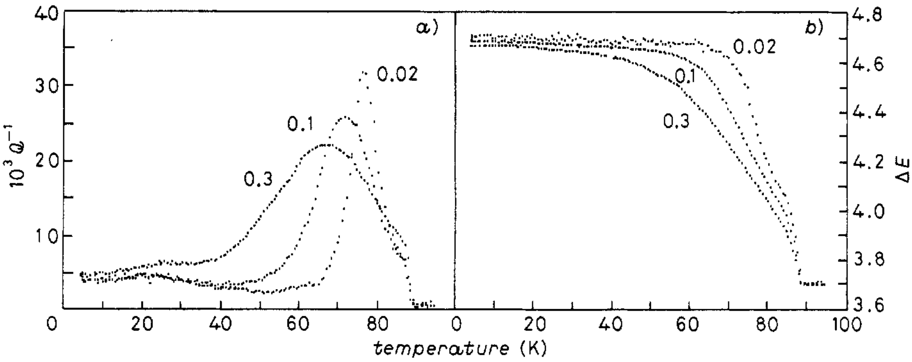

Fig. 3. – Internal friction (*a*)) and modulus change (*b*)) as a function of temperature for three applied stresses (the values on the graph indicate the current, in A, in the excitation coils). The three curves have been measured during a 0.5 K/min heating at 1 Hz and 1800 G.

Figure 6 shows the internal friction and the modulus change as a function of the applied stress for different temperatures, at a fixed magnetic field and frequency; the lowest stress

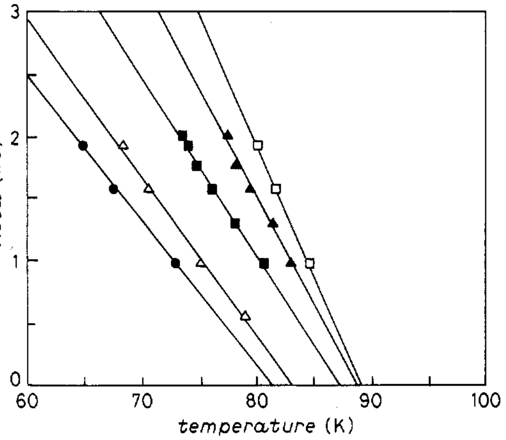

Fig. 4.

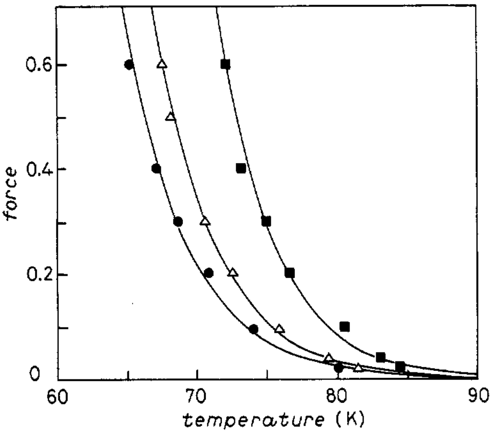

Fig. 5.

Fig. 4. – The phase diagram for flux-lattice P1 peak in YBaCuO for different effective stresses of the applied harmonic excitation signal: □ 0.02, ▲ 0.04, ■ 0.1, △ 0.3, ● 0.6.

Fig. 5. – The dissipation peak temperature T_{P1} as a function of applied stress in YBaCuO, for three magnetic fields: ■ 0.97 kG, △ 1.57 kG, ● 1.93 kG.

corresponds to a mean strain of about $5 \cdot 10^{-4}$, and for the highest stress, the strain is about 10^{-2}. The investigation of the frequency dependence of internal friction and modulus change spectra has shown that the dissipation peak temperature T_{P1} does not change appreciably for frequencies going from 10^{-3} Hz to 10 Hz.

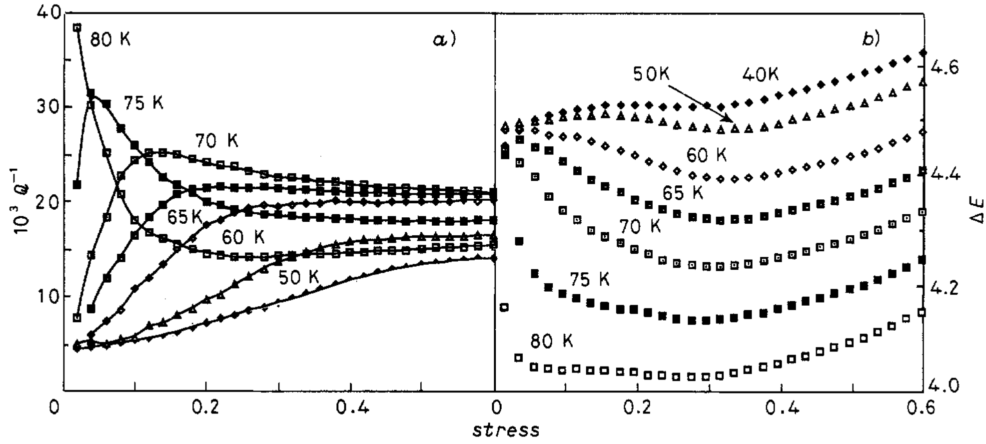

Fig. 6. – Internal friction (*a*)) and modulus change (*b*)) as a function of applied stress for different temperatures at a fixed 1800 G field and 1 Hz excitation frequency.

Discussion. – From the experimental results shown above, some interesting features of the flux-lattice softening picture can be derived. First, the static zero-point measurements are in favour of the concept that flux lines manifest an important increase in their line tension between the transition temperature and a temperature below the dissipation peak, typically 50 K, at which the restoring force becomes constant. Zero-point measurements are obtained by a deformation of the flux-lattice bundle; in our experimental installation, the superconducting ceramic sample can be regarded as completely rigid and thus the deformation of the flux-lattice is a flexion. The experimental results indicate that just at the temperature of the abrupt increase in internal friction, the restoring force is zero. This suggests that the flux lines are «soft» and that they are curved at this temperature. With decreasing temperature, the line tension of flux lines increases and at about 50 K they become «hard»; the process is completely reversible with temperature and thus this effect cannot be due only to a change in pinning force. The geometry of the experiment is thus in favour of 3-dimensional softening rather than of 2-dimensional melting [1, 3, 13]. SQUID measurements in YBaCuO [11], in field-cooled specimens, show a magnetization rate $\mathrm{d}M/\mathrm{d}t$ different from zero in the same temperature range ($50\,\mathrm{K} \div T_c$) as in our results. The linear dependence of ΔF on the field indicates that the work done by the line tension in order to shift the specimen, between T_c and 50 K, is exactly proportional to B^2 [5].

The second interesting point follows from the inspection of the internal-friction measurements as a function of applied stress (fig. 6) as well as a function of temperature (fig. 3); they show a typical depinning behaviour (see for example [8, 14]). However, these curves (*i.e.* fig. 3 and 6) are independent of frequency, in our measurements, in the investigated range of frequencies going from 10^{-3} Hz to 10 Hz. This kind of comportment is in favour of a nonthermal activated mechanism, *i.e.* a hysteretic depinning in which the thermal fluctuation does not aid the breaking-away from pinning centres. If the effect of thermal fluctuations on the breakaway process is not taken into account, the energy dissipated in one complete cycle ΔW, as a function of the applied stress, is zero until breakaway occurs and then jumps to a finite and constant value at the critical stress. The internal friction $Q^{-1} = \Delta W/W$ decreases at higher stress since W continues to increase while ΔW remains constant; the behaviour of our measurements as a function of the stress (fig. 6) is in accord with this picture. For a hysteretic process, however, the critical stress does not depend upon the temperature.

In our picture of hysteretic depinning and softening, as a working hypothesis, the temperature dependence of the critical stress is understood as a consequence of the penetration depth $\lambda(T)$ increasing with the temperature, which diverges as $(1 - T/T_c)^{-1/2}$. A flux line consists of super-electrons moving with a certain density and velocity distribution around the centre of the line. For materials with high Ginzburg-Landau parameters ($\kappa \gg 1$) and in the field range $H_{c1} < H \ll H_{c2}$, the London model is valid as a good approximation; thus the vortex current can be seen as a layer with a typical depth given by λ [15, 16]. This means that the force for extracting a small «pinning centre» (*i.e.* with an interaction range smaller than the penetration depth) from the flux core will be experienced by the layer of super-electrons; this critical force must depend strongly on the layer depth. These «pinning centres» are probably oriented intrinsic defects parallel to the Cu-O planes, as shown by the different results of the internal friction as a function of the orientation in single-crystal experiments [3]; the anisotropy of the material is of considerable importance for the description of flux-lattice dynamics in high-T_c superconductors [1]. Because the critical stress depends upon the temperature via the penetration length, the measurement of internal friction as a function of temperature shows a dissipation peak; a measurement as a function of frequency, at constant temperature, does not show any dissipation peak, as confirmed in our experiment.

At the same time the vortex current acts as a surface tension for the flux line via its kinetic energy; the increase in layer depth corresponds to a decrease of surface tension, or globally to a decrease of flux line tension. The maximum of internal friction as a function of the applied stress is proportional to the inverse of the line tension in a depinning model [8, 14]; thus at high temperature, *i.e.* low line tension, this maximum is higher, as shown in fig. 3 and 6. At the moment we have no complete theoretical understanding of the pinning mechanism, however the experimental results shown in this paper are a starting point for further development.

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