Vibrating-Reed Experiments on Superconducting Suspensions

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The frequency of a vibrating reed made of a superconducting suspension of isolated micrometer-size grains of low- and high-T_c superconductors in a magnetic field *directly* measures the elastic pinning force on the flux line. Four different types of field and temperature dependences of the attenuation caused by moving flux lines are observed and explained. In particular, the shift to lower temperatures of the dissipation peak of small high-T_c grains perfectly agrees with the prediction by thermally activated depinning.

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The physics of vibrating superconductors in the mixed state is a fascinating and, in general, difficult problem. A solvable special case is the superconducting vibrating reed (VR).[1-3] This technique and its variations,[4,5] even as recently reported ultrasonic attenuation,[6,7] use mechanical measurements to provide information on the interaction of the flux-line lattice (FLL) with the atomic lattice via pinning. With the sensitive (low amplitude) VR technique it is possible to measure the elastic pinning force or Labusch parameter $\alpha(B_a,T)$ (B_a is the applied magnetic field and T the temperature), i.e., the curvature of the average pinning potential.[8,9]

In this work we present novel results obtained with micrometer-size superconducting grains in the mixed state embedded in an insulating matrix and performing small rotations relative to B_a. This case is of particular interest for a number of reasons. Two advantages are immediately recognized: There are no limitations on the shape of the superconducting material, and the usually large dissipation[3] by flux jumps is drastically reduced, allowing measurements at very high fields. Since the relative displacement of the FLs in the rotating grains is extremely small, we are able to measure, instead of the usual *hysteretic* dissipation in bulk reeds, purely *viscous* damping. A main result of this paper is that, again due to the small FL displacements relative to the pins, vibrating reeds made of a superconducting suspension *directly* measure the elastic pinning forces. The change in the resonance frequency of the reed with field and temperature is due *only* to the field and temperature dependence of $\alpha(B_a,T)$.

We further show that the FLL dynamics in high-T_c superconductors near and above the depinning temperature T_D is governed by the *diffusion*[10,11] of FLs in agreement with the *thermally assisted flux flow* (TAFF) picture.[12] As a consequence of the diffusive character of TAFF, small grains of a high-T_c superconductor should show a clear *decrease* in T_D as compared with much larger bulk polycrystalline reeds at similar resonance frequencies, because a smaller characteristic diffusion length leads to maximum damping. To our knowledge, the implication of this thermally activated effect in *ceramic* superconductors with decoupled grains has not been recognized yet in the literature.

We have measured granular reeds with both conventional and high-T_c superconductors. $Nb_{66}Ti_{34}$ powder was prepared by arc melting followed by a homogenization heat treatment and powdering after hydrogenation. After dehydrogenation, the powder sample showed a critical temperature of $T_c = 10$ K.[13] For the granular reed an appropriate sieve fraction of the powder was mixed with acrylic powder (Resin 3 by Struers). Thin slabs were cut after thermal polymerization. The high-T_c granular sample was made after mechanical powdering of the ceramic sample of composition $Bi_{1.5}Pb_{0.5}Sr_2$-$CaCu_2O_y$ with $T_c = 70$ K (sample No. 2 of Ref. 14 without any heat treatment). The granular reeds showed approximately Gaussian distributions of the grain dimensions, with $\simeq 37$ μm average grain size and $\simeq 20$ μm standard deviation.

The frequency enhancement of the flexural vibration of a cantilevered superconducting vibrating reed, sufficiently thin and of constant cross section, formally may be ascribed to a *line tension P* exerted on the ends of the reed by the applied field B_a.[1,2] In the following we consider reeds of length l, width w, and thickness d ($l \gg w \gg d$) made of (or containing a suspension of) a superconductor with negligible magnetization. This requires a large Ginzburg-Landau parameter $\kappa \gg 1$, not too small applied field $B_a \gg B_{c1}$, where B_{c1} is the lower critical field, and weak static pinning such that the applied field penetrates almost completely.

In general, one may distinguish four types of line tension pulling at the reed. In cases 1-3 the flux lines are completely pinned *dynamically*, i.e., the very small tilt angle $\varphi \ll 10^{-4}$ of the reed during its flexural vibration does not suffice to unpin the vortices when they tend to stay parallel to B_a. The flux lines thus curve with the reed during the vibration. This means that the internal field stays constant and the reed behaves as if it were in the Meissner state. Then in a *longitudinal* field (case 1) the line tension P_1 is caused by the *complete shielding from the reed's interior* of the small transversal field component $B_a\varphi$, which thus has to flow around the reed like a fluid in laminar flow around a plate. The resulting large stray field causes a line tension

$$P_1 = (\pi w^2/4) B_a^2/\mu_0 . \quad (1)$$

In a *transverse* field (case 2) this shielding does not occur since the small field component $B_a\varphi$ here is *parallel* to the tilted reed (the ac perpendicular component is $\propto \varphi^2$ and therefore negligible). In this case the line tension P_2 is the tilt modulus of the flux-line lattice, $c_{44} = BB_a/\mu_0 \approx B_a^2/\mu_0$, times the cross section of the reed,

$$P_2 = wdB_a^2/\mu_0 . \quad (2)$$

The same line tension P_2 applies also to reeds consisting of isolated superconducting grains for arbitrary field orientation. If the grains are embedded in an isolating matrix with filling factor $f<1$ (case 3), the line tension of the reed is reduced by this factor,

$$P_3 = wdfB_a^2/\mu_0 . \quad (3)$$

The tension P_3 applies to arbitrary shape of sufficiently large grains since our assumption of negligible magnetization assures that $B \approx B_a$; demagnetization effects may thus be disregarded.

Elastic instead of rigid pinning leads to small corrections in cases 1–3 which slightly reduce P_1, P_2, and P_3 and from which the Labusch parameter α may be determined.[1,3]

In case 4, the grains are so small that the flux lines are bound *elastically* to the pinning sites, even if the flux stays parallel to B_a. In this case elastic pinning gives the main contribution to the frequency enhancement with field, in contrast to cases 1–3. The elastic energy density of a vortex lattice displaced by a distance u with respect to the pins is $\alpha u^2/2$. Integrating this over a sphere with radius R using $u(z)=\varphi z$ (B_a along z, sphere centered at $z=0$) the elastic energy is $(\alpha\varphi^2/2)(4\pi/3)R^5/5$. Thus, for small spherical particles embedded with filling factor f in a resin matrix we get a line tension

$$P_4 = wdf\alpha\langle R^5\rangle/5\langle R^3\rangle , \quad (4)$$

where $\langle \cdots \rangle$ denotes the average over all particles. For cubic particles, the factor $\frac{1}{5}$ in P_4 is replaced by $\frac{1}{3}$. Note that $P_4 \propto \alpha$, whereas $P_1 \propto P_2 \propto P_3 \propto B_a^2$.

With P_1 replaced by P_2, P_3, or P_4, the expressions for the frequency enhancement of the reed obtained in Ref. 2 can be applied to any of these cases. In particular, for a small enhancement of the resonance frequency $\nu=\omega/2\pi$ from its field-free value $\nu_0=\omega_0/2\pi$ we get for a reed with small superconducting spheres

$$\omega^2/\omega_0^2 - 1 = (4.65/l^2\rho\omega_0^2)\alpha f g , \quad (5)$$

where ρ is the density and $g=\langle R^5\rangle/5\langle R^3\rangle$ the geometrical factor of the grains.

The condition for the radius of the particles is $R \gg \lambda_{44}$ for case 3 and $R<\lambda_{44}$ for case 4, with $\lambda_{44}=(c_{44}/\alpha)^{1/2}$. This follows from the minimization of the elastic plus tilt energy of the vortex-pin system, which per unit area perpendicular to B and for flux lines of length $2R$ is

$$U = \int_0^R \{\alpha[u(z)]^2 + c_{44}[u'(z)]^2\} dz . \quad (6)$$

The resulting displacement $u(z) = -u(-z)$ of the flux lines with the appropriate boundary conditions $u'(z)=\varphi$ at $|z|=R$ (the flux lines at the surface are parallel to the applied field) and $u(0)=0$ (the z axis is fixed to the material) is

$$u(z) = \varphi\lambda_{44}\frac{\sinh(z/\lambda_{44})}{\cosh(R/\lambda_{44})} . \quad (7)$$

From (7) follow the limits

$$u = \varphi\lambda_{44}\,\mathrm{sgn}(z)\exp[-(R-|z|)/\lambda_{44}]$$

for $R\gg\lambda_{44}$, and $u=\varphi z$ for $R \le \lambda_{44}$ used in (4).

Figure 1 shows the frequency enhancement $\omega^2(B_a) - \omega_0^2$ [$\omega_0 = \omega(B_a=0)$] for *granular* samples with different filling factors $0.025 \le f \le 0.6$ of NbTi alloy versus applied field at constant temperature. In the same figure we show the result for the *bulk* sample of the same composition. At $B_a < 0.8$ T the bulk sample's frequency

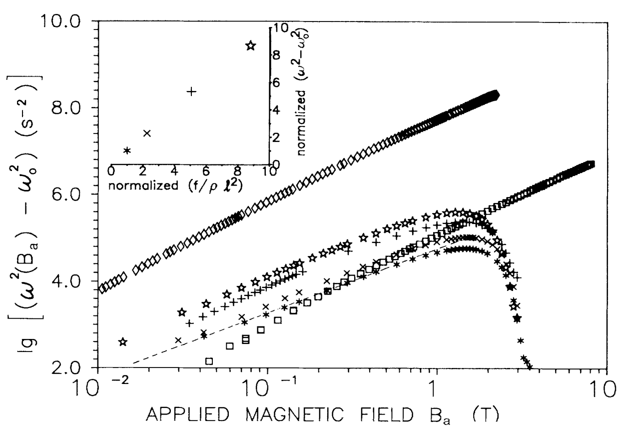

FIG. 1. Deviation of the resonance frequency from its $B_a=0$ value vs applied field at constant temperature. (◇) $Nb_{66}Ti_{34}$ bulk reed at $T=8$ K. Granular reeds with filling factors 0.025 (∗), 0.1 (×), 0.3 (+), 0.6 (★) at $T=8$ K. The resonance frequencies at $B_a=0$ are between 0.2 and 0.6 kHz. The dashed line has a $B^{1.5}$ dependence. (□) High-T_c granular reed ($T_c = 70$ K) with filling factor 0.1, $T=10$ K, and $\omega_0/2\pi = 1.42$ kHz. Inset: Frequency deviation as a function of $f/\rho l^2$ at $B_a=0.1$ T and $T=8$ K normalized to the $f=0.025$ reed.

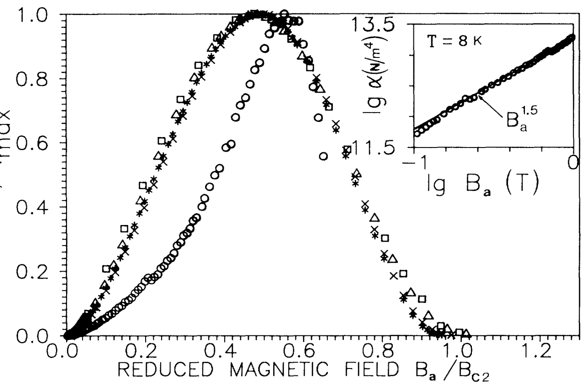

FIG. 2. Normalized Labusch parameter as a function of reduced field. For NbTi grains; at $T=6$ K: $f=0.3$ (*), $f=0.025$ (×) with $B_{c2}\simeq5.8$ T and $\alpha(b=0.5)=7.8\times10^{14}$ N/m^4; at $T=8$ K: $f=0.3$ (□), $f=0.025$ (△) with $B_{c2}\simeq3.0$ T and $\alpha(b=0.5)=7.8\times10^{13}$ N/m^4. For the bulk reed at $T=8$ K (○), $\alpha(b=0.56)=6.5\times10^{13}$ N/m^4. Inset: Double-logarithmic plot of α vs B_a for the bulk reed.

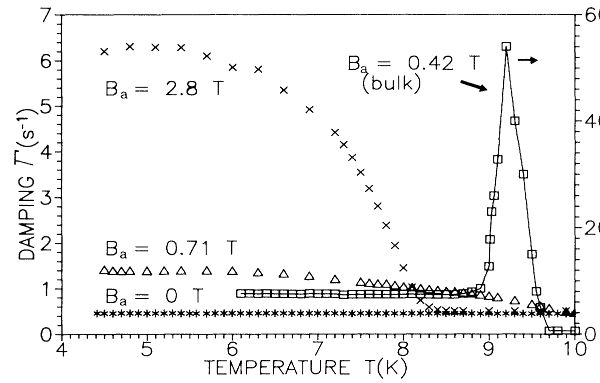

FIG. 3. Temperature dependence of the total damping for the granular NbTi reed with $f=0.1$ at $B_a=0$ T (*), 0.71 T (△, $T_c\simeq9.8$ K), and 2.8 T (×, $T_c\simeq8.3$ K), and for the bulk reed at $B_a=0.42$ T (□, right y axis).

enhancement shows the expected B^2 dependence according to (1); the deviation from this dependence due to nonrigid pinning can be recognized in Fig. 1 at $B>1$ T.

In contrast, the granular samples show an approximate $B_a^{1.5}$ dependence for $B_a<1$ T, independent of the filling factor. This remarkable difference is strong evidence that now the line tension P is not described by cases 1–3, but governed by the Labusch parameter α as expected from (5). Indeed, the absolute value of α and its field dependence obtained for the bulk reed,[15] see Fig. 2, using the standard procedure described in Refs. 2 and 3 are in agreement with those obtained (without adjustable parameter) for the granular samples using (5) with the calculated parameter $g\simeq1.3\times10^{-9}$ m^2. Additional support for this interpretation comes from the fact that the relative frequency increase squared scales with $f/\rho l^2$ as expected from (5) for samples with different dimensions, filling factors, and densities for constant grain-size distribution at fixed field and temperature; see the inset of Fig. 1.

Figure 2 shows the dependences of α on the reduced field $b=B_a/B_{c2}$ at two different temperatures and for two samples with different filling factors and for the bulk reed. As already observed in amorphous $Zr_{70}Pd_{30}$, α scales with field and shows a maximum at $b\simeq0.5$.[3] For the NbTi grains we obtain $\alpha\simeq1.7\times10^{12}B_{c2}(T)^{3.5\pm0.2}$ N m^{-4}T$^{-3.5}$ at $b=0.5$.

In high-T_c superconductors $\alpha\propto B^{2\pm0.2}$.[16,17] This dependence is also observed in the high-T_c granular sample, Fig. 1. For this granular sample we obtain values of α [e.g., α(1 T, 10 K)$\simeq4.3\times10^{13}$ N/m^4] which are in excellent agreement with those determined for bulk reeds.[16,17] This agreement illustrates that the VR technique probes the *pinning of intragranular vortices* in polycrystalline materials.[14]

We will now discuss qualitatively the dissipation mechanisms in the vibrating reed. Four cases may be distinguished: (a) In low-T_c superconducting bulk mechanical oscillators a peak in the damping $\Gamma(T)$ is observed near $T_c(B_a)$ if the damping is governed by hysteresis losses. This is the usual dissipation mechanism in bulk VRs caused by irreversible flux-line displacements.[3,18] The hysteretic damping can be expressed by $\Gamma_h\propto u(l)^n/\alpha^m$, where $u(l)$ is the amplitude of the reed tip and the exponents $n,m\geq0$ depend on the area of the force-displacement hysteresis loop.[2] Since the pinning forces vanish at $T_c(B)$, the peak in Γ shown in Fig. 3 is due to the depinning of the FLs before the superconductor turns normal. Below the peak the nearly constant

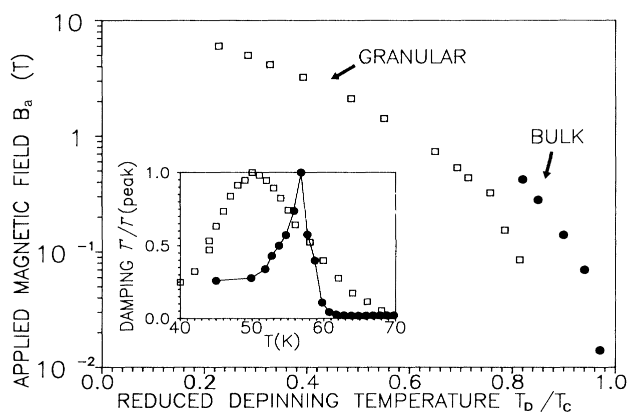

FIG. 4. Depinning lines obtained for the granular ($f=0.1$) and ceramic reeds of $Bi_{1.5}Pb_{0.5}Sr_2CaCu_2O_y$ with $T_c=70$ K. $\nu(B_a=0)=1.42$ kHz (0.36 kHz) for the bulk (granular) reed. The length of the bulk reed is $l=0.8$ cm. Inset: Temperature dependence of the reduced damping at $B_a=0.42$ T for the bulk (●) and granular reed (□) with Γ(peak)$=123$ and 0.2 s^{-1}, respectively.

dissipation is amplitude dependent with $n \simeq 0.3$.[15]

(b) A similar peak is observed in bulk high-T_c reeds; see inset in Fig. 4. This peak occurs, however, at the depinning temperature $T_D(B_a) < T_c(B_a)$ and marks the transition from hysteretic (pinned) to viscous (unpinned) damping.[14,17] The peak occurs when the resonance frequency equals the reciprocal diffusion time τ, i.e., at $\omega \simeq \tau^{-1} = D(B_a,T_D)\pi^2/l^{*2}$, where $D(B_a,T)$ is the FL diffusivity and l^* a characteristic sample length which in bulk reeds in longitudinal B_a is proportional to the reed length.[10,11] The field dependence of T_D yields the depinning line shown in Fig. 4.

(c) For low-T_c granular reeds, a monotonic decrease of damping with increasing temperature is observed independent of the filling factor, as illustrated in Fig. 3 for a granular sample with $f = 0.10$ at different applied fields. This result and the amplitude independence of $\Gamma(B_a,T)$ indicate purely *viscous* dissipation. Near $T_c(B_a)$, Γ depends only on the FL viscosity $\eta(B_a,T) \propto B_{c2}(T)$,[2] which explains its monotonic decrease. With the obtained values of $\alpha(B,T)$ we find that $\lambda_{44} > 50\ \mu\text{m} \geq R$. Deviations from the behavior shown in Fig. 4 may be expected at lower temperatures or higher fields where large values of $\alpha(B,T)$ could reduce λ_{44} and thus affect the temperature dependence of Γ.

(d) In the high-T_c granular reeds $\Gamma(T)$ is nearly amplitude *independent* ($n < 0.03$) but it shows a maximum not observed in the low-T_c granular reeds which is due to thermally activated depinning of the FLs in the high-T_c grains. This maximum in Γ is observed when $D(B_a,T_D) \simeq 4R^2\omega/\pi^2$; now the grain size $2R \approx 40\ \mu$m, and not the length of the reed as in case (b), is the relevant length for the FL diffusion. Since $2R \approx 10^{-2} l^*$, the thermally activated peak in Γ occurs at low temperatures, see inset in Fig. 4, or at

$$\mu_0 D(B,T_D) = \rho(B,T_D) \sim 10^{-4}\ \mu\Omega\,\text{cm}$$

instead of typically 1 $\mu\Omega$ cm in bulk reeds.[17]

Large shifts to lower temperatures and broadening of the dissipation with field are commonly observed for low-density ceramics in ac susceptibility and VR experiments, as well as a considerable sensitivity of Γ or χ'' to the amplitude of the reed or the ac field.[19,20] These effects are likely related to the diffusion and dissipation of FLs on a smaller length scale (grain size) when the superconducting grains are decoupled.

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[1]E. H. Brandt, P. Esquinazi, H. Neckel, and G. Weiss, Phys. Rev. Lett. **56**, 89 (1986).

[2]E. H. Brandt, P. Esquinazi, and H. Neckel, J. Low Temp. Phys. **63**, 187 (1986).

[3]P. Esquinazi, H. Neckel, G. Weiss, and E. H. Brandt, J. Low Temp. Phys. **64**, 1 (1986).

[4]P. L. Gammel, L. F. Schneemeyer, J. V. Waszczak, and D. J. Bishop, Phys. Rev. Lett. **61**, 1666 (1988); see also E. H. Brandt, P. Esquinazi, and G. Weiss, Phys. Rev. Lett. **62**, 2330 (1989).

[5]D. J. Baar and J. P. Harrison, Physica (Amsterdam) **157C**, 215 (1989).

[6]J. Pankert, Physica (Amsterdam) **168C**, 335 (1990).

[7]J. Pankert, G. Marbach, A. Comberg, P. Lemmens, P. Frönig, and S. Ewert, Phys. Rev. Lett. (to be published).

[8]R. Labusch, Cryst. Lattice Defects **1**, 1 (1969).

[9]A. M. Campbell and J. E. Evetts, *Critical Currents in Superconductors* (Taylor and Francis, London, 1972).

[10]A. Gupta, P. Esquinazi, H. F. Braun, W. Gerhäuser, H.-W. Neumüller, K. Heine, and J. Tenbrink, Europhys. Lett. **10**, 663 (1989).

[11]E. H. Brandt, Z. Phys. B **80**, 167 (1990).

[12]P. H. Kes, J. Aarts, J. van den Berg, C. J. van der Beek, and J. A. Mydosh, Supercond. Sci. Technol. **1**, 242 (1989).

[13]D. G. Hawksworth and D. C. Larbalestier, Adv. Cryog. Eng. **26**, 479 (1980).

[14]A. Gupta, P. Esquinazi, H. F. Braun, and H.-W. Neumüller, Phys. Rev. Lett. **63**, 1869 (1989).

[15]J. Kober, Diploma thesis, Universität Bayreuth, 1990 (unpublished).

[16]A. Gupta, P. Esquinazi, H. F. Braun, W. Sadowsky, E. Walker, and D. Shaltiel, in Proceedings of the International Conference on Superconductivity, Bangalore, India, January 1990 [J. Mater. Res. Bull. (to be published)].

[17]A. Gupta, P. Esquinazi, H. F. Braun, H.-W. Neumüller, G. Ries, W. Schmidt, and W. Gerhäuser, Physica (Amsterdam) **170C**, 95 (1990).

[18]P. Esquinazi and E. H. Brandt, Jpn. J. Appl. Phys. **26**, 1513 (1987).

[19]See, for example, H. Küpfer, I Apfelstedt, R. Flükinger, C. Keller, R. Meier-Hirmer, B. Runtsch, A. Turowski, U. Wiech, and T. Wolf, Cryogenics **28**, 650 (1988).

[20]H. Safar, C. Durán, J. Guimpel, L. Civale, J. Luzuriaga, E. Rodriguez, F. de la Cruz, C. Fainstein, L. F. Schneemeyer, and J. V. Waszczak, Phys. Rev. B **40**, 7380 (1990); Physica (Amsterdam) **165C**, 315 (1990).