

## Vibrating-reed studies of flux pinning in the superconducting metallic glass $Zr_{70}Cu_{30}$

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With the vibrating-reed technique we have measured the resonance frequency and damping of superconducting ribbons of a  $Zr_{70}Cu_{30}$  amorphous alloy as a function of magnetic field and temperature for different heat treatments of the same sample. Through these measurements we obtain the Labusch parameter  $a$  in the reduced-field ( $b$ ) range  $5 \times 10^{-4} < b < 0.2$ . At  $b=0.1$ ,  $a = (3.8 \times 10^{11}) B_{c2}(T)^{2.15} \text{ N m}^{-4} \text{ T}^{-2.15}$ , a result which is in agreement with the absolute value and temperature dependence of the pinning force obtained from critical-current measurements. The effect of annealing below the glass transition temperature is to produce changes in  $a$  which depend on field and temperature in a complicated fashion. There are indications that surface pinning could be important in these samples.

The elastic response of the superconducting vortex lattice to very small forces depends on the elastic coupling of the flux lines to the pins (Labusch's<sup>1</sup> parameter  $\alpha$ ). Recently,<sup>2</sup> a novel effect as been discovered which allows the measurement of  $\alpha$  as a function of the applied field  $B_a$  and temperature  $T$ . It was observed<sup>2</sup> that the resonance frequency  $\omega$  and the damping  $\Gamma$  of a superconducting vibrating reed increase dramatically as a function of the longitudinal applied field. The frequency rise is due to the diamagnetic behavior resulting from the pinning of the vortex lattice in the superconductor.<sup>2</sup> Because the pinning is finite, the field-dependent frequency deviates from the ideal diamagnetic (or infinite pinning) result:

$$\omega^2 = \omega_i^2(X) - \omega_{\text{pin}}^2(X). \quad (1)$$

Here  $\omega_i(X)$  is the ideal frequency dependence (with non-local and end corrections included) and  $\omega_{\text{pin}}(X)$  is the pinning-dependent correction. The variable  $X$  is a measure of the applied field:  $X = aB_a$ , where  $a$  is a geometry-dependent constant. The rise in the damping is due to hysteretic and/or viscous losses which are also pinning dependent. For more details see Ref. 2.

The first experiment with this technique has been made with a splat-cooled 60- $\mu\text{m}$ -thick  $Zr_{70}Pd_{30}$  amorphous sample which, because of its relative low pinning turns out to be appropriate for flux pinning studies with the vibrating-reed technique.

In this work we study the frequency and damping dependences of a melt-spun 17- $\mu\text{m}$ -thick  $Zr_{70}Cu_{30}$  amorphous ribbon as a function of  $B_a$  and  $T$  with the vibrating-reed technique. The study of the vortex elastic response is particularly interesting in these samples due to the special behavior of the total pinning force  $F_p$  obtained by critical-current measurements.<sup>3,4</sup> It has been reported<sup>3</sup> that after low-temperature annealing the pinning force  $F_p$  (in a transverse magnetic field) decreases, and for reduced fields  $b (= B_a/B_{c2}) > 0.3$  it follows the two-dimensional collective pinning behavior predicted by Larkin and Ovchinnikov.<sup>5</sup>

Annealing of the  $Zr_{70}Cu_{30}$  amorphous ribbons at temperatures below the glass transition temperature changes

the normal and superconducting properties of the material in a very reproducible way.<sup>7</sup> There are two well-defined regimes. In the first regime the critical temperature decreases  $\sim 10\%$  with almost no change in the residual electrical resistivity. In this regime the sample becomes a more homogeneous amorphous material. In the second regime an opposite behavior is observed and the sample evolves to a crystalline inhomogeneous state.<sup>7</sup>

We have used an experimental arrangement similar to that reported in Ref. 2. Due to the fabrication method<sup>6</sup> which produces slightly bent ribbons, we had to use a relatively short sample (length  $l = 0.34$  cm, width = 0.37 mm) to avoid having to introduce corrections due to a complicated effective magnetic geometry. Such a short sample is not quite convenient for pinning measurements: a long sample would have a smaller effective damping and increase the relative frequency shift, increasing the sensitivity to magnetic field changes.<sup>2</sup>

To avoid changes in geometry with annealing we used a special sample holder with fixed electrodes which allowed thermal treatment without unclamping the sample. High reproducibility has been achieved. In particular it is possible to follow the measured resonance frequency at 4 K which shows the same correlation with the superconducting critical temperature  $T_c$  as the residual resistivity.<sup>7</sup> The resonance frequency at  $B_a = 0$  for the as-quenched state was 638.24 Hz at 4 K.

We followed the evolution of the sample with annealing by measuring  $T_c$  and  $B_{c2}$ . The resonance amplitude when the applied field is negligible in comparison with  $B_{c2}$  ( $B_a = 0.003$  T) has a sharp minimum as a function of  $T$ , which defines  $T_c$ . In a similar way, the field dependence of the amplitude at constant temperature defines  $B_{c2}$ . These critical values agree very well with the values obtained with the electrical resistivity method.

In Fig. 1 we show the Labusch parameter  $a$  as a function of the reduced applied field  $b$  at different reduced temperatures  $t (= T/T_c)$  for the as-quenched sample ( $T_c = 2.67$  K). These values are obtained from the measured resonance frequency  $\omega(B_a)$  using Eq. (1). The ideal diamagnetic frequency dependence  $\omega_i(X)$  is calcu-

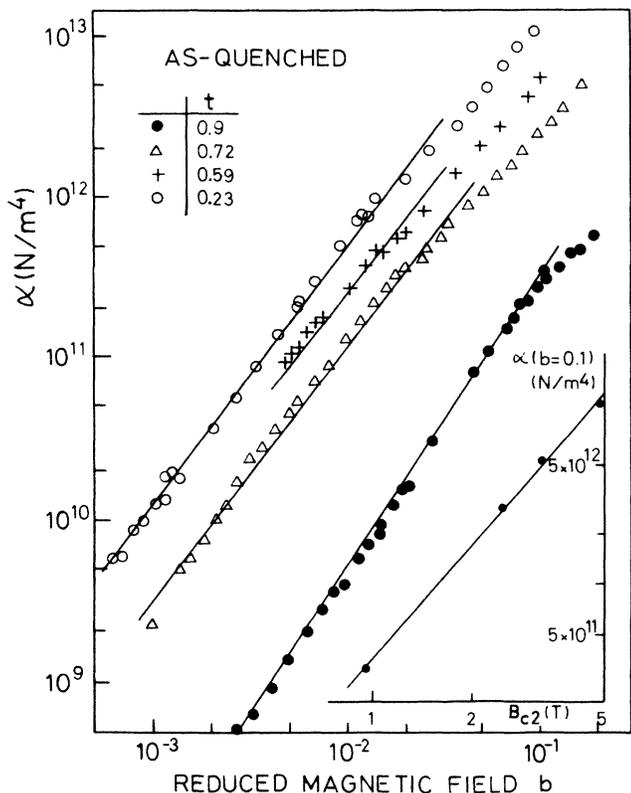


FIG. 1. Labusch parameter  $\alpha$  as a function of reduced magnetic field  $b$  and at different reduced temperatures  $t$  ( $T_c = 2.67$  K) for the as-quenched sample. The straight lines are only a guide. Note the very small values of  $b$ . Inset:  $\alpha$  vs the measured critical field  $B_{c2}(T)$  ( $B_{c2} = 0.9, 2.4, 3.2,$  and  $4.8$  T for  $t = 0.9, 0.72, 0.59,$  and  $0.23$ ). The straight line has a slope of 2.15.

lated with the measured geometry and the resonance frequency at  $B_a = 0$  and  $\omega_{\text{pin}}(X)$  has only  $\alpha$  as a free parameter.<sup>2</sup> In Eq. (1) no magnetization contribution is taken into account.<sup>2</sup> In the measured temperature range we have observed magnetization effects below 0.004 T ( $b < 5 \times 10^{-4}$  at  $t = 0.23$ ) and it is possible to detect the onset of the first penetration field<sup>8</sup>  $B_{c1}$ . We have also measured the change of the resonance frequency with  $B_a$  for different initial ( $B_a = 0$ ) reed amplitudes ( $\leq 1 \mu\text{m}$ ). The temperature and  $b$  dependences and the absolute value of  $\alpha$  do not depend on the amplitude of the reed within the amplitude range used. This amplitude independence of  $\alpha$  supports the assumption of an elastic pinning response.

For  $b < 0.1$  and roughly independent of  $t$  we obtain a  $b^{1.6}$  dependence of  $\alpha$ . Good scaling with temperature is obtained for the whole measurement range,  $\alpha = f(b)B_{c2}(T)^n$ . In particular we plot in the inset of Fig. 1  $\alpha$  at  $b = 0.1$  as a function of  $B_{c2}$ ; the straight line through the points gives  $\alpha = (3.8 \times 10^{11})B_{c2}^{2.15}(T) \text{ Nm}^{-4} \text{ T}^{-2.15}$ . A very different scaling result was obtained in an early experiment<sup>2</sup> for a  $\text{Zr}_{70}\text{Pd}_{30}$  sputtered sample:  $\alpha(b = 0.1) = (1 \times 10^{11})B_{c2}(T)^{3.5} \text{ Nm}^{-4} \text{ T}^{-3.5}$ . Due to rather similar superconducting properties one would expect the same kind of scaling for both samples. It

is probable that the different scaling comes from different surface pinning contributions. In fact, the 60- $\mu\text{m}$ -thick sputtered sample has smoother surfaces than the melt-spun sample. Experiments with different surface roughness and thickness have to be made to clarify this difference.

It is also interesting to compare the total pinning force obtained by critical-current measurements with our results. If we assume that  $F_p = s_p \alpha$ , with  $s_p \lesssim a_0/2\pi$  ( $a_0$  is the flux line spacing),<sup>9</sup> we obtain  $F_p(b = 0.1) = (4.3 \times 10^4)$  and  $(7 \times 10^4) \text{ N/m}^3$  at  $t = 0.72$  and  $0.59$ , compared with  $F_p(b = 0.1) = (2.3 \times 10^4)$  and  $(4.7 \times 10^4) \text{ N/m}^3$  from critical-current measurements.<sup>3,4</sup> With this assumption, we obtain the same  $B_{c2}$  dependence:<sup>3,4</sup>  $F_p \propto B_{c2}(T)^{1.65}$ . The agreement indicates that we are testing the same kind of pinning as in the critical-current measurements. Since there are significant differences between the techniques (with the vibrating reed, the vortices are parallel to the main area of the sample and in the critical-current measurements perpendicular), we find this agreement remarkable.

For  $b > 0.2$  the damping was too high to measure the resonance frequency. We made different experiments with different samples, clampings, and even with different superconducting solenoids. The results were the same. The measured damping is much higher (more than an order of magnitude) than in the  $\text{Zr}_{70}\text{Pd}_{30}$  sample.<sup>2</sup> Assuming that the damping at  $b = 0.1$  is mainly due to the amplitude-dependent hysteretic damping  $\Gamma_h$ , it becomes clear that the high losses result from the particular field dependence of  $\alpha$  and our small length:  $\Gamma_h \propto [u(1)]^m (\Lambda/l)^{m'} \alpha$  where  $\Lambda$  is an effective Campbell penetration depth (which in our case slightly increases with  $b$ ) and  $u(1)$  is the amplitude at the free end of the reed;<sup>2</sup>  $m$  and  $m'$  (both  $> 0$ ) depend on the particular hysteresis loop model. Although smaller amplitudes  $u(l)$  led to smaller hysteretic damping the loss in sensitivity made it impossible to measure a reliable resonance frequency.

At low fields ( $B_a < 0.01$  T) the damping is almost amplitude independent; this result would indicate that visco-loss contributions are important.

In Fig. 2 (circles) we show the Labusch parameter as a function of  $b$  for different temperatures and an annealing treatment which decreases  $T_c$  by 3.4%. This corresponds to the annealing regime where  $T_c$  changes and the resistivity remains constant. We observe that  $\alpha$  changes upon annealing in a rather complicated manner. At  $t = 0.9$ ,  $\alpha$  increases with respect to the as-quenched state (crosses) throughout the measured  $b$  range, at  $t = 0.23$  it decreases, and at  $t = 0.72$  a smaller decrease is obtained.

In general, further annealing decreases  $\alpha$  and also changes its  $b$  dependence at different reduced temperatures so that the scaling present in the as-quenched sample is lost. Surface normal regions (or superconducting regions with  $T_c < 1$  K) induced by annealing in these ribbons have been reported.<sup>10</sup> If these regions nucleate from the beginning of the thermal treatment one could expect such a complicated temperature-dependent behavior for the pinning. In this  $b$  range ( $b < 0.05$ ) and depending on the annealing treatment,  $F_p$  increases,<sup>4</sup> a different behavior from the systematic decrease in the two-dimensional

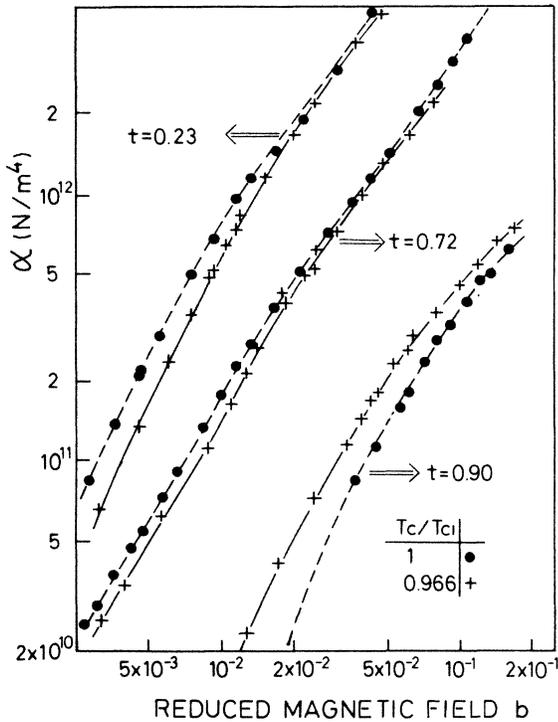


FIG. 2. Labusch parameter  $\alpha$  vs  $b$  at different reduced temperatures for the as-quenched ( $\bullet$ ) and the first annealed state ( $+$ ) with  $T_c = 2.58$  K obtained after 2 h at  $150^\circ\text{C}$ . The dashed and continuous curves are guides for the eye only.

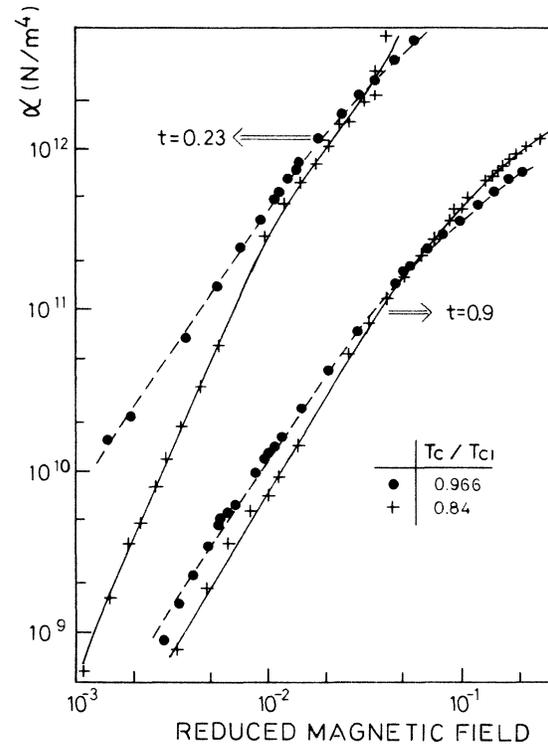


FIG. 3. Labusch parameter  $\alpha$  vs  $b$  for two reduced temperatures  $t$ . Dashed curves ( $\bullet$ ): first annealing state; full curves ( $+$ ): last annealed state with  $T_c = 2.285$  K, reached after 24 h at  $150^\circ\text{C}$ , 18 h at  $220^\circ\text{C}$ , and 46 h at  $280^\circ\text{C}$ .

collective regime observed at higher fields ( $b > 0.1$ ) in the same sample.<sup>3,4</sup>

After longer times and higher annealing temperatures we reach the second and inhomogeneous (partially crystallized) regime where  $T_c$  remains almost constant and the resistivity changes. In this case the behavior of  $\alpha$  is different from that observed in the first annealing treatments. In Fig. 3 we show  $\alpha$  as a function of  $b$  at  $t = 0.23$  and  $t = 0.9$  for the sample where  $T_c$  has been lowered 16% by heat treatments (crosses). For comparison we plot in this figure the results corresponding to the first annealing step (circles). Clearly we see a change in the  $b$  dependence of  $\alpha$ ; there is an increase in  $\alpha$  at  $b > 0.05$ . We observe the same behavior for intermediate temperatures. A big increase of the pinning at  $b > 0.1$  has been observed in the critical-current measurements<sup>3,4</sup> at this annealing stage. But  $\alpha$ , and probably also  $F_p$ , decreases at very low  $b$ . This opposite behavior indicates again the particular

dependence of the pinning in this relatively dilute (small fields) vortex regime. An interpretation of the low-field pinning behavior should be of importance to understand the evolution of the peak in  $F_p$  and the variation of the two-dimensional regime<sup>11</sup> observed in critical-current measurements.

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