Low-frequency response in the surface superconducting state of single-crystal ZrB_{12}

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A large nonlinear response of a single crystal of ZrB_{12} to an ac field (frequency 40–2500 Hz) for $H_0 > H_{c2}$ has been observed. Direct measurements of the ac wave form and the exact numerical solution of the Ginzburg-Landau equations, as well as the phenomenological relaxation equation, permit the study of the surface superconducting state dynamics. It is shown that the low-frequency response is defined by transitions between the metastable superconducting states under the action of an ac field. The relaxation rate that determines such transition dynamics is found.

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I. INTRODUCTION

Nucleation of the superconducting phase in a thin surface sheath in a magnetic field parallel to the sample surface was predicted by Saint-James and de Gennes four decades ago.[1] They showed that for type-II superconductors, for magnetic field higher than H_{c2}, a superconducting phase could exist in a thin sheath near the sample surface. Since that time a lot of experimental and theoretical work has been devoted to this problem.[2–8] All experiments that were performed after the Saint-James and de Gennes publication confirmed the main conclusion of Ref. 1 that nucleation of the superconducting phase occurs in a magnetic field $H_{c3}=2.39\ kH_c$, where H_c is the thermodynamic critical field and k the Ginzburg-Landau (GL) parameter. ac measurements showed that in the surface superconducting state (SSS) the superconductor becomes nonlinear and losses are larger than in the normal and mixed states.[8]

This surface superconductivity has attracted renewed interest from various directions.[9–14] Paramagnetic effects in a superconducting disk,[9] stochastic resonance,[10] and the percolation transition in the field $H_0=0.81H_{c3}$ (Ref. 11) have been observed. It was proposed to use low-frequency response for testing the quality of superconducting resonators in accelerators.[12] Surface states were observed also in single crystals of MgB_2.[13] A different theoretical approach for the study of the SSS based on a generalized form of the GL functional was developed in Ref. 14.

Recently, high-quality superconducting ZrB_{12} single crystals with transition temperatures $T_c=6.06$ K have been grown. Investigation of their physical properties, including electron transport, tunnel characteristics, and critical fields, has shown that the Ginzburg-Landau parameter κ is only slightly larger than the boundary between the type-I and type-II superconductor values.[15,16] In this paper we are concerned with the low-frequency response of a ZrB_{12} crystal when the dc external magnetic field $H_0>H_{c2}$ is parallel to the sample surface. In spite of the fact that the sample is in the SSS,[1] no static magnetic moment is observed, while the ac response in this regime is large and nonlinear, even for an ac amplitude $h_0 \ll H_0$. Indeed, at equilibrium the total surface current equals zero in the SSS, the internal dc magnetic field in the bulk equals H_0, and the magnetic moment of the specimen is small. On the other hand, the ac magnetic field drives the sample into a metastable SSS where the total surface current reaches a finite value. The internal magnetic field deviates from the external one, and as a result, the ac response becomes large. The low-frequency response of superconductors in the SSS was the focus of intensive experimental investigations[4,5,8] since the first prediction of the existence of the SSS in Ref. 1. The observed[8] wave form of the ac response, corresponding to the flux passing through the specimen, explicitly invoked a model similar to the Bean model.[17] Our experimental results presented here show that in a ZrB_{12} single crystal, the Bean critical model of the surface sheath does not give an adequate description of the observed wave form which corresponds to the flux passing through the sample. In the framework of the Ginzburg-Landau theory we calculated the surface current in metastable SSS's which exists under an ac magnetic field. Observing the wave forms, we studied the metastable SSS dynamics and determined the relaxation rate under an ac field. We found that the relaxation time for transition to the equilibrium state is not constant and depends on the surplus of the free energy. This relaxation time is decreased with increasing dc magnetic field, and depends on the driving field frequency.

II. EXPERIMENT

The measurements were carried out at $T=5$ K on a ZrB_{12} single crystal. The sample was grown in the Institute for Problems of Materials Science, Ukraine. Its dimensions are $10.3\times3.2\times1.2$ mm^3 and it was cut by an electric spark from a large crystal of 6 mm diameter and 40 mm length. The surface of the sample was polished mechanically and then chemically etched in boiling HNO_3-H_2O (1:1) for 10 min. X-ray pictures showed that the sample was single-phase material with the UB_{12} structure [space group $Fm3m$, $a=7.407$ Å (Ref. 18)]. The tunnel characteristics of this sample were described earlier.[16] The dc magnetic moment was measured using a superconducting quantum interference device magnetometer. A block diagram of the ac linear and nonlinear setup is shown in Fig. 1.

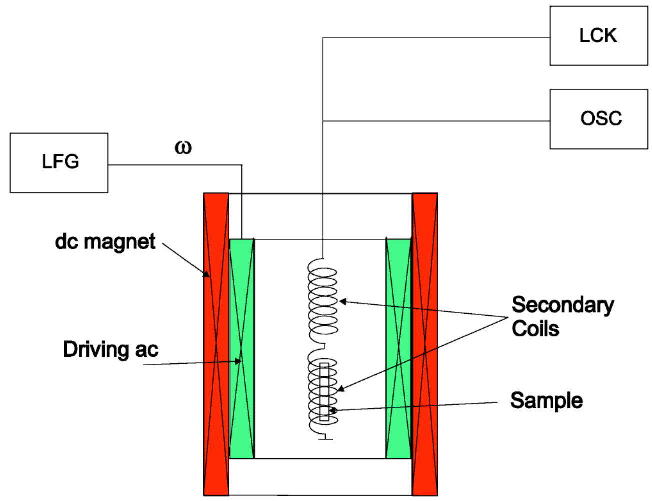

FIG. 1. (Color online) Block diagram of the experimental setup. LFG, low-frequency generator; LCK, lock-in amplifier; OSC, oscillograph.

The ac magnetic field $h(t)=h_0 \sin(\omega t)$ was supplied by the magnetometer copper solenoid. The ac response was measured by an inductive pick-up coil method.[19] The sample was put into one coil of a balanced pair of pick-up coils and the induced voltage $V(t)\propto dM(t)/dt$ was measured with an oscilloscope. Here M is the magnetic moment of the sample. The lock-in amplifier was used in order to measure simultaneously in-phase and out-of-phase signals of the first and third harmonics of the driving frequency. An oscilloscope measured the wave form of the signal in one channel. The second channel of the oscilloscope measured the time derivative of the excitation field.

III. EXPERIMENTAL RESULTS

Figure 2(a) shows the real part of the ac susceptibility at the fundamental frequency χ' and the zero-field-cooled (ZFC) dc susceptibility $\chi_{\rm dc}=M/H_0$ as a function of dc field H_0. The inset to Fig. 2(a) presents the ZFC magnetization curve at 5 K. The field dependencies of the ac susceptibility imaginary part at fundamental frequency, χ'', and the amplitude of the third harmonic, $A_{3\omega}$ are shown in Figs. 2(b) and 2(c), respectively. The amplitude dependence of the third harmonic, $A_{3\omega}(h_0)$ for $H_0=180$ Oe is presented on the inset to Fig. 2(c). It is clear that this dependence is far from cubic as the perturbation theory predicts. Experiment shows that the amplitude dependence of $A_{3\omega}(h_0)$ is not cubic at any dc field H_0.

It is clear that the observed large signal of $A_{3\omega}$ and maximum of χ'' located in a magnetic field $H_{c2}<H_0<H_{c3}$, i.e., in a surface superconducting state, although the zero dc signal indicates that bulk of the sample is in the normal state. The absorption in the SSS exceeds the losses in the mixed and normal states.

Figure 3 shows the time derivative of the magnetic moment of a sample at different applied magnetic fields at $T=5$ K. Note that (i) only in the SSS does the signal not have the sine form; (ii) the amplitude dependence of the third harmonic $A_{3\omega}(h_0)$ presented in the inset to Fig. 2(c) does not exhibit any cubic dependence.

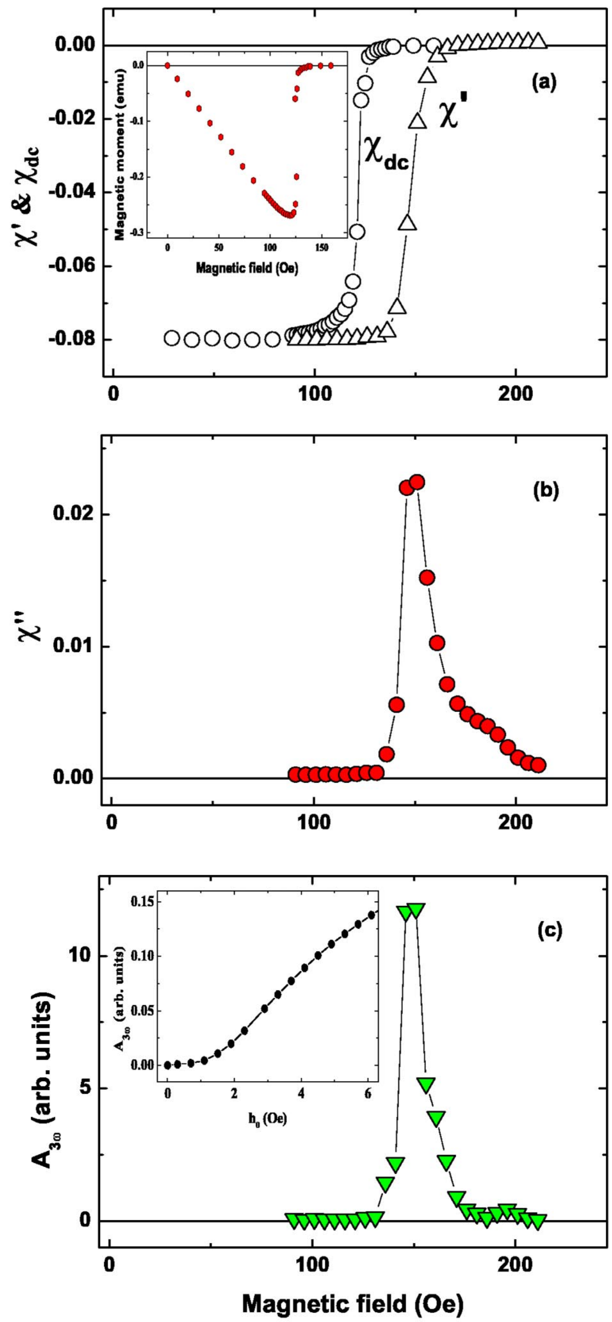

FIG. 2. (Color online) (a) χ' and $\chi_{dc}=M/H_0$ magnetic field dependencies at $T=5$ K. Inset: magnetization curve after ZFC. (b) Magnetic field dependence of χ''. (c) Field dependence of the amplitude of the third harmonic $A_{3\omega}$. Inset: amplitude dependence of $A_{3\omega}(h_0)$ at $H_0=180$ Oe. ac measurements were carried out at frequency $\omega/2\pi=170$ Hz and $h_0=0.4$ Oe.

The experimental data presented in Figs. 2 and 3 are complex and the theoretical model that explains these observations is given in the next section.

IV. THEORETICAL MODEL

Our theoretical approach is based on the numerical solution of the two Ginzburg-Landau equations[22] for the order

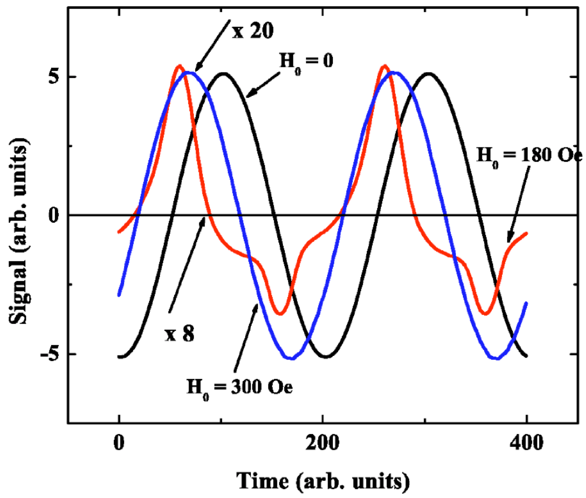

FIG. 3. (Color online) Oscillogram of dM/dt for three different magnetic fields in the Meissner state ($H_0=0$), in surface superconducting state ($H_0=180$ Oe$<H_{c3}$), and in normal state ($H_0=300$ Oe$>H_{c3}$) at $h_0=4.75$ Oe.

parameter and vector potential, which in the normalized form are as follows:

$$-(i\nabla/\kappa+\vec{A})^2\Psi^2+\Psi-|\Psi|^2\Psi=0,$$

$$-\text{curl curl}\vec{A}=\vec{A}|\Psi|^2+i/2\kappa(\Psi^*\nabla\Psi-\Psi\nabla\Psi^*). \qquad (1)$$

The order parameter Ψ is normalized with respect to its value in zero magnetic field, the distances with respect to the London penetration length λ, and the vector potential $\vec{A}$ with respect to $\sqrt{2}H_c\lambda$, where H_c is the thermodynamic critical field, $\kappa=\lambda/\xi$ is the Ginzburg-Landau parameter, and ξ is the correlation length. In the second equation for the vector potential we neglected the normal current, assuming that the skin depth exceeds both the London penetration length and the sample thickness. It is assumed that the sample form is a slab with $2d$ thickness and that the external magnetic field is parallel to its surface. The chosen coordinates are the x axis normal to the slab (thus the symmetry plane is $x=0$), and the z axis directed along the magnetic field. It is assumed that the external magnetic field $H>H_{c2}=\kappa$.

Assuming the surface solutions have the form of

$$\Psi(x,y)=f(x)\exp(i\kappa ky); \qquad (2)$$

therefore, Eqs. (1) reduce to

$$-\frac{1}{\kappa}\frac{\partial^2 f}{\partial x^2}+(A-k)^2f-f+f^3=0,$$

$$\frac{\partial^2 A}{\partial x^2}=f^2(A-k), \qquad (3)$$

where k is constant. The boundary conditions at $x=\pm d$ are $\partial f(\pm d)/\partial x=0$, $\partial A(\pm d)/\partial x=H$; at $x=0$, $f(0)=0$ and $A(0)=0$; $H=H_0+h_0\sin(\omega t)$. An additional condition for the surface states $\partial f(0)/\partial x=0$ is satisfied only asymptotically for $d\to\infty$. For the equilibrium state the value of $k=k_{eq}$ can be obtained by minimizing the Gibbs free energy defined as

$$\tilde{F}=\int dV\left\{\frac{1}{2}|\Psi|^4-|\Psi|^2+|i\nabla\Psi/\kappa+A\Psi|^2+B^2-2BH\right\}, \qquad (4)$$

where B is the magnetic induction. Using Eq. (3) one then obtains

$$\tilde{F}=-HA(d)-\int_0^d dx\left\{\frac{1}{2}f^4(x)+A(x)[A(x)-k]f^2(x)\right\}. \qquad (5)$$

The two coupled Eqs. (3) can be solved by numerical methods. The order parameter for surface solutions deviates from zero only near the sample boundary, and we can consider comparatively small $d/\lambda\leqslant 10$. The actual sample thickness exceeds λ by four or five orders of magnitude. The solutions for large d could be found from the ones for small d by the transformation $k=k_s+H_i(d-d_s)$, where $H_i=H_s(0,H,K)$ is the magnetic field at $x=0$ in the problem for $d=d_s\cong 10\lambda$. The index s corresponds to the solution for this small d. This choice of d_s is sufficient for numerical calculations and provides the solutions with $f_s(0)=0$, $\partial f_s(0)/\partial x=0$. The free energy is transformed as

$$\tilde{F}=\tilde{F}_s-H_i(d-d_s)\left(H-\int[A_s(x)-k_s]f_s^2(x)dx\right). \qquad (6)$$

To simplify the calculations we use below variable k_s and omit the index s. The properties of the equilibrium solutions for a semi-infinite half space have been discussed in Ref. 20. The order parameter, the supercurrent, and the internal magnetic field were calculated. In these states the total surface current equals zero and the free energy reaches a minimum value. The ac magnetic field drives the superconductor into a metastable state. These states correspond to the solutions of Eqs. (3) for $k\neq k_{eq}$. The solution of Eqs. (3) shows that surface states exist in a wide range of k near k_{eq} as shown in the upper panel of Fig. 4, but only for $|k-k_{eq}|\ll k_{eq}$ is the free energy of these states lower than the energy of the normal state. Moreover, this range shrinks with increasing sample thickness (Fig. 5).

This is due to the increase of the contribution of the first term in Eq. (5) which is on the order of the Gibbs energy of the normal state $\tilde{F}_0=-H^2d$. The total surface current equals zero for equilibrium k and increases with increasing $|k-k_{eq}|$ as is shown in the lower panel of Fig. 4. For a given k, the free energy versus magnetic field does not exhibit any minimum in the equilibrium. Only the difference between the Gibbs energy of the superconducting and normal states exhibits a minimum near equilibrium field as was discussed in Ref. 8, but for such a representation the reference point moved with the changing field.

The magnetic moment of the sample actually depends on the total surface current J, because the current is localized in a thin surface layer. This current is a function of the external magnetic field H and k, $J=J(H,k)$. The response of the sample to the ac magnetic field depends on the dynamics of k. *A priori*, one can assume that the equation that governs the dynamics is

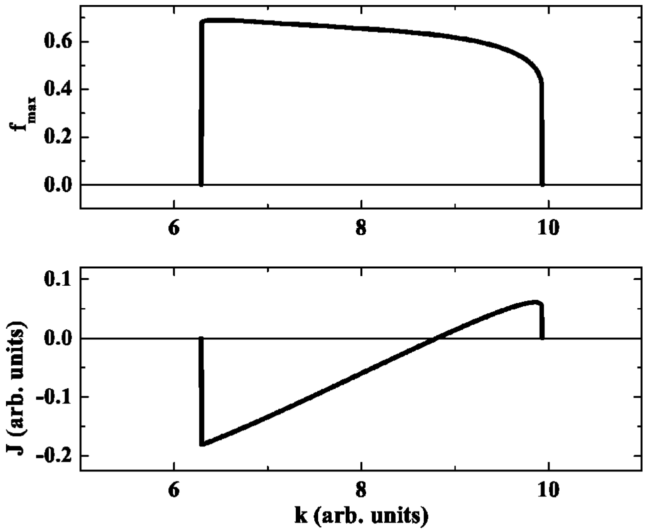

FIG. 4. Upper panel: The maximal value of the order parameter, f_{max}, as a function of k. Lower panel: Total surface current J as a function of the k parameter. The equilibrium value of $k=8.8$ corresponds to zero surface current.

$$\frac{dk}{dt} = -\nu[k - k_{eq}(H)], \tag{7}$$

where $k_{eq}(H)$ is k in the equilibrium corresponding to the instantaneous value of the magnetic field H and $\nu=\nu(k-k_{eq})$ is the relaxation rate. The function $k_{eq}(H)$ has to be found from Eqs. (3) and for $|H-H_0| \ll H_0$ is well approximated by a polynomial of the third order of $h=H-H_0$. Using the function $J(h,k)$ calculated from Eqs. (3) and (7) one can obtain the time evolution of the surface current in an ac field and compare with observed wave forms. The time derivative of the surface current is proportional to the observed signal V. The coefficient $\alpha=(1/V)/(dJ/dt)$ depends on the experimental apparatus parameters. Actually we can obtain $k(t)$ directly from experimental data and test the correctness of Eq. (7). We may write

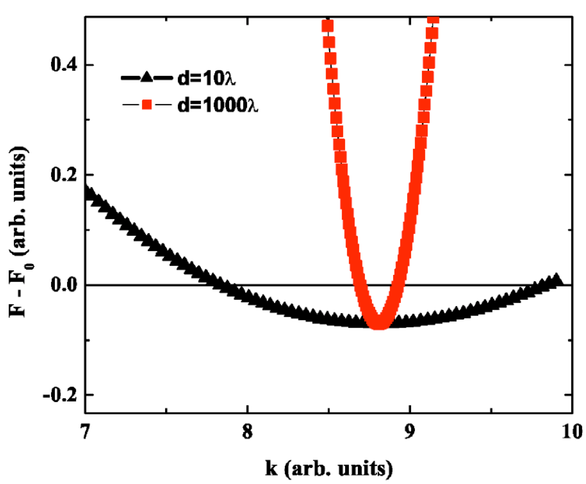

FIG. 5. (Color online) Free energy of the surface superconducting states relative to the free energy of normal state (F_0) in magnetic field $H_0=1$ and $k=0.75$ for two values of the sample thickness.

$$\frac{\partial J(h,k)}{\partial h}\frac{dh}{dt} + \frac{\partial J(h,k)}{\partial k}\frac{dk}{dt} = \alpha V(t). \tag{8}$$

This expression permits us to obtain $k(t)$ from the observed wave form. It is a first order differential equation for $k(t)$. To evaluate $k(t)$ we have to know k at $t=0$ and α, since the derivatives $\partial J/\partial h$ and $\partial J/\partial k$ can be calculated from Eqs. (3). The $k(0)$ value can be found from the condition when the maximal current value during the period is minimal. In order to find α we calculated $J(t)$ assuming that ν in Eq. (7) is constant. Then, we choose the ν value in order to minimize the difference between the calculated and experimental data. This procedure gives both ν and α. To be sure that ν is actually constant, one has to collect the weak ac field data. The observed signal during one period of the ac field and the result of a simulation with Eqs. (3) and (7) are shown in Fig. 4. The data in this figure were collected in a dc field of 130 Oe, and an ac field with amplitude 1.78 Oe and frequency $\omega/2\pi=733$ Hz. In our calculations we took $\nu/\omega=0.05$ and the Ginzburg-Landau parameter $\kappa=0.75$.[16] The good correlation between the calculated and experimental data permits one to find the scale coefficient α that is used below.

V. DISCUSSION

As was shown above the losses are small in both the mixed and normal states and have a maximum at $H_0 > H_{c2}$ [see Fig. 2(a)]. $H_{c2}=126$ Oe is determined from the dc magnetization curve [inset to Fig. 2(a)]. The oscillogram, Fig. 3, in both the Meissner and normal states ($H_0=0$ and 300 Oe) has a sine shape, and for $H_{c2} < H_0 < H_{c3}$ the wave form deviates from a sine shape. We do not observe any clear plateau

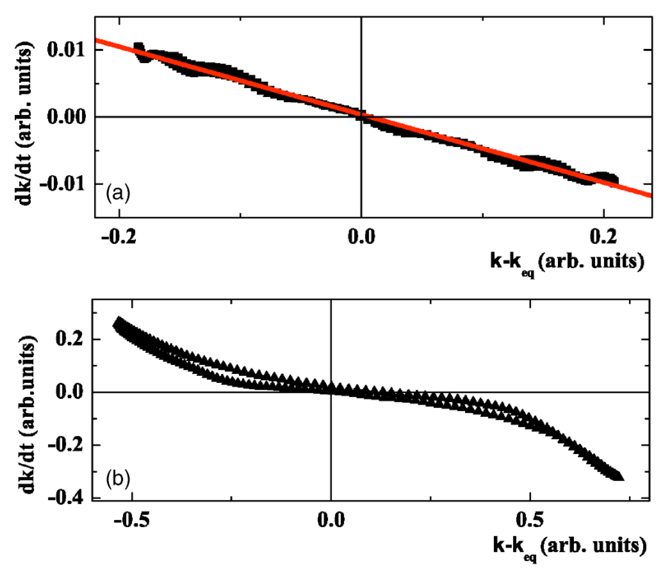

FIG. 6. (Color online) The time derivative dk/dt plotted as a function of $k-k_{eq}$ for $H_0=130$ Oe and $\omega/2\pi=733$ Hz. (a) $h_0=1.78$ Oe and (b) 5.9 Oe. The linear fit of the experimental curve (a) gives $\nu/\omega=0.051$. The hysteresis at large amplitudes (b) shows that, generally speaking, dk/dt depends on k and the instantaneous magnetic field not only through $k=k_{eq}$.

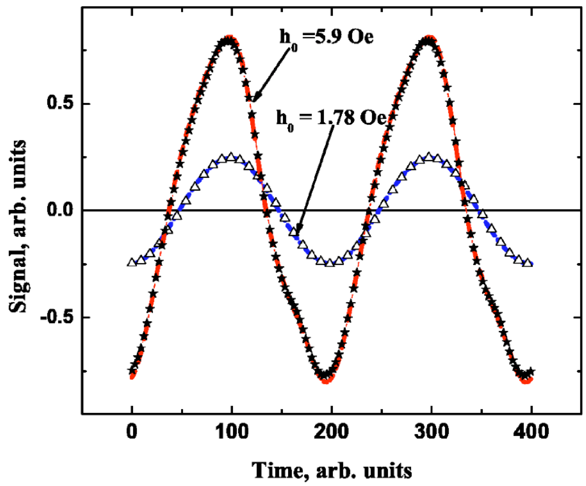

FIG. 7. (Color online) The observed and calculated (solid lines) oscillogram for $H_0 = 130$ Oe, $\omega/2\pi = 733$ Hz at $h_0 = 1.78$ Oe and 5.9 Oe.

for dM/dt in the ac period. Such a plateau is a peculiarity of the Bean model when it is applied to surface currents.[8,21] Using the experimental data and the model developed in the previous section one can calculate dk/dt as a function of $k - k_{eq}$. Figure 6 shows dk/dt plotted as a function of $k - k_{eq}$ obtained from the wave forms that were observed for $H_0 = 130$ Oe and $\omega/2\pi = 733$ Hz. The linear fit of dk/dt at $h_0 = 1.78$ Oe yields $\nu(0)/\omega = 0.051$ which agrees well with the $\nu/\omega = 0.05$ used in Eq. (7) when the scale coefficient has been found. The visible hysteresis in Fig. 6(b) indicates that at a high amplitude of excitation the relaxation rate in Eq. (7) ν depends on k and on the instantaneous value of $h(t)$ not only through $k - k_{eq}(h)$.

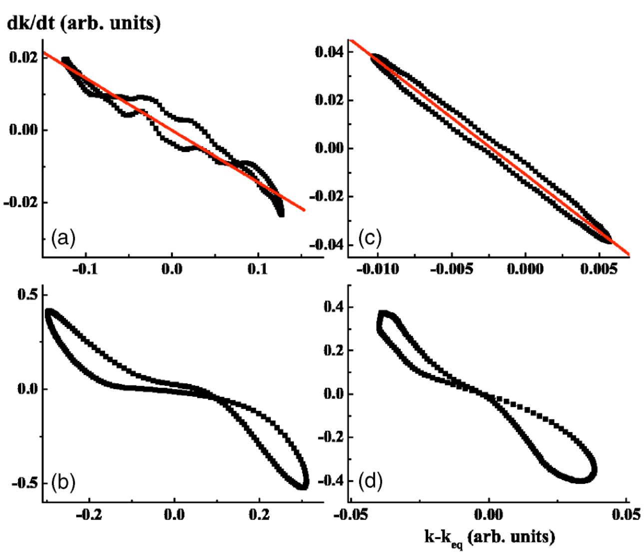

FIG. 8. (Color online) dk/dt as a function of $k - k_{eq}$ at $\omega/2\pi = 733$ Hz. (a) $H_0 = 138$ Oe, $h_0 = 0.59$ Oe; (b) $H_0 = 138$ Oe, $h_0 = 5.9$ Oe; (c) $H_0 = 180$ Oe, $h_0 = 0.59$ Oe; (d) $H_0 = 180$ Oe, $h_0 = 5.9$ Oe. Linear fit at low amplitude of excitation [(a) and (c) panels] gives $\nu/\omega = 0.144$ for $H_0 = 138$ Oe and $\nu/\omega = 4.73$ for $H_0 = 180$ Oe.

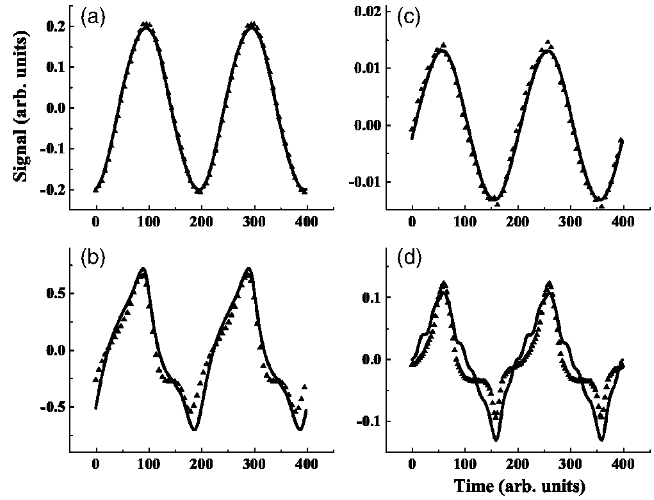

FIG. 9. Calculated (solid lines) and experimental (triangle) oscillograms at $\omega/2\pi = 733$ Hz. (a) $H_0 = 138$ Oe, $h_0 = 0.59$ Oe; (b) $H_0 = 138$ Oe, $h_0 = 5.9$ Oe; (c) $H_0 = 180$ Oe, $h_0 = 0.59$ Oe; (d) $H_0 = 180$ Oe, $h_0 = 5.9$ Oe.

The expression for $\nu(k - k_{eq})$ can be found from fitting of dk/dt by the polynomial of $k - k_{eq}$. The approximation expression, which has the form

$$\nu(x)/\omega = 0.051 - 0.117x + 1.323x^2 + 0.184x^3 - 0.747x^4, \tag{9}$$

provides the calculated data, which with an accuracy of better than 10% reproduce the experimental data for a dc field of 130 Oe and a frequency of 733 Hz as is shown in Fig. 7.

Increasing the dc field leads to the increasing of the relaxation rate ν. We found that for $H_0 = 138$ and 180 Oe, the relaxation parameters for weak ac amplitudes are $\nu(0)/\omega = 0.145$ and 4.725, respectively (see Fig. 8).

The calculated wave forms with the help of the proposed model reproduce experimental data only for a weak ac field as shown in Fig. 9. This is due to the increase in the differ-

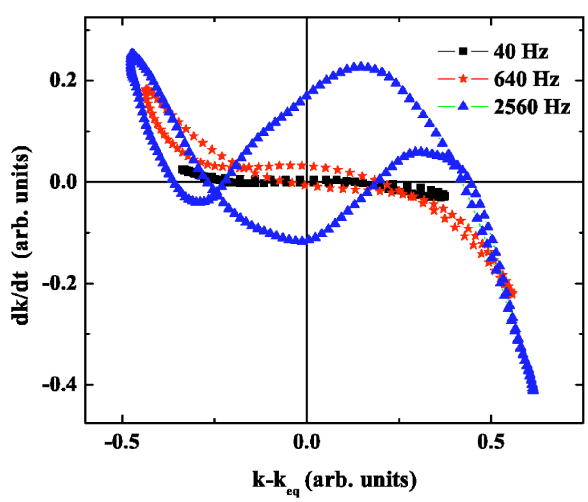

FIG. 10. (Color online) dk/dt as a function of $k - k_{eq}$ at $H_0 = 130$ Oe and $h_0 = 4.75$ Oe for different frequencies.

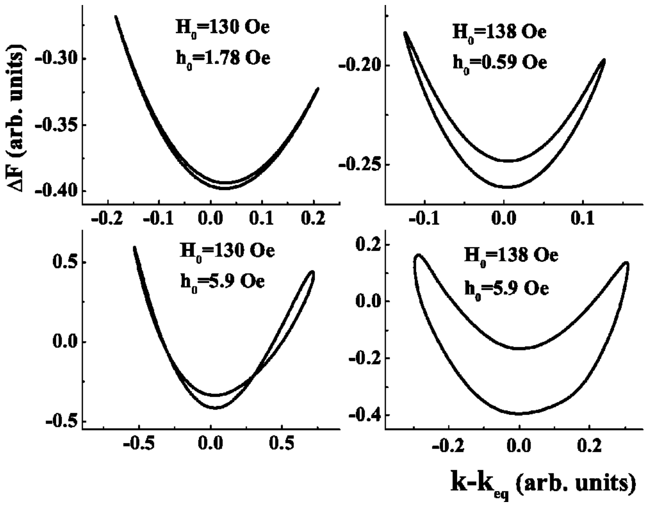

FIG. 11. Calculated dependence of the Gibbs energy as a function of the $k-k_{eq}$ parameter during the ac cycle for different values of the dc field H_0 and amplitude of excitation h_0 at frequency $\omega/2\pi=733$ Hz.

ence between the two values of dk/dt for the same $k-k_{eq}$ at larger ac amplitudes [see Figs. 8(b) and 8(d)].

Figure 10 shows the $dk/dt(k-k_{eq})$ dependence for different frequencies at $H_0=130$ Oe and $h_0=4.75$ Oe. One may conclude from this figure that the relaxation rate ν [if Eq. (7) could be applied] increases with excitation frequency ω.

It is clear that the model equation (7), where the relaxation rate ν depends on the one variable $k-k_{eq}$, is valid only for small ac amplitudes. A transition from the surface state with one k to another k requires changing the order parameter in the whole sample. Possibly, this happens through the nucleation of a new phase. This process is governed by excess energy. In general the relaxation constant in Eq. (7) may depend on the energy of the state. Approximately it could be taken into account by the assumption that the relaxation constant depends on $k-k_{eq}$. We see that for small ac fields and not far away from H_{c2}, this assumption is correct. But increasing the ac amplitude and/or the dc field results in the explicit dependence on both k and the instantaneous value of the magnetic field H_0. The straightforward calculations of the Gibbs energy F, exhibited in Fig. 11, shows that when this energy is a single-valued function of $k-k_{eq}$, the simulation with Eq. (7) gives acceptable results.

When F becomes a multivalued function, the calculated wave forms differ from the experimental data. In order to obtain a proper theoretical description, one has to take into account that the energy of the SSS is not expressed only through $k-k_{eq}$.

VI. CONCLUSION

We experimentally investigated the dynamics of the surface metastable superconducting states of ZrB_{12} in ac fields at low frequencies (40–2500 Hz). It was shown that for low ac amplitudes of excitation this dynamics is governed by a simple relaxation equation. The relaxation rate depends on the deviation from the equilibrium state. Decreasing the frequency of the applied ac field results in increasing the relaxation time.

ACKNOWLEDGMENT

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