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MAGNETISM AND FERROELECTRICITY

Influence of the transport current on the magnetoelectric properties of $La_{0.7}Pb_{0.3}MnO_3$ single crystals with giant magnetoresistance in the microwave region

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The results of experiment on the influence of a direct current and a low-frequency alternating current, as well as a magnetic field, on the microwave-range conductivity σ_{MW} of $Ln_{0.7}Pb_{0.3}MnO_3$ single crystals with giant magnetoresistance are presented. The greatest sensitivity of the samples toward the effects of a current is observed in the temperature range corresponding to the dc magnetoresistance maximum. The response signal of a sample in the microwave range to the effects of an alternating current of a low frequency f_0 has a nonlinear character. As f_0 is varied in a magnetic field, the amplitude of the response signal varies with the appearance of resonance peaks. The results obtained are interpreted within an approach based on the coexistence of two phases having different conductivities in the doped manganite crystals. This two-phase interpretation is supported by data from magnetic-resonance investigations, which demonstrate the existence of two magnetic phases over a broad temperature range in $Ln_{0.7}Pb_{0.3}MnO_3$ single crystals. © *1999 American Institute of Physics.* [S1063-7834(99)02111-5]

The heightened interest in manganese oxides with a perovskite structure and the general formula $R_{1-x}A_x\mathrm{MnO}_3$ (where R is a rare-earth ion, and $A=$Sr, Ba, Ca, Pb) is due to their unusual magnetic and electrical properties. This is mainly because the materials in this family have a large isotropic negative magnetoresistance, which has been termed giant magnetoresistance (GMR). At the same time, it has been established that the magnetic and electrical properties of manganites undergo significant changes as the composition, concentration, pressure, and temperature are varied. There is a hope that materials with acceptable GMR parameters for use in practical magnetic data storage devices and other microelectronics devices will be obtained. Nevertheless, the concentrated research efforts employing every possible method in recent years have not lead to a complete understanding of the mechanisms which account for the entire spectrum of observed magnetic and transport properties and the significant correlation between them.

The mechanism of electronic phase separation has been discussed as one of the possible models which accounts for GMR in substituted manganites.[1–3] In the case of such separation, a sample consists of regions with different carrier concentrations under thermodynamic equilibrium, while the sample is homogeneous from the crystallographic standpoint. The state of such a two-phase system is strongly sensitive to the effects of external factors, such as the temperature, external magnetic field, and pressure. It should probably be expected that such a system will be sensitive to the effects of direct and, especially, alternating currents of various frequencies and amplitudes (we are referring to appreciable probe currents) either without a field or in an external magnetic field. A phenomenon associated with the effect of a current pulse on the behavior of a system undergoing separation of phases has been observed in the degenerate semiconductor EuTe,[4] whose two-phase AFM–FM state is considered proved. As for the investigation of manganites, most of the data presented in the literature refer to resistance and magnetoresistance measurements in a direct current (with small probe currents), but there are scarcely any data from investigations of the magnetoresistive effect in alternating currents in different frequency ranges. The series of publications devoted to the investigation of the magnetoresistive effect at microwave frequencies[5–7] should be noted. One characteristic observation in these investigations is the existence of significant differences in the temperature and magnetic-field dependences of the samples measured in a direct current and in the microwave range.

In this communication we report the results of investigations into the influence of direct and alternating currents on the magnetic and electrical properties of manganite single crystals exhibiting GMR. The changes in the state of the materials investigated was monitored by determining the variation of their losses in the microwave frequency range.

1. EXPERIMENTAL

The investigations were performed on $Ln_{1-x}Pb_xMnO_3$ single crystals grown by spontaneous crystallization from a molten solution. One special feature of the technology was that PbO and PbF_2 served as the solvent and, at the same time, acted as the source of Pb in the crystals. The mixture of La_2O_3, MnO_2, and the solvent was prepared in such a manner that a Pb concentration $x \sim 0.3$ would be ensured. The crystals were grown in a platinum crucible. The technological regime selected ensured the formation of crystals with the mean dimensions $5 \times 5 \times 5$ mm^3. X-ray analysis of the single crystals obtained confirmed the structure and lattice constants corresponding to $x \sim 0.3$.

The samples for the investigation were prepared in the form of wafers measuring $4 \times 2 \times 0.1$ mm^3, and the surfaces of the wafers were polished. The conductivity in the microwave range σ_{MW} was measured using a magnetic-resonance spectrometer[8] with a working frequency of 10.6 GHz. The sample was placed within a three-dimensional rectangular cavity at the maximum of the electric component of the microwave electromagnetic field. Clamped contacts in the form of thin needles were used to pass a current through the sample.

The power reflected from the cavity with the sample P_c or its variation as a function of temperature, external magnetic field, and the amplitude and frequency of the voltage applied to the sample at a constant value of the input microwave power P_{in} was measured during the experiments.

We note that, in the procedure usually used to measure the microwave conductivity σ_{MW}, a sample having a high conductivity is placed at the maximum of the magnetic component of the microwave electromagnetic field. In this case it is not difficult to obtain a dependence which directly relates σ_{MW} to the power reflected from the cavity P_c. However, if the sample is ferromagnetic, the situation is complicated to a considerable extent, because in that case it is necessary to take into account the contribution of the dynamic permeability μ, which depends on the frequency of the electromagnetic radiation, the external magnetic field, and the magnetization of the sample, to the microwave absorption.

In the case where the dimensions of the sample are much smaller than the wavelength of the electromagnetic radiation and the thickness of the skin layer δ is larger than the smallest dimension of the sample or of the same order, we can utilize the procedure for measuring σ_{MW} in which the sample is placed at the maximum of the electric component of the microwave field in the cavity.[9] Using perturbation theory, we can obtain the relationship between the complex dielectric constant of the sample $\varepsilon = \varepsilon' - i\varepsilon''$ and the parameters of the cavity. For conductive materials $\varepsilon'' = \sigma_{\mathrm{MW}}/\omega$, and, as a rule, $\varepsilon'' \gg \varepsilon'$. In the absence of depolarizing factors

$$\frac{1}{Q} - \frac{1}{Q_0} \approx (\varepsilon'' E_m^2 \Delta V)\left(\varepsilon_0 \int_{V_0} E_0^2 \, dV\right)^{-1} = (\sigma_{\mathrm{MW}} E_m^2 \Delta V)\left(\varepsilon_0 \omega \int_{V_0} E_0^2 \, dV\right)^{-1},$$

where Q_0 is the Q factor of the cavity without a sample, Q is the Q factor with a sample, ΔV is the volume of the sample, V_0 is the volume of the cavity, E_m is the strength of the electric field at the antinode, E_0 is the unperturbed electric field in the cavity, and ε_0 is the dielectric constant of the cavity medium. Thus, σ_{MW} is related to the Q factor of the loaded cavity, and while measurement of the absolute value of σ_{MW} raises certain difficulties, it is simple to evaluate its variation in response to the influence of external factors from the change in the microwave power reflected from the cavity ΔP_c.

The use of such a method for measuring σ_{MW} was substantiated in our case, since the skin depth for the samples was $\delta > 0.6$ mm over the entire temperature range, and the contribution of μ to the microwave power absorbed by the sample was essentially eliminated.

The measurements were performed in comparatively weak magnetic fields up to $H = 7$ kOe. The direction of H coincided with the direction of the current in the sample. The resistivity of the materials in a constant current and in a low-frequency alternating current was measured by the traditional four-point probe technique. The measurements were performed in a flow-through nitrogen cryostat in the temperature range 100–360 K.

2. RESULTS AND DISCUSSION

As was noted above, the variation of the parameters of the microwave response (the power reflected from the cavity P_c) contains information on the variation of the conductivity of the material σ_{MW}. Therefore, the magnitude of the relative change in P_c was taken as the principal parameter to be monitored.

Figure 1a presents the dependence of the power reflected from the cavity P_c on the constant voltage U_- applied to the sample in a zero magnetic field and in a field of intensity $H = 7$ kOe. Our attention is drawn to the extremely nonlinear course of $P_c(U_-)$, the slight asymmetry of the dependence relative to $U_- = 0$, and the increase in the relative influence of the external magnetic field with increasing U_-. Such a dependence cannot be attributed to simple heating of the sample. First, the dc current–voltage characteristic is essentially linear at the same values of U_- (Fig. 1b). Second, we directly monitored the temperature of the sample, and the maximum heating did not exceed 1 K even after prolonged passage of current. Finally, under the effect of an alternating current, in the case of heating, everything would be reduced to a change in P_c by a constant amount.

We performed experiments to determine the parameters of the microwave response ΔP_c with a low-frequency alternating voltage in the range from 20 Hz to 10 kHz acting on the sample. These results turned out to be very interesting and informative. When the sample was excited by an alternating current of frequency f_0, the microwave response signal consisted of a set of even and odd harmonic components:

$$\Delta P_c(t) = \sum_n A_n \cos(n f_0 t).$$

The component at $2f_0$ had the largest amplitude, and the even harmonics were generally more intense than the odd harmonics. The amplitudes of each harmonic component in the microwave response signal and the ratio between the amplitudes of the harmonics depended on the amplitude $U_\sim$ of the voltage applied to the sample. Figure 2 presents plots of the dependence of the amplitude of the even and odd harmonics in the microwave response on $U_\sim$ for $f_0 = 1$ kHz.

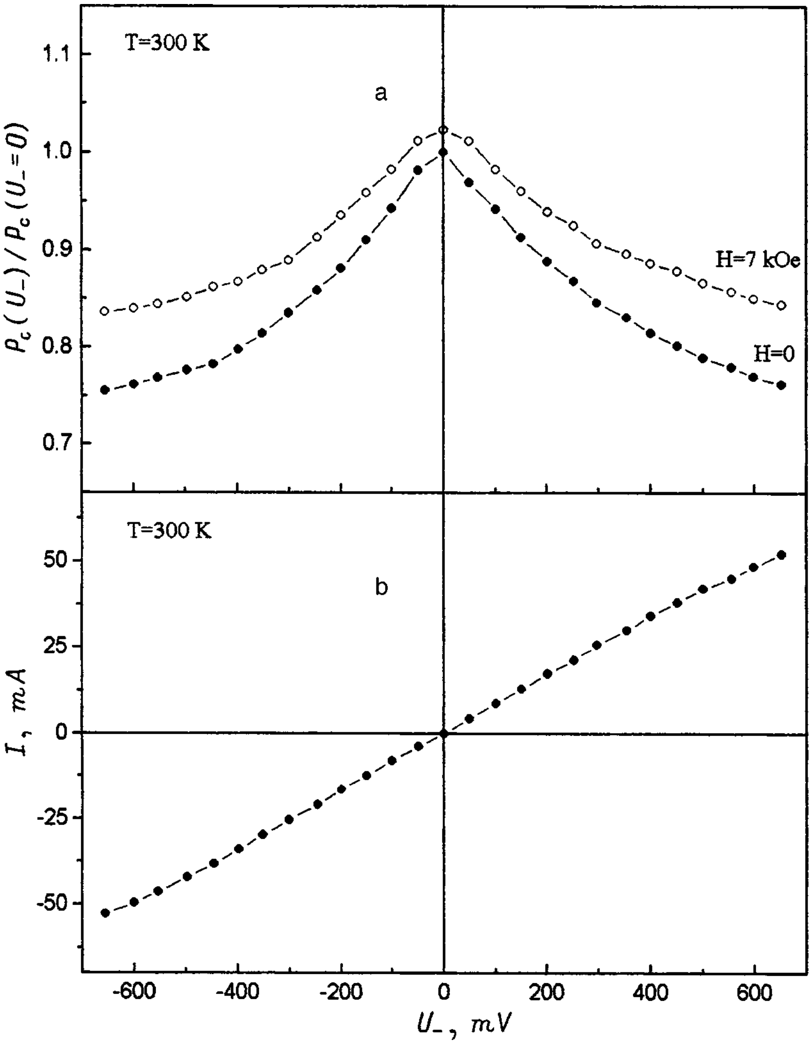

FIG. 1. a—Dependence of the power reflected from the cavity P_c on the dc voltage U_- passed through a $La_{0.7}Pb_{0.3}MnO_3$ single crystal. The value of the power is normalized to the quantity $P_c(U_-=0, H=0)$. $T=300$ K. b—Current–voltage characteristic of the sample. $T=300$ K.

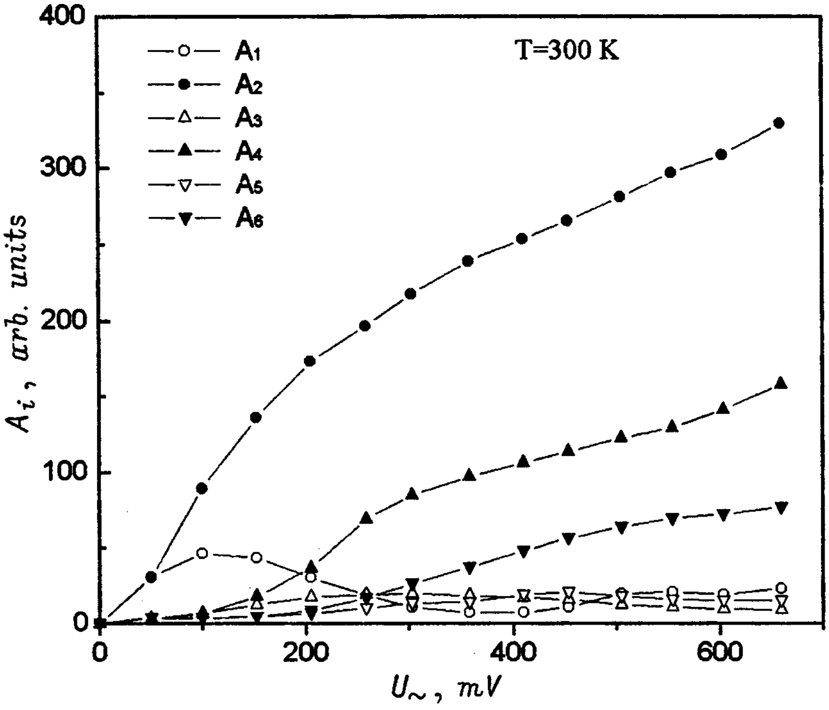

FIG. 2. Dependence of the amplitudes of the odd (A_1, A_3, and A_5) and even (A_2, A_4, and A_6) harmonics in the microwave response signal on the alternating voltage $U_\sim$ with a frequency $f_0=1$ kHz applied to the sample. $T=300$ K.

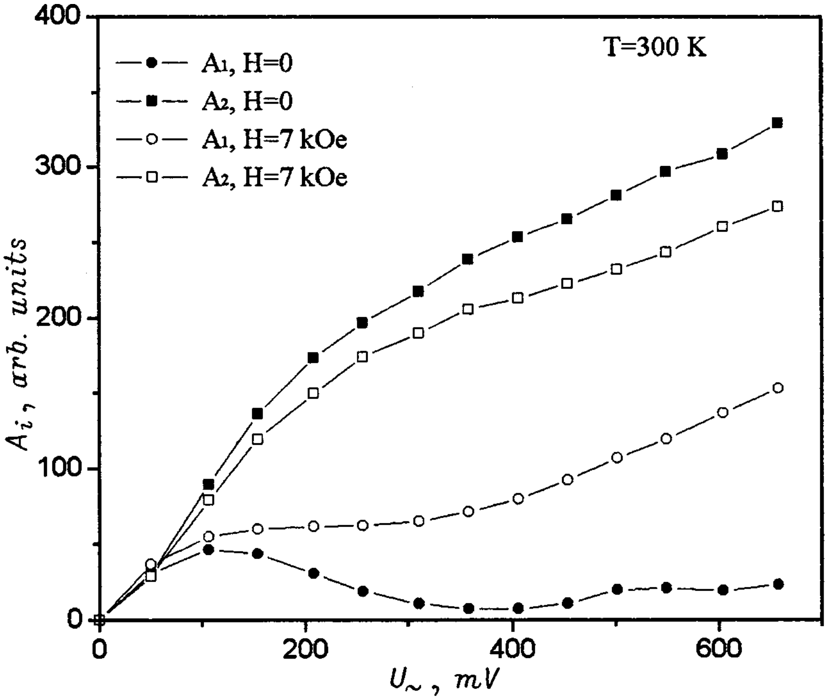

FIG. 3. Dependence of the amplitude of the first (A_1) and second (A_2) harmonics in the microwave response signal on the alternating voltage $U_\sim$ ($f_0 = 1$ kHz) applied to the sample in magnetic fields of intensity $H=0$ and $H=7$ kOe. $T=300$ K.

The harmonics from f_0 to $6f_0$ are shown, although our instrumentation allowed us to observe a component in ΔP_c with a frequency of $12f_0$. Measurements by the four-point probe technique at $f_0=1$ kHz did not reveal any nonlinear effects, and the current–voltage characteristic, as in the case of a direct current, was essentially linear. The linear current–voltage characteristic and the special additional experiments that we performed with variation of the shape and material of the electric contacts allow us to rule out any hypothesis that the effects observed are due to contact phenomena.

Next, for simplicity, let us dwell on the behavior of the first and second harmonics in the microwave response signal. Figure 3 shows the influence of the external magnetic field on the behavior of the amplitude of the first (A_1) and second (A_2) harmonics as a function of $U_\sim$ at $T=300$ K. The value of A_1 depends most strongly on the strength of the external magnetic field H (A_1 increases with increasing H), and the amplitude of the second harmonic A_2 decreases slightly. Figures 4a and b present the temperature dependences of A_1 and A_2 for $H=0$ and $H=7$ kOe. An alternating voltage of constant amplitude $U_\sim=500$ mV with a frequency $f_0=1$ kHz was maintained on the sample over the entire temperature range. For comparison, Fig. 4c presents the temperature-dependent behavior of the resistivity ρ and the magnetoresistance $\Delta\rho/\rho_0=(\rho(0)-\rho(H)/\rho(0))\cdot 100\%$ of a sample in a field of intensity $H=7$ kOe, which was obtained from dc measurements by the four-point probe method.

The temperature dependence of the amplitude of the second harmonic A_2 in the microwave response signal qualitatively mimics the behavior of the dc magnetoresistance. The maximum of A_2 and the strongest influence of the magnetic field H are observed at $T\sim 325$ K, and the maximum of $\Delta\rho/\rho_0$ corresponds to the same temperature. In the temperature range from 250 to 330 K a decrease in A_2 is observed in an external magnetic field. At $T<250$ K and $T>330$ K, A_2 rises slightly with increasing H.

The unusual temperature dependence of the amplitude of the first harmonic in the microwave signal A_1 became more understandable after we recorded the dependences of A_1 and A_2 on the frequency f_0 of the modulating voltage applied to the sample for various temperatures (Fig. 5). In general, the amplitude of the microwave response signal at a fixed temperature varied only slightly for frequencies of the alternating voltage on the sample up to 5 kHz. When f_0 was increased further, the amplitude of the signal quickly decreased, and above 10 kHz no microwave response signal was observed.

When $H=0$, the amplitude of the first harmonic A_1 depends weakly on f_0 and has a smooth curve at all temperatures. In an external magnetic field there are resonance peaks of A_1 at certain frequencies (in the range from 200 Hz to 2 kHz; see Fig. 5). The height of the peaks on the plot of A_1 versus f_0 increases with increasing H without shifts along the frequency scale. Features in the form of two peaks in the spectrum of A_1 ($H=7$ kOe) begin to be displayed at $T=355$ K (Fig. 5c). As the temperature is lowered, their height increases, and they undergo slight frequency shifts (Fig. 5b). Below $T\sim 300$ K the peaks transform into a single maximum (Fig. 5a), whose height decreases as the temperature is lowered further. The features on the plot of $A_1(f_0,H=7$ kOe$)$ vanish at $T\sim 100$ K, and it then coincides with the $A_1(f_0,H=0)$ curve. The maximum sensitivity of A_1 toward the magnetic field is observed at $T\sim 325$ K, which corresponds to the maximum of the dc magnetoresistive effect.

Observing A_2 in the frequency range from 20 Hz to 2 kHz did not reveal any features. Increasing the external

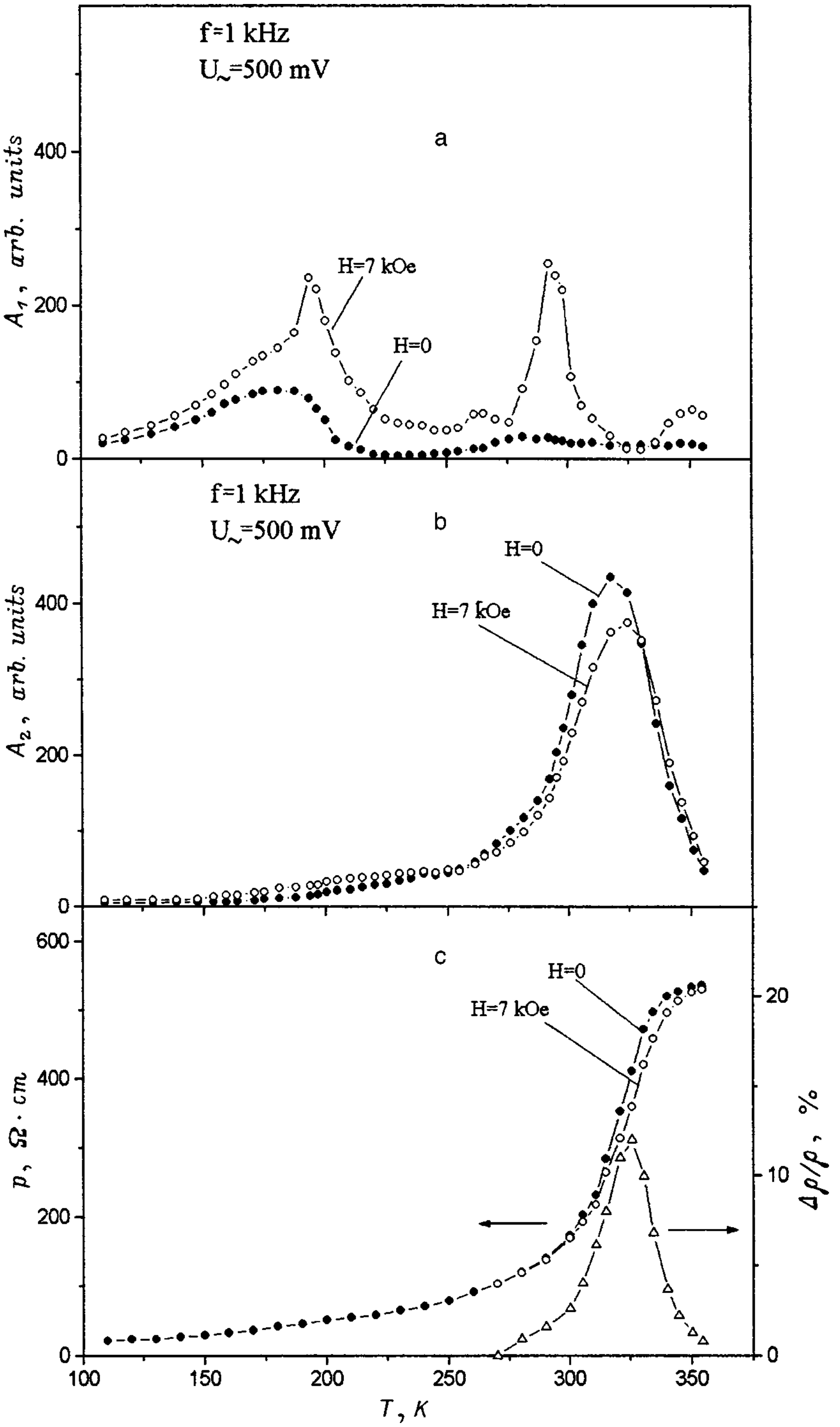

FIG. 4. Temperature dependence of the amplitude of the first A_1 (a) and second A_2 (b) harmonics in the microwave response signal with an alternating voltage $U_{\sim} = 500$ mV ($f_0 = 1$ kHz) on the sample in magnetic fields of intensity $H = 0$ and 7 kOe. Temperature dependence of the resistivity ρ and the magnetoresistance $\Delta\rho/\rho_0$ in a magnetic field of intensity $H = 7$ kOe recorded under dc conditions (c).

magnetic field changed A_2 by the same amount over the entire frequency range considered.

As was shown above, the variation of the microwave response signal ΔP_c is associated with changes in the conductivity in the microwave range σ_{MW}. Our data provide evidence that the passage of a constant current through a sample of a $\mathrm{La_{0.7}Pb_{0.3}MnO_3}$ single crystal leads to a decrease in its conductivity in the microwave range σ_{MW}. As would be expected, an external magnetic field H increases σ_{MW}, the relative influence of H being enhanced as the constant voltage on the sample U_{-} is increased. The change in σ_{MW} in response to the passage of a current does not depend on its direction in the crystal. The effect of the external magnetic field H is isotropic, i.e., there is no dependence on the relative orientation of the direction of the transport current and H.

The sharp difference between the behavior of σ_{MW} and the conductivity measured under dc conditions σ_{DC} can be understood, in our opinion, by utilizing the model of a sample in a two-phase state.[1] In this case the manganite single crystal is a thermodynamic equilibrium system of coexisting regions with different carrier concentrations. One of the phases corresponds to regions with high conductivity thus to ferromagnetic (FM) ordering, and the other phase

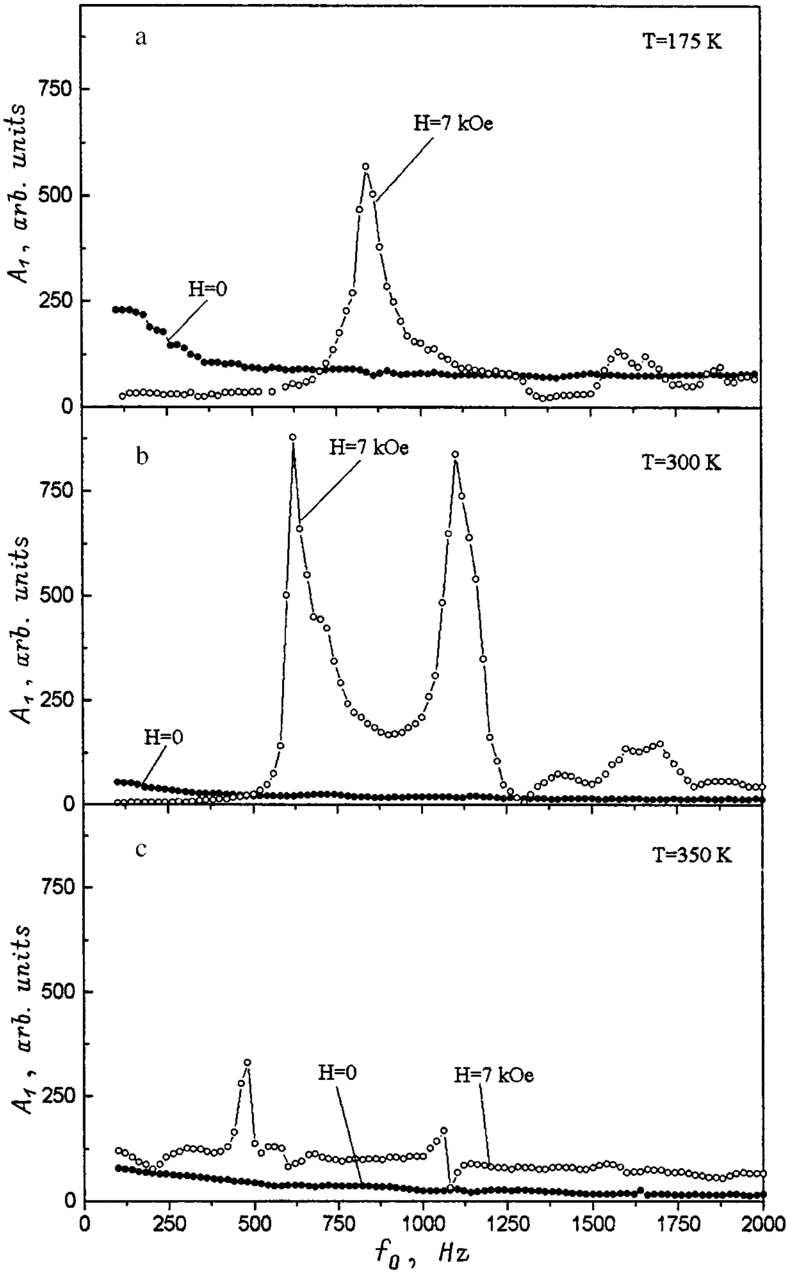

FIG. 5. Dependence of the amplitude of the first harmonic A_1 in the microwave response signal on the frequency f_0 of the alternating voltage on the sample ($U_{\sim}=500$ mV) in magnetic fields of intensity $H=0$ and 7 kOe. a — $T=175$ K, b — $T=300$ K, c — $T=350$ K.

corresponds to regions with a lower conductivity, which are in a different magnetic state, for example, a paramagnetic (PM) state. We shall refer to these phases as the ''metallic'' and ''dielectric'' phases, respectively.

To account for the behavior of the conductivity of a sample in the two-phase state we start with the classical percolation model (see, for example, Ref. 10), in which there are no specific mechanisms for conduction in each of the phases and everything is determined by the ratio between the concentrations of the regions with different conductivities and their topology. At low temperatures the sample consists of a single connected FM ''metallic'' phase, within which regions of the ''dielectric'' phase unconnected to each other can exist. As the temperature is increased, the regions of the ''dielectric'' phase grow, and the volume of the ''metallic'' phase decreases. The ratio between the volumes of the phases at a fixed doping concentration is determined not only by the temperature, but also by the external magnetic field (the magnetic field increases the volume of the FM phase) and probably, as follows from our experiments, by the value of the transport current passed through the sample.

When the conductivity is measured in the microwave range, σ_{MW} manifests itself as the effective averaged value of the conductivities of the ''metallic'' and ''dielectric'' phases. In fact, it is generally known that, if a material is an inhomogeneous mixture and the wavelength of the electromagnetic radiation is much larger than the inhomogeneity scales, such a mixture can be regarded as a homogeneous isotropic medium. However, it will be characterized by definite values of ε and, therefore, of σ_{MW} (Ref. 11). The effective value of ε for an inhomogeneous medium can be calculated exactly in a general form only for certain limiting cases.

When the measurements are performed under dc condi-

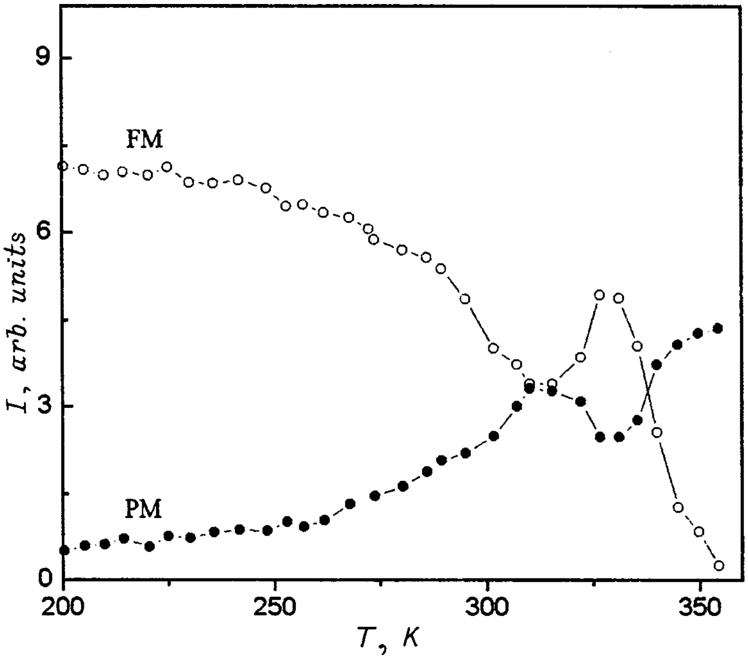

FIG. 6. Temperature behavior of the intensities of the magnetic resonance lines ($\nu = 10.6$ GHz) corresponding to the PM and FM phases in a $La_{0.7}Pb_{0.3}MnO_3$ single crystal.

tions, for which there is percolation through the ''metallic'' phase, the conductivity σ_{DC} is determined mainly by the conductivity of that phase. Therefore, small variations in the ratio between the concentrations of the phases do not have an appreciable effect on the measured value of σ_{DC}. In order to produce an appreciable change in σ_{DC}, a stronger magnetic field must be applied. The same applies to the transport current, whose influence on σ_{DC} should be appreciable at large values of the voltage applied to the sample, but in this case considerable warming of the sample will occur, and correct planning of an experiment is not possible.

As the temperature is increased, the FM order is suppressed, and the concentration of the ''dielectric'' phase increases. Above a certain temperature T_0, the quantity of the ''metallic'' phases decreases so much that it is transformed from a single connected region into numerous separate connected regions, and the flow of current in it through the entire sample ceases. Therefore, the conductivity is determined mainly by the ''dielectric'' phase. It is understood that the strongest conductivity changes and the strongest sensitivity to the magnetic field and the transport current should be manifested within the percolation model in the vicinity of T_0, which can be regarded as the threshold temperature for percolation in the ''metallic'' phase. In our case $T_0 \sim 325$ K, and the maximum of $\Delta\rho/\rho_0$ is achieved at just that temperature.

As an additional argument in support of the idea that two phases coexist in the manganites we investigated, we present the results of magnetic-resonance investigations of $Ln_{0.7}Pb_{0.3}MnO_3$ single crystals. At $T > T_c \sim 355$ K a magnetic-resonance absorption line corresponding to the PM state of the crystal is observed. Below T_c two magnetic-resonance lines were observed down to ~ 100 K. The line corresponding to the PM state underwent strong broadening as the temperature was lowered, the resonance field H_r remained unchanged, and its intensity decreased practically monotonically except in the temperature range from 315 to 340 K (Fig. 6). The intensity of the absorption line corresponding to the ferromagnetic (FM) state increased with decreasing temperature, H_r decreased most strongly at values of T ranging from 355 to 290 K and then reached a plateau. The features of the behavior of the intensities of the FM and PM resonance lines in the vicinity of $T_0 \sim 325$ K, which corresponds to the maximum magnetoresistive effect, are noteworthy. They can be attributed to the fact that the external magnetic field H most effectively increases the concentration of the FM phase in the sample at these temperatures.

The nonlinear form of the dependence of the power reflected from the cavity P_c on the value of the voltage U_{-} applied to the sample (Fig. 1) allows us to postulate some manifestation of this nonlinearity in the microwave response signal upon the application of an alternating voltage $U_{\sim}$ with a low frequency f_0 to the sample, for example, in the form of the appearance of high-order harmonic components. The essentially fully symmetric form of the characteristic allows us to expect that the principal microwave response signal will be observed at the doubled frequency of the modulating voltage $2f_0$ and that the presence of even harmonics of the response signal is possible. This is confirmed by our experiments.

According to Fig. 1, the external magnetic field H should diminish the amplitude of the microwave response, as we observed in the case of A_2 (Fig. 3). The qualitatively similar form of the temperature dependences of A_2 and $\Delta\rho/\rho_0$ corroborates the direct connection between the mechanisms responsible for the influence of the external magnetic field and the transport current on the electrical properties of the manganite single crystals, which has a reasonable explanation within the theory of the two-phase state of the samples.

The reason for the appearance of the f_0 component and the odd harmonics in the microwave response signal and especially for the resonant increase in A_1 in an external magnetic field upon variation of the frequency f_0 of the modulating voltage on the sample is still unclear. This behavior can most probably be associated within the two-phase approach with the dynamics of the displacement of the boundaries of the regions of the ''metallic'' and ''dielectric'' phases or with the mobility of the regions as a whole. When the frequency of the modulating voltage acting on the sample f_0 coincides with natural vibrational frequencies of the boundaries, a resonant increase in A_1 is observed, and the external magnetic field can be regarded as the quantity which effectively influences the dissipation parameters ω_d of the system under consideration and, consequently, the intensity of the resonance lines in the spectrum. The main factor causing the diffuseness of the resonance spectrum of the boundaries is the complex geometry of a two-phase state with a distribution of the regions belonging to the different phases according to size and shape. As the temperature is varied, significant restructuring of the two-phase state of the system occurs, which is reflected in the spectra recorded at different temperatures.

We are intentionally presenting only qualitative arguments, since there are numerous questions which preclude making correct quantitative estimates. First of all, the mechanism of the separation of phases in the materials that we studied is unclear. For example, realization of a mechanism of an impurity separation of phases associated with the inhomogeneous distribution of the impurity in the crystal is possible. On the other hand, in this case, as opposed to the mechanism of an electronic separation of phases, it is difficult to expect strong sensitivity to the effects of external factors, such as the external magnetic field and the transport current. The question of the mechanism underlying the influence of the transport current on the conductivity σ_{MW} of $\mathrm{La_{0.7}Pb_{0.3}MnO_3}$ single crystals that we discovered remains open. The topology of the two-phase state, which varies radically with temperature, is unknown. The concrete mechanisms of conduction in each of the phases must also be taken into account.

Thus, in this report we have presented the results of experiments on the influence of direct and alternating currents on the conductivity of $\mathrm{La_{0.7}Pb_{0.3}MnO_3}$ single crystals in the microwave range σ_{MW}. The investigations have shown that a transport current leads to a decrease in σ_{MW}, while an external magnetic field, as would be expected, increases the conductivity. The strongest influence of a current is observed at the temperature where the maximum of the magnetoresistive effect recorded under dc conditions is observed. Under the effect of an alternating current the crystals display nonlinear properties, which are manifested in the generation of harmonics in the microwave response signal from the cavity containing the sample. A dependence of the microwave response on the frequency of the alternating current in the sample in an external magnetic field has been discovered, and its course has a resonance character.

The results obtained can be described directly within the theory of phase separation in crystals of doped manganites using the classical percolation approach. A separation of phases is also supported by magnetic-resonance investigations, which demonstrated directly the coexistence of FM and PM phases in the samples over a broad temperature range.

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