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Cavity Perturbation Techniques for Evaluation of Microwave Resistivities

Yoshiyuki WATANABE and Masahiro OKA
Department of Physics, Shimane University, Matsue 690, Japan
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The cavity perturbation technique based on the eddy current loss method is modified so as to enable a rather quick measurement of the temperature dependence of microwave resistivities. A heavily doped p-type Si single crystal is employed as a reference to evaluate the resistivities. Some experimental data are given on polycrystalline MnSe specimens.

§ 1. Introduction

The cavity perturbation technique has widely been used for the evaluation of the resistivity of compounds.[1,2,3] Recently, the technique based on the eddy current loss method[4] was improved,[5] and some interest has been directed towards this improved method, since it provides simple means for the determination of the relatively low microwave resistivities.[6,7] Remarkable features of this method are as follows. (1) No direct measurement of Q-value is involved, (2) no explicit knowledge of a geometrical constant C of the resonant cavity is required and (3) measurements are only performed on the relative change of the resonance transmission of the cavity by the sample insertion. By using more than two samples of different size, we can find the value of the microwave resistivities, without any direct evaluation of Q and C. This must serve to the reduction of the experimental error, because the measurement of the change of the Q-value tends to contain in general an appreciable error. A shortcoming of this method, however, may be the long time required to make the actual measurements, especially in low temperature experiments. This is because the replacement of the sample (fixed to the sample holder) disturbs the temperature distribution around the cavity and many hours are required to restore the original temperature. In addition, the sample size suitable for the evaluation of resistivities varies in general with the temperature, i.e., the magnitude of the resistivity. Therefore, we have to perform measurements on many samples with different sizes, for the experiment over a wide range of temperatures.

In the present experiment, we attempted to determine the temperature dependence of the resistivity with use of only one sample of an appropriate size, by comparing it with a reference specimen of known resistivities. This method seems to enable a rather quick measurement of the temperature dependence of microwave resistivities.

In section 2, the formulae of the eddy current loss are reviewed and details of the experimental apparatus will be described in section 3. In section 4, we first demonstrate the measurement of the temperature dependence of the resistivity of Si single crystal with the usual eddy current loss method, i.e., with use of several disk-type samples of different thickness. Then, by employing one of these disks as a reference, we determine the temperature dependence of the resistivity of other materials.

§2. Formulae of Eddy Current Loss

When the sample in the form of sphere or disk is introduced into the microwave cavity and placed at a position of the maximum rf magnetic field, the eddy current loss in the following formulae will be induced within the sample.[5,6)]
(*disk*)

$$P_d = (\pi R^2 L\omega\mu_0/2)\cdot f(\rho/L^2)\cdot|h_0|^2, \qquad (1)$$

where

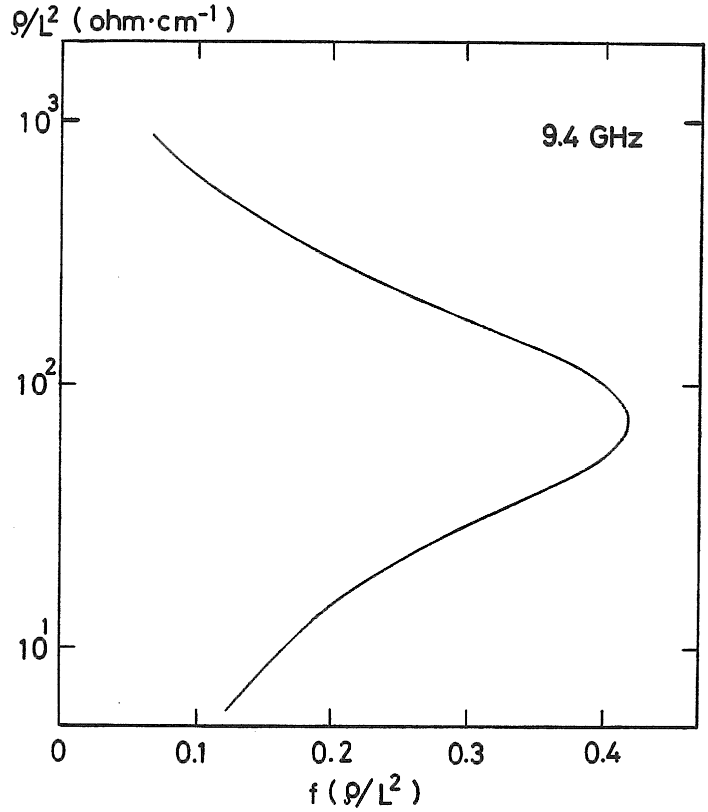

Fig. 1. The quantity f defined by eq. (2) as a function of ρ/L^2; disk-type samples.

$$f(\rho/L^2)=(1/z)\frac{\sinh z-\sin z}{\cosh z+\cos z}, \tag{2}$$

$$z=(\omega\mu_0/2)^{1/2}\cdot(\rho/L^2)^{-1/2}, \tag{3}$$

L : thickness,
$2R$: diameter,
ρ : resistivity,
μ_0 : permeability of free space,
$\omega/2\pi$: frequency of microwave,
h_0 : the maximum value of rf magnetic field.

(sphere)

$$P_s=3\pi a^3\cdot F(\rho/a^2)|h_0|^2, \tag{4}$$

where

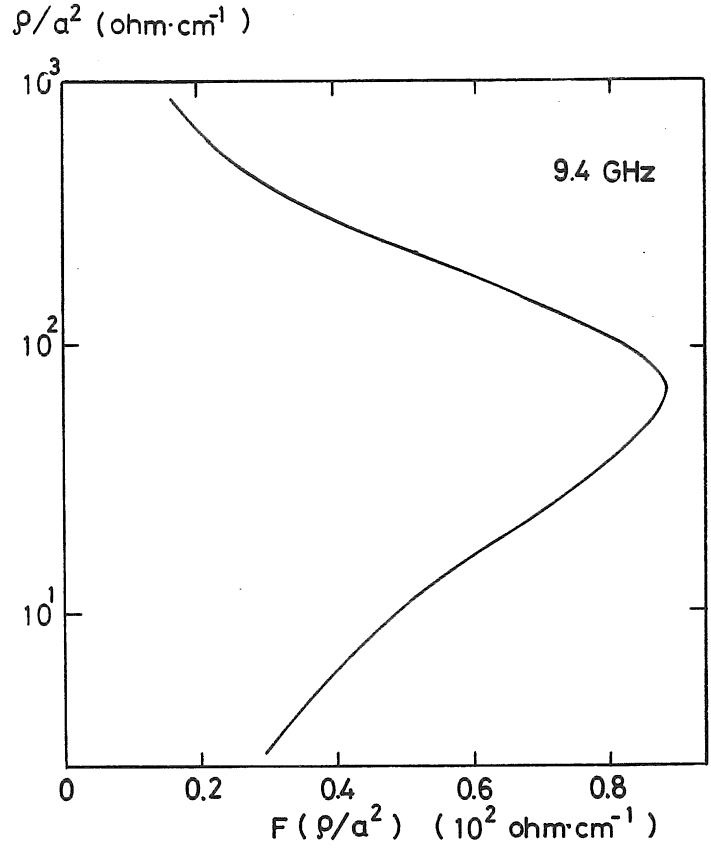

Fig. 2. The quantity F defined by eq. (5) as a function of ρ/a^2; spherical samples.

$$F(\rho/a^2)=(\rho/a^2)\left\{\gamma\frac{\sinh 2\gamma+\sin 2\gamma}{\cosh 2\gamma-\cos 2\gamma}-1\right\}, \quad (5)$$

$$\gamma=(\omega\mu_0/2)^{1/2}(\rho/a^2)^{-1/2}, \quad (6)$$

$2a$: diameter of sphere.

The properties of the cavity vary with the sample insertion, owing to the eddy current loss given by eq. (1) or (4). The ratio Z of the resonance transmission of the cavity with and without sample is then written as

(*disk*)

$$(C/Q_0)(\sqrt{Z}-1)=(\pi R^2 L\mu_0/2)\mathrm{f}(\rho/L^2), \quad (7)$$

(*sphere*)

$$(C/Q_0)(\sqrt{Z}-1)=(3\pi a^3/\omega)\mathrm{F}(\rho/a^2). \quad (8)$$

Here, C and Q_0 are the geometrical constant and the Q-value of the empty cavity. f and F are shown numerically in Figs. 1 and 2 as a function of ρ/L^2 or ρ/a^2.

§ 3. Apparatus

Figure 3 shows the blockdiagram of the present microwave system. The cavity resonates at the TE_{102} mode of the resonance frequency of about 9.4 GHz. The oscillation frequency of the klystron (2K25) is always fixed to the resonance frequency of the cavity by automatic frequency control (AFC) unit.[8] Details of the cavity zone are illustrated in Fig. 4. The sample holder is made of a thin rod of teflon with the diameter of 4 mm, and the sample is fixed to this holder with the adhesive and set in the cavity zone, as shown in the figure. These are inserted into a dewar vessel and the

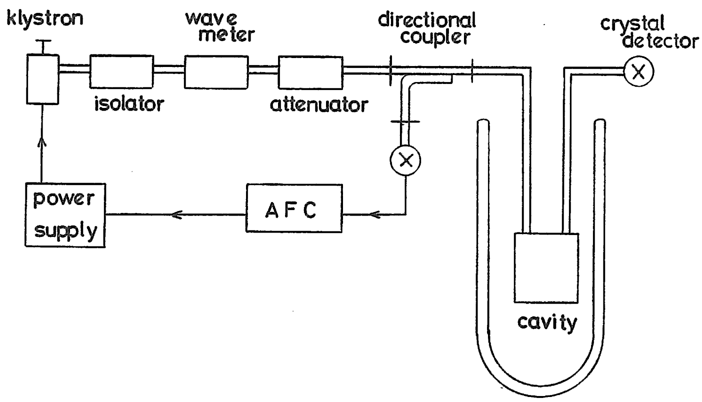

Fig. 3. The blockdiagram of the microwave system.

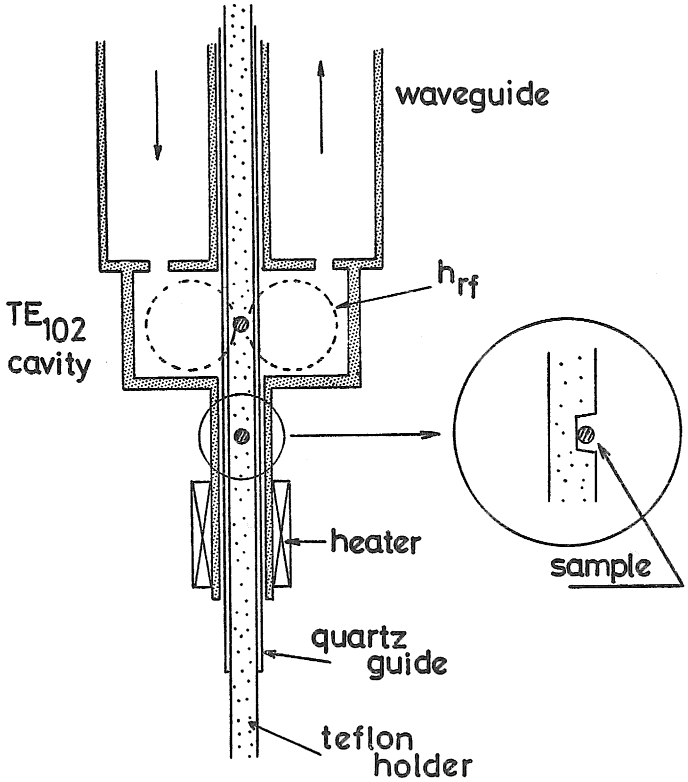

Fig. 4. Rectangular TE_{102} microwave cavity.

temperature of the cavity zone is controlled to a certain value by adjusting the heater-current and the amount of the liquid N_2 in the vessel. By pulling up or down the holder, we can evaluate Z.

§4. Results and Discussion

In this section, we first show the temperature dependence of the microwave resistivity ρ_m of Si single crystal which was evaluated with the eddy current loss method reported in our previous paper.[6] In the earlier eddy current loss method,[4] one had to calculate the C-value and also measure Q_0, in addition to the measurement of Z. In the present case, however, we merely regard the ratio C/Q_0 as an unknown parameter. By measuring Z for several samples of various thickness L(or diameter $2a$) and by substituting these values into eq. (7) (or eq. (8)), we obtain many ρ vs. C/Q_0 curves. Figure 5 shows an example of the ρ vs. C/Q_0 curves obtained at 300 K for disk-type samples of Si single crystal with dc resistivity $\rho_{dc}=1.2\times10^{-2}$ Ωcm (300 K). From the intersection we find the microwave resistivity $\rho_m=1.2$–1.3×10^{-2} Ωcm, that is in

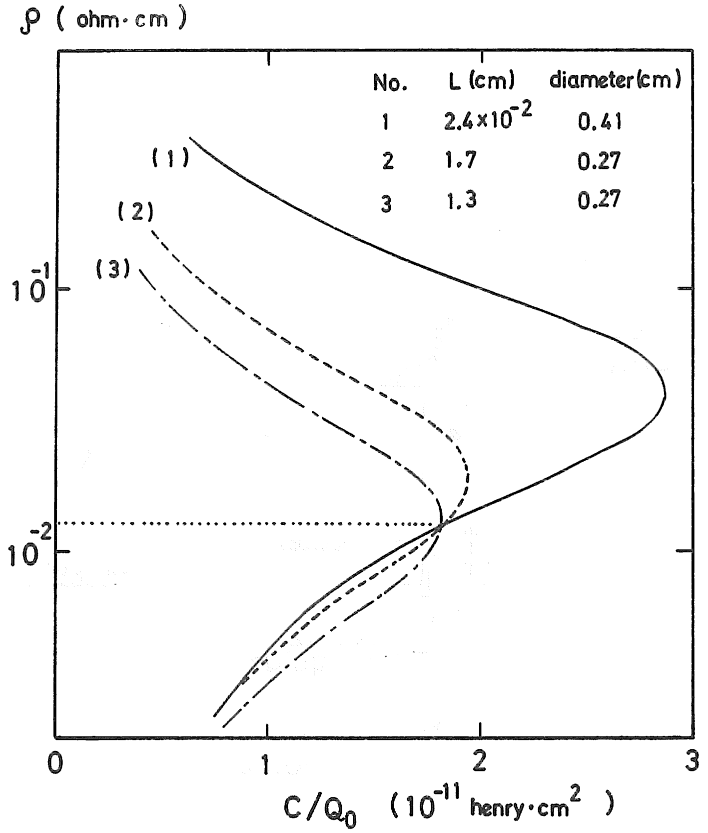

Fig. 5. The ρ vs. C/Q_0 curves obtained at 300 K with disk-type samples of Si of $\rho_{dc}=1.2\times10^{-2}\Omega$ cm (300 K).

agreement with ρ_{dc}. By repeating the similar procedure for various temperatures between 80 and 300 K, we obtain, after a tedious work, the temperature dependence of ρ_m as shown in Fig. 6. In this figure, the open circles denote ρ_{dc} measured with the conventional four point method by using silver paste as a contact material. Although some scattering exists in the measured values of ρ_m, we cannot find any obvious difference between ρ_m and ρ_{dc}. We therefore employ, in what follows, the $\rho_{dc}-T$ curve of the present Si single crystal as a reference in determining ρ_m-values of other materials. As seen in Fig. 6, the present Si crystal (heavily doped p-type one) exhibits only a slight variation with the temperature between 80 and 300 K. This is a favorable behavior for the reference specimen.

Now, we attempt to determine the microwave resistivity of MnSe polycrystals by using one of three disks in Fig. 5 as a reference. Here, MnSe samples are of spherical forms with diameter of $2a=1$–2 mm. We fix both a MnSe sample and a Si reference

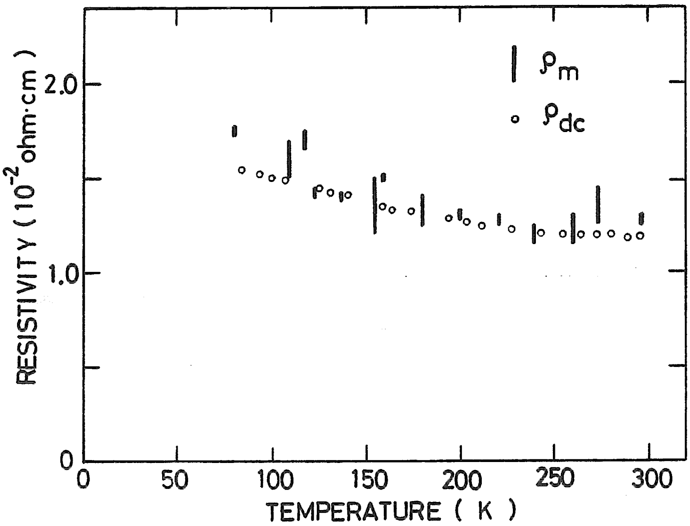

Fig. 6. Microwave resistivities of Si single crystal as a function of temperatures.

on a teflon-holder as shown in Fig. 4. The holder is then inserted into the cryostat and the temperature of the cavity zone containing two samples is kept to a certain value. By pulling up the teflon-holder, we can measure successively the Z-values for both the spherical sample (Z_s) and the reference (Z_r). Z_s and Z_r are related to the resistivities ρ_s, ρ_r by eq. (7) or (8). That is,

$$(C/Q_0)(\sqrt{Z_s}-1)=(3\pi a^3/\omega)\mathrm{F}(\rho_s/a^2), \tag{9}$$

$$(C/Q_0)(\sqrt{Z_r}-1)=(\pi R^2 L\mu_0/2)\mathrm{f}(\rho_r/L^2). \tag{10}$$

We combine eqs. (9) and (10), obtaining

$$\mathrm{F}(\rho_s/a^2)=(\omega\mu_0/6)\cdot(R^2L/a^3)\frac{\sqrt{Z_s-1}}{\sqrt{Z_r-1}}\mathrm{f}(\rho_r/L^2). \tag{11}$$

Substituting $\omega/2\pi=9.4\times10^9$ Hz, $R=0.2$ cm, $L=2.4\times10^{-2}$ cm and $2a=0.175$ cm into eq. (11), we can rewrite it as

$$\mathrm{F}(\rho_m/a^2)=1.80\times10^4\mathrm{f}(\rho_r/L^2)\frac{\sqrt{Z_s-1}}{\sqrt{Z_r-1}}. \tag{12}$$

Since $\mathrm{f}(\rho_r/L^2)$ is known numerically as a function of the temperature (Fig. 7), we can evaluate $\mathrm{F}(\rho_m/a^2)$ for the sample in question directly from the measured values of Z_s and Z_r. We then search out the value of ρ_m graphically through the curve shown in Fig. 2, obtaining two corresponding values of ρ_m at a given temperature. Of course, only one of these is correct and measurements on another sample of the different size distinguish the correct solution of ρ_m. To be emphasized, however, such examinations

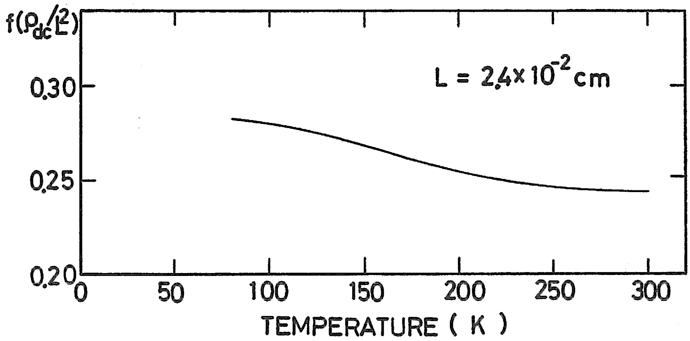

Fig. 7. The temperature dependence of $f(\rho_r/L^2)$ evaluated by assuming $\rho_r = \rho_{dc}$.

only at one or two points of temperatures, *e.g.*, at room temperature, are sufficient for the purpose of the discrimination. Thus, the present procedure to evaluate ρ_m provides a very convenient tool, especially for the measurement over a wide range of temperatures.

In Fig. 8 are shown the temperature dependence of the microwave resistivities of MnSe polycrystal thus obtained (measurements were carried out on the cooling run of temperatures from 300 to 80 K). Resistivities show a sharp change at 180 K, that reflects the structural phase change at this temperature.[9] Details of the $\rho_m - T$

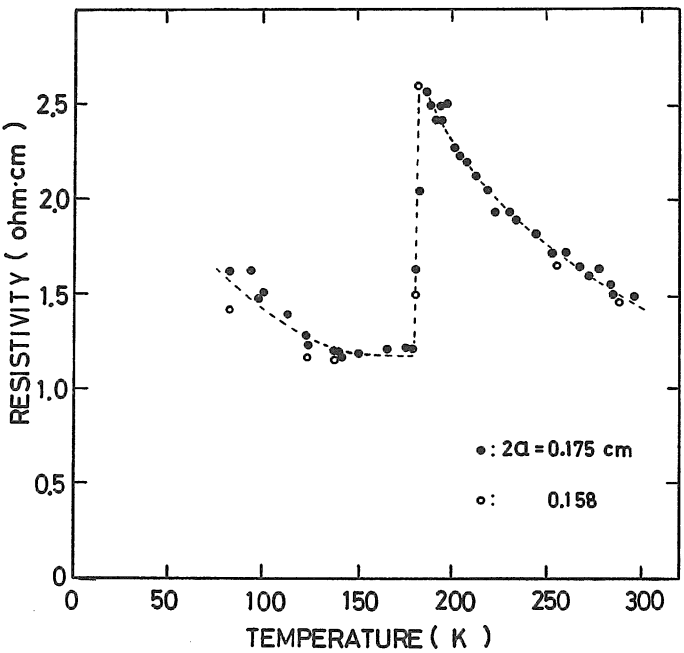

Fig. 8. Microwave resistivities of MnSe.

curve, however, will be discussed elsewhere. Open circles in this figure are the results for another sample of MnSe with a different diameter $2a=0.158$ mm. Agreement between both results is satisfactory, indicating the correctness of the present procedure.

In summary, we first ascertained for our p-type Si single crystal that there is no significant difference between the microwave resistivity and the dc one in the temperature region 80–300 K. Then, by employing this Si sample as a reference, we could determine rather quickly the temperature dependence of the microwave resistivities of another material in question.

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