Ultrasensitive Technique for Microwave Susceptibility Determination Down to 10^{-5}

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An ultrasensitive technique has been developed which allows the measurement of the microwave susceptibility components down to levels on the order of 10^{-5} on small spherical samples of ferrite materials. The novel feature is a feedback scheme to lock the signal klystron onto the 3 dB points of the high-Q cylindrical activity containing the sample. By beating the signal klystron against a crystal-oscillator-stabilized reference klystron, the frequency width of the cavity at half-power can be measured with an error less than a few hundred hertz, leading to the extremely high resolution. The most useful application of the technique to date is the study of microwave relaxation in the form of an effective linewidth several thousand oersteds away from ferromagnetic resonance.

INTRODUCTION

The precise measurement of ferrite microwave susceptibility tensor components at very low levels, on the order of 10^{-3} or smaller, has become extremely important in recent years. On the practical side, such data are required to accurately characterize the losses for devices operated at remanence or at fields far below that required for resonance. In addition, low-level susceptibility measurements have become important in the study of basic microwave relaxation in ferrites. By studying the tail of the resonance absorption, where the susceptibility components are 10^{-3} or smaller, it is possible to determine the intrinsic losses for a given ferrite, even though the actual FMR linewidth is largely due to inhomogeneities of one sort or another.

During the course of an extensive study of relaxation in polycrystals,[1–3] a system has been developed which can easily measure the microwave susceptibility in 2 mm diam ferrite samples down to values on the order of 5×10^{-5}. The ability to make such precise measurements on small spherical samples is extremely useful for three reasons. (1) Very little ferrite material is needed for the measurement. Conventional techniques to measure the susceptibility at low levels have generally required large samples, sometimes completely filling the cavity in order to obtain the needed sensitivity. (2) Only the uniform precession mode is excited, so that the measured susceptibility can be directly related to fundamental relaxation parameters. (3) Because the small sample affects the cavity mode only slightly, perturbation theory is applicable to obtain the susceptibility values and the analysis is greatly simplified. When large samples are used, tedious exact solutions must be used.

I. MEASUREMENT TECHNIQUE

The high sensitivity in the present method results from very high frequency stability and a feedback system to lock the signal klystron on the half-power points of a high-Q cavity. The idea of using feedback to lock the klystron onto the cavity half-power points was first pointed out by Kohane[4] in conjunction with dielectric loss measurements. Very few workers, however, are aware of the extremely high sensitivity of the technique. The stability is such that the half-power frequency-width of a cylindrical TE_{0n} transmission cavity can be determined to within a few hundred hertz at 10 GHz. From the Q and center frequency f_0 of the cavity, the real and negative imaginary parts of the diagonal component of the microwave susceptibility tensor are obtained from standard cavity perturbation theory.

The microwave spectrometer and associated electronics are shown schematically in Fig. 1. The basic transmission cavity spectrometer is of standard design.[4] Attenuators are placed at various points for power level adjustment. A directional coupler samples the klystron output to allow adjustment of the repeller voltage for operation at the top of the klystron mode, and permits wavemeter measurement of the klystron frequency. Another directional coupler feeding a magic T provides a reference output for use in the microwave feedback circuit as well as power to one input of a mixer circuit. The other input comes from a crystal-oscillator-stabilized reference klystron. The beat signal is detected and fed to an electronic counter to measure shifts in the signal klystron frequency. A precision

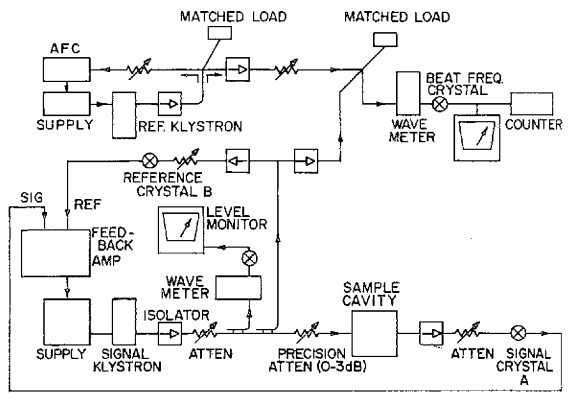

FIG. 1. Schematic block diagram of the microwave instrumentation for ultrasensitive susceptibility measurement.

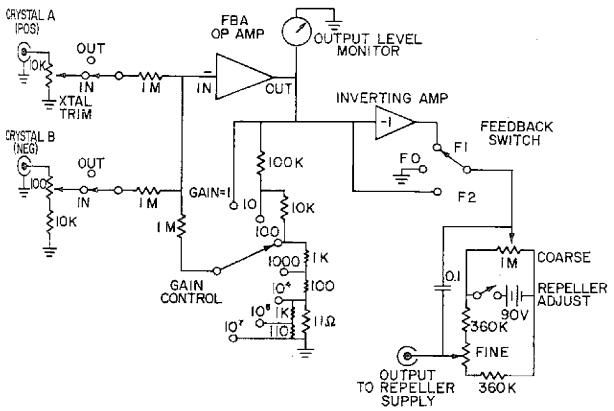

FIG. 2. Schematic diagram of the electronic feedback instrumentation for ultrasensitive susceptibility measurement.

attenuator which can be set at either 0 or 3 dB is placed in the cavity input leg.

A detailed diagram of the feedback electronics is shown in Fig. 2. The gain control adjusts the amount of feedback around the operational amplifier, which has an open loop gain exceeding 10^7. This inner feedback loop should be distinguished from the outer microwave feedback loop which may be closed by means of the feedback switch. In operation, the signal klystron is first tuned to cavity resonance by maximizing the transmitted power, as detected by crystal A (signal crystal) and indicated by the meter on the feedback amplifier. The feedback amplifier (FBA) is set at low gain (100 or 1000) and the outer feedback disconnected (feedback switch in F_0 position). The attenuator in the reference arm is then adjusted to null the amplifier output. The signal crystal (A) and reference crystal (B) are of opposite polarity. This nulling procedure is done with the precision attenuator in the signal arm set for 3 dB attenuation. The "xtal trim" potentiometer should be adjusted so that the null is not affected by small changes in the input power level. (It may be necessary to interchange the inputs to obtain this tracking condition.) The system should be operated with the same crystal output voltages (nominally 100–200 mV) at which these adjustments were made.

The system is now set for zero FBA output when the klystron is set at cavity resonance, with the reference crystal output (as seen at the op amp) equal to the signal crystal output at this point. The next step in the operation is to use this null reference to lock the klystron to one and then the other 3 dB point of the microwave cavity. This is done by removing the 3 dB attenuation in the signal arm by returning the precision attenuator to the 0 dB position, connecting the feedback by putting the feedback switch in position F1 or F2, and increasing the gain of the FBA to greater than 10^6. In the F1 position, the feedback affects the repeller voltage in such a way as to drive the klystron toward the lower frequency 3 dB point of the cavity. The high FBA gain assures that the klystron frequency is locked onto the 3 dB point with negligible error. In the F2 position, the klystron locks on the upper 3 dB point. The half-power frequency width Δf of the cavity is just the difference in counter readings for the two feedback settings. The cavity frequency f is the average of the two values added to or subtracted from the reference klystron frequency, depending on whether the reference klystron is stabilized below or above the cavity frequency. From f and Δf one can determine the microwave susceptibility from cavity perturbation theory.

Perturbation theory[4] yields the following relations for the external microwave susceptibility in terms of measured cavity parameters:

$$4\pi\chi_e''=(2V_cg/V_s)(1/Q_L-1/Q_0), \tag{1}$$

$$4\pi\chi_e'=(4V_cg/V_s)[(f_L-f_0)/f_L]. \tag{2}$$

In the above, χ_e' and χ_e'' are the real and negative imaginary parts of the diagonal component of the susceptibility tensor corresponding to the direction of the microwave field (linearly polarized), and V_c and V_s refer to the cavity and sample volumes with $V_s \ll V_c$. The subscript L indicates that the measurement is made (cavity Q, Q_L, or center frequency f_L) at the field for which the susceptibility measurement is desired. Q_0 and f_0 refer to the same cavity parameters extrapolated to infinite field (see Sec. IV). The quantity g is a factor which relates the cavity energy W to the microwave field amplitude h, according to $W=gh^2V_c/4\pi$ (cgs units). For a cylindrical TE_{011} cavity, g is given by

$$g=\tfrac{1}{4}J_0^2(x_{11})(1+R^2\pi^2/L^2x_{11}^2), \tag{3}$$

where x_{11} is the first zero of J_1. J_0 and J_1 are zero and first order Bessel functions of the first kind. R and L are the radius and length of the cavity, respectively. For the present 10 GHz cavity with $L=3.81$ cm and $R=1.99$ cm, the factor $2V_cg/V_s$ is equal to 1.1×10^3 for a 2 mm diam sample.

II. SYSTEM ERRORS AND RESOLUTION

Ultimately, the sensitivity of the system depends on the accuracy with which the cavity frequency f and half-power frequency width Δf can be measured. The width Δf is given by (f_2-f_1) and f is given by $\frac{1}{2}f_2+\frac{1}{2}f_1+f_{\text{ref}}$, where f_2 and f_1 are the counter readings from the procedure of Sec. I, and f_{ref} is the frequency of the crystal-oscillator-stabilized reference klystron. The $(1/Q_L-1/Q_0)$ term in Eq. (1) can be expressed as

$$\frac{1}{Q_L}-\frac{1}{Q_0}=\frac{(f_{2L}-f_{1L})-(f_{20}-f_{10})}{f_{\text{ref}}}\cdot\frac{(f_{2L}^2-f_{1L}^2)-(f_{20}^2-f_{10}^2)}{2f_{\text{ref}}^2}, \tag{4}$$

TABLE I. Typical data: porous YIG 2.373 mm diameter, $4\pi M_s = 1560$ G, $f_0 = 9.97276$ GHz, $Q_0 = 25132$.

H_0(kOe)	f_0(GHz)	Q	$4\pi\chi_e'$	$4\pi\chi_e''$	ΔH_{eff}(Oe)
0.600	9.97340	24230	−0.08394	0.00096	13.0
0.800	9.97360	24327	−0.10949	0.00086	11.3
1.000	9.97383	24327	−0.14041	0.00086	10.4
1.200	9.97411	24292	−0.17599	0.00090	9.6
1.400	9.97442	24187	−0.21670	0.00101	9.4
1.600	9.97478	24025	−0.26455	0.00120	9.4
1.800	9.97522	23746	−0.32173	0.00151	9.7
2.000	9.97562	23284	−0.39230	0.00206	10.5
2.200	9.97646	22445	−0.48341	0.00311	12.0
2.400	9.97740	20632	−0.60633	0.00566	15.6
2.600	9.97877	16387	−0.78487	0.01384	25.4
2.800	9.98095	9037	−1.07029	0.04620	49.8
5.000	9.96715	23005	0.73354	0.00240	8.8
5.500	9.96851	24095	0.55595	0.00112	7.6
6.000	9.96929	24509	0.45399	0.00066	7.0

where use has been made of the condition $f_{\text{ref}} \gg f_1, f_2$. The observed random error in reading f_1 or f_2 is about ±200 Hz. This error is partly due to the ±100 Hz short term stability for the crystal-oscillator-stabilized klystron at f_{ref}. There is no provision in the system, however, to actually determine f_{ref} itself with this kind of precision, so that the measurement error for f_{ref} is given by the wavemeter resolution of about 1.0 MHz. The reference line is normally operated at 9.90 GHz, while the cavity frequency is about 9.97 GHz. The cavity width is about 400 kHz ($Q \approx 25\,000$), and values on the order of 1 kHz for $\Delta f_L - \Delta f_0$ are normally resolved. With these typical operating parameters, the dominant error comes from the first term in Eq. (4),

$$\delta\left(\frac{1}{Q_L}-\frac{1}{Q_0}\right)\approx\frac{(\delta f_{2L}-\delta f_{1L})-(\delta f_{20}-\delta f_{10})}{f_{\text{ref}}}. \tag{5}$$

For $|\delta f_{1,2}| = 200$ Hz the error in $1/Q_L - 1/Q_0$ is about 4×10^{-8}. This corresponds to an error in $4\pi\chi_e''$ of 4×10^{-5} for a 2 mm diam sample.

In calculating the error in $(4\pi\chi_e')$, it is necessary to remember that the 1.0 MHz error in f_{ref} is not random but is simply a shift due to a lack of resolution in reading the wavemeter. Let this shift be denoted by $\Delta f_{\text{ref}} \approx 1$ MHz (wavemeter resolution). The error in $(f_L - f_0)/f_L$ is given by

$$\delta\left(\frac{f_L-f_0}{f_L}\right)\approx\frac{1}{2f_L}(\delta f_{2L}+\delta f_{1L}-\delta f_{20}-\delta f_{10}) + \frac{\Delta f_{\text{ref}}}{f_L}\left(\frac{f_0}{f_L}-1\right). \tag{6}$$

At fields far from resonance, the quantity $|(f_0/f_L - 1)|$ is generally less than 5×10^{-4} and the dominant error comes from the first term in Eq. (6). The resulting error in $(f_L - f_0)/f_L$ is about 5×10^{-8}, with a corresponding error of 10^{-4} for $4\pi\chi_e'$.

In addition, there is a small systematic error in Δf due to crystal tracking errors. Even after the xtal trim control in Fig. 2 has been set for optimum tracking, there are still small deviations from null as the power level is changed. With the trim adjusted for tracking at 80 mV crystal output, a 0.2 mV deviation as seen at the op amp summing point was noted when the over-all power level was reduced 3 dB. With the system adjusted for coincidence of the mode curve maximum (X-13 klystron) and cavity resonance, this deviation from null results in an increase in the measured Δf by about 100 Hz, well within the limits of the random error. For cavities with lower Q, however, this systematic error in Δf could be substantially larger. In such cases, it is necessary to mechanically retune the klystron to the top of its mode at each 3 dB point.

There is also a possible systematic error due to thermal drift, which results from expansion or contraction of the cavity. If the drift is such that the cavity is shifting to higher frequency, for example, the measurement of F1 (low frequency 3 dB point) prior to measuring F2 (high frequency 3 dB point) will cause the apparent width $\Delta f = F2 - F1$ to be larger than the true width. After an appropriate warmup period, however, this drift is generally small (less than 100 Hz/min). The effect of such slow drift on the measured Δf can be eliminated by alternately reading F1 first and then F2 first, and averaging the (F2 − F1) values so obtained. Over the time required for a typical data run on a given sample (30 min or so, depending on the number of field points), the drift is less than 1 MHz. The drift, therefore, does not appreciably increase the error in $4\pi\chi_e'$, since it is less than the resolution error for f_{ref}.

In the measurement of magnetic microwave susceptibility using cavity perturbation techniques, it is always necessary to determine reference cavity parameters for no magnetic loading as well as the cavity parameters Q and f at the field of interest. In many cases this is done simply by measuring the empty cavity. Where high sensitivity is required, however, the errors due to dielectric loading from the sample holder and the sample itself can be appreciable. The reference values should take such loading into account if the changes in Q and f at the field of interest are to be used as a measure of the magnetic loading only. A somewhat better approach, then, is to leave the sample in place and perform the measurement at very high field. In order to achieve maximum resolution, we adopted an extrapolation procedure to obtain Q_0 and f_L at infinite field.

Typical data for YIG and related materials show that the extrapolated f_0 is several megahertz higher than the value at 12 kOe. The quantity $f_L - f_0$ in Eq. (2) is also on the order of 2 MHz for typical data, so that a proper extrapolation is very important for accurate determination of $4\pi\chi_e'$. Data for Q_0, on the other hand, show that the extrapolated $1/Q_0$ is lower than the 12 kOe value by only 1.3×10^{-7}. This corresponds to an error in $4\pi\chi_e''$ of about

10^{-6}. Consequently, a precise extrapolation to get Q_0 is not very critical for the χ_e'' measurement.

III. APPLICATION TO EFFECTIVE LINEWIDTH

The most fruitful application of the present system and measurement technique to date has been the determination of effective linewidth parameters (ΔH_{eff}) at magnetic fields far from resonance.[2,3] In this way it is possible to obtain a measure of the intrinsic losses, even though the particular ferrite under study has a considerable inhomogeneity-related broadening of the ferromagnetic resonance line. The effective linewidth determination is based on $4\pi\chi_e''$ and $4\pi\chi_e'$ measurements vs applied field H_0, as described in earlier sections. The response can be described analytically by

$$\chi_e=(\chi_e'-i\chi_e'')=\gamma M_s(\gamma H_0+i\eta_0)/[(\gamma H_0+i\eta_0)^2-\omega^2]. \quad (7)$$

Here, γ is the gyromagnetic ratio, $4\pi M_s$ is the saturation induction, ω is the angular excitation frequency, and η_0 is the uniform precession relaxation rate. An approximate solution is first obtained by replacing the γM_s term in the numerator of Eq. (7) with $\gamma_0 M_s$, where γ_0 is the free-electron gyromagnetic ratio. With this replacement, Eq. (7) is simply a complex quadratic equation in $\Omega_0=\gamma H_0+i\eta_0$ which can be solved explicitly for γ and η_0. The exact solution is then obtained by an iterative procedure in which the calculated values of γ are used to estimate γM_s for use in successive iterations. The analysis was carried out on a digital computer and the iteration procedure was terminated when η_0 changed by less than one part in 10^4 on successive iterations.

A common parameter used to describe microwave loss is the half-power linewidth ΔH. It is highly desirable to describe the field dependence of the relaxation rate in units which are compatible with the established convention. The discussion to follow is in terms of an effective linewidth ΔH_{eff} which is proportional to η_0 and is related to ΔH. The meaning of ΔH_{eff} can be understood by considering a simplified susceptibility expression of the form

$$4\pi\chi_e''^{(L)}=\gamma 4\pi M\eta_0/[(\gamma H_0-\omega)^2+\eta_0^2]. \quad (8)$$

Equation (12) characterizes the usual absorption curve for ferromagnetic resonance obtained with a circularly polarized microwave field rotating in a Larmor sense. If η_0 and γ are independent of H_0, then Eq. (8) describes a Lorentzian curve centered at $H_0=\omega/\gamma$ and with a linewidth given by $\Delta H=2\eta_0/\gamma$. Absorption curves which are not Lorentzian can be described by Eq. (8) if η_0 and γ are allowed to be field-dependent and an effective linewidth can be defined by

$$\Delta H_{\text{eff}}=2\eta_0(H_0)/\gamma(H_0). \quad (9)$$

The field dependences of η_0, γ, and ΔH_{eff} provide a measure of the departure of the absorption curve from a Lorentzian line shape. The effective linewidth magnitude and field dependence are determined primarily by $\eta(H_0)$. The gyromagnetic ratio γ does not change appreciably with H_0. For the present purposes, it is simply a normalizing parameter used to express η_0 in linewidth units. The small changes in γ with H_0 are related to an effective line shift discussed previously by Motizuki *et al.*[5] and Vrehen.[6]

Some typical data are shown in Table I for a 2.4-mm-diam sphere of 16% porous yttrium iron garnet with $4\pi M_s=1560$ G and a FMR linewidth of 380 Oe. The tabulated data show cavity frequency, cavity Q, $4\pi\chi_e'$, $4\pi\chi_e''$, and ΔH_{eff} vs applied field. The scatter in ΔH_{eff} at any field point is less than ± 1.0 Oe. When the data are taken extremely carefully, accuracy to ± 0.5 Oe can be obtained. Only data for field points away from resonance have been included. As one approaches resonance, $4\pi\chi_e''$ can become quite large. The present TE_{011} high-Q cavity is much too sensitive for such near resonance measurements. Even at $H_0=2.8$ kOe, where the bias is still 700 Oe from resonance, $4\pi\chi_e''$ on the resonance tail has degraded the cavity Q from 25 000 to 9000. For near resonance measurements it is necessary to use smaller samples and low-Q rectangular cavities. In this region, the susceptibility components are relatively large ($4\pi\chi_e''>0.1$) so that extreme sensitivity is not required.

The physical meaning of the ΔH_{eff} values given in the last column of Table I has been discussed extensively in other reports. In brief, these linewidth values are a measure of the off-resonance losses in linewidth units (oersteds). These specific data show that the off-resonance losses are much smaller than those encountered near resonance. The sample for which the data in Table I were taken has a resonance linewidth of nearly 400 Oe, 40 times larger than the ΔH_{eff}'s of about 10 Oe given in the table. For many materials, ΔH_{eff} in the off-resonance region may be even smaller. It is for this kind of measurement that the extreme sensitivity of the technique is most useful.

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