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Sensitivity analysis of transient measurements using the microwave cavity perturbation technique

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The application of microwave cavity perturbation technique for the study of transients in semiconductors is becoming popular due to its simplicity in measurement procedure and high sensitivity. This paper discusses the effects of quality factor, sample size, and coupling factor on the sensitivity of the measurement. Also, it deals with a measurement approach for the study of triplet state transitions and excited state studies in organic solvents. Finally, a comparison between the cavity perturbation technique and the currently used reflection technique is made. *© 1998 American Institute of Physics.* [S0021-8979(98)00702-6]

I. INTRODUCTION

The microwave technique has been used for carrier lifetime studies in semiconductors since the early 1960s.[1–3] Renewed efforts have been made in using the microwave technique for the characterization of semiconductors[4–10] and organic liquids. Most of the microwave measurements utilize the reflection technique,[12–15] as this does not enforce any limitations on the size and shape of the sample. New techniques have been introduced to improve the sensitivity. Presently, some of the techniques such as the open-ended reflection technique, the free-space technique, and the sample followed by a metallic short are widely used. Due to its high sensitivity, ''the cavity perturbation technique'' is preferred, in addition to the well-established reflection technique. This paper enlights the salient features of the cavity perturbation technique and its application in the study of transients in semiconductors. It also explores the use of this technique to observe the triplet state transitions in organic samples.

The change in the resonant frequency and the quality factor upon introducing a sample inside the cavity is the basis of the cavity perturbation technique. The semiconductor, being a dielectric, is kept at the maximum electric-field position inside the cavity. It is a loaded quality factor that is meant by the term quality factor. The dielectric loss (ϵ'') is expressed in terms of the conductivity of the sample and is given by

$$\epsilon''=\frac{\sigma}{\omega\epsilon_0}, \tag{1}$$

where ϵ'' is the loss measured, σ is the conductivity of the sample, ϵ_0 is the permittivity of free space, and ω is the operating angular frequency in the microwave range.

Using the discrete parallel resonant circuit approximations,[16] for a rectangular TE_{10n} mode (''n'' stands for the number of half-wavelengths in the direction of propagation) reflection-type cavity, it can be shown that

$$\sigma\propto\Delta(1/Q), \tag{2}$$

where $\Delta(1/Q)$ is the change in the quality factor of the cavity due to the insertion of the sample. The constant of proportionality depends on the cavity and sample dimensions.

Under photoillumination, the conductivity of the sample increases from σ_0 to σ, and

$$\sigma\propto\frac{1}{Q_l}-\frac{1}{Q_0}, \tag{3}$$

where Q_0, Q_d, and Q_l are the quality factors of the empty, sample loaded under dark and sample loaded under luminant condition of the cavity, respectively. The excess conductivity factor is given by

$$\frac{\Delta\sigma}{\sigma_0}=\frac{1/Q_l-1/Q_d}{1/Q_d-1/Q_a}. \tag{4}$$

The carrier lifetime is calculated from the transient nature of the quality factor $[Q(t)]$ in the microwave transient photoconductivity decay technique. Generally, the excess conductivity factor will be less than 0.05, and therefore, the variation in the quality factor will also be less. The conventional method of calculating the quality factor involves the measurement of the full width at half-maximum of the resonance curve and the resonant frequency. This method consumes considerable time and is not suitable for transient measurements. Subramanian and Sobhanadri[17] showed that one can calculate the Q factor of the cavity from the normalized reflected power at the resonance. This paper uses this approach to improve the sensitivity of the cavity perturbation technique for the study of transients.

Equation (6) assumes that the increase in the conductivity due to photoillumination does not change the resonant frequency of the cavity. But, for a higher intensity of illumination and in some polar samples, it is possible that the dielectric permittivity of the sample changes as the carrier concentration increases.[18] Therefore, one can also expect a change in the resonant frequency of the sample-loaded cavity. In this case, the transient nature of the microwave signal is the combined effect of the change in the Q factor and resonant frequency. The study of the excited state levels in organic samples is one of the examples. This problem is

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discussed in Sec. IV. While most of the results are analytically obtained, some experimental results are also presented to confirm the theory.

II. EXPERIMENTAL CONDITIONS

All the experiments are conducted using a microwave vector network analyzer (HP5720A) and a klystron microwave test bench operating in the frequency range of 8–12 GHz (*X* band). Two cavities used for the measurements are rectangular and reflection types. One operates under TE_{107} mode (oscillating around 9.9 GHz) and other under TE_{105} mode (oscillating around 9.1 GHz). The test sample used is a standard single-crystal silicon wafer with a dark conductivity of 0.2 mS/cm. The surface area of the sample is $0.4 \times 0.4\ \text{cm}^2$ and the thickness is 500 μm. An infrared light-emitting diode (IR LED) (900–1000 nm: 3 mW/cm^2) is used to irradiate the sample for the study of the photoconductivity. The sample is kept at the maximum electric field in the cavity through a hole drilled at the center. A side hole is provided in the cavity to pass the excitation light.

Theoretical calculations are carried out by assuming a semiconductor sample of dark conductivity 10 S/cm. Excess photoconductivity induced by the illumination is assumed to be 0.5 S/cm. For theoretical purposes, the photoillumination over the surface of the sample is assumed to be uniform. All the analytical calculations entirely depend on $\Delta\sigma/\sigma_0$. The value of $\Delta\sigma/\sigma_0$ is kept as 0.05, but when the analytical and experimental results are compared, same value of $\Delta\sigma/\sigma_0$ is used.

III. SENSITIVITY

The sensitivity (S) in the present context refers to the change in the measured reflected power (k: normalized), with respect to a change in the conductivity,

$$S=\sigma_0\left(\frac{\Delta k}{\Delta\sigma}\right). \tag{5}$$

The sensitivity can also be written as

$$S=A\sigma_0\left(\frac{\Delta(1/Q)}{\Delta\sigma}\right), \tag{6}$$

where

$$\Delta\left(\frac{1}{Q}\right)=\frac{1}{Q_l}-\frac{1}{Q_d}, \tag{7}$$

and

$$A=\frac{1}{Q_d}-\frac{1}{Q_0}=\frac{\Delta k}{\Delta(1/Q)}. \tag{8}$$

From Eqs.(7)–(10), it is very clear that the sensitivity depends on factor A, the relation between $\Delta\sigma$ and $\Delta(1/Q)$, and the relation between $\Delta\sigma$ and Δk. The coupling factor "K" is related to $\Delta\sigma$ through k. Therefore, S can be improved by choosing optimum parameters for the quality factor, the size of the sample, and the coupling factor.

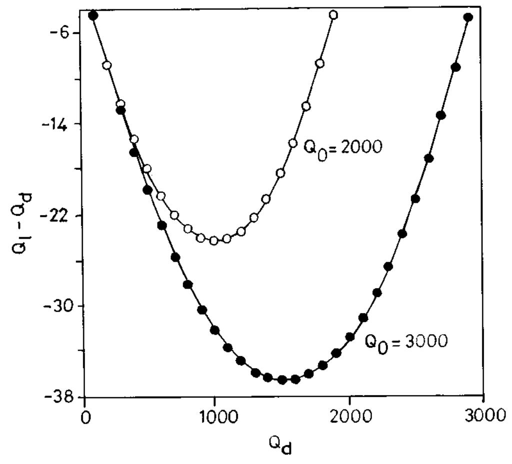

FIG. 1. Variation of $Q_l - Q_d$ with Q_d for two values of Q_0 (3000 and 2000).

A. Effect due to quality factor and sample size

Let the values of Q_0 and $\Delta\sigma/\sigma_0$ be kept constant as 3000 and 0.05, respectively. Different values of Q_d can be obtained by varying the size of the sample. Therefore, one can get the computed values of Q_l for different values Q_d. The calculation is repeated for another value of Q_0 kept as 2000. Figure 1 gives the plots drawn between Q_d and $Q_l - Q_d$ for two values of Q_0 (3000 and 2000). From Fig. 1, it is inferred that for a fixed value of $\Delta\sigma$, $Q_d - Q_l$ is maximum when the size of the sample introduced into the cavity reduces the Q factor by half. It is recommended to increase the illumination area, then the thickness of the sample, since the former increases the total conductance. Further analysis of the variation of $|Q_l - Q_d|$ with Q_0 shows that $|Q_l - Q_d|$ increases with an increase in Q_0. Figure 2 shows this variation calculated analytically based on Eq. (6) and measured experimentally for the silicon sample. The experiment is performed as follows: The sample is illuminated with an IR LED and the value of $\Delta\sigma/\sigma_0$ is measured as 0.217. The initial value of Q_0 is 1504 and Q_d is 1280. The value of A calculated from the change in k is 2585. The sensitivity is 0.3. Q_0 is decreased by inserting a thin metal rod and the values of Q_0, Q_d, and Q_l are measured each time. For a Q_0 value of 600, A is calculated as 1070. This corresponds to the

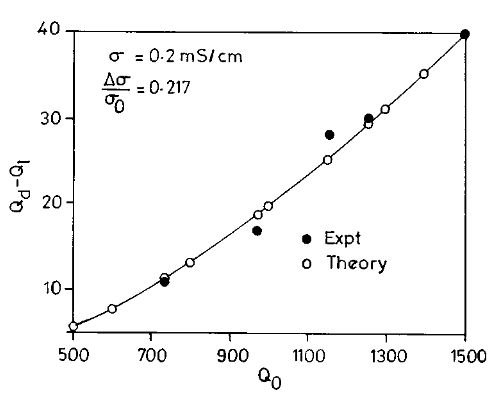

FIG. 2. Variation of $Q_d - Q_l$ with Q_0 for the silicon sample measured experimentally and analytically calculated.

sensitivity of 0.12. Obviously, the sensitivity increases with an increase in the quality factor of the cavity.

B. Effect due to *k* and *K*

The usual procedure of calculating the quality factor by measuring the full width at half-maximum from the resonance curve consumes considerable time, and therefore, cannot be employed for transient studies. It is well known that the area under the resonance curve for a particular cavity-coupling combination remains constant even when perturbed by inserting the sample into the cavity. Under this procedure, Subramanian and Sobhanadri[17] showed that the quality factor of the cavity can be calculated from the normalized reflected power at resonance (k). The area under the resonance curve for the sample-loaded cavity at dark can be written as

$$\text{Area}=\sum 1-(1-k_d)\left(\frac{f_d^2}{Q_d}\right)^2\left[(f_d^2-f^2)^2+\left(\frac{ff_d}{Q_d}\right)^2\right]^{-1}, \tag{9}$$

where k_d is the normalized reflected power at resonance, and f_d and Q_d are the resonant frequency and quality factor of the cavity, respectively. Under illumination conditions, it is assumed that the resonant frequency does not change but the quality factor decreases to Q_l. Also, k_d increases to k_l. Since the area is the same under illumination, Eq. (10) can be written as

$$\text{Area}=\sum 1-(1-k_l)\left(\frac{f_d^2}{Q_l}\right)^2\left[(f_d^2-f^2)^2+\left(\frac{ff_d}{Q_l}\right)^2\right]^{-1}. \tag{10}$$

Therefore, for a fixed span of frequency (variable ''f'' takes the range from $f_d=5\Delta f$ to $f_d-5\Delta f$: Δf is the full width at half-maximum measured from the resonance curve), Q_l can be calculated. Using Eqs. (11) and (12), it can be shown that

$$\Delta k \propto \Delta\left(\frac{1}{Q}\right), \tag{11}$$

and therefore,

$$\Delta\sigma \propto \Delta k. \tag{12}$$

Figure 3 shows that the relation between Δk and $\Delta(1/Q)$ (analytically calculated) is linear, and the slope gives factor A as 2585. By fixing the values of Q_d as 3000 and f_d as 9 GHz, the variation between Q_l and Δ_k are plotted for two different values of k_d (k_d as 0 and 0.05) (see Fig. 4). The values of A and S are calculated as 2682 and 0.88, respectively, when k_d is 0. Similarly, the value of A and S are calculated as 2460 and 0.8, respectively, when k_d is 0.05. These two results show that the sensitivity (S) is maximum when k_d is zero.

The normalized reflected power at resonance (k) is related to the coupling factor (K) as

$$k=\left(\frac{K-1}{K+1}\right)^2. \tag{13}$$

If ''K'' is equal to 1, the cavity is critically coupled and the value of ''k'' will be 0. By measuring K from the standing-wave ratio measurement (using the network analyzer), the corresponding values of k (both analytically and experimentally obtained) are plotted in Fig. 5. The extrapolation shows that as K approaches 0, k approaches 0.96, and as K approaches 1, k approaches zero. It is now clear that the sensitivity is maximum when the cavity is critically coupled.

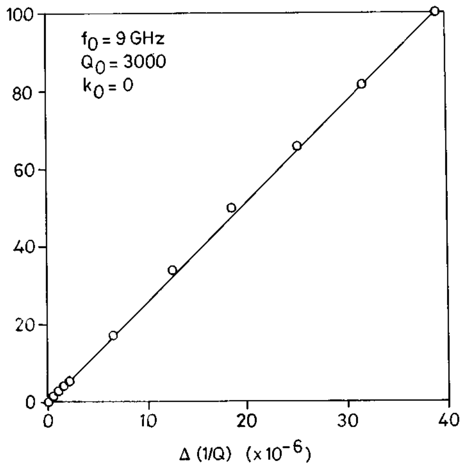

FIG. 3. Variation of Δk with $\Delta(1/Q)$, analytically calculated using Eqs. (11) and (12).

IV. EFFECT DUE TO CHANGE IN RESONANT FREQUENCY

In the case of the excited state studies in the organic samples, an UV laser pulse is used for excitation. The molecules are excited from the ground singlet state to an excited singlet state. The excited molecules either return back to the ground state directly within a few nanoseconds or populate the triplet state and return back to the ground state within a few microseconds. Due to the excitation, the dipole moment of the molecule increases or decreases depending on the sample property. Moreover, due to the excitation, free radicals are formed as in the case of α, α, α-trifluoroacetophenone dissolved in decane.[11] Apart from the microwave power loss, this gives rise to a change in the dielectric permittivity and the resonant frequency of the sample-loaded cavity under excitation. Depending on the magnitude of the

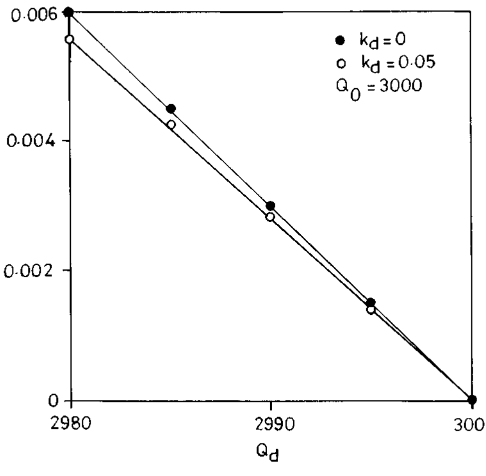

FIG. 4. Variation of Δk with Q_d for two values of k_d (zero and 0.05).

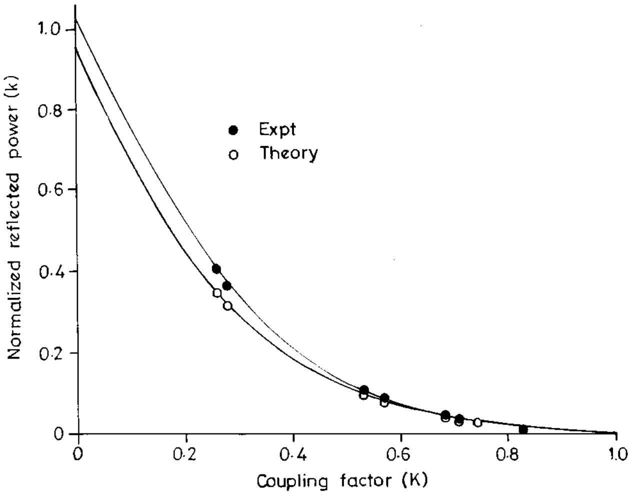

FIG. 5. Variation of normalized reflected power (k) with coupling factor (K).

TABLE I. The value of normalized reflected power measured and analytically calculated using Eqs. (11) and (12) at various shifts in the resonant frequency from that of the at dark condition ($f_0 = 9.890\,875$ GHz: $Q_0 = 1426$).

Resonant freq. shifts to (GHz)	Quality factor	k measured at 9.890 875 GHz	k analytically calculated
9.890 625	1424	0.0118	0.0130
9.890 500	1419	0.0206	0.0194
9.890 375	1416	0.0257	0.0281
9.890 250	1413	0.0398	0.0390
9.890 125	1410	0.0539	0.0521

change in the resonant frequency and its time response, the transient reflected microwave signal deviates from the normal decay pattern (It is assumed that the triplet state decay follows an exponential and the 1/eth value of the maximum of the reflected signal gives the lifetime of that state.) Therefore, it is difficult to calculate the excited state dipole moment of the molecule. But until now, to the best of our knowledge, no literature is available on the experimental measurement regarding this change in the resonant frequency.

From the transient microwave reflected signal due to the pulsed excitation, the excited-state dipole moment and the triplet state lifetime are calculated by Fessenden *et al.*[11] as follows: The peak of the transient signal extrapolated to time $t = 0$ (that is, just the pulse is on) is plotted for different dilutions of the sample in a nonpolar solvent. The slope of this graph is used to calculate the dipole moment at the excited state. Meanwhile, the triplet state lifetime is calculated from the 1/eth decay portion of the transient signal. The above-mentioned procedure may not give accurate values since it did not consider the change in the resonant frequency due to the excitation. An approach, proposed in this paper, can eliminate this error. The following paragraphs explain the detection of change in the resonant frequency and its effect on the reflected signal and on the transient decay.

Assuming a practical change in the resonant frequency and quality factor of the cavity, the k values obtained from both analytical and experimental approaches obtained using Eqs. (11) and (12), agree well, and the values are tabulated in Table I. To ensure whether the resonant frequency is varied or not, the following procedure is carried out. Assume that the resonant frequency under the dark condition (f_d) is 9 GHz, while the quality factor (Q_d) is 3000 while the value of k_d is 0. Under excitation, the new resonant frequency (f_1) is 8.999 95 and Q_1 is 2995. Table II presents the k values calculated analytically using Eq. (11). It is observed that the strength of the signal is maximum positive when the input microwave frequency is kept at f_d. If the input microwave frequency is varied towards the new resonant frequency, the strength of the signal decreases and eventually becomes negative. Meanwhile, the top level of the transient decreases until f becomes f_1 and increases thereafter (refer to Fig. 6). Therefore, if the resonant frequency varies under excitation, then one has to increase or decrease the input microwave frequency until the base of the signal is kept at a minimum. At this frequency (f), the strength of the signal is k_1. On the other hand, if there is no variation in the resonant frequency, then the change in the input microwave frequency results in an increase in the base line of the signal. By knowing f_d, Q_d, f_1, and k_1 using Eqs. (11) and (12), one can compute the value of Q_1 and, therefore, the dielectric permittivity and loss factor can be calculated in absolute terms. The excited state dipole moment can be derived from the loss factor.

By keeping a static change in the resonant frequency due to excitation, one can also get an inverted transient. To check this, a small Teflon piece is inserted step by step while irradiating the silicon sample with the LED. Figure 7 shows the transient microwave photoconductivity signal obtained for the silicon sample while the cavity is at resonance (9.0235 GHz) and at three other off-resonance frequencies (9.0225, 9.021, and 9.0195 GHz). All these plots are obtained while the input microwave frequency is kept at 9.0235 GHz. It is now clear that the signal crosses zero at 9.021 GHz and gets

TABLE II. The values of the normalized reflected power from the cavity at various frequencies when the input microwave frequency is at the resonant frequency of the cavity when the sample is under the dark condition (f_d) and at resonant frequency when the sample is illuminated with light (f_1). The values of f_d is taken as 9 GHz and f_1 is 8.999 95 GHz. Similarly, Q_d is assumed as 3000 with k_d as 0 and Q_1 is 2995.

	Normalized reflected power (k) ($\times 10^{-3}$)		
Input microwave frequency (GHz)	At f_d k_1	At f_1 k_2	Δk ($\times 10^{-3}$) ($k_2 - k_1$)
9.000 00	0.000	2.679	2.679
8.999 99	0.036	2.307	2.271
8.999 98	0.172	1.977	1.805
8.999 97	0.385	1.761	1.376
8.999 96	0.696	1.625	0.929
8.999 95	1.081	1.581	0.500
8.999 94	1.594	1.629	0.035
8.999 93	2.128	1.752	−0.376
8.999 92	2.826	1.986	−0.840
8.999 91	3.556	2.289	−1.267

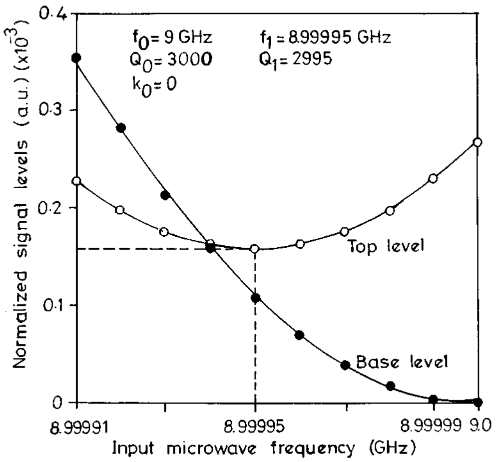

FIG. 6. Variation of the top and bottom level of the transient signal when the input microwave frequency is changed.

inverted. Therefore, when the sample undergoes a change in the resonant frequency with irradiation, there is also a possibility of a change in the polarity of the signal. To detect this possibility, it is better to increase or decrease the input microwave frequency and check the signal amplitude. The amplitude decreases if there is no variation of resonant frequency and increases when the frequency approaches the new resonant frequency. It is to be noted that Fig. 7 shows the transient signal for a static change in the resonant frequency (the time response for the decay is infinity).

Considering a finite time response for the transient frequency decay, the microwave signal can be simulated using Eq. (11). Figure 8 gives two such transient signals obtained for a change in the quality factor from 2500 to 2000 due to photoillumination. One of the signals has a resonant frequency shift by 1 MHz and the other by 0.1 MHz. The resonant frequency under dark is 8.95 GHz. In both cases, the time constant for the frequency is 10 μs, and for the quality factor, it is 20 μs. For a large amount of shift in the resonant frequency, the signal starts from negative and goes to positive, and the decay is not an exponential. As the shift in the resonant frequency decreases, the signal has only positive data. Also, the peak the transients differ. Therefore, the relation between the reflected power and dilution does not provide an accurate value of the excited-state dipole moment. One has to vary the input microwave frequency until it equals the new resonant frequency, and the value of the reflected power can then be used for such calculations. Results resembling Fig. 8 are reported by Fessenden *et al.*[11] and attributed to the formation of radicals due to oxygen in α,α,α-trifluroacetophenone.

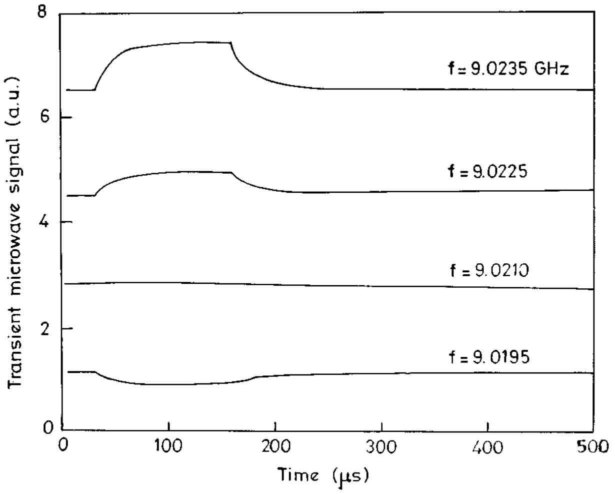

FIG. 7. Transient photoconductivity decay observed for the silicon sample (a) at resonance (9.0235 GHz), (b) resonance shifts to 9.0225 GHz, (c) resonance shifts to 9.021 GHz, and (d) resonance shifts to 9.0195 GHz.

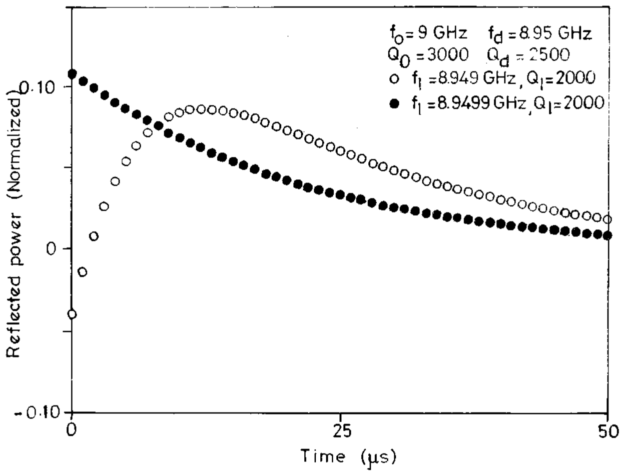

FIG. 8. Analytically calculated transient microwave signal from the cavity when the transient nature of the resonant frequency is also considered. The symbol represents the shift in the resonant frequency by 1 MHz and Δ represents the shift in the resonant frequency by 0.1 MHz. In both cases, the time response for the frequency is 10 μs and the conductivity as 20 μs.

V. COMPARISON BETWEEN REFLECTION AND CAVITY PERTURBATION TECHNIQUES

In the reflection technique, the sample is kept at the open end of the waveguide and the reflected power is measured. The sensitivity is maximum when the sample is backed by a reflection plate kept at an optimum distance decided by the sample thickness, conductivity, and frequency.[19] For a medium conducting sample (between 0.01 and 10 S/cm), it is possible to achieve the sensitivity of 0.4 at 10 GHz. But for high and low conducting samples, the sensitivity is very poor ($S<0.2$). In the cavity perturbation technique, by properly varying the size of the sample such that $Q_d = Q_0/2$, sensitivity around 0.9 can be achieved for Q_0 as 3000 and k_d as 0. This can be further increased by increasing Q_0.

Though both these techniques do not give a simple relation for the change in the dielectric permittivity of the sample due to illumination, the perturbation technique gives us an outline of the measurement and the procedure for calculation.

Due to the ringing time of the cavity ($\sim Q_d/5\omega$),[20] the minimum time constant that can be measured is affected by the operating frequency and the quality factor under the dark condition. This constraint is applicable only to the cavity perturbation technique, whereas the minimum time constant can be almost $1/5\omega$ for the reflection technique.

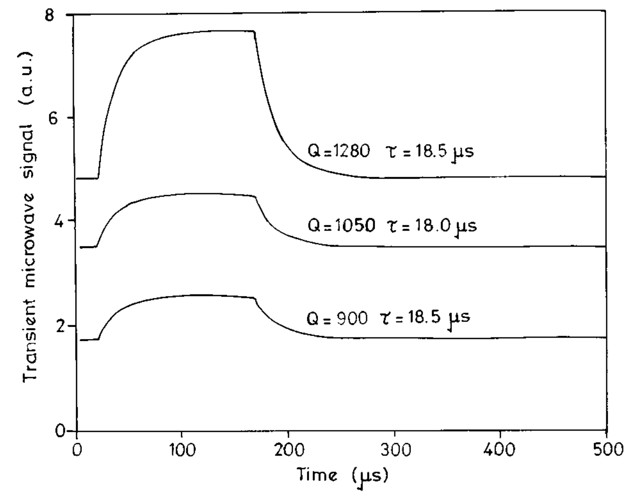

FIG. 9. Transient photoconductivity decay observed for the silicon sample at resonance with various Q values of the cavity.

In the case of the reflection technique, the distance at which the metal backing is kept affects the transient nature of the photoconductivity signal as measured by Martin Schofthaler and Rolf Brendel.[19] But, when the quality factor of the cavity is varied to 1504, 1050, and 900, the transient signals so obtained for the silicon sample are found to be identical, and the lifetime values calculated are 18.5, 18, and 18.5 ms, respectively (refer to Fig. 9).

The size of the sample needed in the perturbation technique is very small (around 2×2 mms: maximum of 1.1 $\times0.2$ cm, if the sample is highly resistive), whereas the reflection technique requires the sample to completely close the waveguide dimension to obtain the maximum sensitivity of 0.4.

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