COMMUNICATIONS

DE LA FACULTÉ DES SCIENCES
DE L'UNIVERSITÉ D'ANKARA

Série A₂ Physique

TOME 29 ANNEE 1980

Design And Construction Of Esr-Endor Cavities

Cüneyt KAPTANOĞLU, Burhanettin ORAL

Faculté des Sciences de l'Université d'Ankara
Ankara, Turquie
La Revue "Communications de la Faculté des Sciences de l'Université d'Ankara" est un organe de publication englobant toutes les disciplines scientifique représentées à la Faculté.

La Revue, jusqu'à 1975 à l'exception des tomes I, II, III, était composé de trois séries

Série A : Mathématiques, Physique et Astronomie.
Série B : Chimie.
Série C : Sciences naturelles.

A partir de 1975 la Revue comprend sept séries:

Série A₁ : Mathématiques
Série A₂ : Physique
Série A₃ : Astronomie
Série B : Chimie
Série C₁ : Géologie
Série C₂ : Botanique
Série C₃ : Zoologie

En principe, la Revue est réservée aux mémoires originaux des membres de la Faculté. Elle accepte cependant, dans la mesure de la place disponible, les communications des auteurs étrangers. Les langues allemande, anglaise et française sont admises indifféremment. Les articles devront être accompagnés d'un bref sommaire en langue turque.
Design And Construction Of Esr-ENDOR Cavities*

by

Cüneyt KAPTANOĞLU, Burhanettin ORAL

Hacettepe University, Department of Physics, Beytepe 83, Ankara, TURKEY

(Received, January 19, 1980; accepted February 22, 1980)

SUMMARY

In the present work are given the design and construction techniques of three different microwave resonance cavities that are used in ESR-ENDOR spectrometers. The mode configuration chosen was the TE_{01, due to its nice fitting in the present experimental conditions such as available magnet gap, suitability for double resonance work etc.

The three cavities produced were tested in a home made ESR spectrometer as well as in a Varian E-9 system and their resonance frequencies were measured to be about 8.858 GHz which is less than that of theoretical value and unloaded Q-factor was estimated to be about 4000-6000.

INTRODUCTION

One of the most important parts of any ESR (Electron Spin Resonance) or ENDOR (Electron Nuclear Double Resonance) spectrometer is the microwave cavity in which the sample to be tested is placed and where the microwave radiation field $H_1$ has the most intense value. For ENDOR work, it is only necessary to wrap the r.f. coils around the sample for inducing NMR transitions while keeping ESR frequency constant throughout the r.f. sweep.

As it is well known for a system which contains a number of well separated levels, the transition probability $W_{nm}$ between any such two levels $n,m$ is proportional to the square of microwave radiation field $H_1$ and therefore, the sample should be placed in a region where this field intensity is most intense and yet, most homogenous. When the resonance absorption phenomena

---

*This work was supported by the Magnetic Resonance Research Unit of the Turkish Scientific and Technical Research Council.
takes place, the change in the effective Q-factor of the cavity is
processed and displayed by means of electronic subunits of the
spectrometer.

Theoretical Background

In the simple ESR theory, when the resonance absorption
takes place in a cavity in which the sample is located, the change
in the cavity Q-factor is given by\(^2\)

\[
\frac{\Delta Q}{Q_o} = 4 \pi \chi'' \eta \frac{Q_o}{P_o}
\]  
(1)

where \(\chi''\) is the imaginary part of magnetic susceptibility \(\chi = \chi' - i \chi''\), \(\eta\) is the filling factor and \(Q_o\) is the unloaded cavity Q-factor.

The signal produced by this change appears as a voltage at
one of the arms of a microwave bridge and its optimum value
is as follows\(^3\)

\[
V_{max} = \sqrt{2} \rho \frac{\pi \chi'' \eta Q_o}{\sqrt{P_o}}
\]  
(2)

where \(\rho\) is the dynamic resistance of detector and \(P_o\) is the mic-
rowave power incident on the detector. Thus, the signal that

carries the necessary information is seen to be directly proportional
to the cavity Q-factor.

In case of nuclei which are coupled to electrons in the sample,
and if one can simultaneously apply NMR power and ESR, then
the term ENDOR represents the double resonance phenomena.
This can easily be applied to a sample around which an NMR
coil is wrapped\(^4,5\). ENDOR may be best explained by considering a
single electron \((S=1/2)\) coupled with a magnetic nucleus \((I=1/2)\)\(^4,6\).
The Hamiltonian describing such a system may be written down as

\[
H = \beta \cdot \vec{S} \cdot \vec{g} \cdot \vec{H} + \vec{S} \cdot \vec{A} \cdot \vec{I} - g_n \beta_n H_o \cdot \vec{I}
\]  
(3)

and in the high field approximation, this becomes

\[
\frac{E}{\hbar} = \gamma_e H_o m_s - \gamma_n H_o m_I - A m_s m_I
\]  
(4)

where \(\gamma_e\)and \(\gamma_n\)are the electron and nuclear gyromagnetic ratios,
respectively, $H_0$ is the external magnetic field, $A$ is the isotropic electron nuclear hyperfine coupling constant and $m_s, m_i$ are the electron and nuclear magnetic quantum numbers which may be diagrammatically shown in Figure-1. In this Figure, $\nu_n > A > 0, S = I = 1/2$, $\nu_n = \left( \frac{\gamma_n}{2\pi} \right) H_0$ and $\nu_e = \left( \frac{\gamma_e}{2\pi} \right) H_0$

conditions are considered to hold.

By using the transition conditions, one finds:

$$\nu_{esr} = \nu_e + A \cdot m_i \quad ; \quad \nu_{nrm} = \nu_n + A \cdot m_s$$

(5)

and for $n$ nuclei with the same $A$ constant, these, then become

$$\nu_{esr} = \nu_e + A \cdot M_i \quad ; \quad M_i = \sum_{i=1}^{n} m_{1i}$$

(6)

For $n$ equivalent nuclei, the number of ESR transitions is $n + 1$ while it is invariant for NMR transitions and there comes the advantage of the ENDOR technique. As one can see from Fig. 1, after having saturated the transition $1 \rightarrow 3$ by means of an intense microwave power, an r.f. power is applied to the level $1 \rightarrow 2$ and thus forming the so called ENDOR line$^{4,5,6}$.

Mode Choice And Cavity Design

Both theoretical and technical necessities led us to make a choice of the TE$_{011}$ mode configuration because, this mode is most suited for placement of r.f. coils for ENDOR and also, it has relatively high Q-factor. In addition, it has a magnetic field configuration such that external magnetic field is always perpendicular to the microwave field in the cavity and has well rotational symmetry with respect to the long axis of the cavity. In this cavity mode, large access holes on top and bottom plates do not appreciably affect the Q-factor which is a very desirable feature when used with low-temperature control systems inside the cavity. The TE$_{011}$ mode configuration is shown in Fig. 2$^4$.

Next important design factor for cavity was that it should be used with the existing E-9 ESR spectrometer whose magnet
has an air-gap of 45 mm. and whose microwave source could be tuned through 8.5 – 9.6 GHz frequency band.

Thus, under these circumstances, the maximum external diameter of the cavity should be 45 mm. and its frequency should lie within the frequency band. The resonance frequency of the cavity is found from the following relation $^9$.

$$\lambda = \frac{c}{v} = \frac{2}{\left( \frac{2J'_{mnp}(k_c a)}{\pi D} \right)^2 + \left( \frac{n}{L} \right)^2}$$  \(7\)

where, for $\text{TE}_{011}$, $m = 0$, $n = p = 1$ and $J'_{01p}(k_c a) \approx 3.82$. For various cavity lengths $L$ and for radius $a = D/2$, the calculated resonance frequencies are tabulated in Table 1. These calculations were made on the basis of $L = 2a = D$ because, for this mode configuration and for the given dimensions, the Q-factor is found to be maximum. So, the last column in Table 1, shows resonance frequencies deduced from the mode graphs $^7$.

<table>
<thead>
<tr>
<th>$a$ (cm)</th>
<th>$L$ (cm)</th>
<th>$f$ (GHz)</th>
<th>$f$ (GHz)</th>
</tr>
</thead>
<tbody>
<tr>
<td>2.07</td>
<td>4.14</td>
<td>9.531</td>
<td>9.525</td>
</tr>
<tr>
<td>2.65</td>
<td>4.1</td>
<td>9.60</td>
<td>9.618</td>
</tr>
<tr>
<td>2.80</td>
<td>4.0</td>
<td>9.86</td>
<td>9.858</td>
</tr>
<tr>
<td>1.95</td>
<td>3.9</td>
<td>10.12</td>
<td>10.111</td>
</tr>
<tr>
<td>1.90</td>
<td>3.8</td>
<td>10.32</td>
<td>10.377</td>
</tr>
<tr>
<td>1.85</td>
<td>3.7</td>
<td>10.66</td>
<td>10.658</td>
</tr>
</tbody>
</table>

Table 1. Calculated resonance frequencies.

As a final decision, we took the values $a = 2.05$ cm. and $L = D = 4.1$ cm. and made the three cavities shown in Fig. 3 through 5.

The cavity block shown in Fig. 3 was simply made of a single copper pipe with proper inner and outer diameters and length. The second one consists of alternate copper and fiber rings epoxied together and final geometry has exactly the same dimensions as before. The last model was again a copper pipe which was made on a lathe and the pitches drilled were then, filled with epoxy. The last two models were thought to be more useful when used in ENDOR, because they tend to decrease eddy cur-
rents and the modulation field could penetrate more easily to the location where the sample is placed. All the cavities were silver plated by a chemical process. The top and bottom plates of the cavities were made on a lathe and their dimensions are shown in Fig. 6 and 7.

The microwave energy incident on the cavity is coupled in by means of a waveguide whose dimensions were reduced and a dielectric plexiglass slab was placed in it. The hole drilled on the top plate and a small hook inside the coupling waveguide provided the necessary microwave coupling. Fig. 8 shows the details of this coupling part. The general view of the cavity in assembled form is shown in Fig. 9. The picture of the complete system and of the two other cavity bodies are shown in the following photographs.

**Design Of Endor Coils**

For ENDOR coils, the two printed circuit boards whose single side was copper plated were used and four holes were drilled on them. The four copper posts with 2 mm. diameter were soldered on these boards thus forming a 4 turn series connected r.f. coil. The geometry of the system can be seen in Fig. 10 a,b and in Fig. 11. And, Fig. 12 shows the r.f. coil system when placed in the cavity.

**Results And Discussions**

After having the cavities completed, the simple ESR spectrometer shown in Fig. 13 was set up and cavity resonance dip was displayed on a scope. In order to measure the resonance frequency, the cavities were connected to the Varian E-9 ESR spectrometer and resonance frequency was measured with a H.P. Frequency Counter (Model 5145 L) and was found to be about 8.858 GHz.

As it can be seen from Table 1, with the given dimensions, the calculated resonance frequency was found to be $\sim 9.6$ GHz and its experimentally measured frequency is seen to be rather low. The discrepancy between the two values is believed to arise
from dimensional errors of the cavities, that is due to poor technical workmanship and careless handling.

Since, the silver plating\(^\text{10}\) was done by a chemical process and it was seen to be rather inhomogenous, that might be another reason for the forementioned difference.

The theoretical unloaded cavity Q-factor is about 12000\(^7\) but the measured value is believed to be close to 4000–6000 and one concludes that in making these types of cavities, good workmanship, good quality material and correct dimensions are essential points for good results.
Figure 1. Energy level diagram of two spins system for $S = 1/2, I = 1/2$ when $\nu_e >> A$.
Figure 2. Field configurations of cylindrical $TE_{011}$ cavity a) microwave magnetic fields $H_1$, b) electrical fields $E$, c) wall currents $I$. 
Figure 3. Cavity block no. 1. Dimensions are in millimeters.
Figure 4. Cavity block no. 2. Dimensions are in millimeters.
Figure 5. Cavity block no. 3. Dimensions are in millimeters.
Figure 6. Bottom plate of the cavities. Dimensions are in millimeters.
Figure 7. Top plate of the cavities. Dimensions are in millimeters.
Figure 8. Coupling system. Dimensions are in millimeters.
Figure 9. General view of the whole cavity system.
Figure 1: The two parts of ENDOR coils. The dashed areas show silver plated copper sheats. Dimensions are in millimeters.

Figure 11. Arrangement of ENDOR coils.
Figure 12. Placement of coils in the cavity. Dimensions are in millimeters.
Figure 13. A simple ESR spectrometer which was constructed in this work.
Photograph 1. Cavity system and other two cavity blocks.
REFERENCES

2. Ibid, 175.
3. Ibid, 178.

ÖZET

Bu çalışmada ESR-ENDOR spektrometrelerinde kullanılan üç farklı mikrodalga rezonans kavitesinin tasarımını ve yapım tekniğini anlatılmaktadır. Faydalananabilir mknatslar arası boşluk, çift rezonans çalısmalarında uygulanabilirliği vs. gibi deneySEL koşullara iyi uyumu nedeniyle, kavitler için TE<sub>01</sub> silindirik mod konfigürasyonu seçilmişdir.

Prix de l'abonnement annuel

Turquie: 15 TL; Étranger: 30 TL.
Prix de ce numéro : 5 TL (pour la vente en Turquie).
Prière de s'adresser pour l'abonnement à : Fen Fakültesi Dekanlığı Ankara, Turquie.