Microwave properties of HTS films: measurements in millimeter wave range

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Received October 21, 2005, revised February 2, 2006

A theoretical and experimental justification of an approach proposed and developed by us for surface impedance standard measurements of HTS films is presented. An analysis of the electromagnetic properties of quasi-optical dielectric resonators with conducting endplates, which provides a theoretical background for studies of HTS films in the millimeter wave range, is performed. With this technique, the highest quality modes, namely whispering gallery modes, are excited in a dielectric cylindrical disc sandwiched between HTS films. Considerable enhancement of the sensitivity of surface resistance measurements in the millimeter wave range is demonstrated, which is important for the fundamental investigation of superconductor physics. It is also shown that the measured frequency shift in the resonator with the HTS endplates as a function of the temperature reveals a possibility for accurate evaluation of the field penetration depth in HTS films.

PACS: 74.25.Nf, 74.78.Bz, 84.40.Dc, 07.57.–c

Keywords: microwave surface impedance, high-temperature superconductors, film, whispering gallery mode dielectric resonator.

1. Introduction

Microwave surface impedance $Z_s$ measurements of high-temperature superconducting (HTS) films are important for fundamental studies and also for technical applications [1]. The resonant systems developed for contactless measurements of unpatterned films are of special interest [2]. A well known technique of surface resistance $R_s$ measurement based on a sapphire (Al₂O₃) resonator with lower mode oscillations was used in the classic microwave band (< 25 GHz) [3]. Various types of the Hakki–Coleman dielectric resonator have the advantage of being able to evaluate the total energy dissipation [4]. The dimensions of dielectric resonators operating in the millimeter wavelength range (> 25 GHz) and utilizing lower-mode oscillations (waves) become unacceptably small. The quality factor $Q$ of the resonators decreases correspondingly. The highest quality factor can be obtained for the resonators using the highest order of azimuthal modes, i.e., whispering gallery (WG) waves [J.K. Wait, Radio Science, 2, 1005 (1967)]. The dimensions of such a resonator in the millimeter wavelength range are acceptable for use in resonant structures with HTS films [5].

We recently reported an accurate technique for the $R_s$ measurement of large-area HTS films using sapphire quasi-optical dielectric resonator (QDR) with conducting endplates (CEP) [6]. In this approach, the WG modes are excited in a dielectric cylindrical disc sandwiched between HTS films or between one HTS film and one copper endplate (Fig. 1). The approach was used in the Ka band and no fundamental limitations for use in a shorter wavelength range were found. It has been shown experimentally that the resonant frequency shift of QDR with HTS film as a function of the temperature can be used for the accurate measurement of surface reactance $X_s$ measurements and, hence, a field...
The electromagnetic fields distribution of monochromatic oscillations in a disc QDR made of an anisotropic uniaxial single crystal with its anisotropy characterized equation (1) and the effective height of the resonator is defined as $l_{\text{eff}} = n\pi/kz$.

The quadrature relations obtained from the Maxwell’s equations for resonant oscillations in resonators with the perfectly and imperfectly conducting endplates are integrated over the entire space. Using the radiation condition, the continuity of the tangential electric and magnetic field components at the curved surface of the resonator, and the impedance boundary condition at the end faces of the resonator, one can obtain an integral equation that defines a shift in the resonance frequency due to imperfect conductivity of the CEP

$$ (\omega - \omega_0^p) \left( \mathbf{H}^* \mathbf{E} + \mathbf{E} \mathbf{H} \right) dV =$$

$$ = 2iZ_s \left[ \mathbf{e}_z \left[ \mathbf{e}_z, \mathbf{H} \right] \right] \mathbf{H}^*_p dS \quad (4) $$

where $\mathbf{e}_z$ is unit vector along the QDR axis; $\omega$, $\mathbf{E}$ and $\mathbf{H}$ are the frequency and the electric and magnetic field vectors for the resonator with the perfectly conducting endplates; and $\omega_0^p, \mathbf{E}_p$, and $\mathbf{H}_p$ are the frequency and the electric and magnetic field vectors for the $p$th mode of the resonator with the perfectly conducting endplates. On the left-hand side of this formula, integration is performed over the entire volume $V$; and on the right-hand side, over the surface $S$ of the CEP having the surface impedance $Z_s = R_s + iX_s$.

When $Z_s$ is small and the mode interaction is neglected ($I_{i, p} = 0$ if $p \neq p$), one can obtain a relationship

$$ (\omega - \omega_0^p) \left( \mathbf{H}^* \mathbf{E} + \mathbf{E} \mathbf{H} \right) dV =$$

$$ = 2iZ_s \left[ \mathbf{e}_z \left[ \mathbf{e}_z, \mathbf{H} \right] \right] \mathbf{H}^*_p dS \quad (4) $$

where $\mathbf{e}_z$ is unit vector along the QDR axis; $\omega$, $\mathbf{E}$ and $\mathbf{H}$ are the frequency and the electric and magnetic field vectors for the resonator with the perfectly conducting endplates; and $\omega_0^p, \mathbf{E}_p$, and $\mathbf{H}_p$ are the frequency and the electric and magnetic field vectors for the $p$th mode of the resonator with the perfectly conducting endplates. On the left-hand side of this formula, integration is performed over the entire volume $V$; and on the right-hand side, over the surface $S$ of the CEP having the surface impedance $Z_s = R_s + iX_s$.
(\alpha - \alpha_p^s) + iZ_s \frac{I_{pp}^2}{W_p} = 0 \quad (5)

where \( I_{pp} \) is the surface current on the finite-conductivity endplates, which is induced by the electromagnetic wave, and \( W_p \) is the energy of the \( p \)th electromagnetic mode in the resonator with the perfectly CEP.

Relationship (5) can be used to calculate the surface impedance \( Z_s \) of the CEP from the measured QDR resonance frequency \( \omega \). We also use (5) for evaluation of the expected frequency shift of the QDR with HTS CEP. This relationship defines the shift between the resonance frequencies of a QDR with the imperfect CEP and that with the perfect CEP for a particular oscillation mode. The shift in the real part of the resonance frequency is specified by the imaginary part of the reactance \( \text{rad} \). The reactance specifies the non-dissipative energy stored in the surface layers of CEP. The surface resistance \( R_s \) is responsible for the period-averaged Joule loss \( I_{pp}^2 R_s \) in the impedance CEP of the resonator.

The evaluations show that resonant frequencies of the QDR made of Teflon with copper CEP differ from those for the resonator with perfect CEP by \( \sim 10 \) MHz. The estimated QDR frequency shift at superconducting-to-normal state transition in HTS CEP is approximately equal to the same value. This shift ensures a sufficient sensitivity for reactance measurements of HTS films.

The surface resistance \( R_s \) of CEP is more convenient to determine using a direct definition of quality \( Q \)-factor in terms of the stored and dissipative energies:

\[ Q^{-1} = k \tan \delta + A_s R_s + Q_{\text{rad}}^{-1}, \quad (6) \]

where \( Q_0 \) is the eigen \( Q \)-factor of QDR with CEP, coefficients \( k \) and \( A_s \) show the contribution of dielectric and conductor losses in the energy total loss in QDR, \( \tan \delta \) is the dielectric loss tangent, \( Q_{\text{rad}} \) is the radiation loss quality. As shown in [6] one can neglected \( Q_{\text{rad}}^{-1} \) in QDR in the case of CEP with a large diameter \( Q_{\text{rad}} = 10^{-5} \) or \( 10^{-10} \).

The expressions for \( k \) and \( A_s \) can be obtained from the electrodynamic solution of the field structure in the QDR with CEP [10]

\[ k = 1/(1 + R_0^Y); \quad (7) \]

\[ A_s = 2/\epsilon_0 \mu_0 l R_Y. \quad (8) \]

The expressions for \( R_0^Y \) and \( R_Y \) (index \( Y \) means HE or EH mode) depend on the dimensions of the resonator and the wave mode within it [13]. In the case of the axially homogeneous HE_{n10} mode, which is excited relatively easily, expressions for \( R_0^Y \) and \( R_Y \) are cited in [6]. Coefficient \( k \) is very close to 1 and coefficient \( A_s \) is shown in Fig. 2 as a function of the azimuthal index \( n \) together with the corresponding values of frequencies for the HE_{n10} and HE_{n20} modes. It can be underlined that (i) \( A_s \) decreases with increasing frequency increasing, and (ii) the values of \( A_s \) are the same for the HE_{n10} and HE_{n20} modes at the same frequency (here, however, the values of \( n \) are different for different modes).

3. The QDR-based approach to surface resistance measurement

The main element of the measurement set-up is a sapphire \( (l = 2.41 \text{ mm}) \) or ruby \( (l = 2.54 \text{ mm}) \) cylindrical disc with diameter \( d = 14.40 \text{ mm} \). The optical \( c \) axes of the single crystals were found at the angles 56° and 15° with respect to the geometric (longitudinal) axis for sapphire and ruby, respectively. Values of the loss tangent \( \tan \delta \) measured at 77 K were found to be 2.9 \times 10^{-6} and 2.3 \times 10^{-6}, respectively. The coupling of the QDR to the transmission lines was formed by dielectric waveguides with one side covered by metal (quasi-image waveguides). The angle between the longitudinal axis of the input and the output waveguides was about 10°. This allowed us to control the coupling between the QDR and the feeder lines by moving the resonator between these lines.

A small splitting of the resonance line was observed at \( R_s \) measurements of high quality HTS films forming CEP (Fig. 3). The \( Q \)-factor and \( R_s \) in the case of a split resonance can be determined also. However, in this case it is necessary to develop the special approach to determine the \( Q \)-factor [6, 14]. At the beginning, test measurements were carried out with annealed Cu which showed very good coincidence of the measured and calculated values of \( R_s \) at \( T = 290 \text{ K} \) (48.8 m\( \Omega \) and 48.4 m\( \Omega \), respectively). The experiment-
As follows from (9), $R_s^\text{min}$ is determined by the resolution of the Q-meter, loss tangent of the dielectric and the filling factor $A_s$ of the superconductor. It can be seen that even at $\delta Q_0/Q_0 = 0.1$ and $\tan\delta = 10^{-6}$, the value of $R_s^\text{min}$ is equal to $4 \times 10^{-3}$ Ω. This value is lower than $R_s$ for the best HTS films in the Ka-band [1], which allows one to perform new experiments on fundamental study of $R_s$ at very low temperatures.

As a result, the QDR-based technique of $R_s$ measurement of HTS films has a number of distinguishing features, namely:

- the technique is convenient for application in the millimeter wavelength range;
- the technique does not demand a calibration procedure, i.e., it is a first principle measurement one;
- for QDR with CEP, axially homogeneous WG modes $HE_{m0}$ are excited the most effectively (where $n$ is the azimuthal index, $s$ means number of field variations along the radius of the dielectric disc and $0$ means field homogeneous distribution along the disc height);
- at excitation of the resonator in a regime of traveling waves, an observed resonant line splits as a rule;
- there is the opportunity to apply distributed and controlled (in a same temperature cycle of measurement) coupling with transmission lines.

The above mentioned features allow us:

- to achieve the highest sensitivity of surface resistance measurement at liquid helium temperatures which opens up a new approach for studying the problem of microwave residual resistance both in HTS materials and other unusual superconductors;
- to measure microwave properties of large-area HTS films;
- to develop sub-millimeter (THz) technique for impedance measurements;
- to consider QDR with CEP as the basis for developing of new microwave devices in millimeter wavelength (for example, high-stability oscillators, filters, etc.).

### 4. Approach to surface reactance study

Surface reactance $X_s$ is the next important impedance characteristic of the HTS thin film. As a rule, the same resonant structure is used for both $R_s$ and $X_s$ measurements. However, this method does not allow us to obtain absolute values of $X_s$ (in contrast to $R_s$) which is due to the impossibility of determining the eigen frequency of resonators with ideal conducting surfaces and insufficient reproducibility of the frequencies upon reassembling the resonator.
Consideration of the above mentioned difficulties allows one to conclude that, evidently, in a given case, analogously to all other resonator techniques, the most appropriate approach can be one at which reactance variation $X_s(T)$ with temperature is determined and the relations

\[
\Delta X_s(T) = \frac{-2\Delta f}{A_s f_0}; \quad (10a)
\]
\[
\Delta\lambda(T) = \frac{\Delta X_s(T)}{2\pi f_0\mu_0}; \quad (10b)
\]

are used, where $\Delta f_0$ is the shift of eigen frequency and $\Delta\lambda(T)$ is the temperature variation of $\lambda$.

Here small air-gaps (microslots) which exist between HTS films, used as CEP, and dielectric disc with WG modes are one of the important considerations affecting accuracy of impedance measurements. It is worth noting that the air-gap effect was negligible in microwave measurements of the dielectric properties of low-loss materials by the lower-mode dielectric resonator method [17]. This problem was later considered numerically in connection with the use of the same lower-mode dielectric resonator for HTS film characterization [18]. It was confirmed that the calculated influence of air gaps between the sapphire disc and CEP on the $Q$-factor can be accepted as insignificant. However, as the calculations showed, the same air gap reduced the resonant frequencies of the resonator rather noticeably. Naturally, the calculated data demand experimental verification, all the more in a given case, where the resonator is a quasi-optical element with WG modes, because a rigorous solution of the electrodynamic problem of QDR with air-gap has not yet been obtained. In addition, it is important to determine the temperature dependence of the air gap effect. We are forced to study this effect in QDR because it is distinct in eigen frequency measurements at reassembling of QDR with CEP.

To clarify the air-gap effect in QDR with CEP for $Z_s$ measurement, the structures shown in Fig. 5 were studied. In a structure shown in Fig. 5,a, a dielectric disc of 14.40 mm in diameter with copper layers sputtered directly onto its end face is placed between bulk copper discs of 30 mm in diameter. The thickness of the copper layers is about 2 $\mu$m, i.e., $t >> \delta \approx 0.35 \mu$m, where $\delta$ is a skin depth. The copper discs are necessary to avoid the edge effect.

Comparison of resonant frequencies of the $HE_{1410}$ mode for the structure shown in Fig. 5,a (the air-gaps are absent) and the structure shown in Fig. 5,b (the air-gaps are present) demonstrates that the air-gap cause a frequency increase of 120 MHz. The use of springs to force the CEP onto the dielectric disc causes an increase of the frequency $\sim 300$ MHz (Fig. 5,c). The latter is analogous to the case of QDR with HTS CEP.

However, despite the variation of an absolute value of the frequency, the temperature dependence of the frequency shift for structures in Fig. 5,a and 5,c is practically identical (Fig. 6). Therefore, the presence

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**Fig. 5.** Three versions of quasi-optical dielectric resonator with conducting endplates in the form of copper films sputtered directly on dielectric disc (a), cooper bulk discs (b), and copper films sputtered directly on dielectric substrates (c).

**Fig. 6.** Temperature dependence of resonant frequency shift of quasi-optical dielectric resonator with copper endplates.
of the air-gaps does not have a noticeable effect on the
temperature dependence of the frequency shift $\Delta f$
which is used for the determination of $\Delta X_s(T)$ in
accordance with (10a). The effect of the air-gaps on $R_s$
measurement can be evaluated easily by using the
coefficient $A_s$ dependence on frequency. This makes
the error of the measured $R_s$ less than 1.5%. Test $R_s$
measurements of the annealed Cu samples verify this
conclusion [6].

In our case the dependence $X_s(T)$ for HTS films
can be determined by using experimental dependence
of the resonant frequency variation $\Delta f_0(T)$ with
temperature for QDR with HTS films as CEP. However,
the observed dependence $\Delta f_0(T)$ is related to both the
variation of reactance $X_s(T)$ and variation $\Delta \varepsilon(T)$ of
permittivity $\Delta \varepsilon(T) = \Delta \varepsilon'(T) + i \Delta \varepsilon''(T)$ of the
dielectric of which the resonator is made of, i.e.,

$$\Delta f_0(T) = \Delta f_s(T) + \Delta f'_s(T).$$

Correctness of (11) follows from the condition
$\Delta f_0(T) \ll \Delta f_s$. One can exclude $\Delta f'_s(T)$ from (11) by
 carrying out additional measurements $\Delta f_{0N}(T)$ of
QDR with CEP made of metal with the normal
skin-effect

$$\Delta f_{0N}(T) = \Delta f'_s(T) + \Delta f''_s(T).$$

From (11) and (12), we obtain the expression

$$\Delta f_{0S}(T) - \Delta f_{0N}(T) = \Delta f_s(T) - \Delta f'_{s}(T) = \Delta F(T).$$

For small variations $\Delta f'_s(T)$ the true relation is

$$\Delta X_s(T) - \Delta X_s^{N}(T) = - \frac{2\Delta F(T)}{A_s f_0}.$$  

In (14) an unknown $\Delta X_s^{N}(T)$ will remain. How-
ever, use of the normal metal allows one to determine
$\Delta X_s^{N}(T)$ by its measured $\Delta R_s^{N}(T)$ because $\Delta X_s^{N}(T) =\Delta R_s^{N}(T)$. It is worth noting that measurements of

\begin{figure}[h]
\centering
\includegraphics[width=0.8\textwidth]{fig7.png}
\caption{Temperature dependence of resonance frequency
variation for quasi-optical dielectric resonator with
cconducting endplates: 1 — Y-123; 2 — Ti; 3 - curve is
difference of curves 1 and 2.}
\end{figure}

\begin{figure}[h]
\centering
\includegraphics[width=0.8\textwidth]{fig8.png}
\caption{Temperature dependence of surface reactance varia-
tion: 1 — Y-123; 2 — Ti; 3 - curve is difference of curves
1 and 2.}
\end{figure}

\begin{figure}[h]
\centering
\includegraphics[width=0.8\textwidth]{fig9.png}
\caption{Comparison of experimental and calculated de-
pendencies of surface reactance of Y-123 thin films.}
\end{figure}

frequency shift depending on temperature are carried
out at rather weak coupling. Here the coupling change is
shown to produce a negligible effect in the frequency
shift, contrary to the change in a loaded quality factor.
The approach described above was used for finding
$X_s(T)$ of Y-123 films, the $R_s(T)$ of which was mea-
sured in a preceding Section. As a normal metal, Ti was
used ($R_s = 203.1 \, \Omega$, $T = 78 \, K$, Ka-band). Figure 7
shows the temperature dependence of $\Delta f_{0S}(T),$
$\Delta f_{0N}(T)$, and their difference $\Delta F(T)$. The Y-123 thin
film dependence $X_s(T)$ obtained from (14) is pre-
sented in Fig. 8. The error of $X_s(T)$ in the temperature
interval from 78 K to 89 K changes from 20% to 2% (at
the absolute value $\approx 8 \mu$ of $X_s$ measurement error).
It is necessary to emphasize that the $X_s(T)$ displayed in
Fig. 8 is the dependence of the variation of the effective
reactance because the film thickness $d_f \approx \lambda_s$ and surface
impedance is a function of $d_f$. A connection of the
effective impedance $Z_s = Z_s(d_f)$ with the intrinsic
value $Z_s(d_f \rightarrow \infty) = Z_s(\infty)$ can be found on the basis of
the impedance transformation rule in view of substrate
properties [1]. This enables us to compare the experi-
mental and calculated dependences of $X_s(T)$ taking
into account the film thickness $d_f$ (Fig. 9).
Restricting the work so far to only phenomenological models for $X_s(T)$ calculation, one can generalize an expression for $\lambda(T)$ on the basis of references [19–21] in the form
\[
\frac{\lambda(T)}{\lambda(0)} = [1 - (T/T_s)^2]^{-\gamma},
\]
where $\lambda(0)$ and coefficients $\gamma$ and $n$ are fitting parameters. The use of different physical models causes different values of $\lambda(0)$, $\gamma$ and $n$ which are obtained by applying a fitting procedure (Table). As follows from Fig. 9 the XY (3D) critical regime approach follows the observed temperature dependence closer than Ginzburg–Landau (GL) behavior and the two-fluid (TF) model. This result seems to be in agreement with a number of measurements of the field penetration depth in single crystals Y-123 for temperatures very close to $T_C$ [19] and is inconsistent with [21], where the experimental data have displayed good agreement with GL theory. However, it might be also to continue the work on the accurate measurement and theoretical analysis of temperature dependence of surface reactance near to $T_C$.

<table>
<thead>
<tr>
<th>Model</th>
<th>$\lambda(T)\text{, nm}$</th>
<th>$n$</th>
<th>$\gamma$</th>
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<tbody>
<tr>
<td>XY</td>
<td>180</td>
<td>1/3</td>
<td>1</td>
</tr>
<tr>
<td>GL</td>
<td>100</td>
<td>1/2</td>
<td>1</td>
</tr>
<tr>
<td>TF</td>
<td>130</td>
<td>1/2</td>
<td>2</td>
</tr>
</tbody>
</table>

4. Conclusion

The QDR-based technique proposed and developed by us allows the investigation of HTS film microwave properties. It permits the highest sensitivity to be achieved in the millimeter wave range. The evaluation of the sensitivity enhancement obtained at very low temperatures indicates the possibility of studying residual microwave surface resistance, which is an important fundamental problem of high-temperature superconductivity. The technique is also useful for the study of reactance properties. No restrictions on developing this QDR-based approach to submillimeter (THz) impedance measurements can now be found.