Accurate Microwave Resonant Method for Complex Permittivity Measurements of Liquids

Kapilevich B. Yu, *Senior Member, IEEE*, S. G. Ogourtsov, *Student Member, IEEE*, V. G. Belenky, *Member, IEEE*, A. B. Maslenikov, and Abbas S. Omar, *Senior Member, IEEE*

***Abstract*—In this paper, a method available for accurate measurements of lossy liquids is presented, tested on standard materials, and compared with the perturbation technique commonly adopted in cavity measurements. The method is based on numerical solving a complex characteristic equation. Realization of the method was done using an E_{010} cylindrical cavity. Results show that measurement errors may be decreased with application of the method presented. The method excludes uncertainties in complex permittivity determination when material has losses and high permittivity.**

***Index Terms*—Biological liquids, cylindrical cavities, losses, permittivity measurement.**

I. INTRODUCTION

ACCURATE microwave (MW) methods for complex permittivity measurement are needed in the investigation of bio-materials, application in medicine, industry, and simulation of interaction a radiation with the human body, etc. [1]–[13].

It is known that bio-materials are often liquids (blood, lymph, physiological solutions, etc.) or presented and investigated in solutions (proteins, carbohydrates, and lipids). Such bio-liquids, having in its basis water, are characterized with high values of permittivity. Also, most bio-liquids have significant losses at MW frequencies. The electromagnetic properties of these materials are sensitive to the temperature [1], [3], [13].

In measurements of complex permittivity of bio-materials at MWs, different modifications of waveguide and transmission-line methods have been successfully used [2], [5]–[9]. As alternatives to the above mentioned, the methods using cavities have also been applied for complex permittivity measurements [10]–[14], [20], [21]. The first group permits processing measurement at continuos and wide frequency band, while the second one is characterized with higher measurement precision and convenient for measurement of small quantities of liquids. Basically, in measurements using cavities, the perturbation approach is commonly applied, which is characterized with limitation on permittivity and losses values as well as sample dimensions [1], [14], [21]. The method suggested is based on the resonator's technique. However, it is freed of the principal limitations inherent perturbation approach due to direct numerical solving of a complex characteristic equation.

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K. B. Yu, S. G. Ogourtsov, and V. G. Belenky are with the Department of Applied Electromagnetics, Siberia State University of Telecommunications and Informatics, Novosibirsk-630102, Russia.

A. B. Maslenikov is with the DNA–Diagnostics Laboratory of Novosibirsk Regional Diagnostic Center, Novosibirsk-630102, Russia.

A. S. Omar is with the Fakultät Elektrotechnik, Otto-Von-Guericke-Universität Magdeburg, Lehrstuhl für Hohffrequenz-und Kommunikationstechnik, D-39016 Magdeburg, Germany.

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II. FORMULATION

A. General Formulation for Model

The perturbation technique and their modifications permit to link via simple formulas changes in the resonant frequency and loaded Q factor determined by sample presence with its complex permittivity [1], [21]. The formulas are obtained by linearization of rigid integral equations, with respect to cavity and sample geometry [18], or expansion into a power series for functions of a nonlinear characteristic equation using first terms only [21]. However, in those papers, estimating formulas for the remainder terms were not be presented. Consequently, a validity of the complex permittivity values is open for a discussion if materials have high permittivity and loss tangent. In these cases, additional systematic measurement errors may reach several percents in permittivity value [15].

A model presented below has no restrictions in complex permittivity values of materials under testing and takes into account losses in cavity walls, as well as an influence of coupling elements.

In this section, the relationship between electrical parameters of a cavity with a dielectric sample to be measured and complex permittivity of the same material is considered.

At first, it is necessary to relate an unloaded resonant frequency with complex natural frequency of the mode resonated from an analysis of a forced oscillation problem of cavity volume with a sample to be tested. In this analysis, normal orthogonal functions of the cavity are used. Relationship between the resonant frequency of an unloaded cavity f and the real part of complex natural frequency f' of the mode resonated may be obtained with respect to losses in the cavity walls [16], [17]. The relationship is valid for a cavity having arbitrary shape and can be written in the following form:

$$f' = f\left(1 + \frac{1}{2}\left(\mathrm{Re}\,\frac{1}{Q_w} - \mathrm{Im}\,\frac{1}{Q_w}\right)\right) \tag{1}$$

where Q_w denotes

$$Q_w = \frac{2}{\delta}\,\frac{\displaystyle\int H_n^2\, dV}{\displaystyle\int H_{n\tau}^2\, dS} \tag{2}$$

and δ is a skin layer depth and H_n is a complex amplitude of the magnetic field being a natural function of a cavity volume with a dielectric sample. H_n is obtained by the field-matching method for the case of walls without losses and $H_{n\tau}$ is a tangential component of H_n at the resonator walls.

The imaginary part of complex natural frequency is calculated from

$$f'' = -\frac{f'}{2Q_S} \tag{3}$$

where Q_S is the Q factor dependent on losses in the sample measured only with

$$\frac{1}{Q_S} = \frac{1}{Q_u} - \frac{1}{Q_{w0}} \tag{4}$$

where Q_u is the unloaded Q factor of the cavity with a sample, Q_{w0} is the partial Q factor determined by losses in cavity walls, and Q_{w0} is determined by

$$Q_{w0} = \frac{2}{\delta} \frac{\int |H_n|^2 \, dV}{\int |H_{n\tau}|^2 \, dS}. \tag{5}$$

It must be pointed out that an imaginary part of complex natural frequency f'' according to (3) is determined by losses in material under test only and does not include losses in cavity walls or external losses.

It should be mentioned that Q_{w0} and Q_w are rigorously identical to each other for a volume without losses or for a volume with uniform lossy filling, but they are different, in the general case, for a nonuniform lossy filling [17].

Note that Q_w and Q_{w0} are depended on the complex permittivity value of a sample to be measured. Hence, behavior of Q_w and Q_{w0} with the complex permittivity value of a sample should be investigated with respect to the type of mode used, sample, and cavity configurations. The situation will be discussed in Section II-B for conditions used in measurements carried out below.

The presence of coupling elements provides additional frequency shift, and neglecting this effect may cause some systematic error in complex permittivity measurements. To evaluate this effect, the method of equivalent circuits is applied [18], [19] For the case of a two-port coupled cavity, it gives

$$\frac{f_m - f}{f} \cong \frac{1}{2}\left(\frac{X_1}{Q_{\text{ex1}}} + \frac{X_2}{Q_{\text{ex2}}}\right) \tag{6}$$

where f_m is the measured resonant frequency of the loaded cavity, X_1 and X_2 are reactances normalized to system impedance, and Q_{ex1} and Q_{ex2} are external Q factors of input and output circuits, respectively.

Secondly, it is necessary to link the complex natural frequency of the mode resonated and the complex permittivity of the material to be measured. It may be done analytically for the cavity with a dielectric sample via a characteristic equation if the cavity volume and sample have a simple geometry, thus, the configurations of the resonator and sample under testing should be chosen to minimize possible measurement error caused by the model approximation.

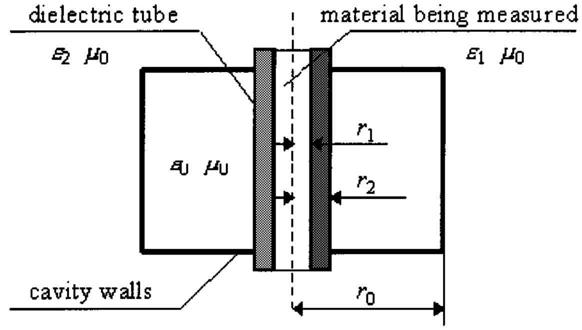

Fig. 1. Cylindrical E_{010} cavity with a sample placed on the axis of the cavity.

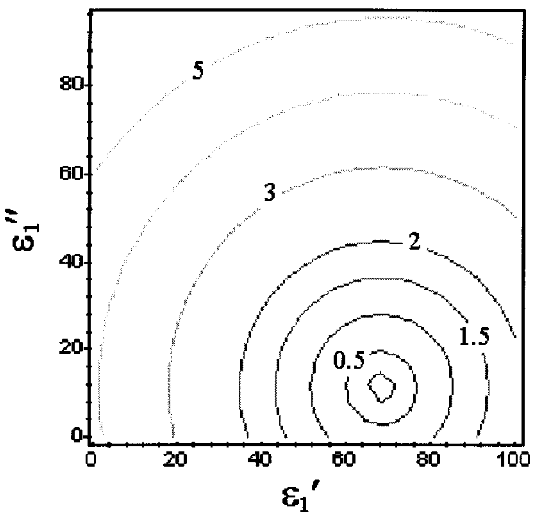

Fig. 2. Example of a location of the minimum for an absolute value of function (7). The value of ε_1 in the minimum is relative complex permittivity of the measured liquid. The liquid is 0.6% gelatin water solution at 23 °C in a glass tube. Complex permittivity of the glass is $\varepsilon_2 = 6.815 - 0.0897j$. Inner and outer tube radii are 0.293 and 0.897 mm, respectively. Cavity radius is 38.189 mm. Value ε_1 in the minimum is evaluated as $68.44 - 11.18j$ for this sample at 3 GHz.

B. Model Realization Using E_{010} *Mode*

For realization of the model satisfying the above demands, the E_{010} mode of the cylindrical cavity is used. The configuration of the resonator containing a material under testing is shown in Fig. 1. The reasons in favor of such a choice are as follows.

1) The configuration permits to obtain a relationship between electrical parameters of the cavity and the complex permittivity of a sample to be measured in the analytical form.
2) E_{010} natural frequency of a hollow cavity is determined by the minimum set of dimensions, namely, radii (Fig. 1). As a result, the measuring error depending on dimensions is reduced.
3) The E_{010} mode has no field variations along the axis. Formulating boundary conditions near holes provides some difficulties for modes having field variations along the axis.
4) Relative error of permittivity measurement due to a sample insertion hole for the E_{010} mode was estimated to be less then 0.1% for the measurements carried out. The estimation was processed with the approach presented in [10].

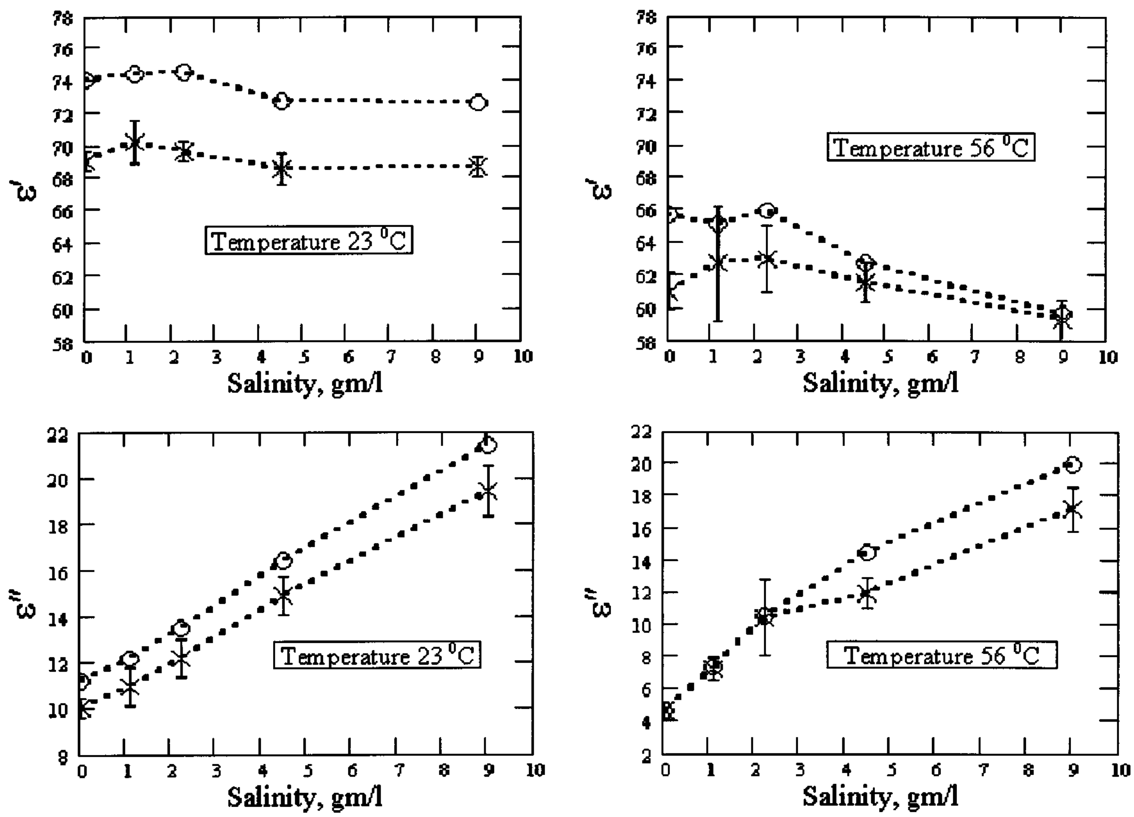

Fig. 3. Measured results for sodium chloride water solutions at 3 GHz. Probability for the confidence interval is 0.9. (○ ○) is the simple perturbation approach. (× ×) is the suggested approach.

The characteristic equation has been obtained using the field-matching method [21] It can be written as follows:

$$\begin{aligned} F(\varepsilon_1) = A_1\Big(&\sqrt{\varepsilon_1}J_{111}(Y_{021}J_{022} - J_{021}Y_{022}) \\ &+ \sqrt{\varepsilon_2}J_{011}(J_{121}Y_{022} - Y_{121}J_{022})\Big) \\ + \sqrt{\varepsilon_2}A_2\Big(&\sqrt{\varepsilon_1}J_{111}(J_{021}Y_{122} - Y_{021}J_{122}) \\ &+ \sqrt{\varepsilon_2}J_{011}(Y_{121}J_{122} - J_{121}Y_{122})\Big) = 0 \end{aligned} \quad (7)$$

where

$$A_1 = J_{000}Y_{002} - J_{002}Y_{000} \quad (8)$$

$$A_2 = J_{000}Y_{102} - J_{102}Y_{000} \quad (9)$$

$J_{nmk} \equiv J_n(x_{mk})$ and $Y_{nmk} \equiv Y_n(x_{mk})$ are the Bessel functions of the nth order of the first and the second kind, respectively; parameter x_{mk} is

$$x_{mk} = \frac{2\pi}{c}\sqrt{\varepsilon_m}f_c r_k \quad (10)$$

$n = 0$ or 1, $m = 0, 1,$ or 2, $k = 0, 1,$ or 2, ε_0, ε_1, and ε_2 are relative complex permittivities of free space, material under testing, and the tube, accordingly, r_0, r_1, and r_2 are the cavity radius, inner, and outer radii of the tube, accordingly, c is velocity of light in a free space, and $f_c = f' - jf''$.

Numerical experiments carried out have demonstrated that the next estimations are valid for lossy samples used in the test measurements

$$0.98 < \frac{\mathrm{Re}(Q_w)}{Q_o} < 1 \quad (11)$$

$$0 < \frac{\mathrm{Im}(Q_w)}{Q_o} < 0.003 \quad (12)$$

$$0.98 < \frac{Q_{w0}}{Q_o} < 1 \quad (13)$$

where Q_0 is the unloaded Q factor of the hollow cavity. Expressions (11)–(13) and the fact that divergences between $\mathrm{Re}(Q_W)$ and Q_0, Q_{W0}, and Q_0 are sufficiently less then the measurement error of Q_0 allowed to use Q_0 as a good approximation for Q_W and Q_{W0} in (1) and (4), respectively. In the general case, a validity of such approximations should be investigated numerically using (2) for the configuration and material used. If these approximations are not valid, the problem should be solved by an iterative process.

Hence, the following expression might be applied to evaluate Q_S:

$$\frac{1}{Q_S} = \frac{1}{Q_u} - \frac{1}{Q_0}. \quad (14)$$

In order to evaluate f', the following formula obtained from (1) is used:

$$f' = f\left(1 + \frac{1}{2Q_0}\right). \quad (15)$$

Formulas (1)–(10) present analytical relationships between f_m, Q_S, measured tube radii r_1 and r_2, and radius of cavity

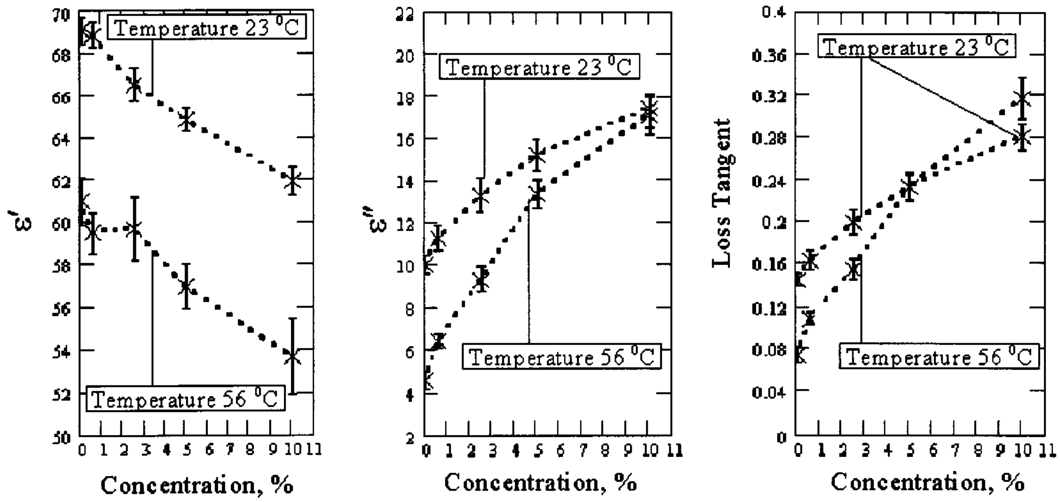

Fig. 4. Measured results for gelatin water solutions at 3 GHz. Probability for confidence interval is 0.9.

r_0 on the one side and the relative complex permittivity $\varepsilon_1 = \varepsilon_1' - j\varepsilon_1''$ on the other side for a known (or preliminary measured) relative complex permittivity of the tube $\varepsilon_2 = \varepsilon_2' - j\varepsilon_2''$. Hence, to determine the relative complex permittivity $\varepsilon_1 = \varepsilon_1' - j\varepsilon_1''$, the system of nonlinear equations

$$\begin{aligned} \mathrm{Re}\big(F(\varepsilon_1'', \varepsilon_1'')\big) &= 0 \\ \mathrm{Im}\big(F(\varepsilon_1', \varepsilon_1'')\big) &= 0 \end{aligned} \tag{16}$$

must be solved numerically. That is equivalent to the problem: minimize $abs(F(\varepsilon_1', \varepsilon_1''))$ with constraints corresponding to the condition discussed, where F is the left-hand-side part of (7).

III. Experimental Setup and Measuring Procedure

The experimental setup consisting of a two-port cylindrical E_{010} cavity connected to a scalar network analyzer was designed for measurements of complex permittivity of liquids at different temperatures. The cavity is placed in a thermostat.

In a general case, the reactances of coupling elements are different. The coupling factors β_1 and β_2 then have to satisfy the following system [22]:

$$\left(\frac{\beta_1 - \beta_2 - 1}{\beta_1 + \beta_2 + 1}\right)^2 = |S_{11}|^2 \tag{17}$$

$$\frac{4\beta_1\beta_2}{(\beta_1 + \beta_2 + 1)^2} = |S_{21}|^2. \tag{18}$$

This system has two solutions being a source of uncertainty. It may be canceled by identically setting the couplings. As a result, it is enough to only measure $|S_{21}|$.

Formula (6) shows that, in order to determine frequency shift caused by reactances of coupling elements, it is necessary to obtain a preliminary estimation for these reactances. In any case, inaccuracy in the determination of coupling element reactances causes a measurement error, which may be estimated with (6).

It is also important to determine a cavity radius with maximum available precision. For example, the cavity used an error in measuring being of ± 0.1 mm leads to an error in determination of distilled water permittivity up to $\pm 10\%$. Again, the cavity radius may be determined with better accuracy via electrical parameters applying

$$r_0 = \frac{\chi_{01} c}{2\pi f'} \tag{19}$$

where χ_{01} is the first root of $J_0(\chi) = 0$ and, here, f' is the real part of the complex natural frequency of a hollow cavity. Numerical experiments have shown that such an approach provides the uncertainty in a cavity radius less then ± 0.005 mm. It corresponds to the error in permittivity less then 0.5%.

To determine complex permittivity of liquids, the parameters of a hollow cavity (unloaded Q factor, radius) and the complex permittivity of a dielectric tube material must be evaluated preliminary for temperatures at which liquids are investigated. Tubes have different inner and outer radii. Inner radii were varied from 0.25 to 0.3 mm. Outer radii were varied from 0.8 to 0.9 mm. Inner and outer radii of the tubes have been measured with a microscope providing errors less then $\pm 1\%$.

As an example, the typical behavior of contour lines of $abs(F(\varepsilon_1', \varepsilon_1''))$ is given in Fig. 2 in a space of ε_1' and ε_1''. One can see that their regularity permits to apply traditional methods for searching extremum in a process of reconstruction of ε_1' and ε_1''.

According to the method presented herein, the complex permittivity for measured materials are determined with numerical solving of the system (16). The starting guess for the numerical procedure and the problem of uniqueness of the solution are easily solved using contour plots similar to those shown in Fig. 2.

IV. Verification of the Technique Proposed and Discussion

To verify the method proposed and to investigate its ability to resolute small variations in complex permittivity, the materials imitating lossy biological liquids have been used [3], [13]. They are sodium water solutions (see Fig. 3) and gelatin water solutions (see Fig. 4). Measurements were carried out at 3 GHz. Results of measurements were processed to yield the mean values of complex permittivity with confidence intervals of 0.9.

The method suggested was compared with a simple perturbation technique [1], [21] using the same test samples, cavity, and

MW setup. Results of the comparison are presented in Fig. 3. Divergence in the real part of the complex permittivity between the method proposed and the simple perturbation is observed. Agreement in the imaginary part of the complex permittivity values is better. Simple perturbation formulas give higher values for the real part of the complex permittivity in comparison to those measured with the method proposed. It agrees with theoretical analysis of the perturbation technique [23].

Analysis of the data given in Fig. 4 allows to formulate the following comments.

1) The measurements of ε' seems to be more informative compared to ε'' since a higher resolution can be reached with a temperature variations for the liquid measured.
2) The temperature behavior of ε'' demonstrates uncertainty for concentrations over 8%. In this case, a discrepancy in ε'' is very small for different temperatures.
3) The intersection point is observed in a behavior of loss tangent near the gelatin's 5% concentration. Such existing points are undesirable phenomena in diagnostic studies.

One can see from Figs. 3 and 4 that behavior of complex permittivity of complicated materials with temperature is strongly determined by concentrations of its components.

V. Conclusion

Results presented in this paper show that systematic measurement errors can be decreased in cavity complex permittivity measurements with the application of the proposed method.

Taking into account applications associated with measurements of biological liquids, the following advantages of the proposed method should be pointed out:

- small quantity of measured material is needed, less then 0.02 μL;
- measured samples are simple in preparing; hence, the high number of samples may be measured per a unit of time;
- in comparison to transmission-line methods, there is no uncertainties in determination for material with high permittivity and losses;
- there are no model limitations associated with a value of complex permittivity and dimensions of a sample under testing.

The method is available for complex permittivity measurements at different temperatures. Experimental setup can also be easily modified for continues monitoring of the complex permittivity of lossy liquids. The method proposed is intended to be used in developing some medical diagnostics based on precise investigation of the behavior of the fundamental property of bio-materials, such as their complex permittivity.

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Kapilevich B. Yu (SM'97) received the M.S. degree in radio physics from Tomsk State Technical University, Tomsk, Russia, in 1961, the Ph.D. degree from Novosibirsk State Technical University, Novosibirsk, Russia, in 1969, and the Dr.Sc. degree from the Moscow Power Energetic University, Moscow, Russia, in 1986.

He was with Microwave Industry until 1970. He then joined the Siberia State University of Telecommunications (SibSUTI), Novosibirsk, Russia, as an Assistant Professor (1972), Full Professor, and the Head of the Microwaves and Antennas Department (1988). From 1993 to 1996, he was a Visiting Professor at the University of Technology of Malaysia. He is currently the Head of the Applied Electomagnetics Department, SibSUTI. He has authored or co–authored four books and over 120 papers dedicated to both active and passive MW devices, theory guided waves, electromagnetics, and some related topics. He holds 14 patents.

S. G. Ogourtsov (S'99) received the Diploma degree in physics from the Novosibirsk State University, Novosibirsk, Russia, in 1993, and is currently working toward the Ph.D. degree in applied electromagnetic at the Siberia State University of Telecommunications and Informatics, Novosibirsk, Russia.

His research interests are in electromagnetic modeling, MW measurements, and its application.

V. G. Belenky (M'99) received the Diploma degree in radio-technics and the Ph.D. degree in microwave engineering from Novosibirsk State Technical University, Novosibirsk, Russia, in 1985 and 1995, respectively.

He is currently an Associate Professor in the Siberian State University of Telecommunications, Novosibirsk. His research interests include MW passive and active filter design, precision measurement methods, and numerical electromagnetics, with particular emphasis on biomedical problem and diagnostics.

A. B. Maslenikov received the Diploma degree in medicine from the Novosibirsk State Medical Institute, Novosibirsk, Russia, in 1985, and the Ph.D. degree in medical science from the Scientific Research Institute of Medical Genetics (SB RAMS), Tomsk, Russia, in 1999.

Since 1994, he has been the Head of the DNA–Diagnostics Laboratory, Novosibirsk Regional Diagnostic Center, Novosibirsk, Russia. His research interest is in molecular genetics, inherent diseases, metabolic process in human organisms, and medical diagnostics.

Abbas S. Omar (M'87–SM'89) received the B.Sc. and M.Sc. degrees from the Ain Sham University, Cairo, Egypt, in 1978 and 1982, respectively, and the Dr.-Ing. degree from the Technical University of Hamburg–Harburg, Hamburg, Germany, in 1986, all in electrical engineering.

Since 1990, he has been a Professor of electrical engineering and, since 1998, the Head of Chair of Microwave and Communication Engineering at the Otto-Von-Guericke-Universität Magdeburg, Magdeburg, Germany. He has authored and co-authored over 130 technical papers extending over a wide spectrum of research areas. He recently directed his search activities to the solution of inverse problems related to remote sensing and MW tomography. His current research fields cover the areas of remote sensing and MW imaging, high-speed multimedia satellite and mobile communication, electromagnetic bullets and their applications to secure low-power wide-band communications and subsurface tomography, stochastical electromagnetics and their meteorological, environmental, and biomedical applications, field theoretical modeling of MW systems and components, MW measurements, and submillimeter-wave signal generation and processing.