

Dielectric Properties of Substrates for Deposition of High-T_c Thin Films Up to 40 GHz

Janusz Konopka and Ingo Wolff, *Fellow, IEEE*

Abstract—Dielectric properties of CaNdAlO₄, LaAlO₃, SrLaAlO₄, SrLaGa₃O₇ and NdGaO₃ monocrystals, prospective substrate materials for the deposition of thin films of high-temperature superconductors, were measured with high accuracy at frequencies up to 40 GHz in the temperature range 10 to 300 K. Most materials exhibit uniaxial anisotropy with ϵ' ranging from 8.88 (SrLaGa₃O₇ along optical c-axis) to 24.18 (LaAlO₃). A decrease of ϵ' with temperature decrease was observed in all materials except CaNdAlO₄ (perpendicular to c-axis) where ϵ' increases. Microwave losses in LaAlO₃, SrLaAlO₄ and SrLaGa₃O₇ decrease with temperature while CaNdAlO₄ and NdGaO₃ pronounced loss increase was found at temperatures below 100 K. We suggest, that in the latter materials at lower temperatures, neodymium ions become magnetically ordered and are responsible for the observed effects.

I. INTRODUCTION

A SUBSTANTIAL effort has been already made to characterize the dielectric properties of prospective monocrystalline substrates for growing thin film of high-T_c superconductors ([1], [2] and references therein). However, the design of microwave superconducting devices in both coplanar and microstrip configurations [3], [4] requires much more accurate data than those as yet published. In this paper we present the results of a systematic study of the dielectric properties of CaNdAlO₄, LaAlO₃, SrLaAlO₄, SrLaGa₃O₇ and NdGaO₃ monocrystals in the frequency range from 4 GHz to 40 GHz and in the temperature range from 10 K to 300 K, based on the measurement of self-resonances of a rectangular cavity completely filled with the investigated material. Up till now only LaAlO₃ was widely used as high-T_c substrate. Other studied materials having also perovskite-like crystalline structures are interesting as prospective substrates not only because their lattice constants are close to that of YBa₂CuO₃O₇, but also because (except for NdGaO₃) they exhibit no twinning. In most cases our data, in particular concerning the anisotropy, are the first reports. In the case of LaAlO₃ and NdGaO₃ the existing data [1], [2] are considerably improved. The frequency and temperature range in which these materials are characterized is also much extended. It is emphasized that microwave measurements of anisotropic materials are difficult and require special

care in setting-up the experiment, and interpretation of the experimental data.

II. THEORY

Resonant frequencies of a rectangular cavity with perfectly conducting walls, completely filled by anisotropic but lossless medium, whose permittivity and permeability tensors are diagonal in the coordinate system set by the cavity axes, were calculated by Lewandowski [5].

The cavity size along optical axis is denoted by c and other two sizes as a and b . The relative permittivity tensor components are ϵ_a , ϵ_b , ϵ_c , and the relative permeability tensor components μ_a , μ_b , μ_c . If uniaxial anisotropy is assumed, then

$$\epsilon_a = \epsilon_b = \epsilon, \quad \mu_a = \mu_b = \mu.$$

For this case the resonant modes become true TM _{m} and TE _{m} modes with respect to c -axis and their frequencies are determined by the set of equations [5], [7]:

$$f_{TM_m}^2 = \frac{1}{4\mu_0\epsilon_0\mu} \left(\frac{k_{mc}^2}{\epsilon} + \frac{k_{ma}^2}{\epsilon_c} + \frac{k_{mb}^2}{\epsilon_c} \right), \quad k_{mc}^2 \neq 0, \quad (1a)$$

$$f_{TE_m}^2 = \frac{1}{4\mu_0\epsilon_0\epsilon} \left(\frac{k_{mc}^2}{\mu} + \frac{k_{ma}^2}{\mu_c} + \frac{k_{mb}^2}{\mu_c} \right), \quad k_{ma,b}^2 \neq 0, \quad (1b)$$

where k_{ma} , k_{mb} and k_{mc} are the wave numbers indexed by mode number $m = (m_a, m_b, m_c)$.

Observe that if the relative permeabilities $\mu \neq \mu_c \neq 1$, then the resonant frequency measurements allow to determine only three quantities e.g. $\mu\epsilon_c$, μ_c/μ and $\mu\epsilon$ instead of the four unknowns μ , μ_c , ϵ and ϵ_c . This is the same limitation as in the scalar case, where from resonant frequency data only the product of $\mu\epsilon$ can be determined.

As was already mentioned, (1a) and (1b) are valid for a cavity completely enclosed by perfectly conducting walls and filled with lossless medium. Deviation from these conditions introduces, in general, a coupling between the cavity modes and changes their resonant frequencies in a manner for which no detailed theory is available. Relatively simple is the treatment if only medium losses are present. In this case a “principle of detailed balance” [6] can be formulated which states that

Manuscript received March 31, 1992; revised August 3, 1992.

J. Konopka is with Instytut Fizyki PAN, Warszawa, Poland.

I. Wolff is with Universität Duisburg ATE-SFB254, 4100 Duisburg 1, P.O. Box 101503, Germany.

IEEE Log Number 9204297.

the loss of energy of any resonant mode due to the excitation of other modes is exactly balanced by the energy contributions made by these modes. As a consequence, medium losses can be accounted for as in the isotropic case, by admitting complex ϵ and μ quantities and complex resonant frequencies. As the materials treated in this paper are low-loss materials, it is assumed that other analogies to isotropic cavities are also maintained and the usual equivalent circuit of the cavity can be constructed for each cavity mode.

Therefore, it is justified to use (1a) and (1b) to determine the real parts of the permittivity tensor elements, provided that appropriate corrections for the coupling losses and wall losses will be used [8]. This procedure was followed, by inserting successively to (1a) and (1b), an appropriate pair of measured resonant frequencies of the identified modes.

The change of the resonant frequency due to a coupling aperture was calculated using the relation [8]

$$\delta_c f_m = -\beta_m \frac{s}{S} \frac{\sqrt{s}}{\lambda_{om}}, \quad (2)$$

where β_m denotes a coefficient depending on transmission line geometry and mode, s and S the areas of the coupling aperture and cavity wall, respectively and λ_{om} is the free-space wavelength corresponding to the resonant frequency. β_m are to be determined experimentally. Similarly, the effect of finite conductivity of cavity walls can be assumed to be equivalent to an additional detuning [8]

$$\delta_{walls} f_m = -1/2Q_{walls}. \quad (3)$$

Microwave losses for each mode were calculated from the measured half-power line-width $2\Delta F$ and the relation

$$\tan \delta = \frac{1}{Q_{eff}} - \frac{1}{Q_{walls}} - \frac{2}{Q_{coup}}, \quad (4)$$

where $Q_{eff} = F_{res}/2\Delta F$. Loss tangent contains generally the total effect of dielectric and magnetic losses. With the measuring method used in this study it is difficult to distinguish whether the electromagnetic energy is lost to the lattice by interactions of the electric fields with electronic charge or by interactions of magnetic fields with magnetic moments. The situation becomes clear only for the materials which do not contain magnetic ions.

III. EXPERIMENTAL

The samples of CaNdAlO_4 , SrLaAlO_4 , and $\text{SrLaGa}_3\text{O}_7$ were prepared from single crystals, exactly X-ray oriented and cut along the crystallographic axes to form a set of 4 rectangular parallelepipeds (with dimensions of about $6.04 \times 5.05 \times 2.56 \text{ mm}^3$), which were polished to get an optical quality surface on all 6 faces. For anisotropic crystals, two of the parallelepipeds were *c*-axis oriented along the narrowest side (referred further to as samples A), while the other two had *c*-axis directed along the wider side (samples B). The samples of LaAlO_3 and NdGaO_3 had the form of $10 \times 10 \times .5 \text{ mm}^3$ rectangular parallel-

epipeds. Small deviations from perfect geometry were found on the level of 0.05% and averaged final dimensions were used in the calculations. The samples were covered on all 6 faces by a thin ($\sim 30 \text{ nm}$), sputtered gold film on which a thicker ($\sim 30 \mu\text{m}$), continuous layer of the same metal was electrolytically deposited. Gold instead of silver was chosen for cavity walls because of its lower thermal expansion coefficient (closer to that of CaNdAlO_4), giving better adherence to the crystal surface during thermal cycling. Resonant frequencies can be significantly changed by plastic deformation of contracting metallic walls during cool-down, which produces air pockets at the crystal-metal interface during warm-up cycle. No such effect was observed with our choice of cavity design. In the final step of sample preparation, the gold layer was cut to expose small $0.4 \times 0.4 \text{ mm}^2$ coupling apertures in opposing cavity walls.

IV. MEASUREMENTS AND DISCUSSION

The resonant frequencies of particular modes and the cavity transmission losses were measured using either a network analyzer or alternatively a computer controlled, GPIB driven set of separate instruments, composed of approximate frequency sources (sweepers), detectors, lock-in amplifier, and a data acquisition and plotting system. One of our major concerns was to avoid parasitic coupling around the cavity and parasitic resonances inside the hermetic low-temperature enclosure of a closed-cycle refrigerator, housing the cavity and microstrip coupling lines. Parasitic couplings, difficult to avoid in such broad-band measurements, may cause errors by shifting the position and distorting the shape of the cavity resonance lines. In order to avoid the possible temperature measuring errors—especially below 50 K, where in the closed-cycle refrigerating system the heat capacity of the cooling head drastically decreases—the data were collected for both cooling-down and warming-up cycles.

The samples were placed with coupling holes facing very small ($\sim 0.3 \text{ mm}^2$) coupling loops terminating two microstrip input and output lines. The size of the coupling apertures was experimentally adjusted until first resonant transmissions become detectable. The coupling apertures were then subsequently increased and the shift of resonant frequencies and the decrease of the effective Q-factor measured. In such a way not only the coefficients β_m for particular modes could be determined, but also the influence of the cavity loading by coupling apertures could be estimated and distinguished from the cavity filling material losses and the wall losses. Depending on the sample, resonant frequencies of 20 to 32 different cavity modes were identified and the effective Q-factor measured, though some of them were too weakly coupled to assure good accuracy of the measurements in the full temperature range.

Wall losses were calculated using the conductivity of gold $\sigma = 4.17 \times 10^7 [1/\Omega\text{m}]$ multiplied by a coefficient associated with the deposition technology, which from our

experience with similar gold films on GaAs was taken to be equal to 0.25. As can be seen from (4) this procedure may introduce an appreciable error in determination of microwave losses. However, the temperature dependence of the losses will be maintained.

In Fig. 1 dielectric permittivities ϵ' of LaAlO_3 and NdGaO_3 versus frequency is presented, demonstrating the accuracy of our measurements. Up to the highest frequencies at which measurements could be performed, the permittivities were found to be constant within the experimental error. The investigated samples were supplied by one producer only. It would be interesting to measure the samples coming from different sources and to determine whether or not the dielectric properties are influenced by the method of growth, by annealing at temperatures close to that at which thin films of high-T_c materials are deposited and by the degree of twinning.

Fig. 2 shows the variation of ϵ' with temperature for the same materials. Upper curve for LaAlO_3 , marked with full triangles, has been corrected for the thermal contraction of the resonator, using thermal expansion coefficient $\alpha = 10 \times 10^{-5}$ [10]. The thermal expansion coefficient for NdGaO_3 was not found in the available literature.

The measurements were performed at X-band frequencies but no appreciable deviations from the $\epsilon'(T)$ curves were found at higher frequencies.

In Fig. 3 the dielectric permittivities of ϵ'_c and $\epsilon'(\epsilon'_{ab})$ for CdNdAlO_4 and SrLaAlO_4 versus frequency are presented. Here also ϵ'_c and ϵ' are within experimental errors constant over the microwave region from 8 to 40 GHz. Our results and in particular strong anisotropy of ϵ' are first reported for these materials at microwave frequencies. It is interesting to note that in $\text{SrLaAlO}_4 \epsilon'_c > \epsilon'$, while in $\text{CaNdAlO}_4 \epsilon' > \epsilon'_c$.

In Fig. 4 and Fig. 5 the temperature dependence of the resonant frequencies of the fundamental mode of CaNdAlO_4 and SrLaAlO_4 for samples A (TE₁₁₀ with respect to c-axis) and B (TM₀₁₁ with respect to c-axis) are compared. For these modes the electric field is directed respectively along and perpendicularly to c-axis of the crystal. Surprisingly, the frequency shift for the principal mode of CaNdAlO_4 with E field perpendicular to the c-axis has the opposite slope as compared to CaNdAlO_4 with E parallel to c-axis, as well as compared to the slopes obtained for both orientations of E in SrLaAlO_4 . This behaviour was verified on all CaNdAlO_4 samples for all resonant modes with E fields perpendicular to the optical (c) axis.

The increase of the resonant frequency of the dielectric resonator, with the decreasing temperature, when corrected for all experimental errors (neglecting the irregularities of the experimental curves of CaNdAlO_4 below 50 K), can be explained by a small decrease of the dielectric constant with temperature. Indeed, a similar behaviour was found in LaAlO_3 , NdGaO_3 and $\text{SrLaGa}_3\text{O}_7$. However, no reasonable mechanism related to electric polarization, which could cause the decrease of the resonant frequency (i.e. an increase of ϵ') of a resonator with de-

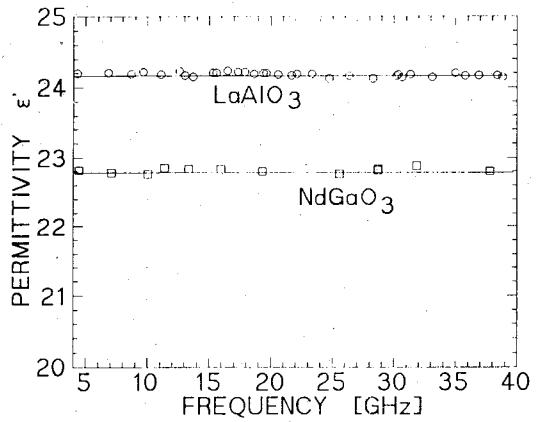


Fig. 1. Real part of the permittivities of LaAlO_3 and NdGaO_3 versus frequency at 298 K.

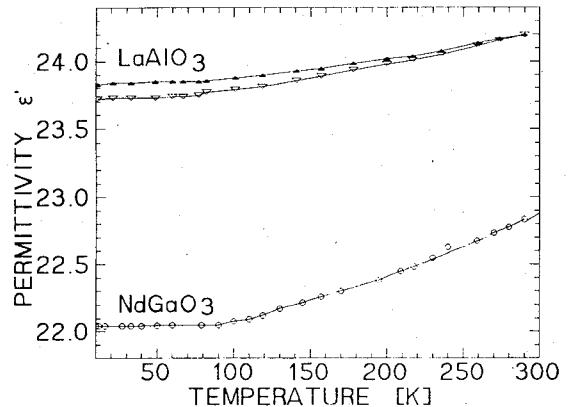


Fig. 2. Temperature variation of the permittivities of LaAlO_3 and NdGaO_3 . Upper curve for LaAlO_3 was corrected for thermal contraction of the sample (thermal expansion coeff. $\alpha = 10 \times 10^{-5}$) [10]. Data on α for NdGaO_3 was not available.

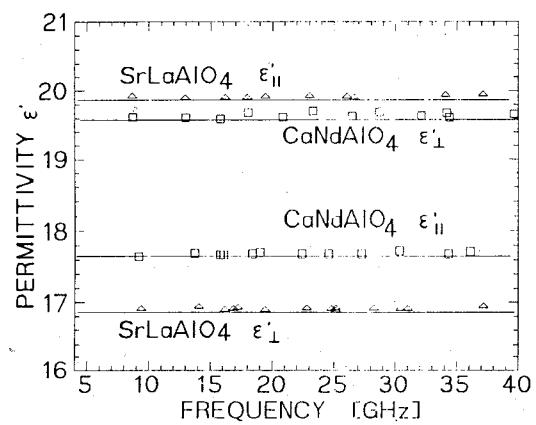


Fig. 3. Dielectric permittivities of CaNdAlO_4 and SrLaAlO_4 versus frequency. Note much higher anisotropy of the SrLaAlO_4 and the reversal of magnitudes of ϵ'_c and ϵ' in both materials.

creasing temperature as shown in Fig. 5 for CaNdAlO_4 , can be suggested. Thermal contraction of the cavity as a possible cause of such an effect can be easily ruled out. The thermal expansion coefficients for both materials has been recently reported by Byszewski *et al.* [11]. These

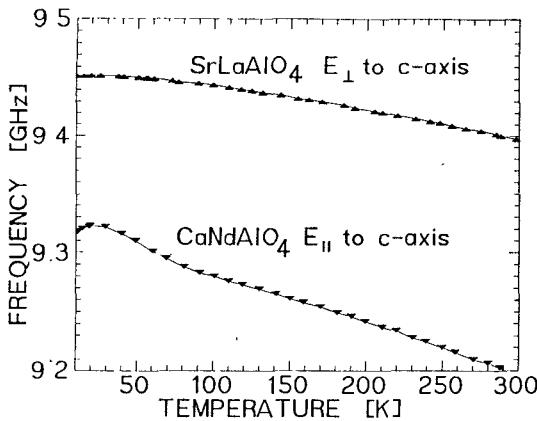


Fig. 4. Resonant frequency of the fundamental mode of the cavity filled with SrLaAlO₄ (geometry B) and with CaNdAlO₄ (geometry A).

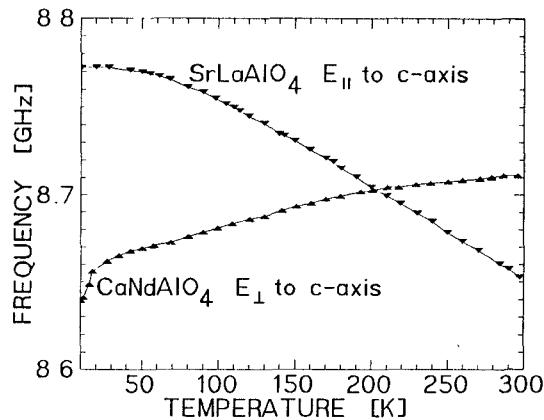


Fig. 5. Resonant frequency of the fundamental mode of the cavity filled with SrLaAlO₄ (geometry A) and with CaNdAlO₄ (geometry B). Note opposite slope of the latter curve.

coefficients show an appreciable anisotropy, but they are all positive and comparable in magnitude, so that they influence the resonance frequencies in a similar way.

One explanation which was considered, is a small increase of the permeability μ_c which can be due to the orientation of the spins of the neodymium Nd³⁺ ions in 4f³ configuration. Insulating crystals containing rare earth ions with partially filled electronic f-shells are found to obey well Curie's law. The (isotropic) permeability in this case can be expressed as

$$\mu'_c = \mu' = 1 + 4\pi \frac{N\mu_B^2 p^2}{3Vk_B T}, \quad (5)$$

where N is the number of magnetic ions in the sample, V is the sample volume, μ_B denotes Bohr magneton, k_B Boltzmann constant, and p is the effective Bohr magneton number. Substituting $p = 3.62$, [9], $V = 0.07778 \text{ cm}^3$ and $N \sim 4.7 \times 10^{20}$, we find that in the case of noninteracting ions the maximal expected effect can be estimated as $\mu' = 1 + 0.16/T$, or $\mu' = 1.00053$ at 300 K and $\mu' = 1.016$ at 10K. In the vicinity of room temperature, the correction on μ' as calculated from (5) does not exceed our experimental errors. At low temperatures this contribution can account for a large part of the apparent

increase of the permittivity ϵ' . However, it cannot change the slope of the $\epsilon'(T)$ curve. It can be seen also from (1a) and (1b) that μ_c , rather than μ , plays a dominant role in the determination of ϵ' . Therefore, the observed effect, especially between 160 K and 50 K, would require a magnetic anisotropy with $\mu'_c > \mu'$. Another explanation which was also viewed, is a crystallographic phase transition in CaNdAlO₄ below 100 K. The physics of these effects is, however, beyond the scope of this paper and will be published elsewhere.

Fig. 6 shows the variation of ϵ' with temperature assuming that relative permeabilities $\mu'_c = \mu' = 1$. The curves marked with squares were calculated from (1a) and (1b) with appropriate corrections for detuning effects due to coupling susceptances and wall losses, while those marked with full triangles have been corrected for the thermal contraction of the resonator, using thermal expansion coefficients $\alpha_c = 15.7 \times 10^{-6} \text{ K}^{-1}$ and $\alpha \equiv \alpha_{ab} = 8.67 \times 10^{-6}$, [11].

We would like to emphasize, that the described above behavior of the dielectric permittivities of CaNdAlO₄ will cause different frequency shift with temperature in resonators designed in coplanar and microstrip geometries, respectively. In particular, the increase of permittivity with the decrease of temperature, may be advantageous and should lead to partial compensation of the troublesome temperature frequency shift of superconducting filters. The latter is due to the inherent and inevitable temperature change of the kinetic inductance of the superconducting strips.

Fig. 7 shows the variation of ϵ' with temperature for SrLaAlO₄. Here also the curves marked with squares were calculated from (1a) and (1b) with appropriate corrections for detuning effects due to coupling susceptances and wall losses, while those marked with full triangles have been corrected for the thermal contraction of the resonator, using thermal expansion coefficients $\alpha_c = 17.1 \times 10^{-6} \text{ K}^{-1}$ and $\alpha = 7.55 \times 10^{-6}$, [11].

The dielectric permittivities of SrLaGa₃O₇ (geometries A and B) are shown in Fig. 8. This material exhibits the highest anisotropy, offering at the same time to the designers of superconducting microwave components the lowest dielectric permittivity (for E field parallel to c-axis) even lower than that of MgO.

The temperature behaviour of the dielectric permittivities for both crystal orientations of SrLaGa₃O₇ is shown in Fig. 9. It is quite similar to that of SrLaAlO₄. Again the curves marked with squares were calculated from (1a) and (1b) with appropriate corrections for detuning effects due to coupling susceptances and wall losses, while those marked with full triangles have been corrected for the thermal contraction of the resonator, using thermal expansion coefficients $\alpha_c = \alpha = 12.8 \times 10^{-6}$, [12].

Microwave losses versus temperature for LaAlO₃, SrLaAlO₄ and SrLaGa₃O₇ are jointly presented in Fig. 10. All these materials do not contain magnetic ions, so the losses as measured can be considered as dielectric losses. All materials exhibit quite similar $\epsilon''(T)$ dependence.

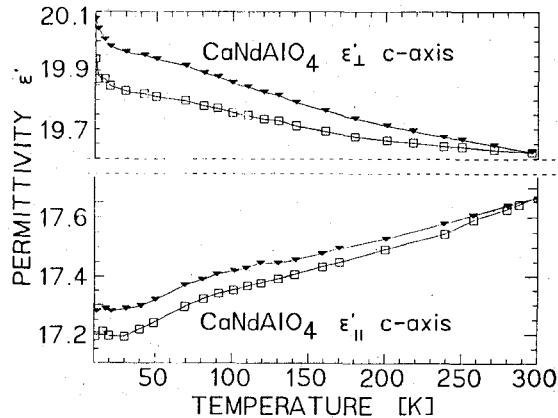


Fig. 6. Temperature variation of the permittivities of CaNdAlO_4 along and perpendicular to optical c-axis $\mu'_c = \mu' = 1$. The curves marked with squares were calculated from (1a) and (1b) with appropriate corrections for detuning effects due to coupling susceptances and wall losses, while those marked with full triangles have been corrected for the thermal contraction of the resonator, using thermal expansion coefficients $\alpha_c = 15.7 \times 10^{-6} \text{ K}^{-1}$ and $\alpha \equiv \alpha_{ab} = 8.67 \times 10^{-6}$ [11].

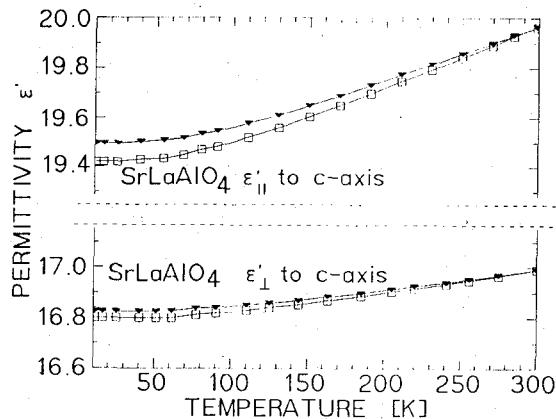


Fig. 7. Temperature variation of the permittivities of SrLaAlO_4 along and perpendicular to optical c-axis. The curves marked with squares were calculated from (1a) and (1b) with appropriate corrections for detuning effects due to coupling susceptances and wall losses, while those marked with full triangles have been corrected for the thermal contraction of the resonator, using thermal expansion coefficients $\alpha_c = 17.1 \times 10^{-6} \text{ K}^{-1}$ and $\alpha = 7.55 \times 10^{-6}$ [11].

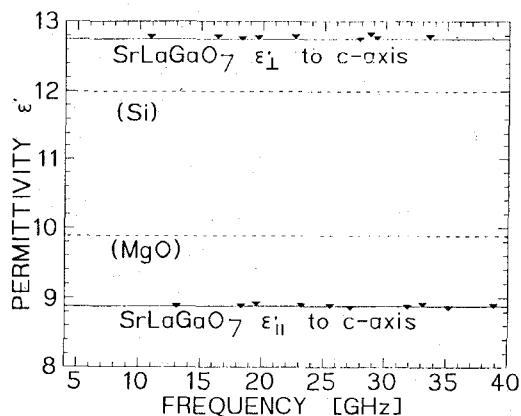


Fig. 8. Frequency dependence of the permittivities of $\text{SrLaGa}_3\text{O}_7$ (geometry A and B). Permittivities of Si and MgO, marked with dotted lines, are shown for comparison.

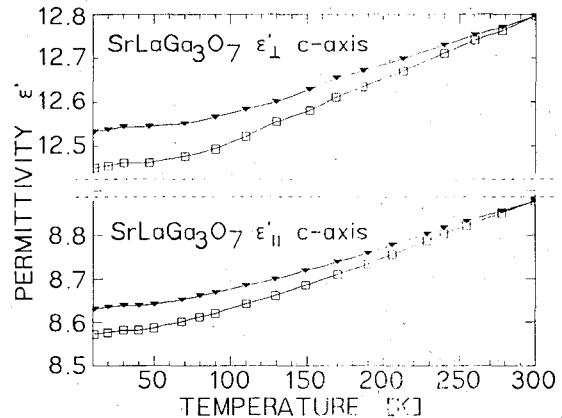


Fig. 9. Temperature variation of the permittivities of $\text{SrLaGa}_3\text{O}_7$ along and perpendicular to optical c-axis. The curves marked with squares were calculated from (1a) and (1b) with appropriate corrections for detuning effects due to coupling susceptances and wall losses, while those marked with full triangles have been corrected for the thermal contraction of the resonator, assuming thermal expansion coefficients $\alpha_c = \alpha = 10 \times 10^{-6}$ [12].

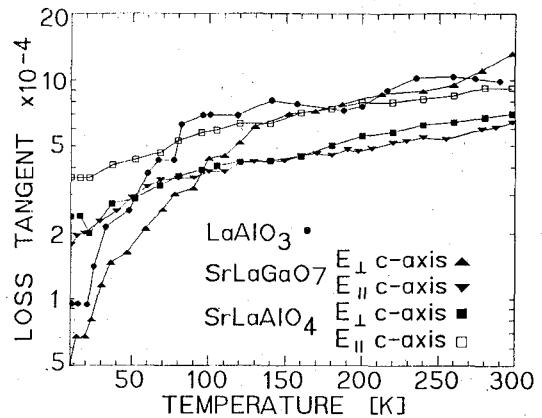


Fig. 10. Loss tangent (ϵ''/ϵ') of LaAlO_3 , SrLaAlO_4 and $\text{SrLaGa}_3\text{O}_7$.

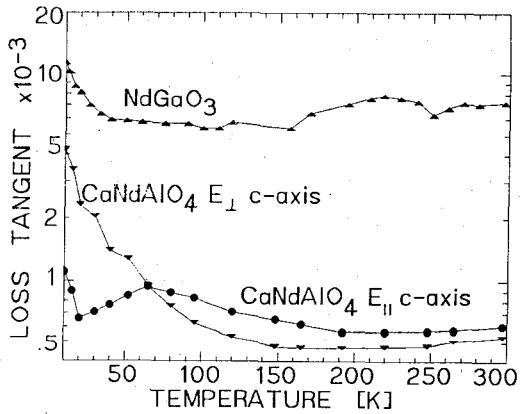


Fig. 11. Loss tangent of CaNdAlO_4 and NdGaO_3 . Note the increase of dielectric losses at low temperatures and the local maximum of losses for E field parallel to c-axis in CaNdAlO_4 .

However, the lowest values have been measured for ϵ''_c in $\text{SrLaGa}_3\text{O}_7$.

Fig. 11 shows the loss tangent versus temperature for CaNdAlO_4 and NdGaO_3 . These materials were placed on the drawings together, because they exhibit a peculiar be-

haviour of the losses. In both cases the loss tangent increases at low temperatures. The increase of microwave losses in NdGaO_3 below 170 K at 6.5 GHz was recently observed by Konaka *et al.* [1] and also at 16 GHz by Klein [13]. Both materials contain Nd^{3+} ions in $4f^3$ configuration. As was already observed each ion has the magnetic moment equal 3.62 Bohr magnetons. We suggest that the collective interaction of these magnetic moments with microwave fields can be responsible for the increase of losses at low temperatures in these materials. [7].

V. CONCLUSIONS

In conclusion, dielectric properties of CaNdAlO_4 , LaAlO_3 , NdGaO_3 , SrLaAlO_4 , and $\text{SrLaGa}_3\text{O}_7$ monocrystals have been measured at microwave frequencies from 4 to 40 GHz and at temperatures from 10 to 300 K with the accuracy sufficient for precise designing of microwave superconducting components. Most materials were found to be uniaxially anisotropic, exhibiting at 300 K permittivities ϵ' ranging from 8.88 for $\text{SrLaGa}_3\text{O}_7$ (for E field parallel to the optical c-axis) to 24.18 for LaAlO_3 . In all materials ϵ' decreases with temperature with the exception of CaNdAlO_4 where ϵ' (along optical c-axis) increases with decreasing temperature. In NdGaO_3 and in CaNdAlO_4 microwave losses unexpectedly increase below 100 K. It is suggested that the magnetic interactions within the sublattice of neodymium ions with microwave fields can be responsible for the observed effects.

ACKNOWLEDGMENT

The authors would like to thank S. J. Lewandowski for many valuable remarks and for providing us with computer program for identification of resonant modes in anisotropic cavities, to A. Pajaczkowska for many helpful discussions and to J. Albrecht, R. Bertenburg, N. Koster, H. Watzel and L. Westerhausen for helping to solve many technological problems.

This work was performed under contract No. TK0332/2 of the German Ministry of Research and Technology. The authors only are responsible for the content.

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Janusz Konopka received the M.Sc. degree in electronics from Warsaw Technical University, the Ph.D. in physics (1964) and the habilitation degree (1970) from Polish Academy of Sciences.

In 1961 he joined the Instytut Fizyki PAN in Warsaw working successively as a group leader, head of the division, deputy director for scientific affairs and full professor. His research interests included dielectrics, narrow-gap semiconductors and superconductors (Josephson voltage standard). In 1967-68 he worked in Paris at Ecole Polytechnique on conduction electron spin resonance in semiconductors. In 1990 he joined the E.E. Department of the University of Duisburg, Germany where he works on physics and applications of high- T_c superconductors.

Dr. Konopka is a member of Polish and European Physical Societies. Ingo Wolff (M'75-SM'85-F'88), for a photograph and biography, see this issue, p. 2227.