

# Beam Waveguide Spectroscopy of Materials in Millimetre and Submillimetre Waves Ranges

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For wavelengths  $\lambda$  longer than approximately 6÷8 mm effective waveguide and resonator technique of low-loss materials properties measurement is elaborated . On the other hand for wavelengths shorter than 0.5 mm very good Fourier transform and laser spectroscopy methods are available..

However, there are some difficulties in carrying out material investigation in the wavelength interval from 5÷4 mm to 0.6÷0.5 mm. The reason is that the waveguide technique is ineffective due to an decrease of the waveguide dimensions, gaps between waveguide and sample walls , on the other hand the optical technique is ineffective due to diffraction effect affecting the field structure and not allowing the use of geometrical laws of optics.

The best way for the measurement of material properties is to use quasi-optical lens beam waveguide transmits only the fundamental low-loss Gaussian type mode with enough small cross-sectional dimensions of a wave beam and large losses for higher order modes [13,14]. In this case the incident on the plane-parallel specimen wave and the wave on the receiving aperture are the same and it is possible to estimate the measurement errors due to a thickness  $l$  of a plane-parallel specimen, inclination of its boundaries to the beam waveguide axis  $\Psi$  and cross-sectional dimensions of the specimen  $a$  and receiving aperture  $b$ . So for beam waveguide consisting of non-reflecting lenses with  $a \approx b > 10\lambda$ ,  $l \leq 1$  cm,  $\psi \leq 0.2$ , refractive index  $n < 5$  the magnitude of errors in transmission  $|t|^2$  and reflection  $|r|^2$  coefficients are less than  $5 \cdot 10^{-3}$  [14], whereas in conventional free space measurements these values may be in this case more than  $10^{-1}$  [18]. Using this quasi-optical set up it was measured complex refractive indexes  $n + ik$ , in very wide intervals  $n$  from 1.05 to 20,  $|t|$  from approximately 1 to  $10^{-6}$ ,

$\tan \delta = \frac{2n\kappa}{n^2 - \kappa^2}$  from  $10^{-5}$  to 1, and  $|r|$  from  $\approx 1$  to  $10^{-4}$

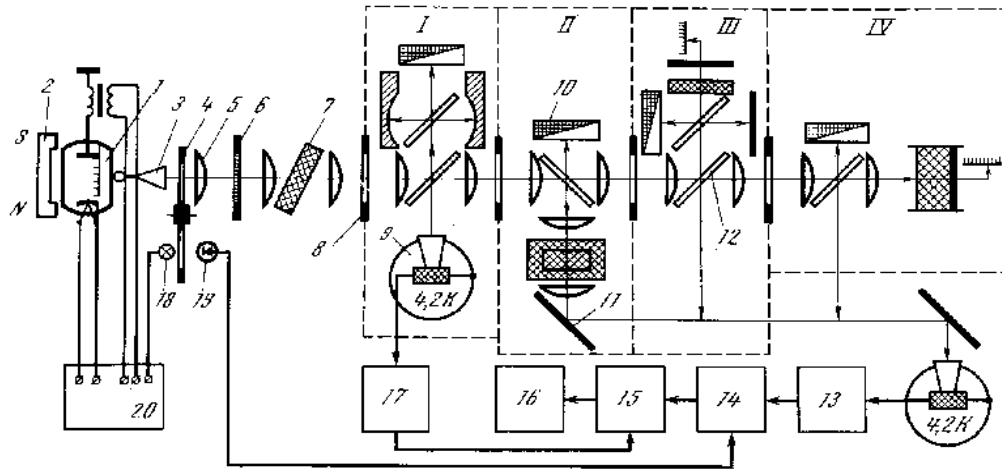


Fig. 1

Here I - resonator for low loss material properties measurement, II - transmission measuring circuit, III - Michelson or Max - Zender interferometer, IV - reflectometer. 1 - BWO, 2 - magnet, 3 - horn, 4 - modulator, 5 - lens, 6 - polarizer, 7 - attenuator, 8 - iris, 9 - receiver, 10 - absorber, 11 - mirror, 12 - beam splitter, 13 - amplifier, 14 - synchronous detector, 15 - digital voltmeter, 16 - storage unit, 17 - voltmeter, 18 - light source, 19 - LED, 20 - power supply.

Measurements of the  $\vec{\mu}$  tensor components in ferrites were performed with the aid of an interferometer with circularly polarized waves which are the natural waves of the longitudinally magnetized material. The diagonal, ( $\vec{\mu}$ ) and non-diagonal ( $\mu_\alpha$ ) components of  $\vec{\mu}$  are related to the  $\varphi_{r,l}$  of right and left circularly polarized waves. The most accurate method of determination  $\mu_\alpha$  is by measuring  $\Delta\varphi = \varphi_r - \varphi_l$  by reversing the direction of the permanent longitudinally magnetic field. Alternately,  $\mu_\alpha$  was determined by measuring the Faraday rotation angle  $\theta$  of a linearly polarized wave:  $\theta = 2(\varphi_r - \varphi_l)$ .

Of special interest are the measurements birefringence and dichroism of crystals  $\Delta n_{i,j}$ ,  $\Delta \kappa_{i,j}$  ( $i, j=x, y, z$  and  $i \neq j$ ). At small  $\Delta n_{i,j}$ , and  $\Delta \kappa_{i,j}$  when  $n_i$ ,  $n_j$ ,  $\kappa_i$  and  $\kappa_j$  cannot be determined individually with adequate accuracy rotating a sample between polarizers and frequency sweeping were used. There are two adjacent frequencies  $\nu_{1,2} = 1/\lambda_{1,2}$  corresponding to a circular polarization waves at the crystal output. These frequencies and the angle of axis  $i$  rotation relative to the electrical field vector of the incident wave, and reflection coefficients  $r_i$ ,  $r_j$  allow us to find  $\Delta n_{i,j}$ , and  $\Delta \kappa_{i,j}$ .

The experimental set up for very low-loss specimens is based on a hemispherical Fabry-Perot resonators (see I, Fig. 1). The quality factor of these resonators is more then 50000.

### 3. Some comments concerning low-loss materials

Low-loss materials are non-polar. Their absorption features are of four types [19,21].

1. Sharp single-phonon absorption due to lattice vibrations in the crystalline regions. Usually the frequencies of such vibrations have been observed below  $\lambda \leq 0.5$  mm.
2. A multi-phonon absorption continuum arising from the vibrational density of states in the crystalline regions and extending into the MM and microwave region.
3. A continuum absorption arising from vibration in the amorphous region. This absorption may extend into MM and microwave region too.
4. An absorption due to impurities and degradation products.

Practically at MM and SMM wavelengths the dielectric losses in non-polar materials both lattice losses and losses due to impurities, charge carriers plays a large role.

At a fixed temperature the theoretical limit of the lattice losses in a solid state dielectrics in the MM wavelength range is determined by a multi-phonon mechanism primarily by two-phonon losses in corresponding ideal crystal. The crystals with low coefficient of heat expansion and high lattice heat conduction, high Debye frequency and sound velocity, high crystal symmetry must have very low losses. The crystals with diamond structure (diamond, silicon) satisfy these criteria to the highest degree. Crystals with Zinc blende and wurtzite structure, sapphire, polyethylene, and some others belong to them too.

Practically losses depend also on material processing, material purity and homogeneity.

#### 4. Material properties

Non-polar liquids. Non-polar liquids are of interest as low-loss substances for MM region applications (immersion and cooling liquids, substances under test in aquametry, etc.)

The use of non-reflecting lens beam waveguide measurement techniques improves the accuracy of determination  $n$  and  $k$  of low-loss liquids because of lesser errors associated with wave beam defocusing effects and standing waves in the measuring channel [14,22]. Some  $n$  and  $\tan\delta$  values for the most transparent non-polar liquids are listed in Table 1. Table 1 presents characteristics of chemical pure liquids (with the exception of crude oil). Practically all these liquids have  $\tan\delta \sim 10^{-4} \div 10^{-3}$  in centimetre wavelength region. At MM waves fast increase of not only absorption  $\alpha$  (dB/cm) but also increase of  $\tan\delta$  is observed. This increase is connected with so named induced dipoles in non-polar liquids [23]. Additional absorption in practically used non-polar liquids arises also due to their humidity. Water is a very lossy liquid at MM and SMM wavelengths

Table 1 (T=18°±20°C)

liquid	$n \pm 0.3\%$	$\tan\delta \times 10^3 \pm 10\%$	$\lambda$ , mm	note
cyklohexane	1.424	0.50	0.63	
octane	1.396	0.74	0.63	
decane	1.407	0.83	0.63	
nonane	1.405	0.93	0.63	
carbon tetrachloride	1.490	3.5	1.0	[23]
cysdecalin	1.474	4.2	1.0	[23]

transdecalin	1.461	0.6	1.0	[23]
benzene	1.510	5.2	1.0	[23]
pentane	1.380	1.9	1.2	
toluene	1.510	5.4	1.2	
1,4 dioxan	1.343	5.0	1.0	
fluorinert cooling liquid FC-43	1.380	1.3	1.0	[24]
CS <sub>2</sub>	1.343	3.0	1.0	[22]
crude oil	1.470-1.570	0.8-1.4	2.0	

Table 2 presents  $\epsilon_1$ ,  $\epsilon_2$  and  $\alpha$  for liquid water [8].

Table 2

$\lambda$ , mm	T, grad C	$\epsilon_1$	$\epsilon_2$	$\alpha$ , dB/mm
2	20	6.28	8.81	41.1
	30	6.86	10.5	46.0
4	20	9.10	16.0	29.5
	30	10.9	18.9	31.9
8	20	18.1	28.0	18.8
	30	23.5	30.8	18.9

Therefore even very small water content in non-polar liquids evokes additional losses, e.g. at  $\lambda=2$  mm the dependence  $\alpha(W)$ , where  $W$  is the concentration of water by volume, for crude oil is about 2 dB/cm per 1% water [8]. Table 1 gives for dry crude oil  $\alpha(0) \approx 0.2$  dB/cm.

Low-loss solid state dielectrics. These dielectrics (polymers, ceramics, crystals, composite materials) have in MM and SMM regions  $n$  from 1.05 to 10 and  $\tan\delta < 10^{-2}$ . Some results of polymers investigations are summarized in Table 3. The measurements of  $n$  and  $\tan\delta$  was carried out at approximately 20°C on polymers the highest commercially purity.

Table 3

material	$n \pm 0.5\%$	$\tan\delta \times 10^3 \pm 10\%$	$\lambda$ , mm	note
polytetrafluorethylene (PTFE), unsintered	1.25-1.44 1.42	0.23-0.26 0.05	0.63	density 1.3-2.2 g/cm <sup>3</sup>
PTFE, sintered, teflon	1.43	0.7	1.3	
polyethylene	1.52	0.6	0.63	
polypropylene	1.51	0.6	0.63	
TPX	1.45	0.7	1.0	

Rexolite	1.59	1.0	2.2	
Duroid	1.48	1.0	3.0	
polystyrene	1.59	2.9	1.0	
teflon-4MB	1.42	1.2	0.63	

Unsintered PTFE features the lowest losses. Investigations of PTFE physical and chemical properties [14] indicate that unsintered PTFE has a higher crystallinity than sintered PTFE (teflon), approximately 95% and 60% respectively). The observed difference in losses can be ascribed to the absorption in the polymer's amorphous phase analogous to the absorption in non-polar liquids.

The refractive index of PTFE is related to the material density  $d$  as

$$n = 1 + 0.196 d,$$

where  $d$  is in  $\text{g/cm}^3$ ,  $1.15 < n < 1.3$ .

The value of  $d$  can be varied either by changing the pressure at which the PTFE powder is moulded, or else by making the PTFE porous.

It should be stressed that for accurate measurements of  $n$  and  $\alpha$  for materials with  $n = 1.3 \div 1.5$  we have used low-loss immersion liquids featuring practically identical refractive indices (see Table 1).

The dependence of  $\alpha$  on frequency  $\nu = 1/\lambda = 2 \div 50 \text{ cm}^{-1}$  for low-loss polymers is in a good accordance with law [2]

$$\alpha_{Nepers} = 8.10^{-4} \nu + 1.4.10^{-4} \nu^2.$$

Tables 4 and 5 present the characteristics of the most interesting low-loss materials with  $n > 1.6$ : ceramics and glasses (Table 4) and crystals (Table 5).

It should be stressed that values of  $n$  in Table 4 belong to the concrete specimens under test and these values may change to material density and technology of preparation.

Table 4 (T=18°±20°C)

material	$n \pm 0.5\%$	$\tan \delta \times 10^3 \pm 10\%$	$\lambda, \text{ mm}$	note
BN	1.73	1.0	0.95	$d=1.45 \text{ g/cm}^3$
BN	2.07	0.64	1.22	$d=1.9 \text{ g/cm}^3$
SiO <sub>2</sub>	1.92	2.0. 0.67	0.65 0.26	
MgF <sub>2</sub>	2.16	0.9 0.6	1.0 1.2	
BeO	2.63	1.2	1.0	
AlN	2.88	4.0 7.0	3.0 1.4	
Al <sub>2</sub> O <sub>3</sub>	3.10	0.26 1.3	2.18 0.95	
BaTiO <sub>3</sub> +TiO <sub>2</sub> (50%)	6.1	1.1	0.95	
SrTiO <sub>3</sub>	15.1	35	1.0	
TiO <sub>2</sub>	9.4	10	1.4	
MgAl <sub>2</sub> O <sub>4</sub>	3.14 2.90	1.5 0.6	1.0 2.5	[16]
La <sub>7/12</sub> Na <sub>1/4</sub> TiO <sub>3</sub>	9.3	12	2.2	
ZnS	2.89	1.9	3.0	

ZnSe	3.02	2.2	3.0	
Na <sub>2</sub> O 6Al <sub>2</sub> O <sub>3</sub>	3.6	2.0	2.3	
SiO <sub>2</sub> (fused)	1.95	1.4	0.85	

In frequency range 100÷400 GHz  $\tan\delta$  for materials presented in Table 4 increases depending on  $f$  as  $f'$  ( $\gamma = 0.5\div 1.0$ ).

Table 5 shows the characteristics of the low-loss crystals.

The results presented in Table 5 show that the lowest losses among solid state materials at room temperature have high resistive silicon and artificial (made using the method of chemical vapor deposition, CVD, [31,32]) diamond. This result is in accordance with theoretical predictions [20,21].

The best results were achieved in the gold-doped silicon ( $\tan\delta = 3\times 10^{-6}$  at  $\lambda \approx 2$  mm). These losses are due to free carriers of charges, because according to [28, 31] the lower limit of loss tangent value due to intrinsic lattice loss in silicon is equal to about  $3\times 10^{-8}$ . For Ge this value is equal  $2\times 10^{-7}$ . The same estimation of the lower limit of lattice loss tangent values have been obtained for the diamond, GaAs, GaP, and InP respectively:  $\tan\delta \approx 10^{-9}$ ,  $10^{-4}$ ,  $2.5\times 10^{-4}$  [31]. In GaAs, GaP and InP the experimental values of  $\tan\delta$  are practically equal to the theoretical predictions for lattice loss. The observed losses in diamond can be explained by the inclusions of non-diamond phases containing amorphous carbon and nanographite.

Table 5 (T=18°÷20°C)

material	$n \pm 0.5\%$	$\tan\delta \times 10^3 \pm 10\%$	$\lambda$ , mm	note
SiO <sub>2</sub>	$n_e=2.14$	0.55	2.18	
	$n_o=2.10$	0.56	2.18	
		0.4	0.66	
Al <sub>2</sub> O <sub>3</sub>	$n_e=3.40$	0.15	2.14	[26]
	$n_o=3.07$	0.25	2.14	[26]
		0.60	0.61	[27]
GGG	3.51	1.3	1.1	[30]
		0.7	2.18	
LiNbO <sub>3</sub>	$n_e=6.7$	6.0	1.0	
		2.0	2.2	
	$n_o=5.1$	4.7	1.0	
		1.5	2.2	
LiTaO <sub>3</sub>	$n_e=6.30$	10	1.2	
	$n_o=6.45$	7.0	1.2	
TGS	$n_y=2.31$	8.4	1.1	
	$n_x=2.91$	15	0.9	
	$n_z=2.74$	620	2.0	
Ge ( $\rho=400$ Om.cm)	3.15	3.3	2.0	
GaAs( $\rho=10^8$ Om.cm)	3.61	0.2	2.24	
InP ( $\rho=10^7$ Om.cm)	3.55	0.2	2.16	[28]
GaP ( $\rho=10^8$ Om.cm)	3.34	0.1	2.16	[28]
Si ( $\rho=25$ kOm.cm)	3.42	0.08	1.4	[27]
Si ( $\rho=40$ kOm.cm)	3.42	0.025	2.0	
Si ( $\rho=150$ kOm.cm)	3.42	0.003	2.0	[29]

diamond, CVD	2.40	0.05	2.1	[32]
diamond, CVD	2.39	0.008	1.95	[31]

The dependence of  $\tan\delta$  for diamond and Si on frequency can be approximated by  $f^{-1}$  law due to conductivity of these crystals, whereas  $\tan\delta$  for InP, GaAs, GaP practically invariable in frequency range 100÷500 GHz.

In Table 5 also some low-loss anisotropic crystals properties are presented.

These crystals are of interest for MM and SMM polarizers and polarization convertors [33]. So TGS crystal is a very good polarizer for short MM and SMM region due to very large dichroism in this material.

The photoconductivity in low-loss silicon and germanium allows to create MM-wave optically controlled attenuators and modulators, e.g. for dielectric waveguides [34].

Low-loss ferrites. Ferrites are very important materials for non-reciprocal devices. Here we present some results of investigation of over 50 types of polycrystal ferrites made by our group [35]. Table 6 shows characteristics of the ferrites with the lowest losses among each type of ferrites.

Table 6 (T=18°±20°C)

material	$n \pm 0.3\%$	$\tan\delta \times 10^3 \pm 10\%$	$\lambda$ , mm	Saturation magnetization, Gauss
YIG ferrites	9.68-3.82	0.75	2.16	<2200
		1.4	0.85	
		2.5	0.6	
LiZn ferrites	3.85-4.0	1.2	2.16	<4800
NiZn ferrites	3.54-3.95	1.0	2.16	<5200
		1.5	1.10	
		3.5	0.60	

The best ferrites have the Faraday rotation angle  $\theta$  about 8÷11 grad/mm for YIG ferrites and 10÷16 grad/mm for NiZn ferrites.

Low-loss composite materials. Now there are many low-loss materials for microwave and MM wave region are available. These materials usually based on PTFE or polyethylene as a matrix and Al<sub>2</sub>O<sub>3</sub>, MgO, TiO<sub>2</sub> as a admixture. Table 7 shows the characteristics of composite material based on PTFE at frequency 480 GHz depending on concentration of admixture by weight,  $W$ , %.

Table 7

admixture	$n \pm 0.5\%$		$\tan\delta \times 10^3 \pm 20\%$	
	$W=5\%$	$W=10\%$	$W=5\%$	$W=10\%$
MgO	1.45	1.48	3.1	7.6
Al <sub>2</sub> O <sub>3</sub>	1.48	1.51	9.1	17.0
TiO <sub>2</sub>	1.58	1.68	10	23

Here dimensions of admixture powder grains are smaller than 0.1 mm.

In porous PTEF losses are lower than 0.5 dB/mm up to  $\lambda=0,6$  mm if grains are smaller than 0.25 mm. Composite materials with  $n>1,7$  where obtained by doping

polystyrene with high-permittivity powders of  $\text{TiO}_2$  [36]. The permittivity of such composite materials is good described by Lichtenecker expression

$$\ln \varepsilon^* = \varphi_m \ln \varepsilon_m + \varphi_a \varepsilon_a,$$

where  $\varepsilon_m$ ,  $\varepsilon_a$  and  $\varphi_m$ ,  $\varphi_a$  are polystyrene and  $\text{TiO}_2$  permittivities and concentrations by volume respectively. Experimentally it was achieved at frequencies near 70 GHz  $\varepsilon=4$  and  $\tan \delta = 1.4 \times 10^{-3}$  for  $\varphi_a = 10\%$  and  $\varepsilon_a = 11$  and  $\tan \delta = 1.3 \times 10^{-2}$  for  $\varphi_a = 40\%$ .

The next practically important composite materials are glass plastics for antenna covers.

Some results of investigation of many types of glass cloths and resins used for preparing glass plastics were presented in [37].

So cloths based on non-alkaline and quartz glass fibers have  $\varepsilon = 3.6 \div 6.3$  and  $\tan \delta$  from  $(0.2 \div 2.0) \times 10^{-3}$  at frequencies 30 ÷ 35 GHz to  $(0.3 \div 4.0) \times 10^{-3}$  at frequencies 300 ÷ 350 GHz.

Resins used for glass plastics (epoxy and silicon-bounded types) have  $\varepsilon = 2.8 \div 3.1$  and  $\tan \delta$  from  $(1.2 \div 2.5) \times 10^{-2}$  at frequencies 30 ÷ 35 GHz to  $(2.5 \div 3.5) \times 10^{-2}$  at frequencies 300 ÷ 350 GHz. The permittivity  $\varepsilon_1$  for each material is practically invariable at frequencies 10 ÷ 350 GHz.

Antenna covers materials based on porous  $\text{SiO}_2$  and  $\text{Al}_2\text{O}_3$  have  $\varepsilon = 1.15 \div 3.8$  and  $\tan \delta$  from  $(1 \div 5) \times 10^{-3}$  at frequencies 150 ÷ 300 GHz.

For some applications it is of interest composite dielectrics with dispersion  $n$  connected not with material properties but with dimensions of insertions into composite material matrix. For example the artificial dielectric [38] consisting of teflon with cylindrical periodical holes (hole diameter  $d = 2.5$  mm, grating period 5 mm) has in the interval of  $\lambda$  2.5 mm  $> \lambda > 4$  mm  $n$  increasing from 1.36 to 1.4 with two resonant regions where  $n_{\min} = 1.31$  and  $n_{\max} = 1.39$  and  $\tan \delta \approx 3 \times 10^{-3}$  instead of  $\tan \delta < 10^{-3}$  at the other frequencies.

Natural, building and common use materials. These materials are of great interest for communications and traffic applications as well as for instruments for non-destructive test of materials, manufactured articles and environment [38, 30].

Table 8 shows building and natural materials and substances properties.

Table 8 ( $\lambda = 2$  mm,  $T = 20^\circ\text{C}$ )

material	$n \pm 1\%$	$\tan \delta \times 10^2 \pm 10\%$	$\rho$ , g/cm <sup>3</sup>	note
brick (red)	1.78	3.5	1.5	
brick (silic.)	1.82	4.2	1.8	
concrete	2.40	5.5	1.7	
ashalt	1.50	8.0	1.3	
sand	1.55	2.5	1.8	
soil	1.60	3.8	1.5	18 <sup>0</sup>
	1.60	2.5	1.5	-39 <sup>0</sup>
snow	1.12	0.65	0.23	-36 <sup>0</sup>
		2.6	0.23	-1 <sup>0</sup>
pine tree wood	1.4	3.4/2.0	0.5	
glass, window	1.45	5.0		
organic glass	1.60	1.5		
marble	1.50	1.0		
ebonite	1.67	1.1		$\lambda = 0.6$ mm

cardboard	1.80	6.0		
cautchuck	1.66	30		
glues	1.57-1.72	1.0-2.0		$\lambda=1.7$ mm
phenolone	1.8	3.9		$\lambda=1.7$ mm
polymyde	1.66	1.8		$\lambda=1.7$ mm
veneer	1.5	10		$l=7.6$ mm
plaster	1.7	0.7		

Here less value of  $\tan\delta$  for pine tree wood corresponds to electrical field perpendicular to wood fibers. Water content in wood materials is less than 8%.

In Table 9 clothes materials are presented. Here  $|t|^2$  and  $|r|^2$  are power transmission and reflection coefficients,  $n_{eff}$  - effective refractive index for materials  $n_{eff}=\lambda \arg t / 2\pi d$ , where  $d$  - material thickness.

Table 9 ( $\lambda=1.6$  mm)

material	$ t ^2, \%$	$ r ^2, \%$	$n_{eff}-1$	$l, \text{mm}$
cloths for tents	82-94	$\leq 1.3$	0.2- 0.3	0.3 - 0.5
cloths for coat, wool	77- 84	$\leq 3.0$	0.1- 0.2	2- 4
cloths for suits, wool	85- 98	$\leq 1.0$	0.2- 0.3	0.5- 1.0
silk	89- 93	$\leq 1.0$	0.28- 0.35	0.15- 0.25
leather, natural	79- 85	$\leq 5.0$	0.22- 0.28	0.9- 1.5
leather, artificial	75- 89	$\leq 5.0$	0.22- 0.28	0.7- 0.8
fur, artificial	75- 89	$\leq 3.0$	0.04- 0.07	4.0- 12
astrakhan	71	$\leq 1.0$	0.14	35
cloths for shirts	92- 95	$\leq 5.0$	0.18- 0.23	0.2- 0.3

Frequency dependence of  $n$  for materials in Tables 8 and 9 is weak but  $\tan\delta$  and  $|t|^2$  vary considerably as frequency changes. For example  $\tan\delta$  of brick at  $\lambda=6\div 7$  mm is only  $10^{-2}$ ,  $\tan\delta$  of concrete is  $6\times 10^{-3}$ . Transparency of clothes decreases as frequency increases from  $98\div 75\%$  at  $\lambda=1.6$  mm to  $76\div 30\%$  at  $\lambda=0.5$  mm.

All materials in Table 9 have strong dependence of frequency on moisture for practically dry materials (see Table 9).  $|t|^2=98\div 75\%$  in temperature interval  $T=5^\circ\div 20^\circ\text{C}$ , for moisture  $W=28\%$   $|t|^2=80\div 25\%$ .

## 5. Conclusion

This short review shows modern situation with low-loss materials for applications in MM and SMM wavelengths region. This situation is continuously changed: material properties becomes better, new, first of all artificial, materials are created, new information concerning frequency, temperature dependence of  $n$  and  $\tan\delta$  is appeared as well as information how these values are changed under different external effects..

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