

## **SILICON AS AN ADVANCED WINDOW MATERIAL FOR HIGH POWER GYROTRONS**

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### **ABSTRACT**

The absorptivity of high-purity grades of silicon (Si) and its reduction by subsequent doping procedures are investigated.

The dielectric data are given for the wide range of frequencies (30 - 330 GHz) and temperatures (30 - 330 K) in comparison with the data set for sapphire. The advanced material performance in high power window applications is discussed taking into account both dielectric properties of the optimized silicon grades and thermal conductivity.

## INTRODUCTION

Gyrotrons for plasma heating are one of the greatest challenges in the present development of millimeter wave generators as they require the output power about 1 MW in the frequency range 100-200 GHz in CW operation regime [1,2].

The cardinal problem for reaching the CW regime is caused by the gyrotron output window, because up to present there are no windows available that tolerate the transmission of megawatt power levels.

In the most advanced window concept for CW gyrotrons a cryogenically-cooled sapphire window is being developed [3,4] because only at cryogenic temperatures, sapphire reaches the required levels of high thermal conductivity and low dielectric absorption. But up to now the upper limit for sapphire is 1 MW at 140 GHz [3,5].

At the same time for the ITER project for heating of fusion plasmas required the gyrotrons with the frequency 170 GHz, where the power transmission capability of sapphire windows is reduced by about a factor of 1.4.

However, we can denote the set of homopolar substances which combine the lower absorption losses and the higher thermal conductivity.

Substances for which these properties are expected are the first of all elements of group IV of the periodic system. But also compounds with a diamond-like crystal structure of the group IV elements and even of the III-V group elements have these potentials inherently. Their outstanding thermophysical and millimeter wave properties as compared to all other dielectric elements and compounds are due to their crystal structure with high symmetry and the weakness or absence of (first order) dipole moments in the collective lattice vibrations.

Preliminary estimates show that the lattice contribution to the dielectric loss tangent ( $\text{tg}\delta$ ) ranges in diamond, Silicon (Si) and Germanium (Ge) between  $10^{-9}$  to  $10^{-7}$  [6].

The contribution from thermally excited charge carriers over the band gap (intrinsic conductivity at 290 K) increases the total limit in  $\text{tg}\delta$  to  $\sim 4 \cdot 10^{-6}$  in Si and  $\sim 2 \cdot 10^{-2}$  in Ge.

The losses in single crystal diamond by intrinsic conductivity are negligible.

In the first experimental investigations it was found that at room temperature commercial silicon grades range among the low loss materials which have been inspected so far at MM and especially at SubMM wavelengths [7,8,9,10].

Problems for their application were identified by their marked loss minimum around room temperature which implied a marked instability under thermal loads which could not be compensated by cryogenic cooling.

The present studies are aimed at identifying more promising silicon grades by selecting of the compensated industrial silicon and by modifying them by special doping procedures for the reducing loss level [11].

#### EQUATIONS

As discussed above the lattice absorption contributes negligibly to the loss tangent, therefore  $\text{tg}\delta$  can be expressed in the form [10]:

$$\text{tg}\delta = 4\pi e^2 N / m' \omega \nu \epsilon = 4\pi \mu e N / \omega \epsilon = 2N \mu e / f \epsilon \quad \text{with:}$$

$$N = 2.8 \cdot 10^{-13} \cdot f \cdot \text{tg}\delta, \quad f[\text{GHz}], \quad \epsilon = 11.7$$

for n-type of Si

$$N = 8.1 \cdot 10^{-13} \cdot f \cdot \text{tg}\delta, \quad f[\text{GHz}], \quad \epsilon = 11.7$$

for p-type of Si

where:

$N$  is the free charge carrier concentration;  
 $\mu$  is the mobility, ( $\sim 500 \text{ cm}^2/\text{V}\cdot\text{sec}$  for hole,  
 $\sim 1450 \text{ cm}^2/\text{V}\cdot\text{sec}$  for electron, at 300K).

$e$  is the electron charge =  $4.8 \cdot 10^{-10}$ .

$\nu$  is the frequency of collision,  
 ( $\sim 10^{13}$  Hz at 300K,  $\sim 10^{12}$  Hz at 80K).

$m'$  is the effective mass.

( $10^{-27}$  g for hole,  $0.33 \cdot 10^{-27}$  g for electron)

## INSTALLATIONS

For the dielectric parameters investigation two installations based on high-Q Fabry-Perot resonators were used.

One installation which was used to measure the loss tangent and refractive index at room temperature as the frequency function was based on a symmetrical arrangement of the spherical mirrors (with radius of curvature ( $R$ ) = 240 mm) forming a resonator of the length = 300- 420 mm. The sample was placed in the center. The measurements were performed at the frequencies for which the sample thickness was resonant. It means that the number of half-wavelengths ( $m$ ) contained in the specimen is integer [12].

The other installation was used to measure the dielectric parameters as the temperature function around 145 GHz. It was based on a (quasi-) hemispherical geometry composed of a spherical mirror ( $R = 122$  mm) and a plane mirror separated by  $L = 114$  mm. The sample was placed on the plane mirror [13] and investigated at arbitrary thickness (non-integer  $m$ ).

The dielectric parameter studies were flanked by the following investigations:

The free carrier concentration (by Hall's

method) and DC resistivity ( $\rho$ ) were measured at room temperature and compared with results from the C-V [14] and PTIS (Photo Thermal Ionization Spectroscopy [15]) method.

Deep level impurities were determined by the DLTS (Deep Level Transient Spectroscopy [16]) technique.

Independently, the free carrier concentration was calculated from loss tangent measurements [10]. It is worth noting that apart from aforementioned methods the concentration determination was the most convenient and not contradictory exactly by this way. The results normalized to the frequency 150 GHz are given in the Table.

Table. The characteristics of the Silicon grades investigated in this study.

	LR-Si initial	dLR-Si doped	HP-Si	HR-Si initial	eHR-Si e-irr.
Hall's measurements					
conductiv.	n	p	p	n	n
$N/10^{11} \text{ cm}^{-3}$	30		4.5	4	
$\rho$ , kOhm·cm	1.3		35	11.4	
$\text{tg}\delta \times 10^5$					
290 K	102	2	2.1	6	2.4
150-180 K	300	0.6	8	15	1.3
calculated from $\text{tg}\delta$ measurements [10] (290 K)					
$N/10^{11} \text{ cm}^{-3}$	43	2.43	2.55	2.52	1.01
$\rho$ , kOhm·cm	1	51	48	17	42

## MATERIALS CHARACTERISTICS

The silicon crystals used for this study were all grown by the floating-zone (FZ) technique.

The sample LR-Si was n-type "Low Resist" Si. Its conductivity type was caused by phosphorus as the major impurity according to contact free PTIS measurements [15]. The compensating impurity was boron. The samples contained an impurity center with the ionization energy  $E_i = 0.164$  eV and a concentration  $\sim 20\%$  of the free carrier concentration (according to DLTS results).

The sample dLR-Si was LR-Si doped with gold. The doping was produced by Au diffusion at the temperature of  $\sim 1100$  C. The Hall measurements indicated that the doping procedure resulted in the conversion of initial n-type Si into p-type.

It is known, that the Au produces in Si both acceptor ( $E_c - 0.54$  eV) and donor ( $E_v + 0.35$  eV) levels. The p-type conductivity of doped Si was connected just with the Au acceptor level [17].

The sample HP-Si was p-type "High Purity" Si. It was the most pure of all samples used in this work. Its conductivity type was caused by boron and compensating impurity was phosphorus.

The sample HR-Si was n-type "High Resist" Si from Wacker (Burghausen, Germany). Its conductivity type was caused by phosphorus as the major impurity. The compensating impurity was boron.

The free carrier concentration, according to C-V measurements, did not change from 300 K down to 77 K, which underlined the low concentration of deep impurities.

The sample eHR-Si was an electron irradiated n-type "High Resist" Si cut out of a big block purchased from Wacker. The electron irradiation was made at CIEMAT, Madrid, with 2 MeV electrons [18] to a total dose of  $7 \cdot 10^{15}$  Gy/s.

## RESULTS

The refractive index data for Silicon were obtained by the standard procedure of solving resonator equation for different types of Fabry-Perot resonators.

The refractive index varies from sample to sample between 3.416 to 3.423. At 290 K, the refractive index decreases practically linearly over inspected frequency range from 30 GHz to 330 GHz. For instance, in the HR-Si specimen it drops from 3.426 to 3.415. The permittivity variation down to cryogenic temperatures is consistent with previous data for HR-Si [11].

A general overview of the loss tangent data obtained at room temperature is given in Fig.1. It is clearly seen that  $\text{tg}\delta$  decreases with increasing frequency in all samples following well the  $f^{-1}$  law. The loss data obtained with two measuring systems agree within experimental accuracy. Different samples of the same material even when cut out of a different big block places showed individual losses varying within 30%. The same situation was found for the losses at different places of disk with a big diameter.

At room temperature all silicon samples have the losses at least by a factor of 5-10 lower (except LR-Si) than in sapphire around 145 GHz. At this frequency this attractive feature was further inspected at the cryogenic temperatures.

As shown in Fig.2, the  $\text{tg}\delta$  increases below room temperature for the HR and HP samples. A maximum is reached around 130 K. Below 50 K  $\text{tg}\delta$  strongly decreases with temperature decreasing.

The LR-Si sample follows similar curve, but it is shifted to a much higher level so, it falls out of the scale.

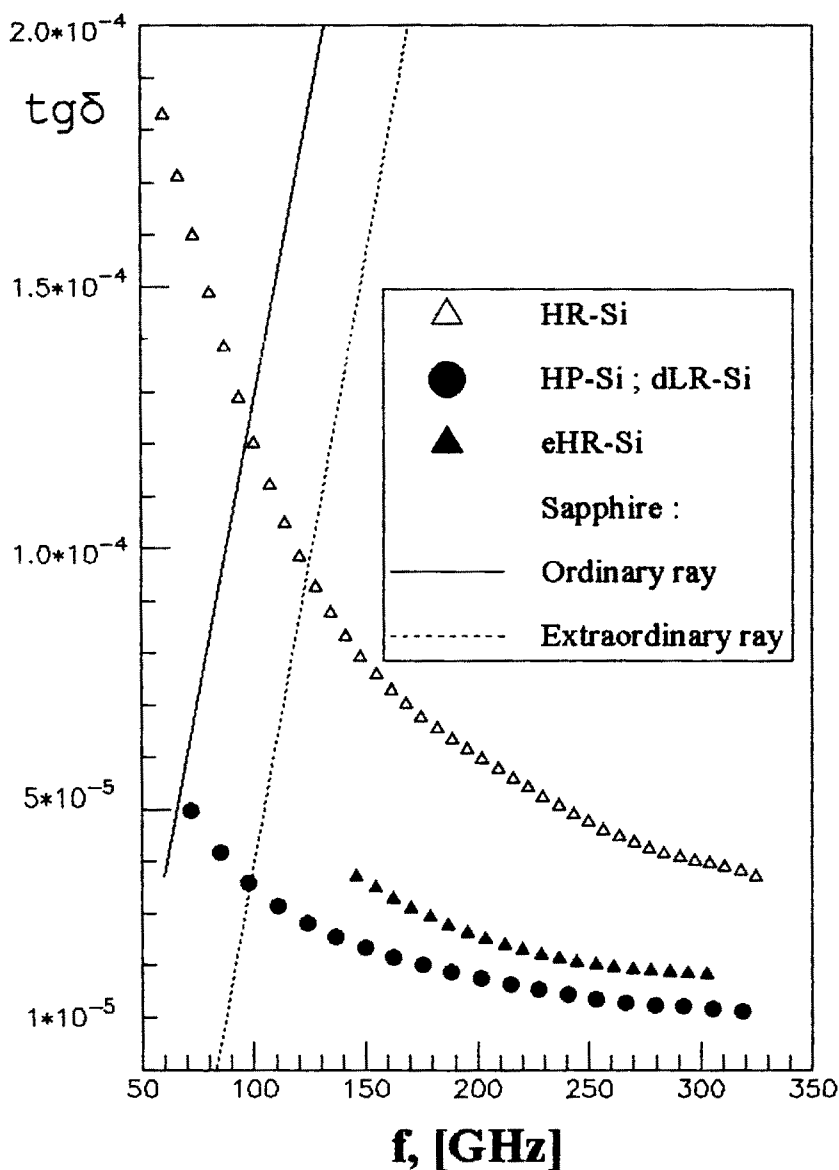


Fig.1 The frequency dependence of dielectric loss in selected Silicon materials compared to high quality Sapphire



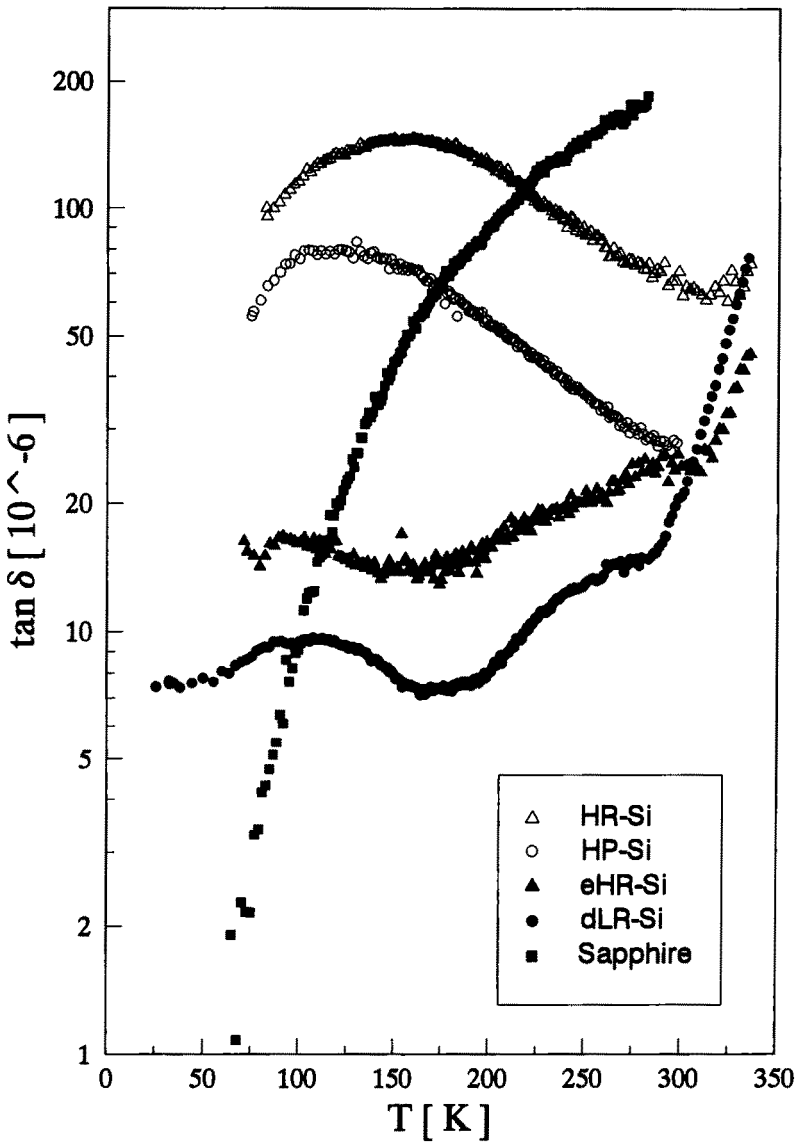


Fig. 2 The temperature dependence of dielectric loss at 145 GHz in selected Silicon materials compared to high quality Sapphire

The temperature function can be understood in terms of conductivity in Silicon compensated by shallow impurities. From 25 K to 50 K the shallow impurities are ionized and create free charge carriers increasing the loss strongly.

Above 130 K, the saturation of free carrier concentration produced by shallow impurities, take place and the  $\text{tg}\delta$  curve becomes dominated by the temperature function of carrier mobility which falls with increasing temperature. Above room temperatures the main loss mechanism arises from intrinsic charge carriers created by the ionization of Si atoms. ( $N \sim T^{1.5} \cdot \exp[-E_g/2kT]$ )

The temperature behavior of Au doped silicon is essentially more complicated. The consistent picture can be found in terms of the Au impurity levels in the Si [17].

The strong loss tangent drop with temperature decrease from 350 K to 200 K is connected with the freezing of Au level with  $E_i = 0.54$  eV.

Intermediate increasing of losses below 150 K can be caused by an enhancement of the mobility.

The final loss tangent drop at the lowest temperatures is connected with the freezing of shallow levels of Phosphorus with  $E_i = 0.045$  eV.

The  $\text{tg}\delta$  temperature dependence of electron irradiated Si shows the deep level presence with a lower activation energy in comparison to Au doped Si. With high probability it is related to the acceptor level with  $E_i = 0.16$  eV [17].

In total, the free charge carrier concentration could be reduced at room temperature by trapping process at defect centers (deep traps).

#### THERMAL MODELING OF HIGH POWER WINDOW

The principal difference of doped silicon in comparison to initial silicon occurs at low tem-

peratures: The low losses attained in the two Si grades permit to expect that doped Si windows will be able to work without using LN cooling. Edge cooling appears to be feasible with the low temperature refrigerators (possible coolant CHF<sub>3</sub> at 191 K, CF<sub>3</sub>Cl at 192 K) and may be with other devices even closer to room temperatures.

Apart from low  $\text{tg}\delta$  in this temperature range, as can be seen from Fig.3, Si has high thermal conductivity. This is in positive contrast to the thermal conductivity and losses for Sapphire which has acceptable parameters only at the low cryogenic temperature and at low frequency end of the MM wave range (Figures 1-3).

With the afore mentioned data base, the first technology exploratory calculations for the 1 MW CW transmission capability of the new silicon materials were made.

The calculations were based on the following design conditions:

Edge cooling by liquid nitrogen (80 K);  
Disk thickness = 3.1 mm. Diameter = 80 mm;  
Gaussian wave-beam structure; Frequency 170 GHz;  
In this case the CW dissipated power is ~ 370 W;  
The equilibrium temperature of window center is ~ 110 K, which is reached after 0.7 sec.

Even more striking are the results for the following conditions:

Disk thickness = 3.1 mm. Diameter = 80 mm;  
Cooling with edge temperature fixed to 200 K;  
Two peak flattened wave-beam structure; In this case, the CW dissipated power is ~ 620 W; The equilibrium temperature of center ~ 240 K, which is reached after 10 sec. Further increasing of edge temperature leads to the unstable regime.

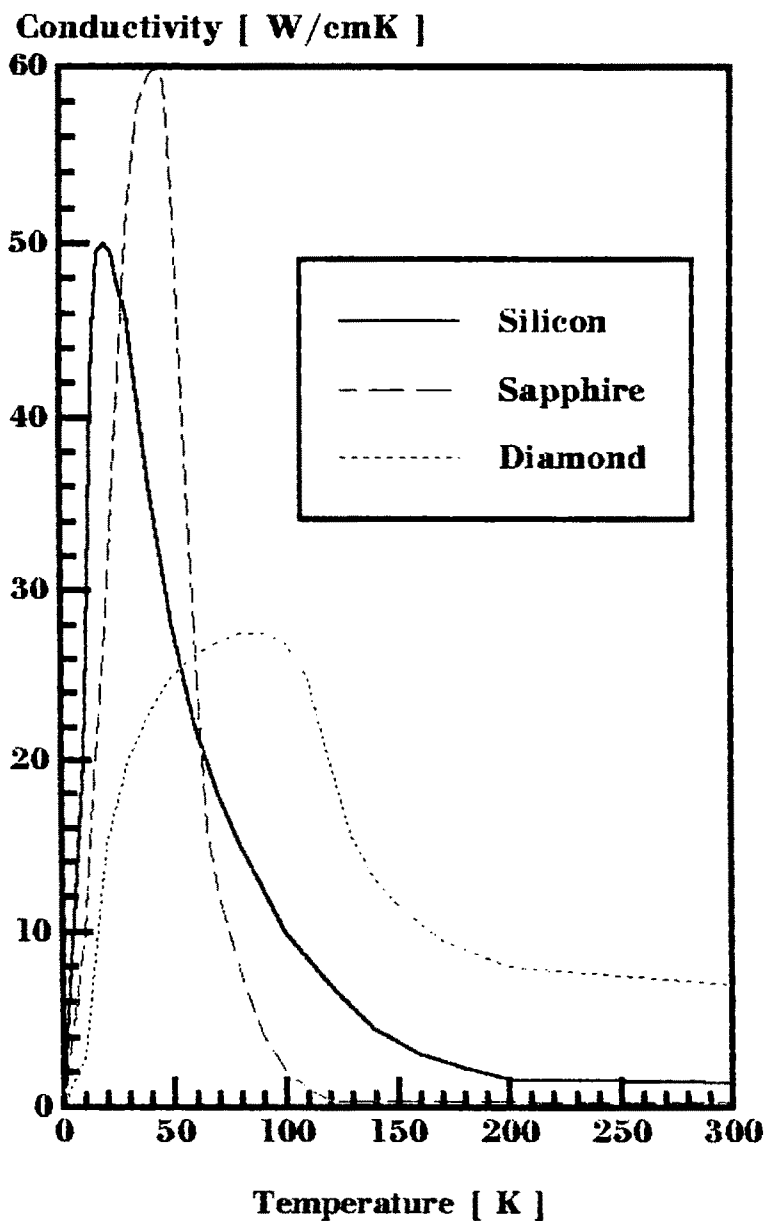


Fig.3 The thermal conductivity of Silicon compared to Sapphire and Diamond

In the light of these results and from view point of the materials development the task of constructing megawatt windows appears to have found a practical solution.

## CONCLUSION

The feasibility of relatively simple doping methods to reduce the free carrier concentration in commercial silicon grades was demonstrated.

By this method, the silicon disk with extremely low losses was produced which can work as a 1 MW gyrotron window in CW operation by edge cooling with low temperature refrigerators at temperatures near 200 K.

At the same time, there is a further chance to minimize the losses by technology improving of Au doping and electron irradiations and by this way to increase the cooling temperature.

The high-purity silicon with the same performance is not yet realized and will cost at least on two orders of magnitude more.

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## REFERENCE

1. V.A. Flyagin, A.L.Goldenberg, and V.E. Zapevalov. Advanced Russian Gyrotrons for Plasma Investigations. The 19th Int. Conf. on IR and MM Waves. Japan, 1994, pp. 77-78.

2. M.Q.Tran et al. Feasibility of EU Home Team on the Manufacture of a Gyrotron for ECRH on ITER. The 19th Int. Conf. on IR & MM Waves. Japan, 1994, pp. 67-68.

3. H.Haefner, E.Bojarsky, K.Heckert, P.Norajitra, H.Reiser. Liquid nitrogen cooled window for high frequency plasma heating Journal of Nuclear Materials, 212-215, 1994, pp. 1035-1038.

4. A.Kasugai, K.Yokokura, K.Satamoto, M.Tsuneoka, T.Yamamoto, T.Imai, Y.Saito, K.Ito, T.Yoshiyuki, K.Ebisawa. High power tests of the cryogenic window for millimeter waves. Dig. 19th. Int. Conf. on IR & MM Waves, Sendai (J), 1994, JSAP Cat. Nr. AP941228, pp. 295-6.

5. P.B.Sushilin, A.Sh.Fix, V.V.Parshin. Perspectives of increasing the gyrotron output window capacity. "Gyrotrons" In. of Apl. Ph. Sc. A. USSR, Gorky, 1989, pp. 181-194.

6. B.M.Garin. Russian Conf. Dielectric-93, St.Petersburg, 1993.

7. M.N.Afsar, H.Chi. Millimeter wave complex refractive index, complex dielectric permittivity and loss tangent of extra high purity and compensated silicon, Int. J. of IR & MM Waves, 15(7), 1994, pp. 1181-1188.

8. R.Heidinger, A.Kumlin. Frequency and temperature dependence of the MM-wave dielectric properties of silicon with high d.c.resistivity. Digest of 14th Int. Conf. on IR & MM Waves. Florida, 1990. VSPIE, V.1576, pp. 274-278.

9. V.V.Parshin. Dielectric materials for gyrotron output windows. Int. J. of IR & MM Waves. Vol.15, N.2. 1994, pp. 339-348.

10. V.V.Parshin, V.N.Shastin. The non contactive technique for investigations of high purity semiconductors in MM range of wavelengths. Int. Symp. "Physics & Engineering of MM and SubMM Waves" Kharkov, Ukraine. vol.3, 1994, pp.656-658.

11. R.Heidinger, A.Kumlin. The impact of extrinsic conductivity on the mm-wave dielectric loss in high resistivity silicon. Dig. 16th Int. Conf. of IR & MM Waves, Lausanne (CH), 1991, SPIE Vol. 1576, pp. 450-1.

12. Yu.A.Dryagin, V.V.Parshin. A method to measure dielectric parameters in 5-0,5 MM wavelength band. Int. J. of IR & MM Waves. 1992, Vol. 13, N.7, pp.1023-1032.

13. R.Heidinger, G.Link. Dielectric loss measurements between 25-300 K with a hemispherical Fabry-Perot resonator. Dig. 18th Int. Conf. of IR & MM Waves. Colchester (UK), 1993, SPIE V. 2104, pp. 64-65.

14. C.T.Sah, L.Forbes, L.L.Rosier, A.F.Tasch. Solid-St. Electron. 1970, Vol. 13, p. 759.

15. B.A.Andreev, V.B.Ikonnikov, E.B.Kozlov, T.M.Lifshits, V.B.Shmagin. Materials Science Forum, V.143-147. 1994, pp. 1365-1370.

16. D.V.Lang. J. Apply. Phys. V.45. N.7, 1974, pp. 3023-3032.

17. A.G.Milns. Deep impurities in semiconductors. New York, Wiley Interscience (1973).

18. J.Molla, A.Ibarra, R.Heidinger, E.R.Hodgson. Electron-irradiated silicon: An optimized material for gyrotron windows. J. of Nuclear Materials, 1994, in press.