

BROADBAND MEASUREMENTS OF DIELECTRIC PROPERTIES OF LOW-LOSS MATERIALS AT HIGH TEMPERATURES USING CIRCULAR CAVITY METHOD

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Abstract—We describe a broadband microwave test system that can measure dielectric properties of microwave low-loss materials at high temperatures using circular cavity method. The dielectric constants and loss tangents of samples at different temperatures were calculated from measured shifts of resonant frequencies and unloaded quality factors of the multimode cavity with and without sample. Detailed design and fabrication of the circular cavity capable of working at temperatures up to 1500°C are discussed. The measurement theory and new calculation method of the radius and length of the cavity at different temperatures are presented. The hardware system was built to measure dielectric properties at wide frequency band from 7 to 18 GHz and over a temperature range from room temperature to 1500°C. Measurement results of the dielectric properties of quartz samples are given and show a good agreement with the reference values.

1. INTRODUCTION

Microwave low-loss materials have been widely applied in microwave devices and circuits, communication devices, antenna windows, and so on [1, 2]. It is important to accurately characterize the dielectric properties of materials and their variation with temperature before using them, especially in antenna windows and high power microwave

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devices [3, 4]. The working temperature of antenna window may be over 1000°C when applied in space shuttles. It is a requirement of the antenna window to maintain a stable performance at different temperatures [5]. Therefore, knowledge of the dielectric properties of low-loss materials at high temperatures is necessary to the application of materials and also helpful to the research and development of materials. The direction of electric field in the materials during measurements should be the same with the one in their applications if the materials under test are anisotropic. The materials used in antenna windows are mainly discussed in this paper. Because the direction of electric field is parallel to the flat surface of antenna window, the direction of electric field in the chosen method should be parallel to the flat surface of the material.

Transmission/reflection method, and free space method have been generally used to measure electromagnetic properties of microwave materials at variable temperatures, and the temperatures can be over 1000°C [6–9]. Open-ended coaxial probe method is frequently used to measure complex permittivity at high temperatures due to the advantages of simple measurement process and broad frequency band [10–12]. Dielectric properties at variable temperature are also measured by using Short-circuited method which requires a tight contact between sample and waveguide surface [13, 14]. But while being used to measure low-loss materials these methods have higher measurement uncertainties compared with cavity resonance methods.

Quasi-optical resonator method has been applied to the measurement of dielectric properties at Ku band over the temperature range from room temperature to 1200°C [15]. Cavity perturbation method is reported to measure complex permittivity at temperatures as high as 2000°C [16]. Sample and cavity can be heated together, or only sample is heated to the required temperature and then inserted into the cavity [14, 16–18]. Circular cavity method and split-cylinder resonator are commonly preferred to measure complex permittivity at room temperature due to their low measurement uncertainty. Compared with circular cavity method, split-cylinder resonator requires bigger and thinner sample and more complicated theoretical analysis. Because the sample is inserted between the two halves of the cavity, it has no support under the sample and is easy to be out of shape during elevated temperature measurements. While applied in very high temperature measurements, it is also less convenient than circular cavity method. The methods have also been applied to high temperature measurement at millimeter band at temperatures near 200°C [19, 20] and at microwave band at the temperatures 1200°C [21]. There are also other resonant

cavity methods, such as reentrant cavity method and helix cavity method [22, 23]. Cavity resonance method can measure the dielectric properties at resonant frequencies by using different cavities. Each cavity has advantage and disadvantages. But, some of the cavities if working at temperatures above 1500°C are difficult to fabricate by using high temperature metal due to complexity of the cavity shapes and high tolerance requirement of the cavity.

Circular cavity method was selected in this paper for the high temperature measurement of dielectric properties for the following advantages. One advantage is its relatively low measurement error margin and high reproductivity. The second advantage is that the direction of electric field is parallel to the flat surface of the sample and there is no perpendicular electric field. The third advantage is that the cavity and sample are easier to fabricate compared with other cavity resonance methods. The measurement theory for circular cavity method described in the paper uses frequency-variation method and is different from the paper [21] using length-variation method. Another major advancement to the reported methods [19–21] is that the radius and length of the cavity at high temperatures, which are important to the accurate measurement of dielectric properties, are calculated using new calculation method. In addition, the length of the cavity was fixed during the high temperature measurement, and it's more convenient for measurements to be implemented. The detailed design and fabrication of cavity is discussed. Test system for high temperature dielectric property measurement was built, and measurement results of samples are presented. Because the paper optimized the dimension of the cavity, and the frequency range of the test system can cover 7–18 GHz which is wider than the circular cavity method described in the references [21, 24]. The highest temperature of the test system is 1500°C. The measurement ranges of the system are from 1.2 to 10 for the relative permittivity ϵ'_r , from 5×10^{-4} to 3×10^{-2} for the loss tangent $\tan \delta$. The measurement uncertainties are 2% for dielectric constant and 15% for loss tangent at room temperatures, and 4% for dielectric constant and 25% for loss tangent at high temperatures, respectively.

2. MEASUREMENT THEORY

There are two types of traditional methods to measure dielectric properties by using circular cavity. One is length-variation method, and the other is frequency-variation method. It makes the design of cavity working at high temperature more complex if we choose length-variation method due to the requirement for changing the cavity

length during the measurement process to keep the resonant frequency constant. Therefore, the frequency-variation method is used in the paper. The length of the cavity does not need to be adjusted during the whole process of measurement, and the dielectric constant and loss tangent are obtained from the measurements of the shifts of the resonant frequency and quality factor before and after the load of sample on the bottom endplate of the cavity shown in Figure 1. The cavity works at TE_{01n} modes. R and L are the radius and the resonant length of the circular cavity respectively, and d is the thickness of the sample.

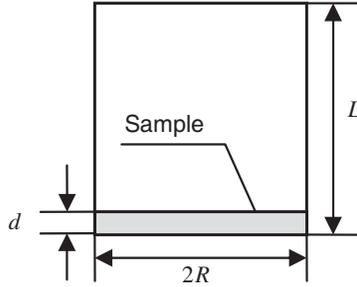


Figure 1. Circular cavity loaded with disk sample.

The dielectric constant ε'_r can be determined using Equation (1) [25].

$$\varepsilon'_r = \left(\frac{\lambda_0}{2\pi}\right)^2 \left[\left(\frac{X_{0m}}{R}\right)^2 + \beta_\varepsilon^2 \right] \quad (1)$$

where λ_0 is the wavelength in free space, X_{0m} is the root of Bessel function, R is the radius of the cavity, β_ε is the propagation phase constant in the sample, λ_0 and β_ε can be calculated from Equations (2) and (3) respectively.

$$\lambda_0 = \frac{c}{f_s} \quad (2)$$

$$\frac{\tan \beta_\varepsilon d}{\beta_\varepsilon} = \frac{\tan[\beta_0 (L - d)]}{\beta_0} \quad (3)$$

where c is the light speed in free space, f_s is the resonant frequency of the cavity loaded with sample, d is the thickness of the sample, L is the length of the cavity, β_0 is the propagation phase constant in free space and can be determined from Equation (4).

$$\beta_0 = \left[\left(\frac{2\pi f_0}{c}\right)^2 - \left(\frac{X_{0m}}{R}\right)^2 \right]^{1/2} \quad (4)$$

where f_0 is the resonant frequency of the empty cavity.

The loss tangent of the sample can be determined from Equation (5).

$$\tan \delta = \left(1 + \frac{u}{pv\varepsilon'_r} \right) \left[\frac{1}{Q_s} - \frac{1}{Q'_0} \right] \quad (5)$$

where

$$\frac{1}{Q'_0} = \frac{1}{Q_0} \left(\frac{f_0}{f_s} \right)^{\frac{5}{2}} \frac{\left[\left(\frac{X_{0m}}{R} \right)^2 (pv + u) + 2R(p\beta_\varepsilon^2 + \beta_0^2) \right]}{(pv\varepsilon'_r + u) \left[\left(\frac{X_{0m}}{R} \right)^2 \left(1 - \frac{2R}{L} \right) + \left(\frac{2\pi f_0}{c} \right) \frac{2R}{L} \right]} \quad (6)$$

$$p = \left\{ \frac{\sin[\beta_0(L-d)]}{\sin(\beta_\varepsilon d)} \right\}^2 \quad (7)$$

$$u = 2(L-d) - \frac{\sin[2\beta_0(L-d)]}{\beta_0} \quad (8)$$

$$\nu = 2d - \frac{\sin(2\beta_\varepsilon d)}{\beta_\varepsilon} \quad (9)$$

The main difference between the measurements at room temperature and at variable temperatures is that the diameter and length of the cavity is variable and dependent on the temperature. The paper uses fixed ratio of resonant length to diameter to obtain the geometry of the cavity at different temperatures. A is defined as

$$A = \frac{L}{R} \quad (10)$$

R can be determined from the geometrical measurement of the cavity at room temperature. The resonant length L at each mode can be calculated using the resonant frequency of the empty cavity. A is assumed as a constant at high temperature. However, A is different for each mode.

The diameter of the cavity at different high temperatures can be determined from the resonant frequency f_0 at one of the TE_{01n} modes according to Equation (11).

$$R = \frac{c}{2\pi \cdot f_0} \sqrt{\mu_{01}^2 + \left(\frac{n\pi}{A} \right)^2} \quad (11)$$

Here μ_{01} is the first root of zero order Bessel function.

The resonant length of the cavity at a certain temperature can be calculated from the following equation.

$$L = A \cdot R \quad (12)$$

Since the thickness of the sample d is also temperature dependent, it should be calculated at each measurement temperature according to the thermal expansion coefficient of the sample material.

3. CAVITY DESIGN

The cavity used in the paper is a multimode circular cavity, and TE_{01n} is chosen as the working mode due to its high quality factor. In order to reduce the number of the disturbing modes near the working mode, the dimension of the cavity must be carefully designed to make the disturbing modes away from the wanted modes as far as possible using resonant mode chart. According to Equation (13), the original value of cavity dimension covering frequency 7–18 GHz can be obtained by adjusting the values of radius R and length L .

$$f_0 = \frac{1}{2\pi\sqrt{\mu\varepsilon}} \sqrt{\left(\frac{\mu_{01}}{R}\right)^2 + \left(\frac{n\pi}{L}\right)^2} \quad (13)$$

Resonant frequency f_s of the cavity in presence of the sample can be calculated based on the assumption that the original values of dielectric constant and sample thickness are known and the original value of cavity dimension is obtained. Equation (1) can be transformed into Equation (14).

$$f_s = \frac{c}{2\pi\varepsilon'_r} \sqrt{\left(\frac{X_{0m}}{R}\right)^2 + \beta_\varepsilon^2} \quad (14)$$

We can obtain the frequency shift of every working mode with and without sample by introducing the minimum and maximum values of dielectric constant into Equation (14). Adjust the original value of cavity dimension repeatedly until the disturbing modes in the frequency shift range of all the working modes are greatly reduced. Since the cavity will work at high temperatures, the cavity dimension should be adjusted the third time after considering the thermal expansion coefficient of the material used to make the cavity. Finally, we optimized geometrical configuration of the cavity to obtain multimode resonances. The size of the empty cavity is $\Phi 52.02 \times 76.62$ mm at room temperature and $\Phi 52.48 \times 77.29$ mm at 1500°C .

In order to reduce the disturbance of TM_{11n} mode which is the degenerate mode of TE_{01n} , there is a small gap between the bottom endplate and cavity sidewall which does not affect the electromagnetic field of TE_{01n} modes. The schematic structure of the cavity is shown in Figure 2.

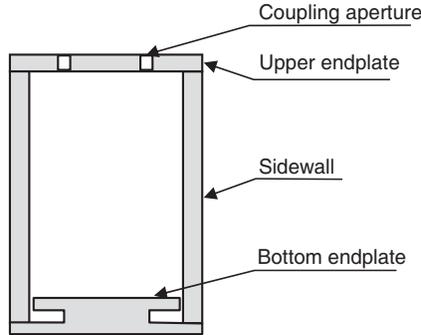


Figure 2. Cross section of the cavity.

It is through the two coupling apertures distributed symmetrically on the upper endplate of the cavity that the electromagnetic field is excited and sampled. The position of the coupling apertures are determined on the basis that microwave energy of the working mode must be coupled as much as possible and the energy of the disturbing mode as little as possible. The radius of the coupling aperture should not be too big to influence the distribution of the electromagnetic field in the cavity, and can be determined after building resonant cavity model in HFSS software. Because of the broad frequency range and many working modes, the amplitude of each working mode should be analyzed before selecting a proper value as the radius of the coupling aperture.

The material used to make cavity is required to work at high temperatures above 1500°C for a long time and have conductance high enough at high temperatures to ensure that the cavity can still resonate. There are several high temperature metal material candidates such as molybdenum, tungsten, platinum, rhodium, and iridium. Metal tungsten is not considered due to its high rigidity, difficult fabrication, and frangibility. Metal iridium is both expensive and difficult to fabricate. Alloy of platinum and rhodium is easy to fabricate and has high conductance, but it costs too much. Metal molybdenum has high conductance at high temperatures and can be fabricated easily, but has the disadvantage to be easily oxidized when exposed in air atmosphere. After comparison, metal molybdenum is

chosen to make the cavity. In order to prevent the cavity from being oxidized, vacuum furnace is applied to heat the sample and cavity, and the surface of the cavity is coated with metal iridium.

4. TEST SYSTEM

The high temperature test system is schematically shown in Figure 3. Adapters are used to connect coaxial cables with cooling waveguides which are cooled by water to ensure the adapters and coaxial cables work in room temperature. The wall of the heat insulation waveguide is thin to prevent significant thermal conduction from the hot cavity to the cooling waveguide. Each coupling aperture is connected with a high temperature waveguide which is made of metal molybdenum. The waveguides are double ridged waveguides for wide working frequency band. The vacuum furnace uses resistive heating rings at the endplates and sidewall of the cavity to ensure that the sample and the cavity are heated isothermal. Temperature is measured with a thermocouple under the sample through the bottom endplate. Computer controls temperature via the temperature controller by reading the thermocouple and varying the current to heat the cavity and sample. The computer also controls the network analyzer Agilent N5230 by setting sweep and reading data. Figure 4 is the photograph of the hardware test system.

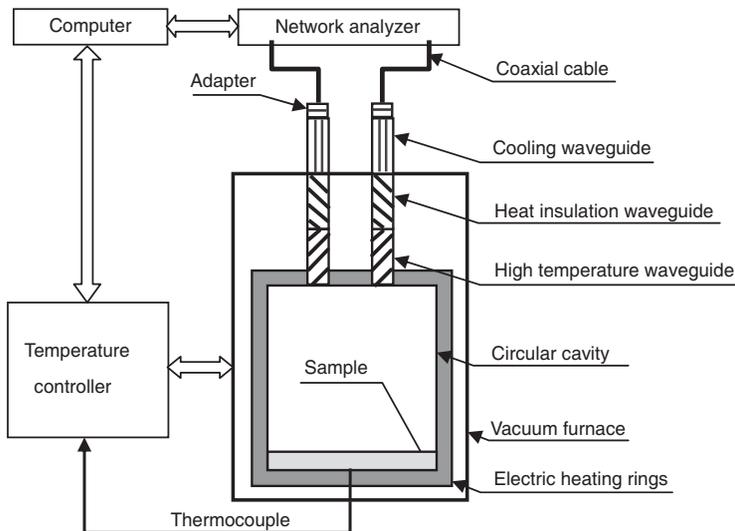


Figure 3. Block diagram of high temperature test system.



Figure 4. Photograph of the entire test system.

The empty cavity is heated and measured at different temperatures at first. When the temperature reaches at a certain temperature, several minutes are needed to heat the whole cavity to the same temperature. Then the resonant frequencies and unloaded quality factors of different modes of the empty cavity are measured automatically. The sample is loaded into the cavity after the test system is cooled to room temperature. After the temperature maintains the same temperature as the empty cavity for several minutes to reach isotherm, the resonant frequencies and unloaded quality factors of the different modes of the loaded cavity are obtained. Dielectric constant ϵ'_r and loss tangent $\tan \delta$ at various temperatures and frequencies can be determined from Equations (1) and (5). The process of the measurement is automated by the computer.

Before high temperature measurement, the sample is checked to make sure that it is in the solid state during the whole process of measurement and does not change into liquid at the highest temperature to prevent the cavity from being contaminated and damaged.

The resonant frequencies and unloaded quality factors of the empty cavity dependent on temperature are shown in Figures 5 and 6 respectively. The resonant mode of the cavity is TE_{01n} and n varies from 1 to 9. The working frequencies shown in Figure 5 cover 7–18 GHz.

The repeated measurement results of frequencies and unloaded quality factors shown in Figures 7–10 refer to the two modes of the cavity respectively. The repeatability of frequencies is less than 2 MHz, and the quality factors less than 300. The good repeatability of the empty cavity ensures the accurate measurement of the dielectric properties.

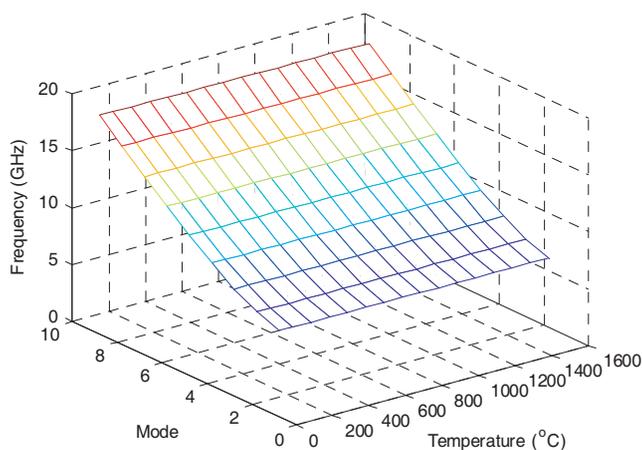


Figure 5. Temperature dependence of resonant frequencies.

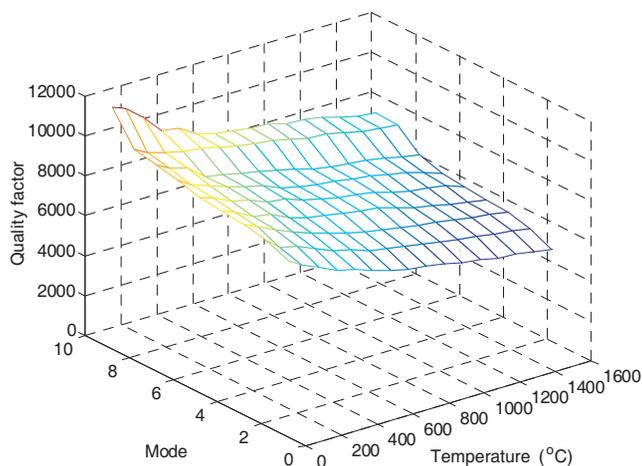


Figure 6. Temperature dependence of quality factors.

5. MEASUREMENT RESULTS

Samples made of homogeneous and isotropic fused quartz have been measured using the hardware system developed in the paper. Due to the isotropy of the sample, we don't need to consider the sample orientation in the measurements. The size of the sample is $\Phi 51 \times 2.81$ mm and the density is 2.195 g/cm^3 . Because the thermal expansion coefficient of fused quartz is smaller than 10^{-6} per $^\circ\text{C}$, the thickness changes of the sample as a function of temperature were

neglected in the measurements. Table 1 shows the impurity content of the quartz samples measured in the paper.

Table 1. Impurity contents of the samples.

Impurity	Na	Li	Ca	Al	K	Mg
Content (ppm)	6.5	1.3	16.0	140.0	1.5	4.4

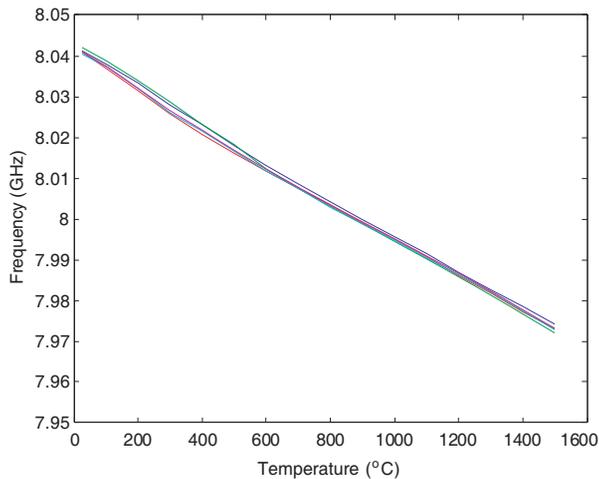


Figure 7. Repeated measurements of the resonant frequency of TE_{012} mode.

Figures 11 and 12 show the measurement results of the dielectric constant and the loss tangent of the sample dependent on temperature and frequency respectively. Tables 2 and 3 show the measured values for the three quartz samples made from the same material. The measurement results indicate the high repeatability of the test system. We can draw the conclusion from the tables that the measurement results of dielectric constant are in good agreement with the ones from MIT and CRC, and the results of loss tangent are between the ones from MIT and CRC [26]. MIT used resonance method and CRC used short-circuited method which has higher measurement uncertainty for loss tangent when measuring low-loss materials. The measurement uncertainties of the implemented system are, respectively, 2% for dielectric constant and 15% for loss tangent at room temperatures, and 4% for dielectric constant and 25% for loss tangent at high temperatures. The uncertainties given are analyzed and based on

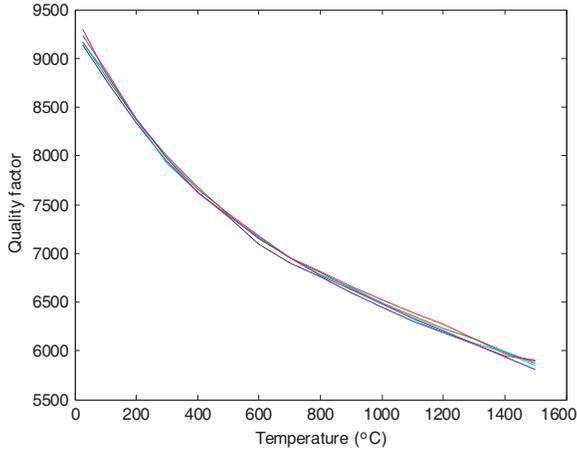


Figure 8. Repeated measurements of the quality factor of TE_{012} mode.

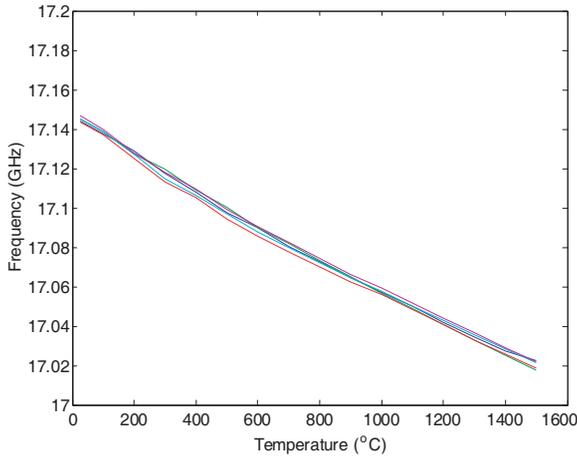


Figure 9. Repeated measurements of the resonant frequency of TE_{018} mode.

the measurement ranges of the system. Because the system is aimed at working at high temperatures, the measurement uncertainty of the loss tangent of extremely low-loss material at room temperatures is relatively higher than other methods. The detailed measurement uncertainty analysis will be reported in the future paper. Compared with the MIT data, the loss tangent differences may be caused by different samples with different impurity and measurement uncertainty.

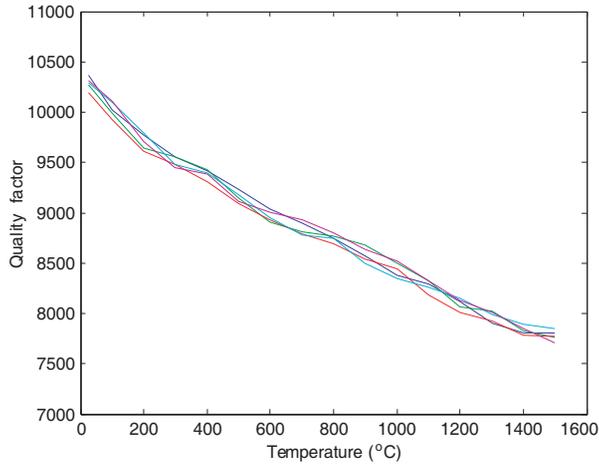


Figure 10. Repeated measurements of the quality factor of TE₀₁₈ mode.

Table 2. Measurement results of ϵ'_r of quartz samples at 9.10 GHz and data from MIT and CRC.

Temperature (°C)	Data using the system in this paper			MIT Data (8.52 GHz)	CRC Data (9.4 GHz)
25	3.850	3.859	3.845	3.82	3.81
100	3.861	3.868	3.852	3.83	/
200	3.862	3.871	3.863	3.84	3.83
300	3.86	3.882	3.868	3.84	/
400	3.876	3.888	3.879	3.86	3.84
500	3.883	3.901	3.889	3.87	/
600	3.893	3.905	3.902	3.88	3.86
700	3.907	3.908	3.909	3.89	/
800	3.917	3.915	3.918	3.90	3.88
900	3.936	3.947	3.925	3.91	/
1000	3.935	3.948	3.934	/	3.91
1100	3.942	3.951	3.938	/	/
1200	3.946	3.956	3.942	/	3.93
1300	3.951	3.959	3.946	/	/
1400	3.957	3.962	3.953	/	3.96
1500	3.967	3.969	3.965	/	/

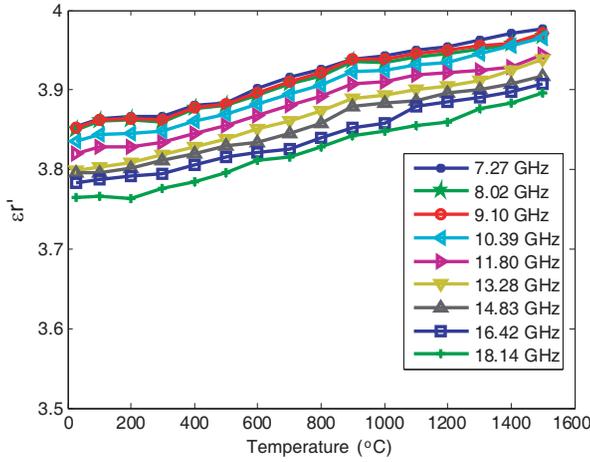


Figure 11. Temperature and frequency dependences of dielectric constant of quartz.

Table 3. Measurement results of $\tan \delta$ of quartz samples at 9.10 GHz and data from MIT and CRC.

Temperature (°C)	Data using the system in this paper			MIT Data (8.52 GHz)	CRC Data (9.4 GHz)
25	0.000333	0.000233	0.000290	0.00015	0.0015
100	0.000344	0.000260	0.000254	0.00014	/
200	0.000371	0.000283	0.000316	0.00012	0.0018
300	0.000372	0.000336	0.000341	0.00012	/
400	0.000428	0.000359	0.000335	0.00012	0.002
500	0.000415	0.000350	0.000350	0.00012	/
600	0.000453	0.000365	0.000353	0.00014	0.0029
700	0.000458	0.000386	0.00036	0.00014	/
800	0.000463	0.000399	0.000370	0.00014	0.0048
900	0.000454	0.000404	0.000387	0.00014	/
1000	0.000474	0.000426	0.000393	/	0.011
1100	0.000515	0.000449	0.000402	/	/
1200	0.000543	0.000502	0.000438	/	0.025
1300	0.000565	0.000521	0.000455	/	/
1400	0.000605	0.000550	0.000488	/	0.046
1500	0.000639	0.000576	0.000524	/	/

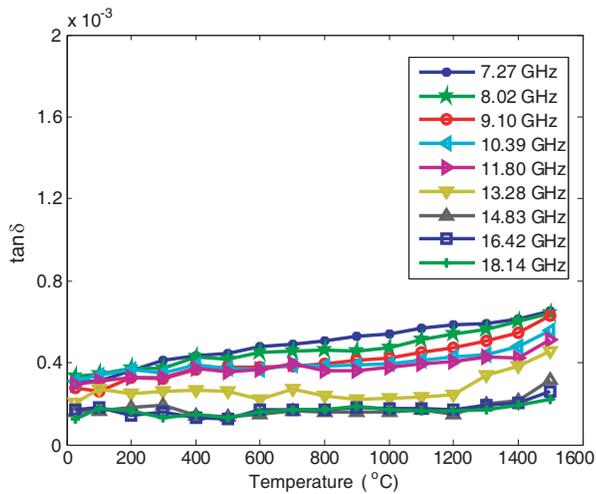


Figure 12. Temperature and frequency dependences of loss tangent of quartz.

6. CONCLUSIONS

In this article, we have presented a test system for the dielectric properties measurements of low-loss materials over wide temperature and frequency ranges using circular cavity method. The test system can provide reliable measurement results at temperatures from room temperature to 1500°C with the broad frequency range 7–18 GHz. The measurement uncertainties of the test system are, respectively, 2% for dielectric constant and 15% for loss tangent at room temperatures, and 4% for dielectric constant and 25% for loss tangent at high temperatures. The measurement results of dielectric constant are in good agreement with the ones from MIT and CRC. In addition, we believe the frequency of the test system can be expanded to other bands and elevated to higher temperature.

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