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Microwave Complex Permittivity of Yttria-Stabilized Zirconia

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ABSTRACT The complex permittivity of 8-mol yttria-stabilized zirconia (8-YSZ) in powder and sintered (i.e., solid) forms were measured from 32 to 40 GHz using a circular waveguide probe. This probe is suitable for measuring both the powder and solid forms of materials. Comparative completely filled rectangular waveguide measurements of the powder were performed at three frequency bands to verify these results and also to extend the measured complex permittivity estimation frequency range. The results indicated good agreement between the two different measurement techniques. The complex permittivity of the 8-YSZ powder was measured to be ($\epsilon_r = 2.45{-}j0.04$). Conductor-backed solid 8-YSZ, representative of an in-service ceramic coating, was also measured using the circular waveguide probe. Complex permittivity was measured to be significantly higher ($\epsilon_r = 29.28 - j0.07$) when the 8-YSZ was sintered into a solid form. This was attributed to densification and other effects occurring during the sintering process.

INDEX TERMS Ceramics, circular waveguides, complex permittivity, microwave materials characterization, thermal barrier coatings (TBCs).

I. INTRODUCTION

YTTRIA-STABILIZED zirconia (YSZ) is a widely used ceramic used in many critical applications, for example, in thermal barrier coatings (TBCs) and in solid oxide fuel cells [1]. In such applications, 7-8YSZ (i.e., yttria doping of 7%–8% by weight in zirconia) is widely used for its desirable mechanical and thermal properties [2]. Degradation of 8-mol YSZ (8-YSZ) coatings includes in-service thinning and microcracking. These can be monitored by measuring changes in its macroscopic electrical properties, namely, the relative complex permittivity ($\varepsilon_r = \varepsilon_r' - j\varepsilon_r''$). Relative (to free-space) complex permittivity, hereon referred to as complex permittivity, is an intrinsic material property expressed relative to free-space. The real part, dielectric constant, indicates the ability of the material to store electric energy, and the imaginary part, loss factor, indicates the propensity of the material to absorb electric energy.

In addition, the evaluation of YSZ powder is also of interest. Complex permittivity estimation of YSZ powder can facilitate monitoring the presence of contaminants, doping levels, particle size, and phase composition [3]. However, when YSZ is sintered or sprayed into a solid form, hereon referred to as solid YSZ, its properties significantly change and so it may be necessary to inspect YSZ in both powder and solid form. As previously mentioned, in solid form thickness and microcracking (e.g., porosity level, which is directly related to its complex permittivity) are of great interest as a means for process control and maintenance of critical structures (e.g., TBC). To this end, microwave materials characterization is well suited for these inspections. Open-ended waveguide techniques are capable of simultaneous estimation of the complex permittivity and thickness of layered dielectric materials [4], [5], such as solid YSZ which is usually sprayed on a metallic substrate. Subsequently, the estimated YSZ complex permittivity can be related back to other properties such as microcracking that manifests itself as an increased porosity level. There have been some previous works in measuring the complex

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permittivity of YSZ in powder (e.g., [6] and [7]) and solid form (e.g., [8], [9], [10], and [11]). Previous methods used to characterize the dielectric properties of solid YSZ were primarily reported at low frequencies [6], [8], [9], [10], [11], or using resonant techniques (i.e., narrow band) [6], [8], [9]. However, none of these works have considered a technique proven capable of complex permittivity estimation in *both* powder and solid form.

This work investigated the utility of a recently developed materials characterization technique using a circular waveguide, operating in the TE_{01} mode from 32 to 40 GHz [5]. This frequency range corresponds to a significant portion of the Ka-band (26.5–40 GHz) frequency range. The technique was used for estimating the complex permittivity of 8-YSZ in both the *powder* and *solid* forms. This particular probe has a small footprint and can be used on electrically thin (i.e., $<\lambda/2$) and low-loss coatings (i.e., $(\varepsilon_r''/\varepsilon_r') \leq 0.05$) under certain conditions, as described in [5]. The circular waveguide probe operating in its TE_{01} mode does not suffer from using a finite flange [5], unlike the more commonly used open-ended rectangular waveguide. The latter type of probe is adversely impacted by the use of a finite-sized flange, causing measurement errors when measuring thin and low-loss samples [12], such as solid YSZ in most applications.

Since there were limited materials characterization results, in the form of complex permittivity, of powder 8-YSZ at microwave frequencies in the literature, measurements performed using the circular waveguide were repeated using the well-known completely filled rectangular waveguide technique to validate the results obtained by the former at Ka-band (26.5–40 GHz) [13]. These results were subsequently compared to those in [7], in which the same technique and frequency band are used. Differences in the estimated complex permittivity of 8-YSZ powder between this work and [7] are discussed in Section II. Given the unavailability of circular waveguide probes at other frequency bands, and the desire to estimate powder complex permittivity over a wide range of microwave frequencies, completely filled rectangular waveguide measurements were extended to at X-band (8.2–12.4 GHz) and Ku-band (12.4–18 GHz), as well.

II. POWDER 8-YSZ RESULTS

The following measurements were performed on commercially obtained fine-grade (i.e., with surface area of ~13.4 m^2/g) 8-YSZ powder [14]. These measurements were performed in ambient conditions (22 °C and 50% humidity). A Denver Instrument S-64 balance was used to measure the mass of each sample for density calculations. The samples, prepared in respective sample holders, were measured using a calibrated Anritsu MS46524B vector network analyzer (VNA), which measured the relevant reflection and transmission coefficients known as scattering parameters (i.e., S-parameters) as a function of frequency. These measurements were performed at the microwave frequency bands mentioned earlier. A short–offset–short–load–thru (SSLT) calibration was performed [15], for the completely filled rectangular waveguide measurements, and the calibration reference planes are shown in Fig. 2. For the circular waveguide probe and as mentioned in [5], a (common) thru–reflect–line (TRL) calibration was performed and the reference plane is shown in Fig. 1(a).

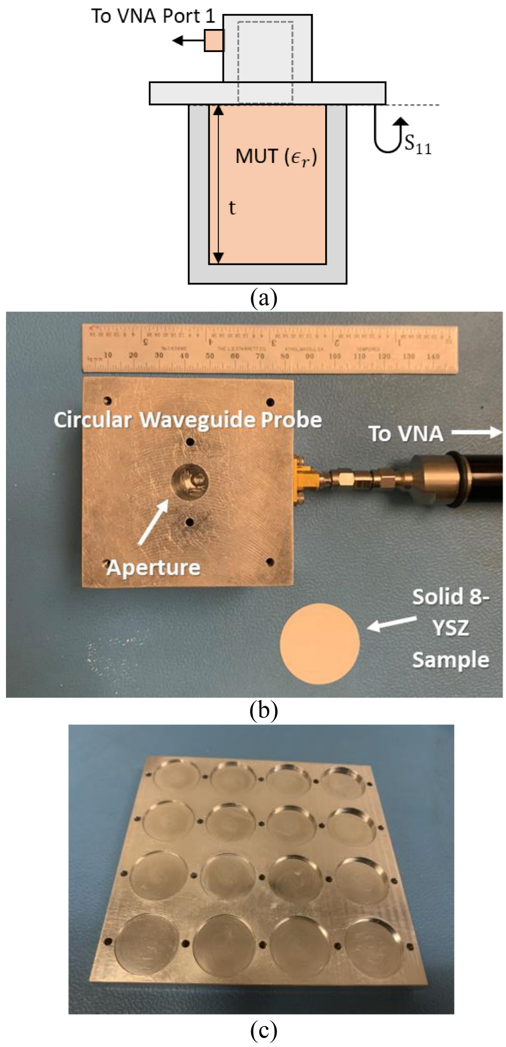


FIGURE 1. (a) Measurement schematic of the circular waveguide TE_{01} mode measurement (not-to-scale); (b) actual probe; and (c) sample holder showing 16 possible depths [i.e., t in (a)].

First, the fine grade 8-YSZ powder [14] was measured using the circular waveguide, as shown in the measurement schematic of Fig. 1(a) and pictured in Fig. 1(b). The circular waveguide probe has an aperture diameter of 5.7 mm. Fig. 1(c) shows a sample holder with 16 different depths, several of which were used in the measurements. As described in [5] and since the complex permittivity of the powder was unknown, multiple measurements were performed using several sample thicknesses (t) to obtain a good average for the complex permittivity estimations. To ensure propagation of the dominant TE_{01} mode, this size probe can

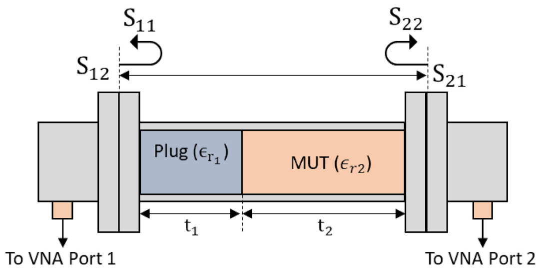


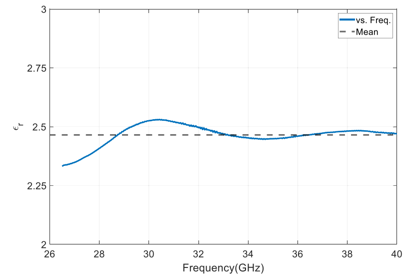
FIGURE 2. Measurement schematic of the completely filled rectangular waveguide measurement setup for a material under test (MUT).

only operate in the frequency range of 32–40 GHz [5], which constitutes a significant portion of the standard Ka-band (26.5–40 GHz) frequency range. Next, completely filled rectangular waveguide measurements were performed at each of the three frequency bands mentioned previously to validate the circular waveguide measurements and also to extend the measurement frequency range. The measurement schematic of the former technique is shown in Fig. 2. A plastic plug was inserted flush to one end of the waveguide section.

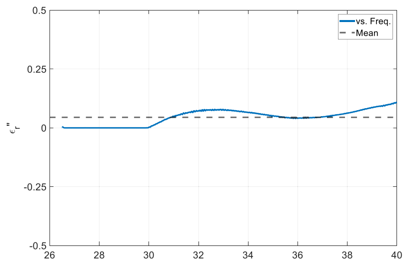
In performing these measurements, the powder was lightly packed into the respective circular and rectangular sample holders ensuring homogenous powder distribution within the volume of the sample holder. The surface was then swept clean using a flat blade. The density of the powder samples was measured to ensure sample-to-sample consistency among all measurements. In addition, the measurements were repeated ten times per sample. Furthermore, two operators independently performed each of the measurements. These steps were implemented to ensure measurement consistency and to reduce overall measurement uncertainty. The measured complex S-parameters were then used to calculate the complex permittivity of each sample [5], [13].

Fig. 3 shows an example of complex permittivity estimation, as a function of frequency, using the Baker-Jarvis technique [16]. This example used a completely filled rectangular waveguide measurement at Ka-band (26.5–40 GHz) with the properties of the plug de-embedded from the measurement. The results for the powder 8-YSZ measurements are summarized in Table 1, where the calculated complex permittivity ($\varepsilon_r = \varepsilon_r' - j\varepsilon_r''$) was averaged over the respective frequency ranges.

There is good agreement between the measured results shown in Table 1, both between the two operators and between the circular and rectangular waveguide measurements. The differences in complex permittivity estimation between operators were likely due to the samples' density, which corresponded to less than half a gram difference in mass in all respective cases. Notably, the estimations of complex permittivity for the 8-YSZ powder are much lower than those reported in [7] (i.e., 32.5–j1.6e-5) which uses the completely filled rectangular waveguide technique at Ka-band (26.5–40 GHz). However, these values were compared with those of sintered YSZ and not powder. It is expected that the complex permittivity of 8-YSZ to be lower in its



(a)



(b)

FIGURE 3. (a) Real and (b) imaginary parts of estimated complex permittivity as a function of frequency, for a completely filled Ka-band (26.5–40 GHz) rectangular waveguide.

TABLE 1. Estimated complex permittivity of powder 8-YSZ at multiple frequency bands, measurement methods, and when using two operators.

	Method	Circular Waveguide	Rectangular Waveguide		
	Frequency (GHz)	32-40	26.5-40	12.4-18	8.2-12.4
Oper. 1	Mean Density (g/cm^3)	1.28	1.29	1.28	1.34
	ε_r'	2.30±0.18	2.48±0.19	2.41±0.20	2.79±0.35
	ε_r''	0.03+0.01	0.06±0.03	0.08±0.03	0.16±0.03
Oper. 2	Mean Density (g/cm^3)	1.07	1.3	1.22	1.27
	ε_r'	2.19±0.02	2.45±0.02	2.47±0.03	2.56±0.06
	ε_r''	0.03±0.01	0.04±0.02	0.04±0.07	0.03±0.03

powder form than when made (i.e., sintered) into its solid form. This is due to the difference in the volume fraction of air in these two states—as will be shown in Section III since the solid samples were ~7 times more dense than the powder samples. This difference can be accounted for when considering an appropriate dielectric mixing model describing the complex permittivity in these two distinct forms (i.e., the presence of air has a significant influence on the measured *effective* complex permittivity) [17]. However, since the true density of the powder particles and its size distribution were not known, such a mixing model could not be directly applied. Nevertheless, the measured complex permittivity values were in the range typical for ceramic

TABLE 2. Estimated complex permittivity (given thickness) of conductor-backed solid 8-YSZ using stacked samples.

Measured Sample Thickness (mm)	Estimated					
	ε_r'			ε_r''		
0.25	32.54	±	1.66	0.00	±	0.01
0.49	28.03	±	0.02	0.08	±	0.02
0.75	28.82	±	0.54	0.08	±	0.00
0.99	28.84	±	0.16	0.08	±	0.01
1.24	28.16	±	0.03	0.11	±	0.00

powders, for which the volume fraction of air is relatively high [6].

III. SOLID 8-YSZ RESULTS

To show the utility of the circular waveguide probe for estimating the complex permittivity of solid 8-YSZ, five samples were measured using the circular waveguide probe shown in Fig. 1(b). The solid samples were sintered from the 8-YSZ material [18] and had a radius of 12.7 mm and a thickness of 0.25 mm. One such sample is shown to the right side of the circular waveguide probe in Fig. 1(b). The mean density of the samples was measured to be 8.99 g/cm^3, which indicates significant compaction occurred during the sintering process (i.e., less air content/porosity compared to the powder samples).

Since there were a limited number of samples available, ten conductor-backed measurements were performed using each of one to five of the samples stacked on top of one another, to generate thickness diversity (i.e., from 0.25 to 1.25 mm) in the solid sample measurements. Although some very slight air gap may have been present between the samples, light pressure was applied to minimize its effect. The estimated complex permittivity values are shown in Table 2. Conductor-backed measurements are representative of in-service type measurements in which 8-YSZ is applied as a coating to a metallic substrate, such as a TBC [1]. Subsequently, a full-wave electromagnetic (EM) model [5] implemented in MATLAB was used to closely estimate the complex permittivity of the solid samples, knowing both the thickness of the samples and the measured reflection coefficient. In other applications, the EM model may also be used to estimate thickness for a given complex permittivity or estimate both complex permittivity and thickness simultaneously, when neither are known a priori.

The estimated complex permittivity of the solid 8-YSZ samples was significantly higher than those measured for the powder samples over the same frequency ranges, as expected. This difference was attributed to the effects of sintering, which significantly densifies the powder in addition to other effects [3], [9]. By comparing the measured densities of the samples, it is clear that the solid samples were significantly more compacted than the powder samples, which reduces the volumetric fraction of air (porosity) in the former samples leading to higher dielectric constant values [10], [17]. As compared to other values found in the literature (e.g., Table 1 for density of 5.89 g/cm^3 in [4]) these measured values were in the range but slightly higher due to the samples having higher density of 8.99 g/cm^3. Note that the estimation of dielectric constant, ε_r', and its measurement uncertainty was higher when only one sample was used (i.e., the 0.25-mm sample). This was primarily due to 0.25-mm sample being electrically too thin over the measured frequency range. This caused a phase change in the measured reflection coefficient that was relatively small compared to the thicker samples, for this measurement method. For a more detailed description of this issue, the reader is referred to [10]. Therefore, the results of this measurement should be ignored. However, the results for the other four thicknesses were consistent and in good agreement. Note that for a certain complex permittivity and thickness range of interest, it is possible to optimize the circular waveguide probe dimensions and the operating frequency to maximize measurement sensitivity to thinner coatings.

IV. CONCLUSION

This work investigated the use of a circular waveguide probe for estimating the complex permittivity of 8-YSZ in both powder and solid form. Estimations of the powdered YSZ were also performed using the completely filled rectangular waveguide technique at three different frequency bands. These results indicated good agreement and indicate a low complex permittivity estimation for the YSZ powder ($\epsilon_r = 2.45 - j0.04$). Conductor-backed solid 8-YSZ, representative of an in-service ceramic coating, was also measured using the circular waveguide probe. Complex permittivity estimation was significantly higher ($\epsilon_r = 29.28 - j0.07$) for solid 8-YSZ. As explained earlier, this difference is primarily due to the effects of sintering. Future work using this circular waveguide probe could include the detection of contaminants, doping levels, particle size, and phase composition in powder YSZ and explore in-situ measurement of solid YSZ coatings.

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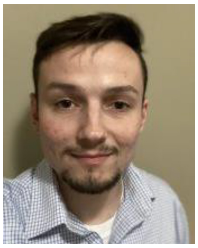

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