MEASUREMENT OF COMPLEX PERMITTIVITY OF SOLIDS UP TO 1000°C

Thorsten Hauschild*, Reinhard Knöchel**

* Technische Universität Hamburg-Harburg; Arbeitsbereich Hochfrequenztechnik
Wallgraben 55; 21071 Hamburg, Germany; email: T.Hauschild@tu-harburg.de
** Technische Fakultät der Christian Albrechts Universität zu Kiel
Lehrstuhl für Hochfrequenztechnik; 24143 Kiel, Germany; email: rk@techfak.uni-kiel.de

Abstract

A technique suitable for measuring the permittivity of solids up to 1000°C in X-band is introduced. Although techniques for permittivity measurements are well known, there exist a lot of technological problems such as calibration and choosing the right material for carrying out measurements at high temperatures. We present a method to determine the complex permittivity over the whole temperature range from room to high temperatures using only one calibration and measurement cycle.

1 Introduction

In [Hau95] a technique suitable for measuring density profiles of solids inside an ecologically friendly circulating fluidized bed coal combustor has been introduced. To calibrate this system it is necessary to know the complex permittivity of the powder compound inside the combustor at high temperatures up to 1000°C. To determine the complex permittivity at room temperature, various methods in example [HP85] or [Bae95] are well known. The accuracy of these methods depends on the calibration of the used vector network analyzer and on the type of material measured.

Mechanical sampling of the material inside a fluidized bed coal combustor in Kassel/Germany shows that the compound's distribution inside the combustor is nearly independent of the place of measurement and the load state of the combustor. Approximately 30 % volume fraction is lime, 30 % is gypsum, 30 % is sand, 1 % is coal and the rest are other components. The average particle size is 200μm. This solid type material has both dielectric and metallic characteristics with a relatively high loss $\tan\delta$. Therefore a method similar to that presented in [HP85] using a HP 8510C network analyzer is well suited for this material. At room temperature the permittivity of $\epsilon_r = \epsilon' - j\epsilon'' = 3.092 - j0.012$ with a standard deviation over frequency range of $\sigma_{\epsilon_r} = 0.002 - j0.002$ is virtually frequency independent in X-band (see fig.1). The shown measurement result has been obtained using an HP 8510C with TRL-calibration. If the combustor is driven into the fluidized state, this value can be used to calculate the dielectric constant versus the concentration of solids in air using the empirical law of refractive indices $\sqrt{\epsilon_r} = \sum_i m_i \sqrt{\epsilon_{ri}}$; m_i =volume fraction of the material i.

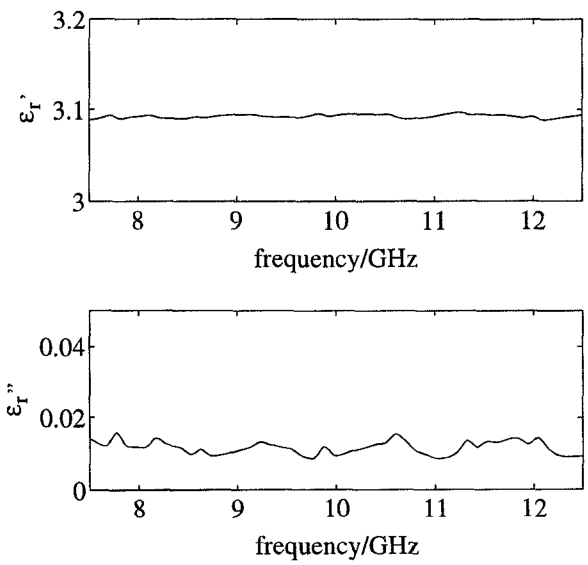

Fig. 1: real and imaginary part of ϵ_r, combustor compound at room temperature, volume fraction of the solids 55.6%, standard waveguide system, internal TRL calibration of HP 8510C

At higher temperatures this simple method can not be used anymore. The waveguide measurement setup,

TH 3F

0-7803-3246-6/96/$5.00 © IEEE

1996 IEEE MTT-S Digest

if it is made from brass or aluminium, is limited to temperatures of approximately 400°C. For every temperature the full internal TRL calibration of the HP 8510C has to be carried out, because one has no access to the calibration data of the HP 8510C during the calibration process.

To solve these problems a high temperature waveguide system has to be built up. In order to be able to measure the complex permittivity over the whole temperature range with only one calibration cycle and one measurement cycle, a new external calibration procedure has to be implemented.

2 Measurement system and calibration

For higher temperatures a suitable waveguide material has to be chosen. Using a high-grade austenitic stainless steel (DIN material No. 1.4841 or X15 CrNiSi 25 20 equivalent UNS S31000 or AISI 310) temperatures up to 1200°C can be reached with the system. This steel has a low coefficient of thermal expansion of $18 \cdot 10^{-6} m/(m \cdot K)$ and is non-scaling.

Windows made from alumina (Al_2O_3) are used to enable partial filling of a waveguide section which is only 10cm long, having a thickness of 1mm and dielectric constant ϵ_r of approximately 10 up to 1100°C with a very low loss tangent. The complete waveguide system is shown in figure 2 and the window region can be seen in detail in figure 3.

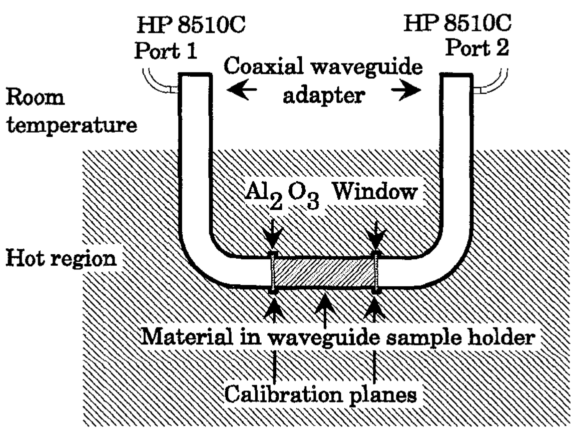

Fig. 2: Waveguide measurement setup

The lower part of the waveguide system can be heated and is isolated towards the rest of the system

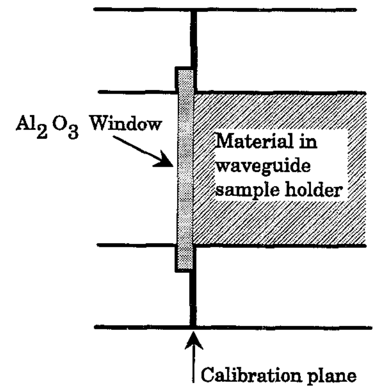

Fig. 3: Detailed graph of the window part of the waveguide measurement setup

and the network analyzer by using waveguides made from stainless steel with low heat conductivity.

The system should be calibrated with calibration planes placed right behind the windows. The HP 8510C TRL calibration routine can be used for that purpose only at constant temperature. When changing temperature the complete calibration routine has to be carried out again. Doing this the waveguide system has to be heated for every calibration standard to be connected. If the permittivity is to be measured every 50°C up to 1000°C twenty calibrations with 3 heatings each have to be performed. Approximately 12 hours are needed for heating and cooling the system with a need of 30 days for calibrating the whole system [1].

So external calibration of the HP 8510C is applied. In literature various methods are known. Using a 3-position method [Heu93], [Bae95] the measurement and calibration can be performed with only 4 heating cycles. But in this case only one material can be measured requiring a new calibration for each material. Therefore we decided to perform a TLS/S (**T**hru, **L**ine, **S**hort/**S**hort) calibration which is a modified TRL-calibration [Heu94]. Here 3 heating cycles are required for a calibration with the 3 standards and then any material can be measured with only one heating cycle within 12 hours. The calibration standards of the TLS/S-calibration may be partly unknown. This can be used for the high temperature calibration because the length of the line-standard is not required to be known exactly. Thus the length variation due to thermal expansion is not to be considered. The reflection coefficients of the S/S-standard can be completely unknown.

[1]The calibration standards can not be changed at high temperatures.

Using the TLS/S procedure at higher temperatures the fact that the standards are not known exactly is not a disadvantage due to the self-calibrating facility. A thru connection, a line with a length of 1cm and a double short are used as calibration standards.

Controlling of the HP 8510C and evaluation of the calibration data have been done using a simple PC 486 with the programms HP VEE and MATLAB respectively.

3 Results

Measurement results for the combustor compound by using the new waveguide system and the new calibration at room temperature are shown in figure 4. Excellent agreement of the permittivity measured with our new system compared to the standard system (figure 1) can be seen.

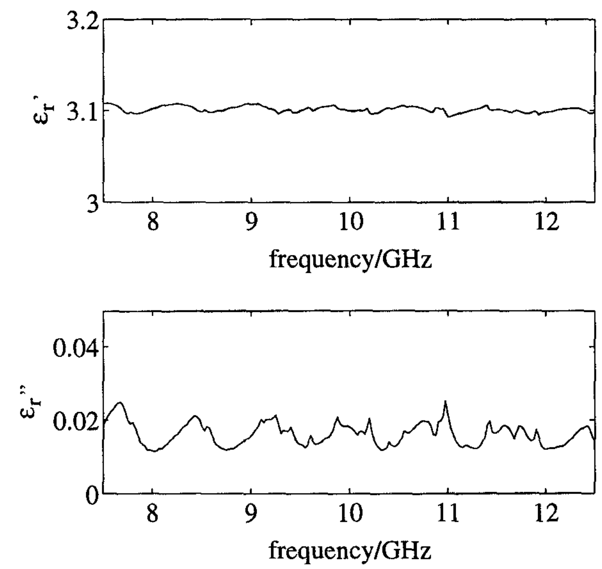

Fig. 4: real and imaginary part of ϵ_r, combustor compound at room temperature, volume fraction of the solids 55.6%, high temperature waveguide system, external TLS/S calibration of HP 8510C

A simple method to check the quality of the calibration at higher temperatures is to measure a simple waveguide filled with air. At 1000°C the permittivity of air was determined with the measurement setup as $\epsilon_r = \epsilon' - j\epsilon'' = 1.015 - j0.001$ (mean value over frequency range, standard deviation $\sigma_{\epsilon_r} = \sigma_{\epsilon'} - j\sigma_{\epsilon''} = 0.005 - j0.001$). This result shows the accuracy of the method and the quality of the calibration. The permittivity of air over the whole temperature range is displayed in figure 5.

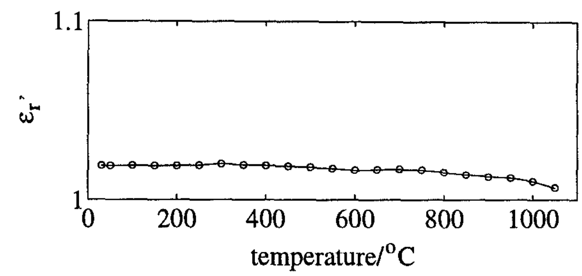

Fig. 5: real part of ϵ_r of air (averaged over frequency range) versus temperature, high temperature waveguide system, external TLS/S calibration of HP 8510C

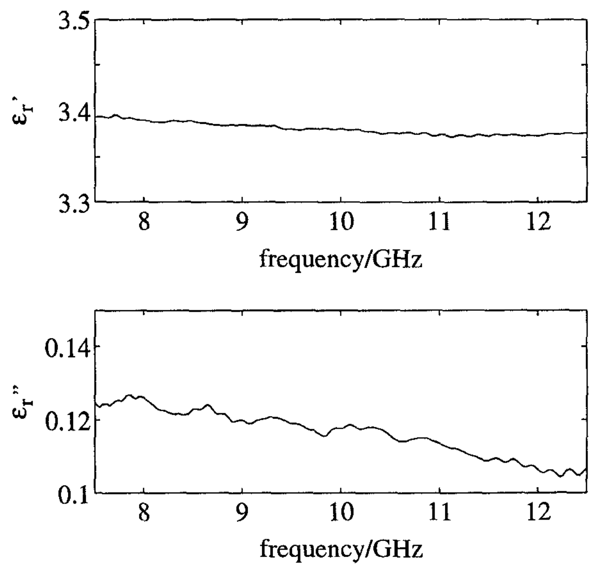

Fig. 6: real and imaginary part of ϵ_r, combustor compound at 1000°C, volume fraction of the solids 55.6%, high temperature waveguide system, external TLS/S calibration of HP 8510C

In figure 6 the permittivity for combustor compound at 1000°C and in figure 7 the permittivity over the whole temperature range is shown. The permittivity varies only slightly over the temperature from $\epsilon_r = 3.100 - j0.016$ with a standard deviation of $\sigma_{\epsilon_r} = 0.003 - j0.003$ at room temperature[2] to $\epsilon_r = 3.381 - j0.117$ (standard deviation $\sigma_{\epsilon_r} = 0.007 - j0.006$) at 1000°C. This mean value of the permittivity has been calculated by taking the thermal expansion coefficient to calculate the length of the waveguide section filled with combustor compound into account. The length changes due to thermal expansion from 100mm to 101.76mm at 1000°C. Measurements of the combustor compound sampled at

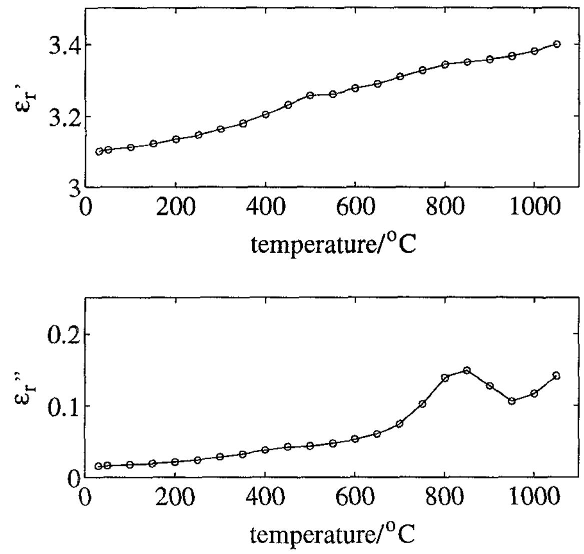

Fig. 7: real and imaginary part of ϵ_r (combustor compound) (averaged over frequency range) versus temperature, volume fraction of the solids 55.6%, high temperature waveguide system, external TLS/S calibration of HP 8510C

different positions inside the combustor have been carried out. The dielectric constant shows the same behaviour for every sampling position. Only the absolute value varies due to a varied volume fraction of solids in the sampled combustor compound.

4 Conclusions

With the new waveguide measurement setup and an external calibration scheme of the network analyzer it is possible to measure the dielectric permittivity of solids and powders from room temperature up to 1000°C. The calibration requires only 3 heating cycles for the whole temperature range. After calibration the complex permittivity of a material sample can be determined in only one heating process.

[2]Due to a new filling of the waveguide sample holder the permittivity is slightly different compared to fig. 4.

5 Acknowledgment

The financial support of the *Deutsche Forschungsgemeinschaft* in the *Sonderforschungsbereich 238* is gratefully acknowledged. The authors would like to thank Holger Heuermann, Franz Menke and Jan Stabe for fruitful discussions.

References

[Bae95] Baek, K.-H.; Sung, H.-Y.; Park, W.S.; *A 3-Position Transmission/Reflection Method for Measuring the Permittivity of Low Loss Materials;* IEEE Microwave and Guided Wave Letters 1995, Vol. 5, No. 1; pp. 3-5

[Hau95] Hauschild, T.; Knöchel, R.; *Microwave Measurement of Density Profiles in Fluidized Bed Reactors with Improved Spatial Resolution;* Proc. IEEE MTT-S International Microwave Symposium 1995 Orlando, Paper TH3F-13; pp. 1467-1470

[Heu93] Heuermann, H.; Schiek, B.; *The In-Fixture Calibration Procedure Line-Network-Network-LNN;* Proc. 23th EuMC Madrid, 1993; pp. 500-505

[Heu94] Heuermann, H.; Schiek, B.; *Robust Algorithms for Txx Network Analyzer Self-Calibration Procedures;* IEEE Trans. on Instrumentation and Measurement 1994, Vol.43, No.1; pp. 18-23

[HP85] Hewlett Packard; *Material Measurement;* Product Note 8510-3, Palo Alto, 1985