#### Loss Tangent and Dielectric Constant of Solder Mask Measured with Split-Post Dielectric Resonators

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#### Abstract

As PCB computing bus frequencies climb above 1GHz, measurement of the high-frequency properties of PCB materials becomes critical for design modeling. Understanding these properties and their dependence on temperature and humidity will enable manufacturers to formulate new materials that will be less subject to variation. This paper will introduce a method of measurement of loss tangent and relative permittivity of solder mask from 1.2 to 10.2 GHz and show the sensitivity of these properties to variability of temperature and humidity. The solder mask is coated on one side of a Corning® 7980 high-purity fused silica (HPFS) disc. This disc is inserted in a split-post dielectric resonator (SPDR). The resonance of the SPDR plus uncoated disc is compared to the resonance of the SPDR plus coated disc to calculate the loss tangent and relative permittivity of solder mask. This coated-disc technique has been used to determine the properties of other microwave materials.<sup>1</sup>

#### Terms

HPFS: high-purity fused silica, Loss tangent: a.k.a Df or tan d.; PCB: printed circuit board; Relative permittivity: a.k.a. Dk or er, SPDR: split-post dielectric resonator

#### Introduction

New high-speed busses such as PCI Express operate at  $\sim 1$ GHz, and higher speeds are planned. They use differential transmission lines to enhance signal integrity. Microstrip differential transmission lines have solder mask between the traces. In differential transmission lines the electric field lines are between the two traces as well as between the traces and the reference plane. Loss tangent and relative permittivity of the solder mask are contributing factors to their transmission line losses.

Figure 1 shows a field-solver simulation of the transmission line loss of a 100-ohm characteristic impedance, 11-inch long micro-strip differential bus constructed over Teflon®. Teflon was chosen because it does not significantly absorb moisture and we intend further investigations with a Teflon PCB substrate. The trace width is 18 mils, space is 12 mils and the dielectric thickness is 10 mils. We used values of loss tangent and relative permittivity from our measurements of solder mask that were made after the solder mask was exposed to three different conditions of temperature and humidity. It can be seen in this simulation that the loss of such a differential bus is influenced by the variation of the loss tangent and relative permittivity of the solder mask.



Figure 1 - Differential Transmission Line Loss as a Function of Change in Environment

This result supports the need to develop and use measurement methods that include control of temperature and humidity. This will yield data that supports modeling of differential micro-strip transmission lines and the variation of their losses with temperature and humidity.

Present in-house testing methods used at Taiyo Ink ®for measuring loss tangent and relative permittivity include a 1 MHz bridge method and a 1 GHz capacitance method. Further testing at a commercial lab is accomplished at 5 and 10 GHz using a perturbation method with cavity resonators. The perturbation method is quite similar to the calculation used with SPDRs in that the shift of Fo and Q is used to calculate loss tangent and relative permittivity. The sample for this method is solder mask alone, which we found leads to handling problems when exploring varying conditions of temperature and humidity. The samples tend to curl and become brittle when dried from the humid state.

SPDRs are resonant cavities designed by their materials of construction and dimensions to resonate at the frequency of interest. The insertion of a dielectric material into the cavity results in a change in the center frequency and quality factor of the resonance. This resonance is measured using a vector network analyzer (VNA). Using tables derived from electromagnetic laws the loss tangent and dielectric constant can be calculated from these changes.<sup>2</sup> The equipment used to make our measurements is shown in Figures 2 and 3.



Figure 2 - Split-Post Dielectric Resonators



Figure 3 - VNA and SPDR in Use

#### **Resonator Operation**

The VNA is operated to sweep the frequency of its measurement energy from just below to just above the resonant frequency of the SPDR as shown in Figure 3 above. A cross-sectional diagram of an SPDR is shown in Figure 4. The input and output of the resonators are two small loop antennas that barely penetrate the walls of the cavity. The penetration depth of these antennae is set using a thumb-wheel on a threaded rod that holds the antenna such that the forward transmission loss at resonance is -40 dB. At the resonant frequency the impedance of the cavity is at a local minimum so the forward transmission of energy is at a local maximum. The VNA is operated to measure S21, the center frequency, and the quality factor of the resonance. Because the calculation of loss tangent and relative permittivity is based on ratios of center frequency and quality factor it is not necessary that the VNA be calibrated as it would be if it were measuring a transmission line.

The measurement of  $F_c$  ( $F_0$  or  $F_s$ ) and Q ( $Q_0$  or  $Q_s$ ) is accomplished by a LabView program provided by Dr. Michael Janezic of NIST. The program operates the VNA to obtain 256 measurements of S21 of the resonating cavity and calculate a best fit value for  $F_c$  and Q. The program determines a weighted least-squares fit of the resonance peak developed by NIST statisticians.<sup>3</sup>

For the purpose of measuring the properties of a coating,  $F_0$  is the resonant frequency of the empty fixture plus uncoated blank.  $Q_0$  is the quality factor of the resonance for the fixture plus uncoated blank.  $F_s$  is the resonant frequency of the cavity+blank+coating and  $Q_s$  is the quality factor of that resonance. The measurement of Q is illustrated in Figure 5. The thickness uniformity of the coating is essential to obtain accurate measurements with low uncertainty.<sup>4</sup> The loss tangent and relative permittivity of the sample is calculated from  $F_0$ ,  $Q_0$ ,  $F_s$ ,  $Q_s$  and sample thickness by software supplied by the SPDR manufacturer.<sup>2</sup>



Figure 4 - Cross-Section of an SPDR and Coated HPFS Disc (not to scale)



Figure 5 - F<sub>c</sub> and Q of Resonance Peak

#### Solder Mask Selection and Coating Process

Four solder masks supplied by Taiyo America were sampled for measurement. The two-part liquid photo-imageable (LPI) systems varied in composition, commercial usage and expected performance. The composition varies by resins systems, additives and fillers, both organic and inorganic. For purposes of this paper, the solder mask products are as LPI A, B, C and D. For reasons of space we will present only the results from the LPI C sample. (See Table 1.)

Product	Reason for choice
LPI A	Extensive Commercial Use
LPI B	Extensive Commercial Use
LPI C	Performance Record
LPI D	Compositional Characteristics

Table 1	-	Solder	Mask	S	elections
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The wafers were processed, as received, without any type of surface preparation or cleaning. Typical solder mask application requires a surface roughening and cleaning of the surface, mechanically or chemically. The wafers required careful handling and, for the testing, no cleaning or changing of the surface characteristics.

The inks and hardeners were mixed at the specified ratios (7:3). The LPI solder masks were coated onto a flexible PET substrate using a drawn down bar. After coating, the films were dried in a convection oven at 80 C for 30 minutes. Draw down bar gap settings of 0.004" and 0.008" gave dry thicknesses of approximately 50 micron and 100 micron respectively. A carrier panel with machined pockets to fit the diameter and thickness of the discs was used to hold the discs during coating to keep the surrounding surface and the surface of the discs coplanar.

The films were laminated to the surface of HPFS discs having a frosted surface finish to enhance adhesion. The plates were heated to 80 C and the vacuum was drawn to  $10^{-4}$  torr for the lamination process. This flattened the mask to give a uniform coat. For increased solder mask thickness, additional laminations stacked easily.

The masks were exposed to ultraviolet light (800 mj/cm<sup>2</sup>) for initial polymerization. Although typical for circuit board applications, the wafers were not developed in a sodium carbonate solution. Given that the light polymerized the entire coating, the developing solution performed no function. Finally, the solder masks were cured in a convection oven at 150 C for 60 minutes. Additionally LPI C received a post thermal cure exposure to UV light of 3 J/cm<sup>2</sup>. Table 2 and Figure 6 show the specific processing steps and parameters.

Process Description	Variable	Value
Surface Preparation	None	NA
Solder Mask Mixing	Hand Mix Time	5 min
Coating	Hand Draw Down Gap	0.008" and 0.004"
Drying	Oven Temperature	80 C
Drying	Oven Time	30 min
Application	Plate Temp	80 C
Application	Vacuum	$10^{-4}$ torr
Application	Vacuum Cycle Time	30 sec
Application	Press Time	30 sec
UV Exposure	Energy	800 mJ/cm <sup>2</sup>
Cure	Cure Time	60 min
Cure	Cure Temperature	80 C
UV Exposure*	Energy	$3 \text{ Joule/ } \text{cm}^2 (\text{as req'd})$

#### Table 2 - Solder Mask Processing Steps



**Figure 6 - Solder Mask Processing Steps** 

#### Design of Experiment

#### Equipment Setup

All samples were conditioned for a minimum of 1 hr to the desired state in a temperature and humidity controlled chamber. Temperature was controlled to  $\pm -0.5$  degrees Fahrenheit and humidity to  $\pm -2\%$  RH.

#### **Standards Measurement**

Polished discs of Corning 7908 HPFS were used as a reference standard. At the beginning and end of sample measurements using each SPDR the appropriate size polished HPFS disc was measured. These discs were also previously measured at NIST. Figures 7 and 8 below show the comparison of these measurements.



Figure 7 - Loss Tangent of HPFS



Figure 8 - Relative Permittivity of HPFS

#### Repeatability

Repeatability testing was done for all four SPDRs using the LPI C sample. The SPDRs and samples were placed in the environmental chamber at 70 degrees F and 15% RH, then stabilized for at least one hour. Each sample was measured 30 times in close succession. The test required about 2 hours for each SPDR. For each measurement the  $F_0$  and  $Q_0$  of the blank HPFS disc were obtained, then the  $F_s$  and  $Q_s$  of the HPFS disc+ sample obtained. The standard deviation of the relative permittivity and loss tangent that resulted from these measurements is shown in Table 3.

Table 5 - Repeatability Test Results						
Fixture	Loss tangent					
1.25 GHz	0.074	0.0006				
3.2 GHz	0.003	0.0002				
7.1 GHz	0.011	0.0002				
10.2 GHz	0.006	0.0001				

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#### **Corner Cases**

The accuracy of SPDRs is impacted by operation in humid air so for this phase of testing they were operated in a dry box that is swept with dried compressed air that kept the box at or below 10% RH. Samples were conditioned in the environmental chamber, removed from the environmental chamber, and measured within less than a minute of being placed in the SPDR.

The corner cases examine the range of our area of interest; a cool dry day in a conditioned space in the temperate zone, to a hot humid day in the tropics. A laptop or desktop PC would be expected to operate reliably in both environments. The PC board in this computer would be expected to absorb moisture based on the condition of the cooling air it was supplied. Table 4 shows the combinations of temperature and humidity we examined. The corner cases are shown in bold blue.

The solder mask samples and polished HPFS standard discs were conditioned in an environmental chamber to the states shown in bold blue in Table 4. The SPDRs were kept in the dry box to eliminate environmental conditions as a metrology variable. The samples were removed from the environmental chamber, quickly measured using the SPDRs in the dry box, then returned to the environmental chamber. The HPFS measurements were consistent with the data shown in Figures 7 and 8. The results of measurements of LPI C are shown in Figures 9 and 10.

Table 4 - Sample Temperature and Kelative Humbury Kanges							
Temp (F) / RH %	15	55	95				
70	70 deg F/ 15% RH	70/55	70/95				
85	85/15	85deg F / 55% RH	85/95				
100	100/15	100/55	100 deg F / 95% RH				

Table 4 - Sample Temperature and Relative Humidity Ranges



Figure 9 - Loss Tangent of LPI C in Varying Environmental Conditions



Figure 10 - Relative Permittivity of LPI C in Varying Environmental Conditions

Clearly, temperature and relative humidity are significant variables in the loss tangent and relative permittivity of this material. LPI C was chosen to report from the study sample because of its typical behavior and to save space. The results of LPI A, B and D have similar behavior of increasing relative permittivity with increasing temperature and humidity. They also show a pattern of increase in loss tangent as a function of increasing temperature and humidity.

#### Limiting Variable

The limiting variable portion of our experiment holds one of the two variables constant, temperature or relative humidity, and sweeps the other. Our limiting variable test applies all the conditions displayed in Table 4. This test uncouples the variables and enables the study to show the individual effects of temperature and relative humidity. The 3.2 GHz SPDR fixture was chosen for this study. Relative permittivity and loss tangent of LPI C for each RH and temperature combination of Table 4 was measured.

Each condition was allowed to acclimate inside the environmental chamber overnight to assure equilibrium condition of moisture in the samples. The chamber reaches equilibrium in just a few minutes, however letting samples sit overnight ensures the moisture ample time to diffuse into the solder mask. During the measurements at each condition, a polished HPFS disk was measured before the experiment run and afterwards. This ensures the SPDRs were providing accurate measurements. The HPFS measurements were consistent with data shown in Figures 7 and 8. The results of measuring LPI C are in Figures 11 and 12.



Figure 11 - Relative Permittivity of LPI C as a Function of Temperature and Relative Humidity



Figure 12 - Loss Tangent of LPI C as a Function of Temperature and Humidity

Clearly, while temperature certainly affects relative permittivity and loss tangent to a measurable degree, relative humidity is the dominant effecting factor. Data in Figures 11 and 12 shows relative humidity to be the dominating factor in the variation of relative permittivity and loss tangent of LPI C.

#### Conclusions

The SPDR method of measurement and the sample preparation method we have used offer advantages in preserving the sample as environmental conditions are varied. Control of temperature and relative humidity are essential in the measurement of loss tangent and relative permittivity of solder mask. With increasing bus frequency accurate data including these environmental effects is essential for modeling transmission line losses. Temperature and relative humidity should be controlled and reported for such work, and the effects on varying them on other materials investigated. Personal computers must consistently perform in a variety of climactic conditions. Data used to model transmission lines for PCB bus design must account for these variations.

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# Loss Tangent and $\mathbf{E}_r$ of Solder Mask from 1 to 10 GHz Using Split Post Dielectric Resonators

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Always on your side.

# Agenda

- Motivation
- Measurement Equipment and Process
- Sample Preparation
- Data and Conclusions
  - HPFS standards
  - Example solder mask material
- Technical Details for Q & A

# 100-ohm, 20 cm long differential bus on Teflon® substrate, coated with "material C"



3

# SPDRs and support equipment



resonators



Environmental Chamber for Sample conditioning





Dry box for resonators

### The measurement

- Characterize resonator + uncoated substrate disc obtaining F<sub>0</sub> and Q<sub>0</sub>
- Characterize resonator + coated substrate disc obtaining  $\rm F_s$  and  $\rm Q_s$
- Measure solder mask sample thickness
- Calculate loss tangent and relative permittivity using F<sub>0</sub>, Q<sub>0</sub>, F<sub>s</sub>, Q<sub>s</sub> & sample thickness





### Sample prep processes

### Sample preparation



### Draw-down bar and solder mask ink



# Making solder mask films with a draw-down bar



### Vacuum laminator



### Data - Results

#### Corner Cases

#### Limited Variable Sweep

Three conditions shown In Blue

Variables – Temp & RH

Temp (F)/RH%	15	55	95	
70	70/15(day1)	70/55 (day 4)	70/95 (day 7)	
85	85/15 (day 2)	<b>85/55 (day5)</b>	85/95 (day 8)	
100	100/15 (day3)	100/55 (day6)	<b>100/95</b> (day9)	

### Corner Case Results: Material C







### **Corner Case Results: Material HPFS**





### Limited variable Sweep – Material C



Max ▲ εr(temp) ~ 0.075, (2.6%) Max ▲ εr(RH%) ~ 0.275, (8.9%)



#### Limited Variable Sweep – HPFS @ 3.2 GHz



### ε<sub>r</sub> Repeatability – Material C









### Loss Tangent Repeatability – Material C



1.25 GHz







10.2 GHz

### Conclusions

- Temp & RH clearly effects  $\varepsilon_r$  & tan  $\delta$ . Control is necessary to push future performance requirements.
- RH is the dominant factor effecting  $\epsilon_r \& \tan \delta$
- $\epsilon_r \& \tan \delta$  dependence on temp & RH should be reported, and new materials researched
- Reduction of RH sensitivity would improve the consistent performance of PCB materials over a full range of environmental working conditions around the world

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### **Backups**

# Calculation of $\epsilon_r$ '

- From electromagnetic principles, dimensions and materials of resonator the fixture manufacturer calculates tables of K<sub>e</sub>, K<sub>1</sub> and K<sub>2</sub> as a function of sample thickness (columns) and ε<sub>r</sub>' (rows). These are supplied as text files "DINP10", "DINP20" and "Qinp" with software to perform the calculations
- SPDR fixture manufacturer checks fixtures and tables against known values for well-studied materials
- Extrapolate/interpolate table for material thickness.
- Solve equation 1 by trial and error, interpolating Ke linearly.

$$\varepsilon_{r}^{'} = 1 + \frac{f_0 - f_s}{h f_0 K_e(\varepsilon_{r}^{'}, h)} \quad \text{Eqn}$$

 $f_o$  = resonant frequency of empty fixture

- f<sub>s</sub>= resonant freq of fixture + sample
- h= thickness of the sample (meters)

 $\varepsilon_r$ '= relative dielectric constant

"Ke" constants in "DINP20"							
	extrapolated		thickness (microns)				
Er'	65	100	200	400			
2	3.482191	3.47706081	3.462402885	3.435585465	3.3901665	3.336494475	3.31968384
5	3.483015	3.478243695	3.46461087	3.43911402	3.3942368	3.335007075	3.287557005
10	3.484440	3.48018033	3.46800978	3.444057615	3.3976839	3.323616405	3.216262305
20	3.487263	3.48376818	3.4737825	3.45038409	3.3927313	3.271748355	3.030305145
35	3.491498	3.488526855	3.48003762	3.451325775	3.3591834	3.14188929	2.718630525

#### **Example for 3.2 GHz fixture**

# Calculation of loss tan

"K1 constants in file "DINP10"							
	extrapolated			width (mic	rons)		
Er	65	100	200	400			
2	6.878251	6.87361	6.860349	6.810238	6.737078	6.641804	6.617596
5	6.911202	6.90575	6.890172	6.855504	6.803876	6.728044	6.655688
10	6.915682	6.91769	6.923428	6.921873	6.900993	6.825097	6.608787
20	6.945795	6.95745	6.99075	7.031179	7.030779	6.850379	6.221865
35	6.986444	7.0107	7.080002	7.148631	7.077461	6.580101	5.371282
50	7.026654	7.05959	7.153694	7.21465	6.983663	6.121364	4.571013

"K2" constants in "Qinp"							
	extrapolated		width (microns)				
Er	65	100	200	400			
2	1.000205	1.000315	1.00063	1.001245	1.002489	1.004859	1.009025
5	1.000863	1.001318	1.002618	1.005103	1.010028	1.019604	1.035439
10	1.001943	1.002974	1.00592	1.011554	1.022757	1.044125	1.075414
20	1.004191	1.00638	1.012635	1.024651	1.048081	1.089983	1.130423

Extrapolate/interpolate tables for K<sub>1</sub> and K<sub>2</sub> for material thickness. Interpolate values for ε<sub>r</sub>' determined in that calculation step

## Calculation of loss tan

 $\rho_{es}$ = h  $\varepsilon_r$ 'K<sub>1</sub>( $\varepsilon_r$ ',h) [eqn2] where:

> $\rho_{es}$  =electric energy filling factor in the sample h=sample thickness in meters K<sub>1</sub> is tabulated in table DINP10

$$Q_p = Q_0 K_2$$
 [eqn 3]

Where:

Q<sub>p</sub>=Q<sub>parasitic</sub>

 $Q_0$  = resonant quality factor of the empty fixture K<sub>2</sub> is tabulated in file named "Qinp"  $\tan \delta = \frac{Q_s^{-1} - Q_p^{-1}}{\sum_{s=1}^{n} \log_s (\log 4)} \quad \text{Where: } Q_s = \text{resonant quality factor} \\ \text{of fixture with test sample}$ inserted

#### Field structure in split-cylinder tuned cavity **Field structure** Z is TE<sub>doz</sub> **Dominant propagation** mode for this measurement Is TE<sub>011</sub> ρ That is: does not vary with $\phi$ , varies with p, varies with z

E field is zero at ends and center axis, increases with  $\rho$ , constant in  $\phi$ 



### **Corner Case Results: Material A**



#### Max Tan δ 🔺 ~0.016, 56%

Max ε<sub>r</sub> **Δ** ~0.25, 7.7%



### **Corner Case Results: Material B**



#### Tan δ 🔺 ~0.007, 1.7%





### Corner Case Results: Material D



ε<sub>r</sub> ▲ ~ 1.85, 65%

