

# What is Impedance and Dielectric Spectroscopy?

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Impedance and Dielectric Spectroscopy (IS/DS) is a broadband characterization tool that can provide a wide range of information not often accessible by using single frequency measurements or dc methods. With IS/DS one can detect and often isolate responses due to the bulk of the materials or devices, the presence of internal and external interfaces as well as capture details associated with charge storage and dissipation. Equipment suitable for making measurements over as many as 12 orders of magnitude in frequency is now easily available, and the technique has found niches in the characterization of batteries, fuel cells and solar cells because all of these devices contain many different material layers and a wide variety of electrical responses. Many studies in the literature describe examples of individual components of these energy related devices. IS/DS has also been used as a method of quality control of capacitors and to monitor aging of electrical and structural components. However, the method is not limited to energy related devices only. Any material and/or device regardless of its end application may be characterized to determine the contributions of the different components/interfaces encountered and the frequency dependencies observed. Responses for dielectric type materials are best interpreted in the dielectric formalism while more conducting materials are best described in terms of conductivity as a function of temperature and/or atmosphere.

In this tutorial (videorecorded as two parts in May 2019 for the IEEE Instrumentation and Measurement Society's Video Tutorial Program <https://iee-ims.org/vt-program>), the technique of impedance and dielectric spectroscopy applied to the solid state characterization of materials and devices (without using liquid electrolytes) was described in the simplest possible terms. The tutorial begins by describing the expected responses for the real and imaginary impedance of a wide range of material and device types. Most insulating or semiconducting materials can be represented by parallel RC circuits while conducting materials may be represented by series or parallel RL

circuits [1]. Since materials and devices contain many interfaces, it is possible that one may detect more than one process which gives rise to more than one semicircle in the complex impedance graph, the sizes of which depend on what the response of the specific conducting path being detected is related to. Impedance spectroscopy is inversely related to dielectric permittivity since charges present in materials and devices can give rise to dielectric relaxation [1]. The reader is referred to more detailed research articles where the conversion of impedance to three other formalisms known as admittance, electric modulus and permittivity can be used as additional input for interpreting information often hidden inside of the partially analyzed impedance data [2]. Although there is not enough room to describe all of the nuances of this technique in this short introductory article, these conversions are necessary to achieve the best fit and capture all of the electrical paths in a given experiment. Nevertheless, it is shown that it is possible to relate the spectra obtained to the presence of certain key responses in the material or device under test such as charge storage, electronic conduction, surface adsorption, switching phenomena and many others. The tutorial ends with examples that demonstrate that this technique is exceptionally good for establishing quality control in a production environment and/or to assess service life of electronic and non-electronic components in a non-destructive way.

## Background and Expected Trends

Impedance Spectroscopy is a technique that involves the measurement of the current, voltage and phase angle over a wide frequency range so that the impedance is given by:

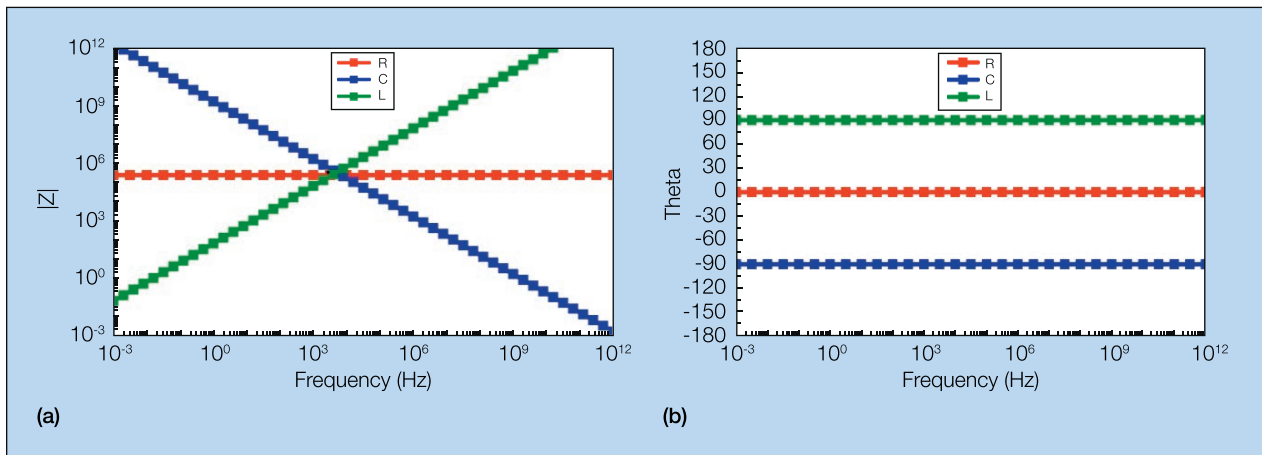
$$Z(\omega) = \frac{V(\omega)}{I(\omega)} \quad (1)$$

where  $\omega$  is the angular frequency,  $V(\omega)$  is the ac voltage and  $I(\omega)$  is the current which lags the voltage by a phase angle  $\theta$ , and  $Z$  is a complex number that consists of  $Z'$ , the real part of impedance, and  $Z''$ , the imaginary part of impedance.

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**Fig. 1.** (a) Impedance Magnitude  $|Z|$ ; and (b) Phase angle theta plotted versus log frequency for a resistor (R), a capacitor (C) and an inductor (L).

$$Z = Z' + jZ'' \quad (2)$$

When making impedance measurements, one often measures the impedance magnitude  $|Z|$  which is given by:

$$|Z| = \sqrt{Z'^2 + Z''^2} \quad (3)$$

and the phase angle is:

$$\theta = \tan^{-1}(Z'' / Z') \quad (4)$$

In addition to varying the frequency of excitation, it is possible to also vary the voltage of the ac signal and change specifics of the electrode contacts (such as composition, size and shape) so there are many ways to affect the measured response.

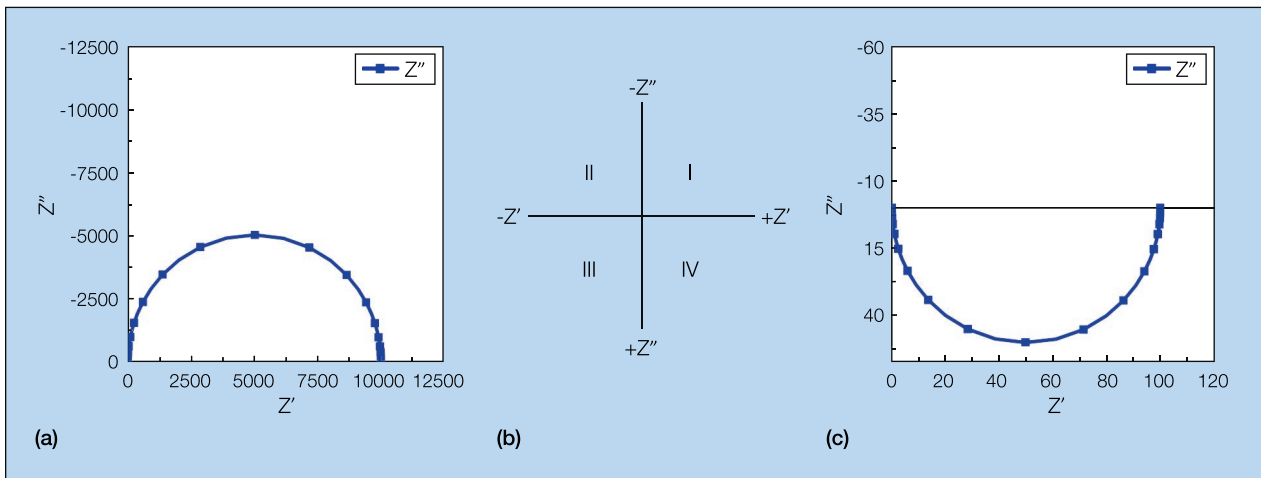
This method has been used to characterize the electrical properties of materials and devices since the late 1960s when high temperature solid oxide fuel cells were first investigated as a function of frequency [3]. With the advent of personal computers and access to increasing amounts of data storage, the method has been applied to a wide variety of applications such as detecting corrosion layers on metallic materials, determining the ionic conductivity for different components in batteries, fuel cells and solar cells, dielectric polarization in capacitors, conductive percolation in composites, etc. The most often used method involves the usage of a liquid electrolyte to evaluate the response of the materials under test to the exposure to a liquid electrolyte. Because this process is an electrochemical process, it is better known as electrochemical impedance spectroscopy. This is the method that is useful for understanding the behavior of chemical sensors for a wide range of applications. What will be discussed here are measurements conducted in the solid state that probe the bulk properties of materials and devices under test and not the surface reactions which are often interpreted in terms of the Randles circuit [4].

Solid state impedance spectroscopy can be used on all classes of materials including bulk ceramics, polymers, metals

and composites and may also be used to characterize complete electronic, semiconducting and superconducting devices, keeping in mind that one needs to know all of the components and physical arrangement of the layers or individual components [5]. Because the resistivity of materials can range over 22 orders of magnitude, ac measurements can provide a plethora of information besides the changes in complex impedance graphs that are often reported in the literature. This presents a challenge and an opportunity to the research community. Luckily, ac measurements can also detect capacitance and inductance in addition to resistance of a measurement. As shown in Fig. 1a and Fig. 1b, these three circuit elements, a resistor (R), a capacitor (C) and an inductor (L) behave quite differently. For a perfect resistor, the impedance magnitude is independent of frequency while it has a phase angle of zero. For a capacitor, the impedance magnitude decreases with increasing frequency and has a constant -90 degrees phase angle. An inductor has a +90 degrees phase angle and its impedance magnitude increases with increasing frequency.

Since most materials and/or devices will normally have more than one circuit element, we need to work with combined parallel and series circuit combinations of these three elements. The simplest case is the parallel RC circuit, which can often represent an insulating material (e.g., ionic conductor, high resistance semiconductor) and the RL parallel or series circuit which can represent a more conductive type material (high conductivity semiconductor, even metallic alloys, etc.). If these circuits are arranged in parallel, they will give rise to a semicircle in the complex impedance plane as shown in Fig. 2. Capacitive circuits will show in the first quadrant while inductive circuits will appear in the fourth quadrant.

Most materials and/or devices will normally have more than one current path present which gives rise to combinations of these parallel circuit elements. In Fig. 3, four cases of sequential parallel RC circuits labeled as  $R_1C_1$  and  $R_2C_2$  where  $C_1 = 10^{-3} C_2$  are shown [1]. The values of the resistors can be:  $R_1$  is greater than  $R_2$ , equal to  $R_2$  or smaller than  $R_2$ . If  $R_1$  and  $R_2$  are sufficiently different such as  $R_1 \ll R_2$  then the presence of the other may or may not be detectable as demonstrated in Fig. 3d.

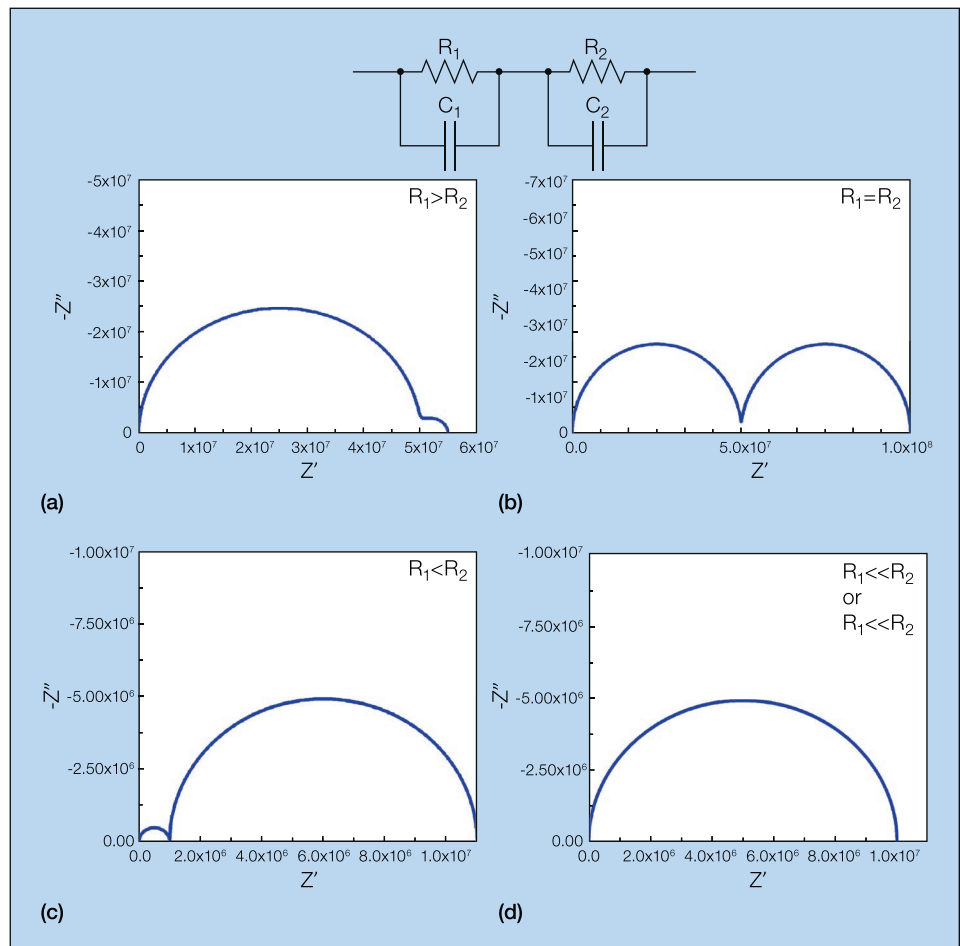


**Fig. 2.** (a) Complex impedance ( $Z''$  vs  $Z'$ ) for an RC circuit in parallel; (b) Sign convention showing complex impedance graph for an RC circuit in the first quadrant; and (c) Complex impedance ( $Z''$  vs  $Z'$ ) for an RL circuit in parallel which must appear in the fourth quadrant since  $Z''$  is positive in this case.

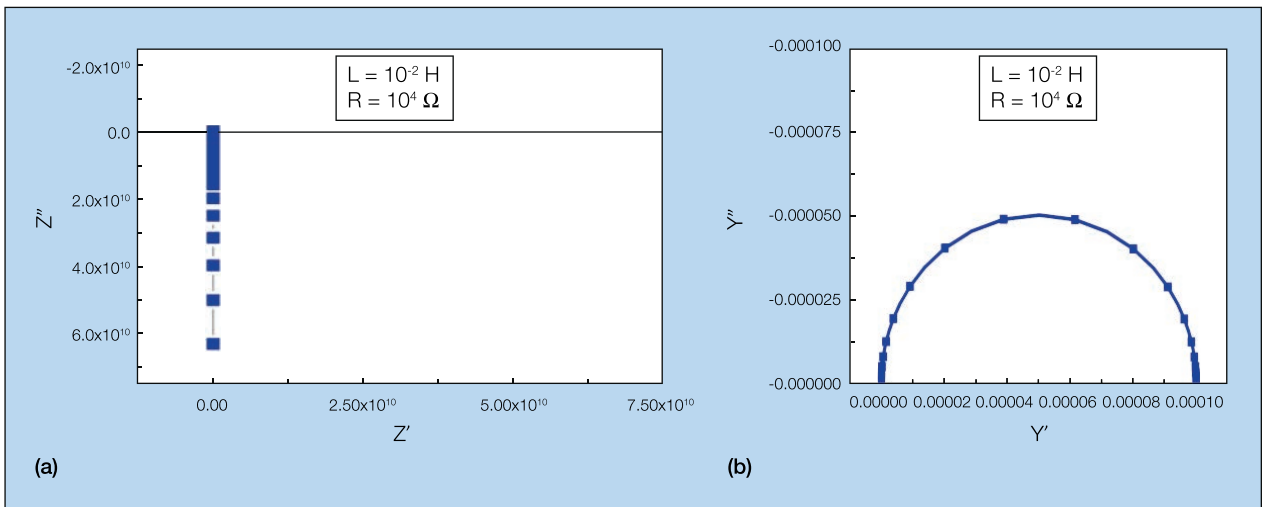
By far, the four spectra given in Fig. 3 are examples of impedance response that are the most often reported in the literature. However, when measurements are conducted, one may or may not always detect a semicircle in the complex impedance plane as the ability to detect them is a function of the specifications of the equipment that is being used, the dimensions of the object one is measuring and the properties of the object or material being probed. This does not mean that impedance spectroscopy is not useful. It just means that the circuit path being detected in such a sample is not parallel RC-like. This is where the conversion to other dielectric functions such as dielectric permittivity, electric modulus and admittance can come in handy to shed more light into what is being probed [2]. Because of space limitations, the reader is referred to the cited references.

Going back to how we can interpret the complex impedance graphs that show semicircles as in Fig. 2a and Fig. 2c, there are several things one can get from them. The intersection of the imaginary  $Z''$  impedance onto the real  $Z'$  impedance axis provides a direct measure of the value of R in every case. If the semicircle is in the first

quadrant, this immediately means it is a parallel RC circuit and the capacitance C may be calculated by taking the apex of the semicircle and setting it equal to  $\omega\tau = 1$  and since  $\tau = RC$  then one can obtain  $C = 1 / 2\pi f$  [6]. Likewise, for the parallel RL circuit, one can get the value of R from the size of the semicircle and the frequency at the apex of the downward semicircle in



**Fig. 3.** Effect of R values in parallel RC circuit elements in series with  $C_1 = 10^{-3}C_2$ , modified from [1].



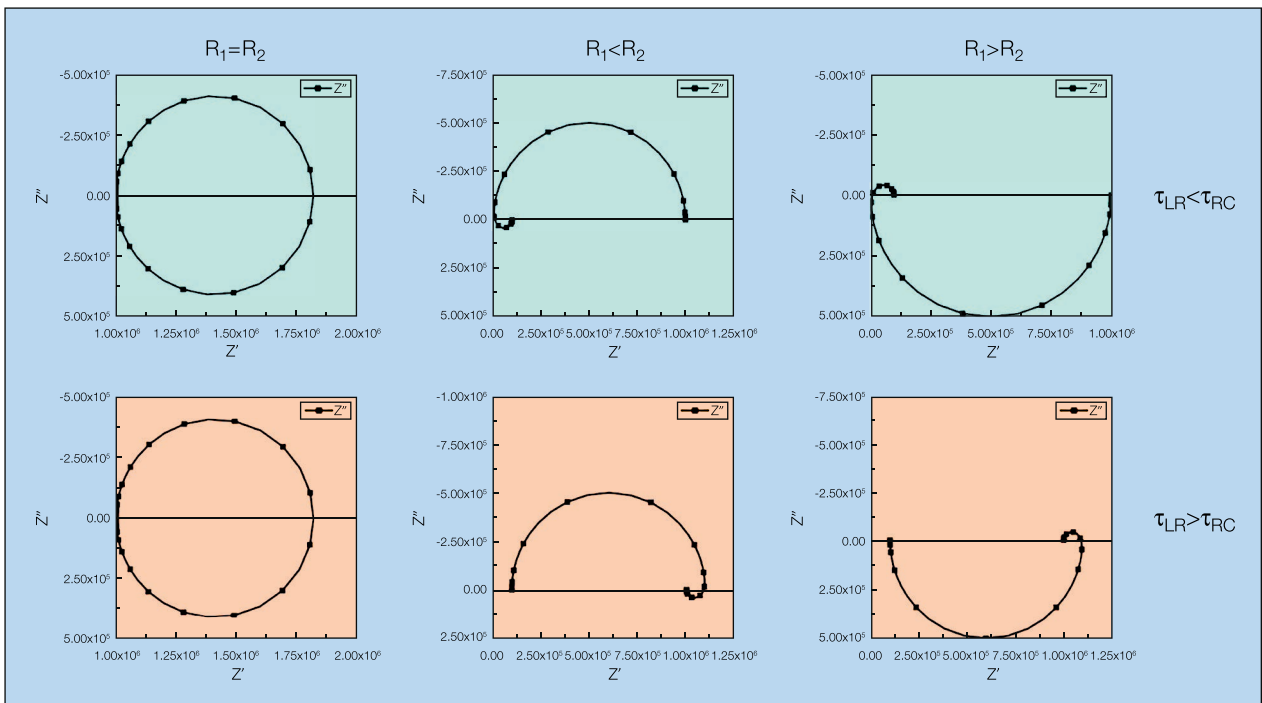
**Fig. 4.** (a) Complex impedance ( $Z''$  vs  $Z'$ ); and (b) Complex admittance ( $Y''$  vs  $Y'$ ) for a series RL circuit.

this case will provide  $L$  since  $\tau = L/R$  and  $L = R/\omega$  [6]. If a measured spectrum provides reasonable values of  $R$ ,  $C$  and  $L$  that are compatible with the dimensions and expected properties of the materials then one can proceed to conduct further analysis.

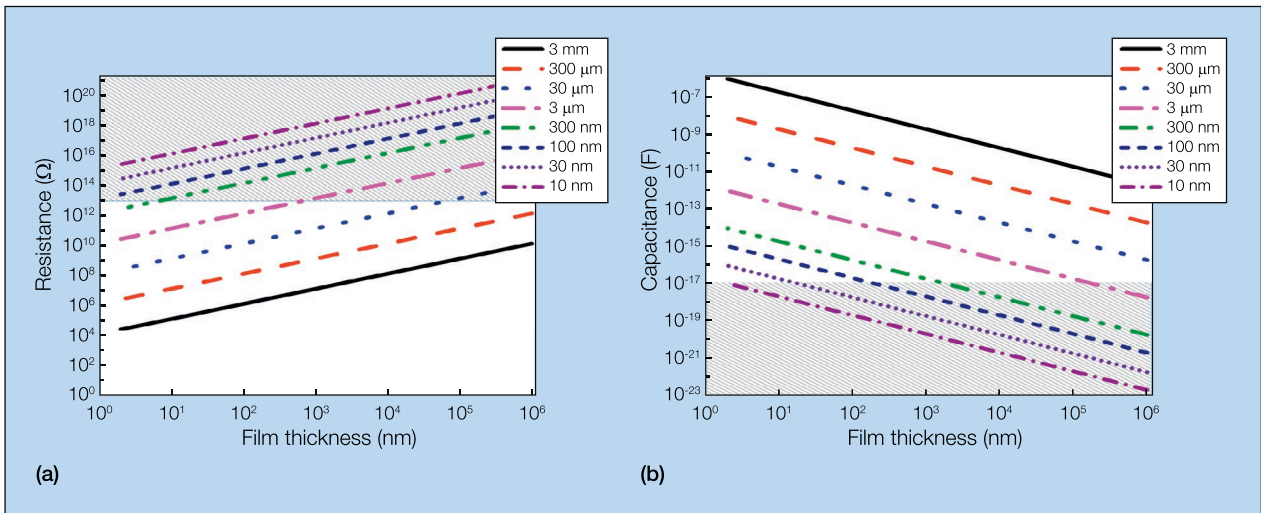
As for the case for multiple semicircles displayed in Fig. 3, interpretation requires some additional considerations. The problem is that there are many possible reasons why a measured spectrum will show two semicircles. Examples include layers with different electrical response (i.e., grain boundaries in polycrystalline materials, segregation of small amounts of impurities into or out of these interface regions [7], formation of Schottky barriers [8], formation of voids, etc.).

For the case of series RL circuits, complex impedance graphs do not result in complete semicircles, and therefore researchers are often confounded by this discovery. However, if one converts the impedance values to admittance values, keeping in mind that  $Y = 1/Z$ , the formation of a semicircle in the complex admittance can be observed as shown in Fig. 4. The intersection of imaginary  $Y''$  with the real  $Y'$  provides the ac conductivity of the specimen being evaluated. Similarly, one may obtain  $R = 1/Y'$  and  $L$  from the apex of the semicircle where  $L = R/\omega$ . This behavior is representative of fairly conducting materials such as conducting oxides [9].

Fig. 5 displays the wide variety of complex impedance responses that one may get when combining parallel RC and



**Fig. 5.** Effect of parallel RC in series with parallel RL on observable spectra which depend on the value of  $R$  and the time constant for the RC and RL portions, modified from [10].



**Fig. 6.** Calculated resistance and capacitance for HfO<sub>2</sub> films with thicknesses ranging from 1 nm up to 1 μm (on the x-axis) using circular top electrodes with different diameters (shown in the legend). Shaded areas are not possible to measure accurately due to equipment limitations.

parallel RL in series [10]. This combined set of graphs indicate that there are two important parameters: one is the value of R in each case, and the second is the value of the corresponding time constant. As Fig. 5 indicates, when the values of  $R_1=R_2$ , the complex impedance graph shows perfect circles made up of the  $R_1$  and  $R_2$ . Even though one cannot see it on these simulations, the order of appearance from low to high frequency is different depending on whether the time constant is  $\tau_{LR} < \tau_{RC}$  or  $\tau_{LR} > \tau_{RC}$ . This is more clearly seen on the parts where  $R_1 < R_2$  or vice-versa.

It should be added that because many materials and devices have components with resistivity that can vary over 20 orders of magnitude (when including the most insulating to the most conducting components), interpretation can be very complicated. In fact, for the same material or device, the complex graphs and frequency explicit spectra can be quite different and lead to confusion unless the user has been trained in the proper analysis methods. Additional characteristics associated with the dielectric, optical or magnetic response of a given material or device can further complicate the analysis and must be taken into consideration.

## Obtaining Trustworthy Responses

Electrical measurements are extremely sensitive to many different factors in addition to the specific R, C and L responses they may have. As already mentioned, because the resistivity of materials can range from  $10^{-6}$  to  $10^{14}$  ohm-cm, it is sometimes difficult to detect the correct properties for either end of the spectrum. This means that when one makes impedance measurements, one must already have an idea of what range of values one expects to obtain based on the base properties of the materials/components involved by taking the geometric corrections needed. For resistivity,  $\rho$ , one can estimate R by taking the expected length  $l$  and electrode contact area A using:

$$R = \frac{l}{\rho A} \quad (5)$$

If the electrode is circular then A is obtained from multiplying  $\pi$  by the radius squared. This can help the researcher determine what size specimens they should consider preparing to ensure that the measurements will provide the widest range of information possible. For a given material or device, the time constants will be representative of the properties of the DUT so any adjustments in the physical dimensions will not affect the location of the inflexion points on the real or imaginary functions [6]. Additionally, one must pay attention to the equipment specifications to ensure that what one wishes to measure is possible. In Fig. 6, calculations to obtain the expected resistance and capacitance were done for hafnium oxide (HfO<sub>2</sub>) films that are expected to be insulating and have a low dielectric constant of about 24 [11]. It is clear that the shaded regions in Fig. 6a show resistance values that would be extremely difficult to measure because most instruments can only go up to  $10^{10}$  ohms or so. Likewise, the shaded capacitance regions in Fig. 6b represent values that are also not easily measurable ( $<10^{-17}$  F). A more detailed version of this issue is described in [12] where silicon dioxide (SiO<sub>2</sub>) films were experimentally measured and their impedance properties were calculated. This is a more serious case because the dielectric constant of SiO<sub>2</sub> is only 4 and places more restrictions on the low end of the measurable capacitance as a function of film thickness and contact electrode size.

Tables 1 and 2 list some of the factors that one needs to be aware of when making measurements of materials or devices. Table 1 focuses on characteristics related to the chemical and physical make up of the objects under test, whether they are individual materials, composites or devices. Fabrication of devices can lead to many defects at the interfaces of layers or the presence of polar molecules which can affect the detected response. Additionally, the effect of aging also needs to be considered. When a material or device is pristine and new, the values will tend to be more reproducible, whereas later in its useful life, the development of

**Table 1 – Material dependent factors for measurements of materials and/or devices**

• Main component conducting or insulating
• Porosity or delamination at interface layers
• Presence of polar molecules (i.e., bulk water, permanent dipoles or organic chains)
• Charge accumulation at interfaces between layers or at contacting electrode
• State of material or device: new, midlife, spent
• Tunneling behavior or dielectric breakdown
• Presence of inadvertent impurities
• Sensitivity to moisture, optical excitation or magnetic excitation

**Table 2 – Measurement dependent factors**

• Equipment specifications
• Relative humidity or other atmospheric conditions
• Frequency and temperature
• Electric field orientation with respect to features
• Electrode material, size, and shape
• Ratio of material conductivities and size features
• Connectivity of the phases or components of interest

defects caused by aging can give rise to substantial changes in the measurement profile. In Table 2, the most important factors related to the measurement dependent factors are given. As already mentioned, the specifications of the equipment one is using should always be considered. The ambient conditions of when the measurements are being conducted also need to be monitored as temperature and relative humidity or even dry or wet air can affect the response of materials, especially more insulating types [13]. In contrast, for more conducting materials or devices, one needs to be aware of potential errors associated with the formation of non-ohmic contacts and the size of the features being measured [14]. For heterogeneous materials, such as composites or multimaterial devices, one needs to consider the size, shape and distribution of the conducting paths and whether they interconnect or not as this can cause changes in impedance of as many as 10 orders of magnitude [15]. Finally, even for mostly conducting materials such as carbon nanotubes, the arrangement of the bundles and junctions between them can affect the resultant response [16].

## Suggested Procedure

Establish most suitable baseline electrical testing set up to obtain the desired response of the materials/device system of interest and develop master curves for the parameters of interest. Model the response using equivalent circuit and electrical interface based microstructural modeling. Corroborate models with complementary experimental characterization

techniques to evaluate the changes predicted. Model the expected changes, taking into account external variables such as temperature, atmosphere, mechanical loading, environment, etc. Monitor the changes against the predicted changes to develop more detailed long-term models. This of course assumes that for every case, the researchers have evaluated at least three replicates of the specimen they wish to evaluate. This is necessary to establish the sample-to-sample variation under ideal conditions. From prior extensive experience, it has been determined that if the samples are well made, the results should be reproducible within 1% for identically made specimens. If different batches of specimens made under nominal identical conditions are evaluated, it is expected that the error may be as high as 5%. If the error is considerably higher than 5%, this may possibly indicate that something else is causing a larger error which needs to be determined and identified. Electrical measurements, when made with identically made specimens under ideal conditions, are extremely reproducible. As such, any differences detected should not be taken lightly as this probably means that there has been some factor that caused the change and must be eliminated or at least minimized.

## Concluding Remarks

- Insulating materials show large frequency dependent impedance responses but minimal frequency dependence of the dielectric properties.
- Insulating materials are extremely sensitive to moisture. This is more so if porosity, bulk water or alkali ions are present.
- Impedance spectroscopy is an excellent method to identify the presence of different interfaces and/or different electrical phenomena.
- One must use lower frequencies in addition to microwave range to detect all bulk, interfacial or space charge effects that are occurring.
- Conducting materials are more susceptible to being affected by the types of contacts used and set ups must use materials that make ohmic contacts or use superimposed dc bias to lower the interfacial barrier.
- Conducting materials may show a large variety of conducting paths in real materials which show very interesting frequency dependences.

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