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SENSING (RIMSS)**

Task Order 0020: High Frequency Eddy Current NDE

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Final Report**

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1.0 SUMMARY

The study includes the development of a prototype system based on the “EddyCus[®]” Eddy Current (EC) device platform to characterize the thickness and electrical conductivity of wet conductive coatings. The prototype system includes special sensors for application on wet surfaces, electronics for Eddy Current measurements and laboratory software with algorithms to determine the coating thickness after complete drying.

For method validation, an extensive sample preparation program was carried out. Two types of wet conductive coatings (low and high conductive) deposited at different thicknesses and with different coated areas on ceramic (Al_2O_3) and carbon fiber–reinforced plastic (CFRP) substrates were investigated. Drying processes were analyzed under real conditions, i.e. drying under normal atmosphere, normal pressure and room temperature.

2.0 INTRODUCTION

To develop a prototype system for characterization of drying behavior for wet conductive coatings, the overall process of the wet coatings has to be analyzed. This includes the coating process and the behavior of different substrates and coatings, different areas and coating thicknesses and different drying conditions.

Due to varying materials of the wet conductive coating experiments, the drying process has to be evaluated. Therefore, different substrates and different lacquers for the coatings were used. The lacquers varied in conductive particles (silver, copper, graphite) and related solvent. The drying process was analyzed under real conditions, i.e., drying under normal atmosphere, normal pressure and room temperature. During the drying of the coatings, the real drying process was evaluated and information about the behavior of electrical conductivity, layer thickness and shrinking of the layer was evaluated. As a result of fluctuating the size of the coated area, additional information about the drying process was evaluated. Working with large area wet coatings allows the simulation of the practical conditions more realistically, especially the drying process, which proceeds differently in small sample areas.

One sensor prototype using three very thin needles for contacting the surface was proposed. The needles are arranged around the coil and guide the sensor mechanically with a constant lift-off to the sample surface. The pins will touch the wet coating, but when measurements are performed in the early stage of the coating, the coating will not be damaged. The pins can be stored in a tissue of solvent during the interval between the measurements to remove any residue from the coating.

An Eddy Current prototype system for measurements on wet conductive coatings was designed based on the EddyCus[®] electronics developed at Fraunhofer IKTS-MD. The system will initially be tested under lab conditions and, if successful, in field conditions at real structures.

3.0 METHODS, ASSUMPTIONS, AND PROCEDURES

3.1 Percolation Behaviors of Wet Conductive Coatings

Electrically conductive pastes, also called wet conductive coatings, consist of a matrix of the carrier material (polymer matrix) filled with electrically conductive particles (mostly silver, but other electrically conductive materials are also used). The formation of the conductive areas in defined structures with a random distribution of the conductive particles in the matrix is described by the percolation theory [1]. From a certain amount of conductive particles in the polymer matrix, the individual particles are in contact with each other and form individual interconnections. The electrical conductivity of the system is realized when the conductive particles come into contact with each other, building a network of conductivity passing through the entire volume. After depositing, the coating is soft and mostly non-conductive. The polymer matrix is hardened during the drying of the coating, whereby the filler particles are fixed in their position. Through the evaporation of the solvent and crosslinking processes, the layer can shrink during drying. The shrinkage process improves final electrical conductivity of the coating, because particles are more strongly pressed together.

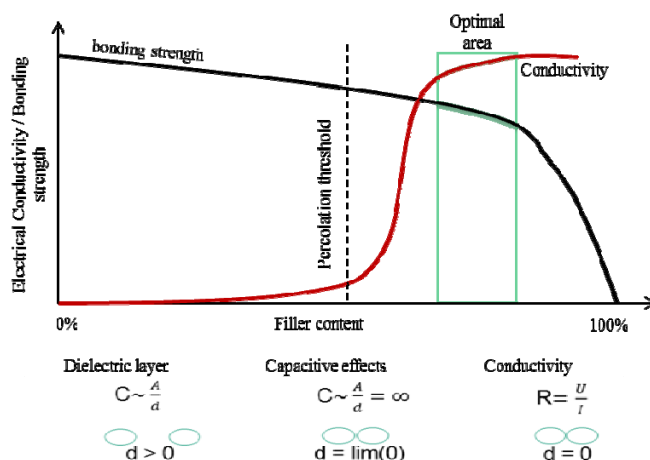


Figure 1. Schematic of the Percolation Behaviors in Wet Conductive Coatings

The drying process for wet conductive coatings is shown in Figure 1 and can be described (simplified) as follows. First, the particles are uniformly distributed in a volume. At this time, the layer has a maximum thickness, while forming no or a very low electrical conductivity. During the drying of the coating, the polymer matrix shrinks depending on its composition. Drying decreases the layer thickness and increases the conductivity of the layer, because conductive particles are pressed together by the shrinkage of the layer and come into better contact with each other. At that time, the drying is complete, and the layer has reached its maximum electrical conductivity and minimum thickness. During the drying of conductive coating, two parameters are changing, i.e. the thickness of the coating decreases and its conductivity increases; both parameters affect the Eddy Current signal. According to the functional capability of the wet conductive coatings, the final sheet resistivity is a combination of final thickness and conductivity.

3.2 High-Frequency Eddy Current Principle

The lower the conductivity of the specimen being tested, the higher the measurement frequency should be selected for an optimal trade-off between sensitivity and penetration depth.

When using Eddy Current testing, a primary electromagnetic field is induced around the induction coil. By reacting with a conductive sample, this field excites Eddy Currents in the sample, which cause a secondary field in opposition to the primary field. Impedance changes are recorded in the pickup coil. The higher the conductivity of the sample, the lower the Eddy Current penetrates into a specimen:

$$\delta = \sqrt{\frac{2}{\omega \mu \sigma}} \quad (1)$$

where ω is the angular frequency $2\pi f$, μ is the permeability and σ is the electrical conductivity.

The density of the Eddy Current is strongly affected by the frequency of the exciting field as given by Faraday's law

$$U_{ind} = -\frac{d\Phi}{dt} \quad (2)$$

where U_{ind} is the induced voltage and $\frac{d\Phi}{dt}$ is the rate of change of magnetic flux.

Both, equation (1) and (2) show that the lower the conductivity of the specimen the higher the frequency required for the desired higher sensitivity [2]. However, conductivity should not be considered solely for the choice of an optimal frequency. The higher the frequency, the lower is the penetration depth of the Eddy Current. Therefore, by the choice of the optimal frequency for Eddy Current testing, the conductivity, thickness and measuring depth of the target sample need to be considered.

3.3 Description of the Sample Plan

Properties of the wet conductive coatings as well as their drying behaviors are strongly affected by the type of used particles, carried matrix, chemical composition, environmental conditions [3], etc. The type of the conductive particles and their ratio in a carried matrix are responsible for the final conductivity of the coating. Chemical composition (type of carried matrix, thinner) defines the shrinkage ratio, while environmental conditions influence the rate of processes during curing.

To evaluate the prototype system based on Eddy Current techniques for characterizing drying behaviors of the wet conductive coatings, the influence of each parameter of coatings on the curing process should be studied. This is only possible by varying the one parameter, while others parameters remain fixed. Therefore, it is most prudent to produce the wet conductive coating instead of buying them. Thus, by changing the type of conductive filling, the type of matrix and thinner remains the same. To eliminate the influence of environmental conditions, coatings are dried at room temperature.

As drying behavior of the wet conductive coatings should be analyzed by the Eddy Current method, not only are the processes in them influencing measurements, but also the material used as substrate has an impact. In order to only acquire information about behaviors of the wet

conductive coatings during curing, a non-conductive substrate should be used. Knowing the drying behavior of the wet conductive coating, the influence of the substrate type on the Eddy Current measurements can be investigated.

Considering the above information, a sample plan was defined. The plan (Table 1) considered single-layered samples where the following parameters were varied:

- Type of conductive particles
- Thickness of coating
- Coated area
- Type of substrate

Table 1. Final Sample Plan

Name	Type of conductive particles	Thickness	Coated area	Substrate
Coating (1)	Silver coated Copper	$d_1 = 80\mu m$ $d_2 = 160\mu m$ $d_3 = 240\mu m$	4cm×4cm	Ceramic
Coating (2)	Silver coated Copper		4cm×4cm	CFRP
Coating (3)	Silver coated Copper		2cm×2cm	Ceramic
Coating (4)	Silver coated Glass		4cm×4cm	Ceramic

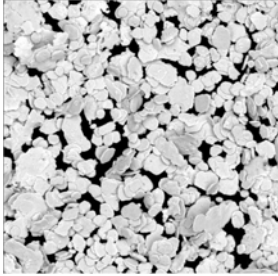
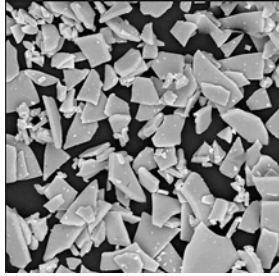
3.4 Experimental Setup

3.4.1 Producing Wet Conductive Coatings

Two types of wet conductive coatings are used in this work: low and high conductive pastes. Low conductive pastes are polymer-based lacquers, reinforced by silver-coated glass conductive particles; high conductive pastes are polymer-based lacquers as well, but reinforced by silver-coated copper conductive particles (Table 2). The curing time for both types of coatings is 24 hours at room temperature under normal conditions. Wet conductive coatings are deposited on the ceramic and CFRP substrates by the screen printing technique, but using the frame instead of the mesh, at three thicknesses (80μm, 160μm, 240μm) in accordance with the thickness of the copper frame used by the application.

Ceramic substrate is used because it does not have its own conductivity and the EC measurements are influenced only by the changes of the wet conductive coatings. Knowing the drying behaviors of the wet conductive coatings on ceramic substrate, the influence of the CFRP substrate on EC measurements can be investigated.

**Table 2. Properties of Additional Particles for Completing the Sample Plan
(allocated by Potters Industries)**

	Silver-coated Copper	Silver-coated Glass
Form of Particles	Flakes	Flakes
Averaged Size	3 μm – 15 μm	23 μm
Photograph from datasheet		

In accordance with requirements, next wet conductive coatings were deposited and measured:

1. High conductive coatings with coated area of 4cm $\times$ 4cm on ceramic substrate.
2. High conductive coatings with coated area of 4cm $\times$ 4cm on CFRP substrate.
3. High conductive coatings with coated area of 2cm $\times$ 2cm on ceramic substrate.
4. Low conductive coatings with coated area of 4cm $\times$ 4cm on ceramic substrate.

3.4.2 Application of the Wet Conductive Coatings

After preparation, wet conductive coatings are deposited on a ceramic substrate using the frame printing technique (Figure 2). The thickness and coated area of the coating depends only on the thickness and form of the frame. The copper frame is the same size as a ceramic substrate of 70mm $\times$ 70mm. In the center of the copper frame, a rectangular opening of 40mm $\times$ 40mm is etched; with this, the coated area is controlled and remains constant for all deposited coatings. The thickness of the frames are 80 μm , 160 μm and 240 μm , corresponding to the desired coating thickness. Wet conductive coatings are applied to the substrate using a scraper.

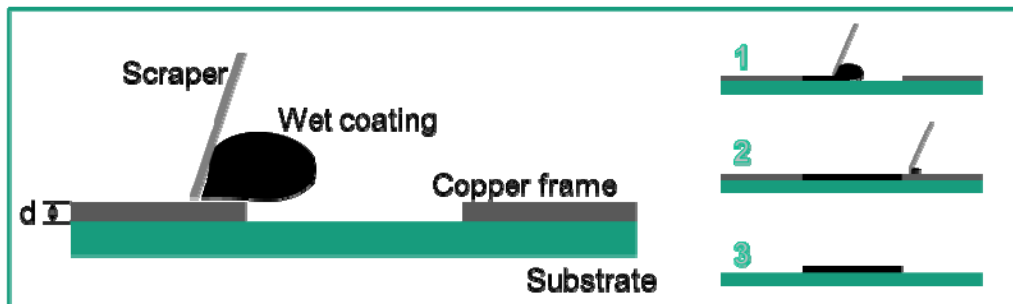


Figure 2. Schematic of the Frame Printing Technique

After application, wet conductive coatings are placed under the High-Frequency Eddy Current sensor and are cured for 24 hours at room temperature at normal conditions. At the same time, Eddy Current measurements are being performed at 30 second intervals over 24 hours (from beginning to the end of the drying); the lift-off remains constant at 80 μ m.

Due to the manual application process, the thickness of the coatings deviates from the desired value. Therefore, additional reference measurements were performed on the conductive coatings and used for analysis.

3.4.3 Measurements on Wet Conductive Coatings

High-Frequency Eddy Current Measurements

Based on the EddyCus[®] High-Frequency Eddy Current testing system designed and manufactured by Fraunhofer IKTS, a special system for wet coating characterization was developed that operates in a frequency range of 100kHz up to 20MHz (Figure 3).



Figure 3. Photograph of the Eddy Current Testing System for Laboratory Investigations

Figure 4 below illustrates how EddyCus[®] works in the radio wave range. This allows recording of the smallest conductivity changes of the specimen. Moreover, the High-Frequency Eddy Current system allows automatic data acquisition over more than 24 hours at 30 second intervals.

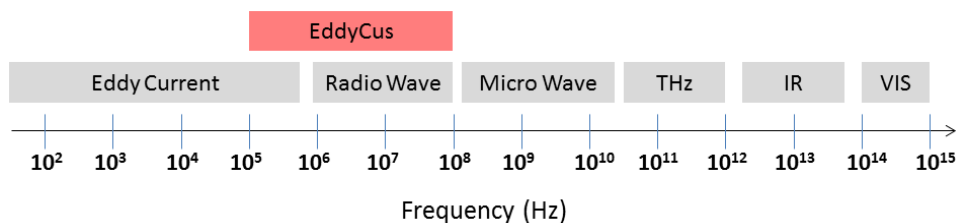


Figure 4. Drawing of the High Frequency Eddy Current Based Testing System

To prevent measurement drift over time, the EddyCus[®] has a self-referencing option that allows switching of the sensor with a bypass circle for reference measurements. Drifts caused by a shift in the room temperature or other environmental influences can be compensated for using this method. The self-referencing procedure is carried out before each measurement.

It is known that lift-off strongly influences Eddy Current measurements, which means that the lift-off should be as small as possible or should stay constant for each sample [4–6]. Therefore, the High-Frequency Eddy Current sensor is integrated into a precision positioning table that provides an exact lift-off adjustment of 10µm.

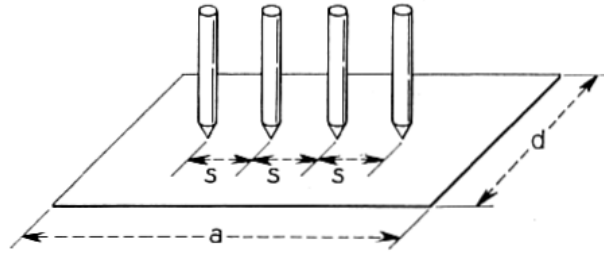
3.4.4 Reference Measurements

The final sheet resistivity and thickness of the conductive coatings were measured after curing and are listed in Table 2 to 5. The thickness was measured using the Laser Profilometer (Figure 5) and the sheet resistivity using the Keithley multi-meter (Figure 6) with a four-point probe [7].

Electrical resistance of the dried silver-coated copper particle samples was measured using Four-Point Probe process in the center of each coating. In accordance with the geometry of the coated area and distance between electrodes, which we use for measurements, the sheet resistivity is obtained as:

$$R_s = \left(\frac{V}{I}\right) \cdot C, \quad (1)$$

where C is the correction factor for various geometry $C\left(\frac{a}{d}; \frac{d}{s}\right)$. In our case $\frac{a}{d} = 1$, $\frac{d}{s} = 20$. In [7] correction factor C for 4cm×4cm geometry is given as $C = 4.4516$, for 2cm×2cm $C = 4.2209$.



$\left(\frac{V}{I}\right)$ is a value, that we obtain from the measurements using 2182A nano-voltmeter and 6221 DC and AC current source (KEITHLEY).

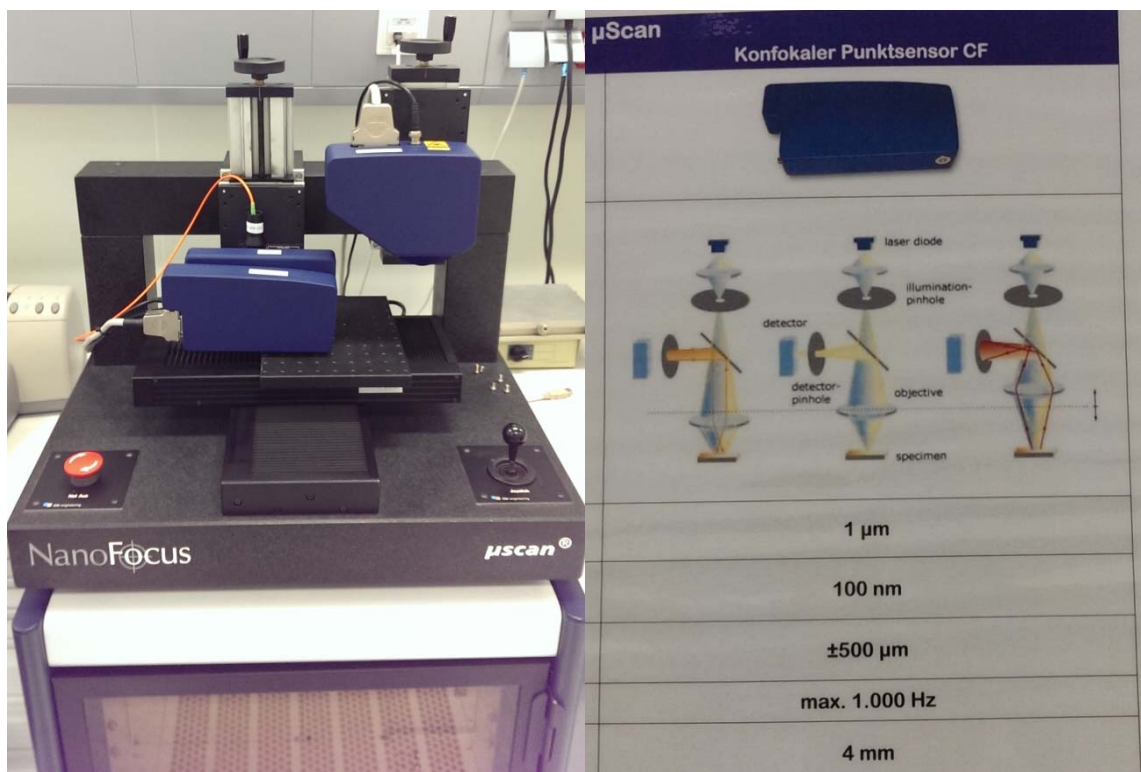


Figure 5. Photograph of the NanoFocus μ scan Laser Profilometer

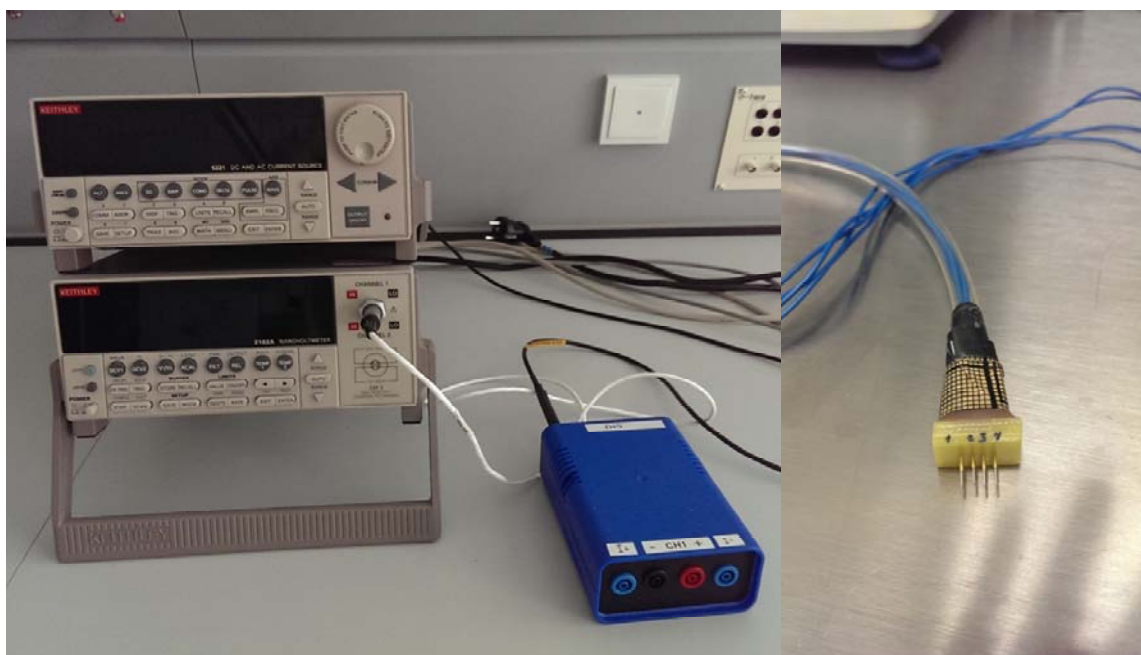


Figure 6. Photograph of the 2182A Nano-Voltmeter and 6221 DC/AC Current Source (KEITHLEY)

Table 3. Silver Coated Copper-based Layers on Ceramic Substrate with a Coated Area of 4×4cm

Screen printing frame thickness	Final thickness of the conductive coatings	Final sheet resistivity of the conductive coatings
Thin d=80μm	d ₁₋₁ =56-61μm	R _{F1-1} =71.67-138mΩ/□
Middle d=160μm	d ₁₋₂ =97-117μm	R _{F1-2} =28.05-43.18mΩ/□
Thick d=240μm	d ₁₋₃ =138-160μm	R _{F1-3} =22.7-27.16mΩ/□

Table 4. Silver Coated Copper-based Layers on CFRP Substrate with a Coated Area of 4cm×4cm

Screen printing frame thickness	Final thickness of the conductive coatings	Final sheet resistivity of the conductive coatings
Thin d=80μm	d ₂₋₁ =47-59.6μm	R _{F2-1} =67.7-89mΩ/□
Middle d=160μm	d ₂₋₂ =95-120μm	R _{F2-2} =40.1-54.8mΩ/□
Thick d=240μm	d ₂₋₃ =140-175μm	R _{F2-3} =26.7-44.5mΩ/□

Table 5. Silver Coated Copper-based Layers on Ceramic Substrate with a Coated Area of 2cm×2cm

Screen printing frame thickness	Final thickness of the conductive coatings	Final sheet resistivity of the conductive coatings
Thin d=80μm	d ₁ =82.1-88.1μm	R _{F3-1} =51.9-65.4mΩ/□
Middle d=160μm	d ₂ =88.3-101.6μm	R _{F3-2} =45.6-57.8mΩ/□
Thick d=240μm	d ₃ =144.5-167.5μm	R _{F3-3} =30.8-32.1mΩ/□

Table 6. Silver Coated Glass-based Layers on Ceramic Substrate with a Coated Area of 4cm×4cm

Screen printing frame thickness	Final thickness of the conductive coatings	Final sheet resistivity of the conductive coatings
Thin d=80μm	d ₁ =46-69.6μm	R _{F4-1} =436.3-934.8mΩ/□
Middle d=160μm	d ₂ =94-98μm	R _{F4-2} =287.6-338.3mΩ/□
Thick d=240μm	d ₃ =105-119μm	R _{F4-3} =164.7-267.1mΩ/□

4.0 RESULTS AND DISCUSSION

The data obtained by High-Frequency Eddy Current measurements are represented as real ($\text{Re}(U)$) and imaginary ($\text{Im}(U)$) part of the complex voltage over drying. For offset compensation, the Eddy Current drying curves were normalized to a value that was measured in the very first measurement of the drying period (after the sample is placed under the High Frequency Eddy Current Sensor). The Eddy Current signal shifts with the changing conductivity of the wet conductive coatings while drying. By matching the Eddy Current signal at different drying times with the final parameters of coatings, called references, a characterization of the long time drying behavior becomes possible, and is described in detail below.

The High-Frequency Eddy Current testing system operates at a frequency range of 100kHz to 20MHz, but only some frequencies provide an optimized possibility to characterize the drying behavior of the coatings deposited on ceramic and CFRP. Each frequency, ranging from 100kHz to 20MHz, was analyzed and only those that provide correlations to the final parameters of coatings are selected.

4.1 Frequency Selection

By choosing an optimal frequency for coatings analysis, two parameters are playing the important roles: the resistivity of the coatings and the lift-off. As mentioned above, the resistivity of the wet conductive coatings are being changed from the beginning of the drying until the coatings are completely dry. The lift-off is also changing as a byproduct of the decreasing thickness of the coatings during drying. Even slight variations in the lift-off, even that of 1 μm , deviation can cause changes in EC measurements. To find the frequency that provides measurements with minimal lift-off influences while the amplitude of the signal is high enough to record the resistivity changes over the duration of the drying process, an additional experiment was performed.

Two thicknesses (thin and thick) of each type of wet conductive coatings were deposited (high conductive coatings with coated area of 4 $\times$ 4cm on ceramic substrate, high conductive coatings with coated area of 4 $\times$ 4cm on CFRP substrate, high conductive coatings with coated area of 2 $\times$ 2cm on ceramic substrate, low conductive coatings with coated area of 4 $\times$ 4cm on ceramic substrate), EC measurements with a lift-off of 1mm were performed after curing. Then a certain coefficient was artificially created and is given by:

$$K_1 = \frac{Re_1 - Re_2}{Re_3 - Re_2} \quad K_2 = \frac{Im_1 - Im_2}{Im_3 - Im_2},$$

Re_1 – Real Part complex Voltage after first measurement at a lift-off of 100 μm ;

Re_2 – Real Part complex Voltage at the end of the drying at a lift-off of 100 μm ;

Re_3 – Real Part complex Voltage at the end of the drying at a lift-off of 1mm;

Im_1 – Imaginary Part complex Voltage after first measurement at a lift-off of 100 μm ;

Im_2 – Imaginary Part complex Voltage at the end of the drying at a lift-off of 100 μm ;

Im_3 – Imaginary Part complex Voltage at the end of the drying at a lift-off of 1mm.

From a physical standpoint, that coefficient means that the smaller the difference of EC Amplitudes $Re_3 - Re_2$ and higher $Re_1 - Re_2$, the higher is the value of characteristic coefficient, and thereby the lower is the influence of lift-off on EC Signals. Figures 7 through 10 show characteristic coefficients K1 and K2 over the frequency for different coatings.

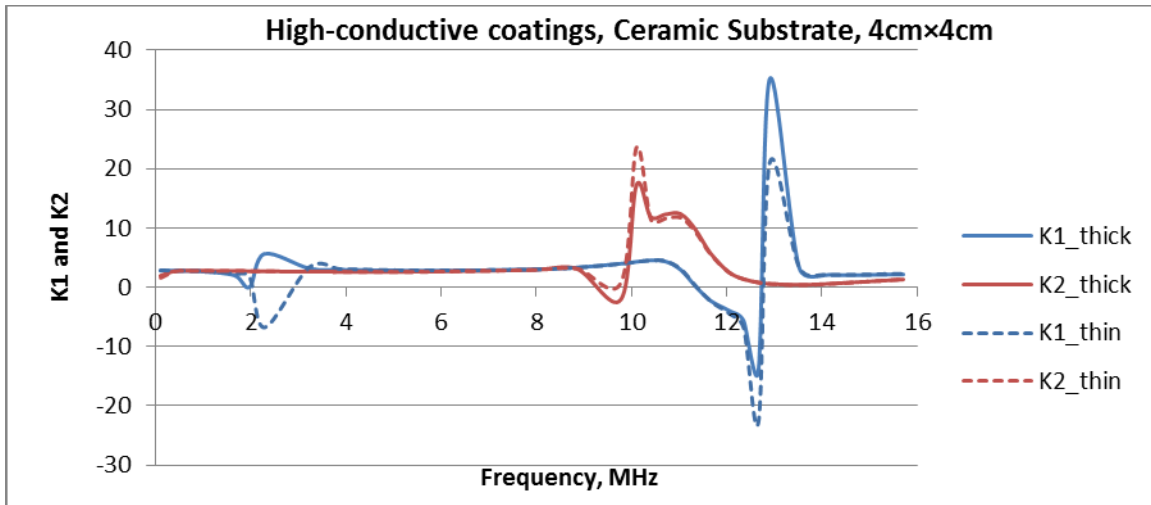


Figure 7. Characteristic Coefficients K1 and K2 over the Frequency for High Conductive Coatings with Coated Area of 4cm×4cm on Ceramic Substrate with Two Thicknesses, Thin and Thick

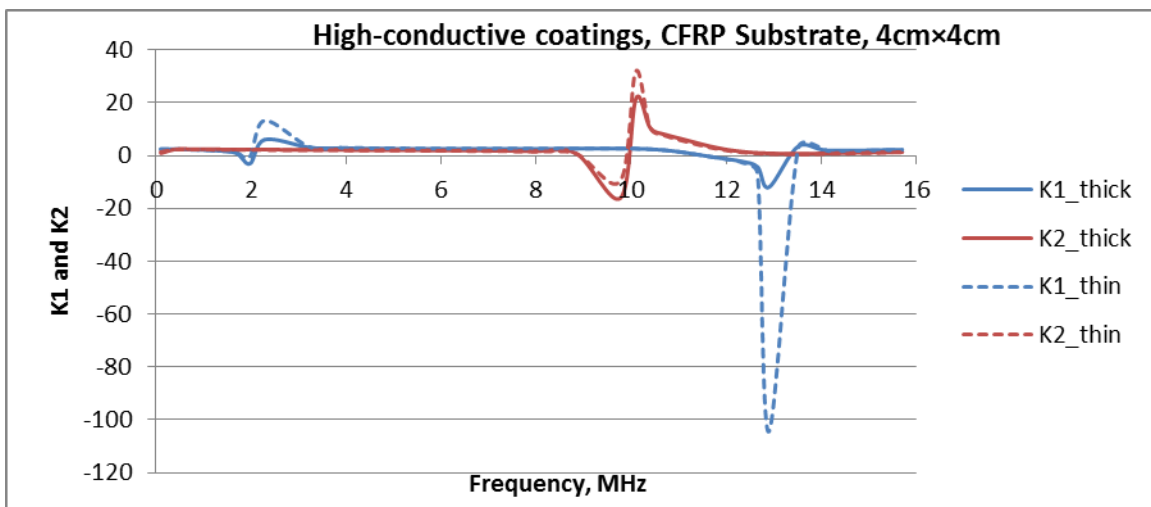


Figure 8. Characteristic Coefficients K1 and K2 over the Frequency for High Conductive Coatings with Coated Area of 4cm×4cm on CFRP Substrate with Two Thicknesses, Thin and Thick

Figure 7 and Figure 8 above show that at a frequency of 9.8MHz and 10.1MHz the K2 (imaginary part) has the higher value, whereas K1 (real part) is staying the same for layers with different thicknesses; moreover, these two frequencies are equal for high-conductive coatings with a coated area of 4cm×4cm on ceramic and CFRP substrates. The higher absolute value of K1 (real part) is present at frequencies of 12.664MHz and 12.9MHz for layers on CFRP substrate and of 12.9MHz and 13.523MHz for layers on ceramic substrate. The frequency of the 12.9MHz allows the better opportunity for separating layers having different thicknesses on different substrates due to layers on ceramic substrate K1>0 and it is its maximum, and for layers

on CFRP $K1 < 0$ and it is its minimum. It was experimentally proven that exactly these frequencies provide measurements with the highest accuracy of correlations. These frequencies were selected for the analysis. In the same way, an optimal frequency can be selected for two other coatings: high conductive with coated area of $2\text{cm} \times 2\text{cm}$ on ceramic substrate and low conductive with coated area of $4\text{cm} \times 4\text{cm}$ on ceramic substrate (Figures 9 and 10).

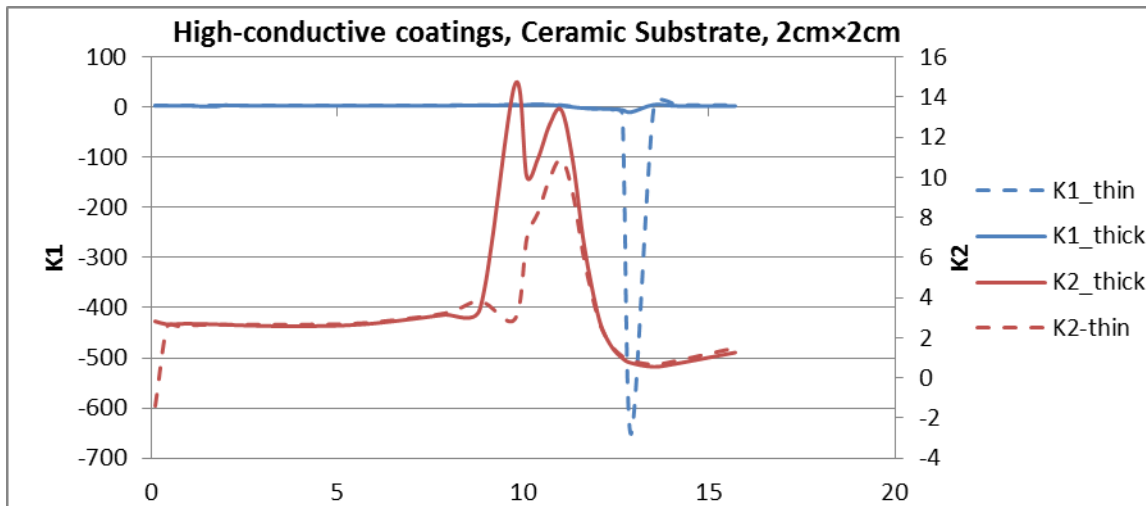


Figure 9. Characteristic Coefficients K1 and K2 over the Frequency for High Conductive Coatings with Coated Area of $2\text{cm} \times 2\text{cm}$ on Ceramic Substrate with Two Thicknesses: Thin and Thick

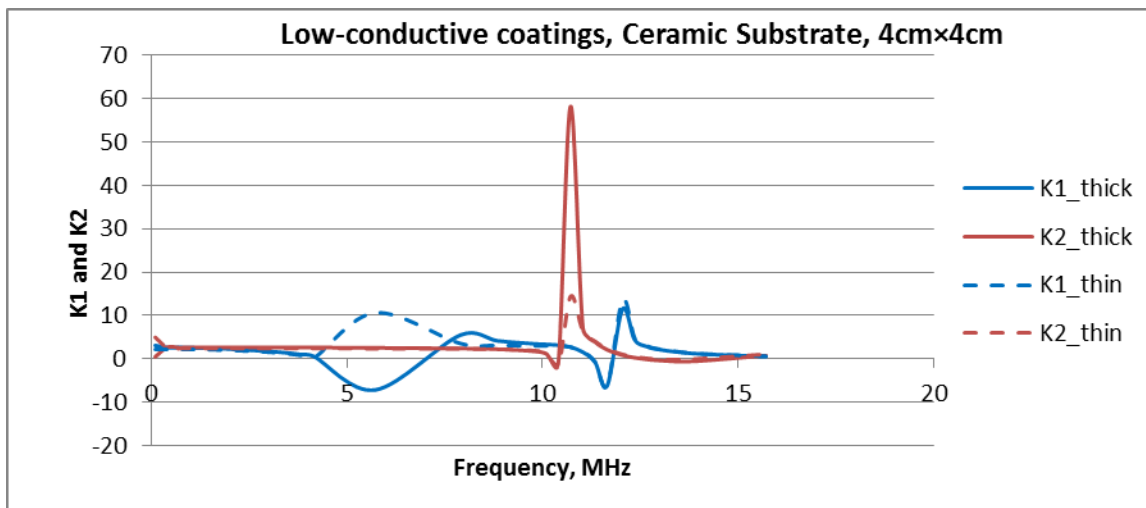


Figure 10. Characteristic Coefficients K1 and K2 over the Frequency for Low Conductive Coatings with Coated Area of $4\text{cm} \times 4\text{cm}$ on Ceramic Substrate with Two Thicknesses: Thin And Thick

This is the method to select an optimal frequency automatically.

4.2 Drying Behavior of Wet Conductive Coatings

Based on EC measurements, the drying behaviors of the wet conductive coatings can be visualized and described as follows: there are three dominating states during drying and these three states have both, high and low conductive coatings (Figure 3):

- State 1 occurs when the coatings are liquid and the chemicals used as a thinner evaporate and particle percolation starts.
- State 2 occurs while the coatings are still wet, the added chemicals are evaporated and polymerization is in progress.
- State 3 characterizes the end of the polymerization processes of the coatings.

State 1 can also be called a “percolation threshold” [1]. This is the period of time during the drying, when the properties of the coating are strongly changed according to the rapid evaporation of the chemicals. High conductive coatings have a distinctive feature in State 1, a “characteristic point”, whereas low conductive coatings do not. It was experimentally established, that the “characteristic point” provides an opportunity to characterize coatings while they are liquid in accordance to the correlations between amplitude & final sheet resistivity and time of the “characteristic point” & final thickness. Correlations for the silver-coated glass-based layers are not possible in this state.

State 2 occurs after the percolation threshold, when the added chemicals are evaporated and properties of the conductive coatings are changing slowly. During this state the polymerization process is in progress.

The coatings are wet in State 2, so repairs are possible. There are correlations for all measured conductive coatings and the characterization of each of them is possible.

Sometime after polymerization process has started, the coatings are hardened enough and their properties are almost unchanged or are not changed at all. This characterizes State 3, the end of the polymerization. In this state, no repairs are possible but correlations for all measured conductive coatings and the characterization of each of them is possible.

The time when the States 1, 2 or 3 occur depends on the chemical composition, environmental conditions, temperature of the curing, thickness of the coating, coated area, etc.

Figure 11 shows drying behaviors for silver-coated glass-based (top) and silver-coated copper-based (bottom) layers. Schematic representations show States 1, 2 and 3 for these coatings. For high conductive coatings it is observed, that the thicker the coating, the later a “characteristic point” occurs and the lower the amplitude is of the EC signal. It can be seen in Figure 3 that the type of the particles used as a conductive filling strongly influences the percolation processes in the wet coatings during drying and their final conductivity. It is also recognized, that the time when States 1, 2 and 3 occur stays the same for layers based on copper and glass particles. This means that the time of percolation, polymerization start and end does not depend on the type of the particles, but on the type of polymer matrix and the chemicals used as a thinner.

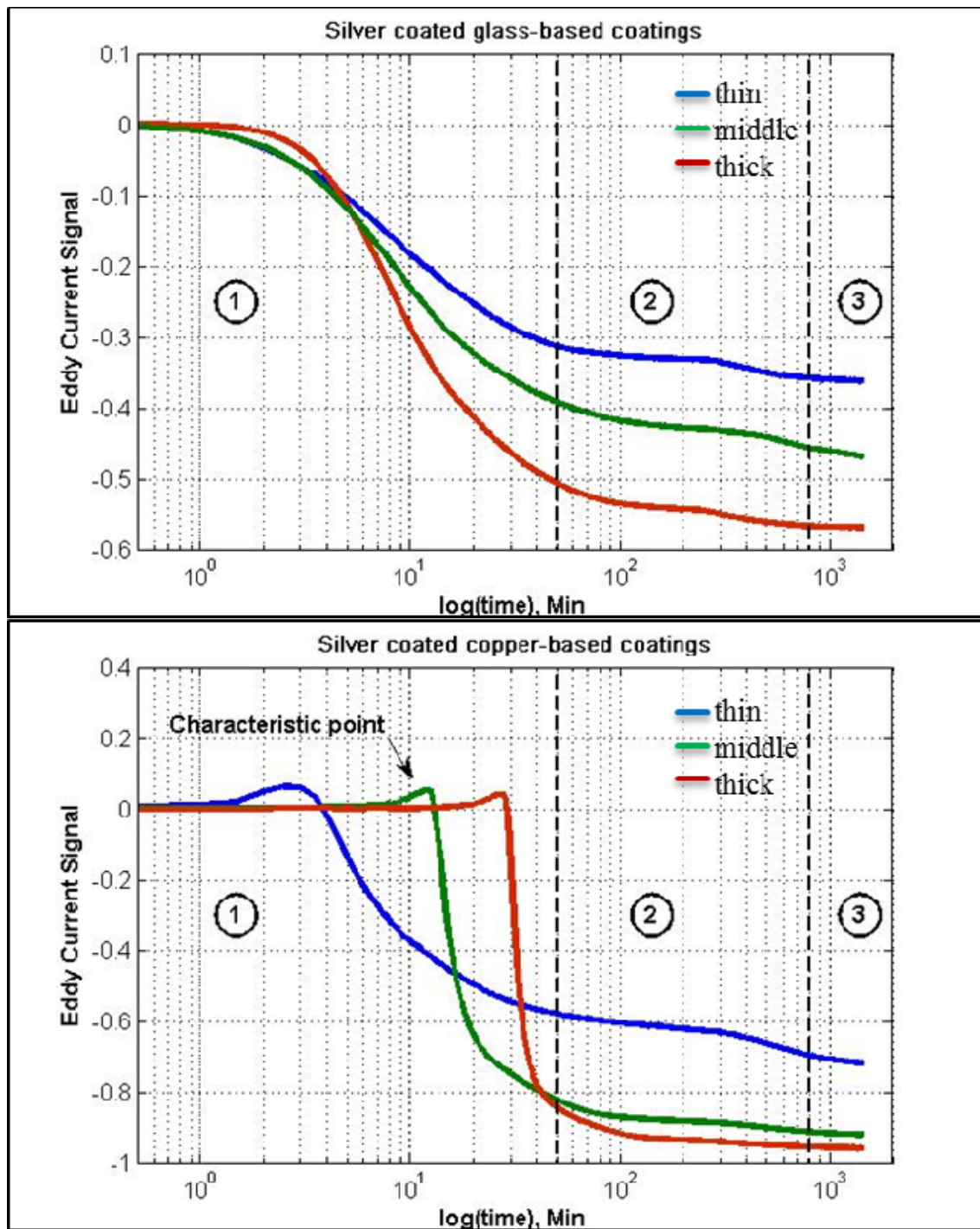


Figure 11. Schematic Representation of the Drying Behaviors for Low (Top) and High (Bottom) Conductive Coatings

Conclusion: final sheet resistivity and drying behaviors in State 1 depends on the type of particles; the duration of each state and the time when the “characteristic point” occurs, depends on the type of the polymer and thinner. Knowing the drying behaviors, the application process of the wet conductive coatings can be controlled.

4.3 Characterization in State 1

Characterization in State 1 is only possible for layers having a characteristic point. Using the reference measurements, the following correlations are possible.

4.3.1 High Conductive Coatings with Coated Area of 4cm×4cm on Ceramic Substrate

- Correlations between the real part of the complex voltage at the characteristic point at a frequency of 10.1MHz and the final sheet resistivity (Figure 12).
- Correlations between the time of the characteristic point of the real part of the complex voltage at a frequency of 10.1MHz and the final thickness (Figure 13).
 - ❖ Absolute deviation, 18.26 mΩ/□
 - ❖ Relative deviation, 26.11 %

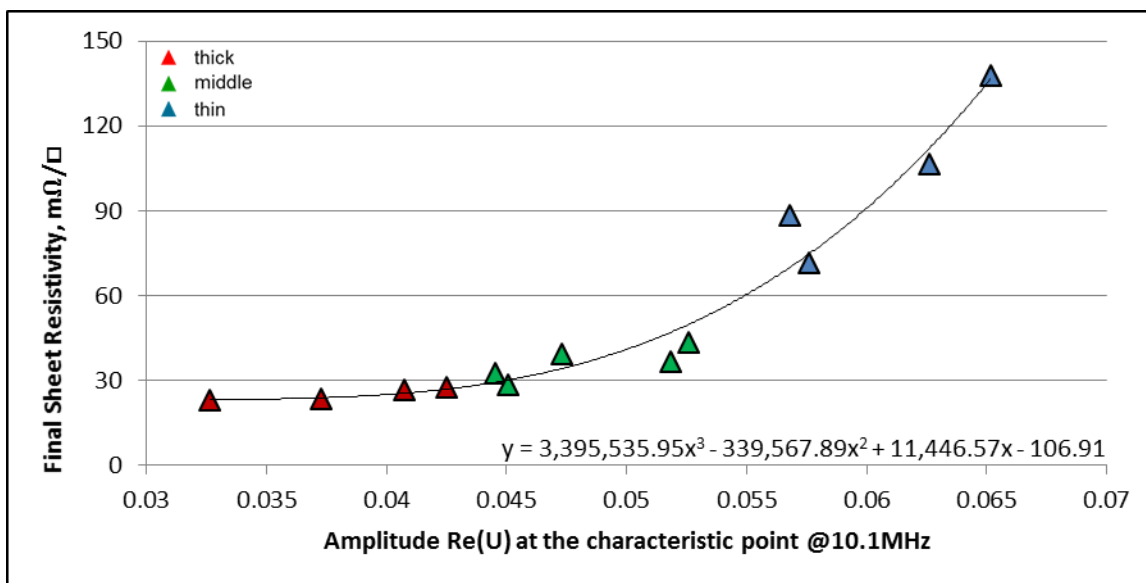


Figure 12. Final Sheet Resistivity as a Function of the Amplitude Re(U) of the Complex Voltage at the Characteristic Point at a Frequency of 10.1MHz for High Conductive Coatings on Ceramic Substrate with a Coated Area of 4cm×4cm

- ❖ Absolute deviation, 12.09 μm
- ❖ Relative deviation, 20.64 %

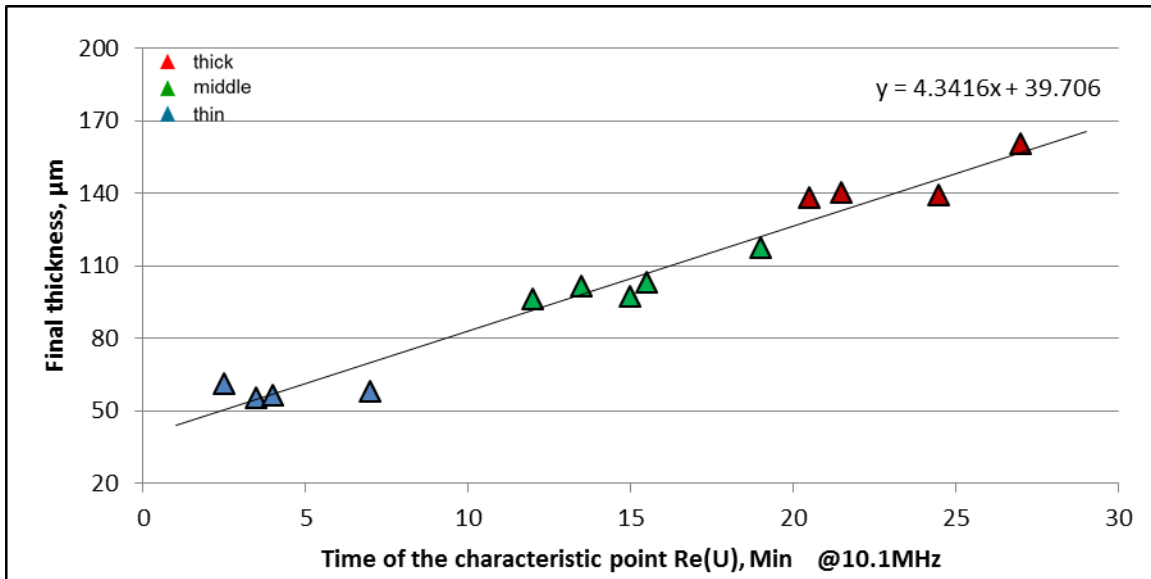


Figure 13. Final Thickness as a Function of the Time of the Characteristic Point Re(U) of the Complex Voltage at a Frequency of 10.1MHz for High Conductive Coatings on Ceramic Substrate with a Coated Area of 4cm×4cm

4.3.2 High Conductive Coatings with Coated Area of 4cm×4cm on CFRP Substrate

- Even though the coatings have a characteristic point, there are no correlations in State 1 because CFRP substrates have their own conductivity and the conductivity of wet coatings in the beginning of drying is very low, which strongly influences the EC measurements.

4.3.3 High Conductive Coatings with Coated Area of 2cm×2cm on Ceramic Substrate

- Results show that it is possible to characterize these conductive coatings using the correlations between a time of the real part of the complex voltage at the characteristic point at a frequency of 9.8MHz and final sheet resistivity (Figure 14).
- Correlations between a time of the real part of the complex voltage at the characteristic point at a frequency of 9.8MHz and final thickness (Figure 15).

- ❖ Absolute deviation, 3.7 mΩ/□
- ❖ Relative deviation, 6.01 %

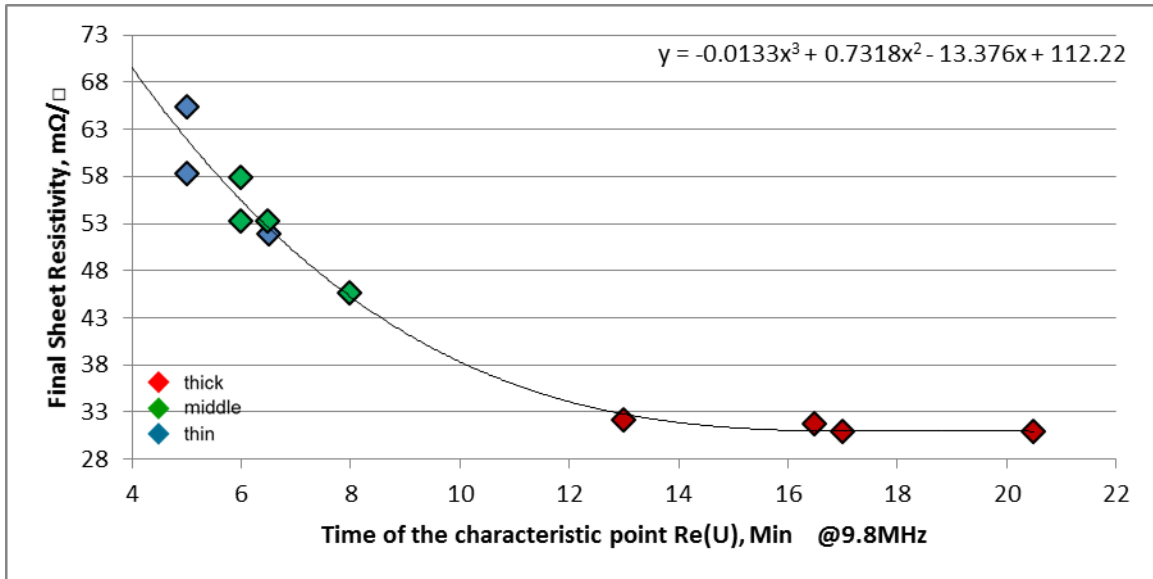


Figure 14. Final Sheet Resistivity as a Function of The Time of the Characteristic Point Re(U) of the Complex Voltage at a Frequency of 9.8MHz for High Conductive Coatings On Ceramic Substrate with a Coated Area Of 2cm×2cm

- ❖ Absolute deviation, 7.48 mΩ/□
- ❖ Relative deviation, 5.45 %

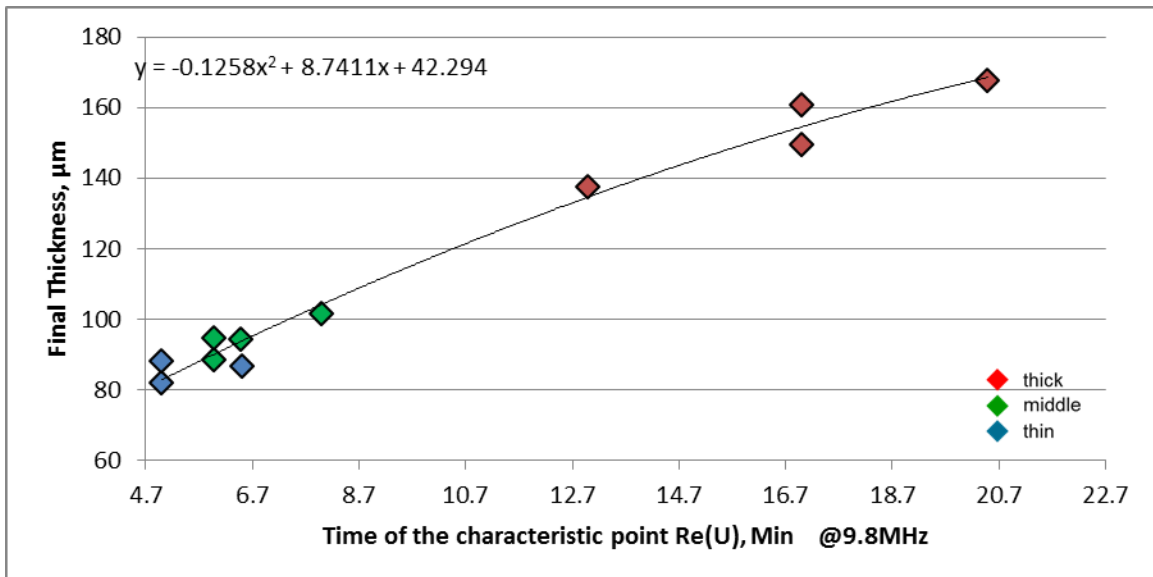


Figure 15. Final Thickness as a Function of the Time of the Characteristic Point Re(U) of the Complex Voltage at a Frequency of 9.8MHz for High Conductive Coatings on Ceramic Substrate with a Coated Area of 2cm×2cm

4.3.4 Low Conductive Coatings with Coated Area of 4×4cm on Ceramic Substrate

- Low conductive coatings do not have a “characteristic point” during drying and thereby, no correlations are possible in State 1.

4.4 Characterization in State 2

Characterization of each of coating is possible in State 2. The time when State 2 occurs is approximately 70 minutes after deposition for all coatings used in this work. EC measurements can be performed 70 minutes after depositing, without performing them during the first hour.

4.4.1 High Conductive Coatings with Coated Area of 4cm×4cm on Ceramic Substrate

- Correlations between the amplitude of the real part (Figure 16) and/or the amplitude of the absolute value of the complex voltage (Figure 17) at a frequency of 13MHz and the final sheet resistivity.
- ❖ Absolute deviation, 6.75 mΩ/□
 - ❖ Relative deviation, 20.63 %

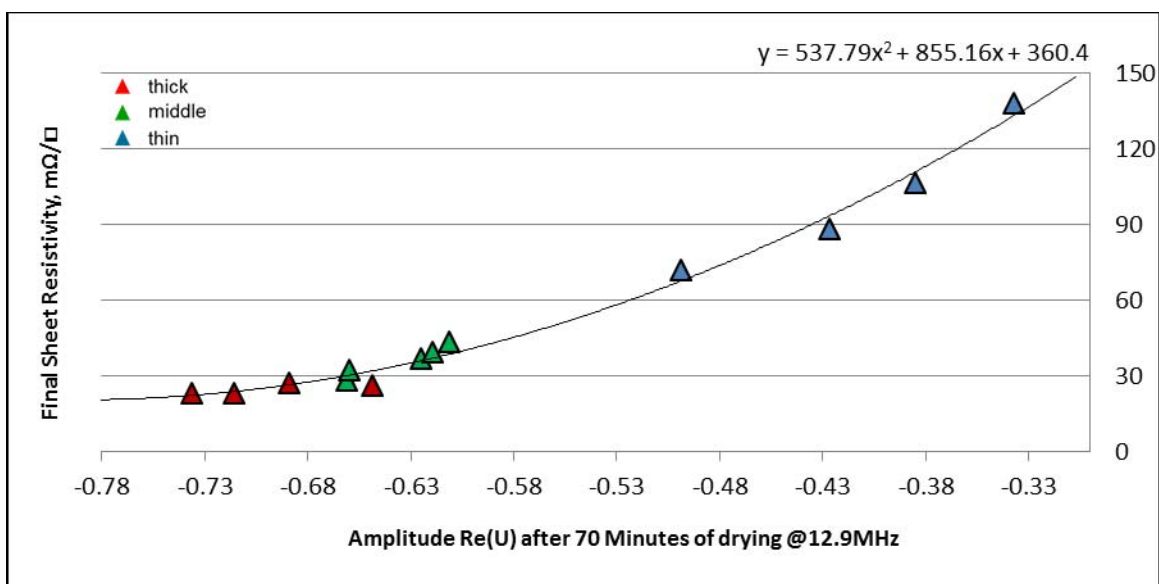


Figure 16. Final Sheet Resistivity as a Function of the Amplitude Re(U) of the Complex Voltage After 70 Minutes of Drying at a Frequency of 12.9MHz for High Conductive Coatings on Ceramic Substrate with a Coated Area of 4cm×4cm

- ❖ Absolute deviation, 5.23 mΩ/□
- ❖ Relative deviation, 19.53 %

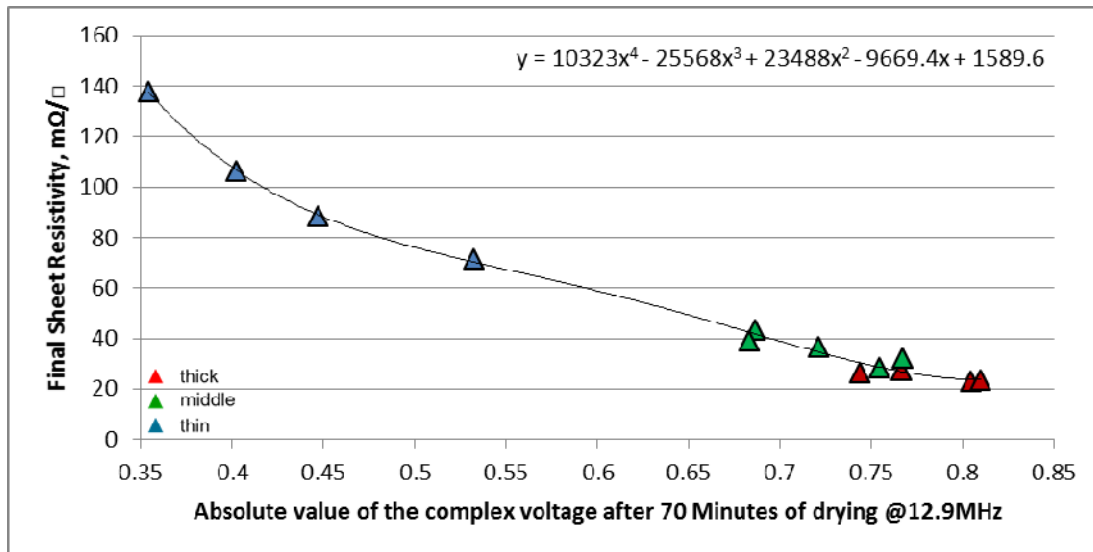


Figure 17. Final Sheet Resistivity as a Function of the Absolute Value of the Complex Voltage After 70 Minutes of Drying at a Frequency of 12.9MHz for High Conductive Coatings on Ceramic Substrate with a Coated Area of 4cm×4cm

4.4.2 High Conductive Coatings with Coated Area of 4×4cm on CFRP Substrate

- Correlations between the amplitude of the real part (Figure 18) and/or the absolute value of the complex voltage (Figure 19) at a frequency of 13MHz and the final sheet resistivity.

- ❖ Absolute deviation, 7.88 mΩ/□
- ❖ Relative deviation, 21.51 %

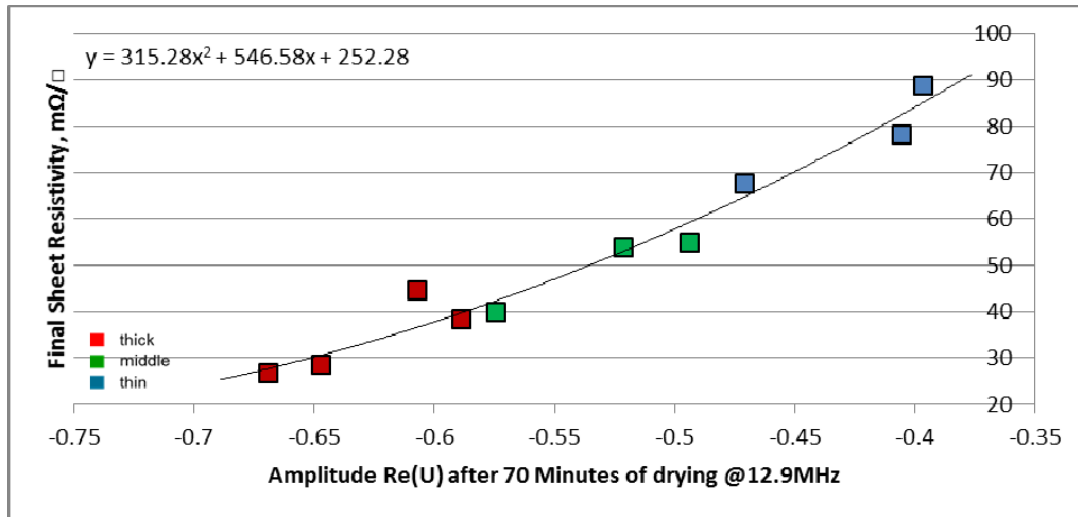


Figure 18. Final Sheet Resistivity as a Function of the Amplitude Re(U) of the Complex Voltage after 70 Minutes of Drying at a Frequency of 12.9MHz for High Conductive Coatings on CFRP Substrate with a Coated Area of 4cm×4cm

- ❖ Absolute deviation, 5.48 mΩ/□
- ❖ Relative deviation, 7.99 %

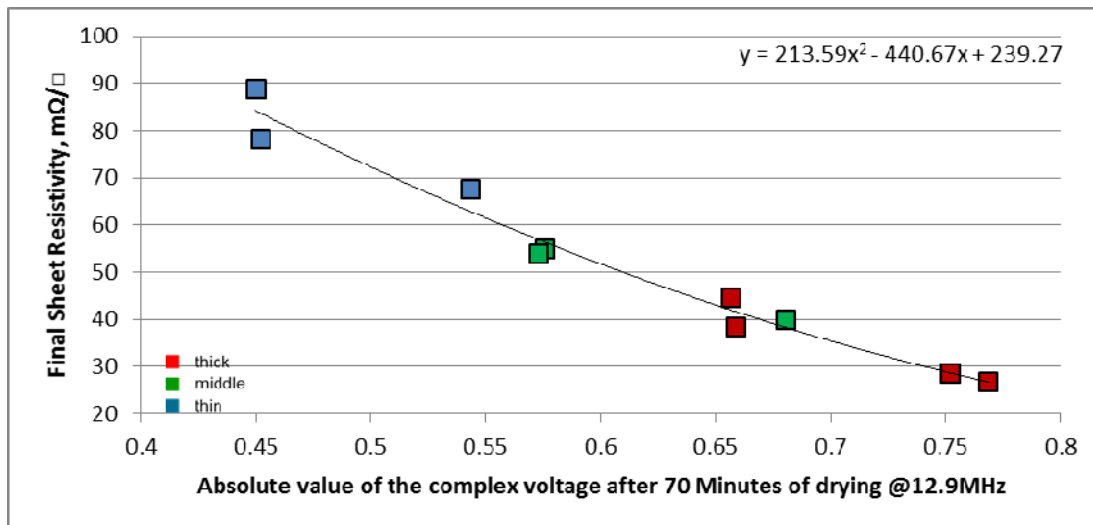


Figure 19. Final Sheet Resistivity as a Function of the Absolute Value of the Complex Voltage after 70 Minutes of Drying at a Frequency of 12.9MHz for High Conductive Coatings on CFRP Substrate with a Coated Area of 4cm×4cm

4.4.3 High Conductive Coatings with Coated Area of 2cm×2cm on Ceramic Substrate

- Correlations between the absolute value of the complex voltage at a frequency of 12.9MHz and the final sheet resistivity (Figure 20).
- Correlations between the amplitude of the imaginary part of the complex voltage at a frequency of 10MHz and the final sheet resistivity (Figure 21).

❖ Absolute deviation, 4.36 mΩ/□

❖ Relative deviation, 8.7 %

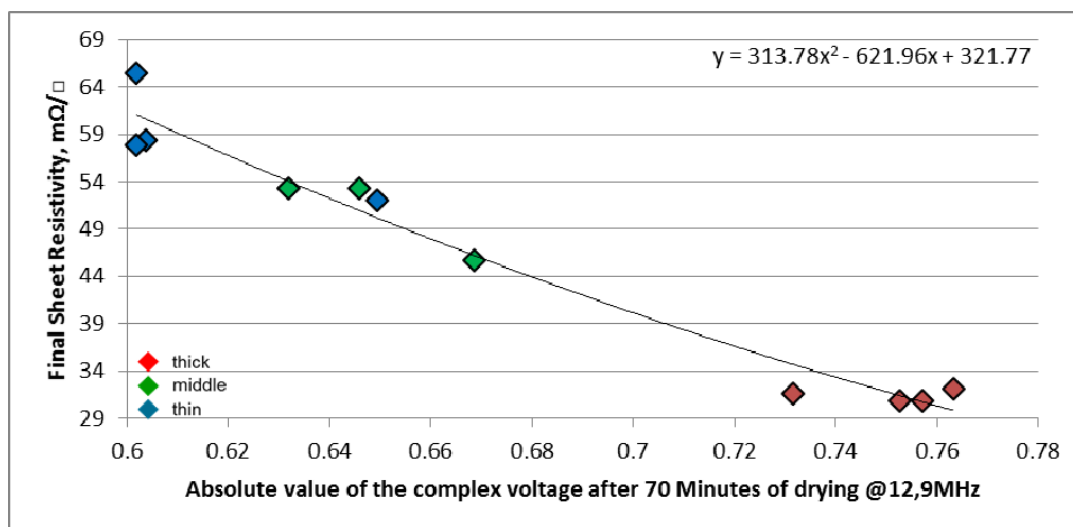


Figure 20. Final Sheet Resistivity as a Function of the Absolute Value of the Complex Voltage after 70 Minutes of Drying at a Frequency of 12.9MHz for High Conductive Coatings on Ceramic Substrate with a Coated Area of 2cm×2cm

❖ Absolute deviation, 4.25 mΩ/□

❖ Relative deviation, 9.6 %

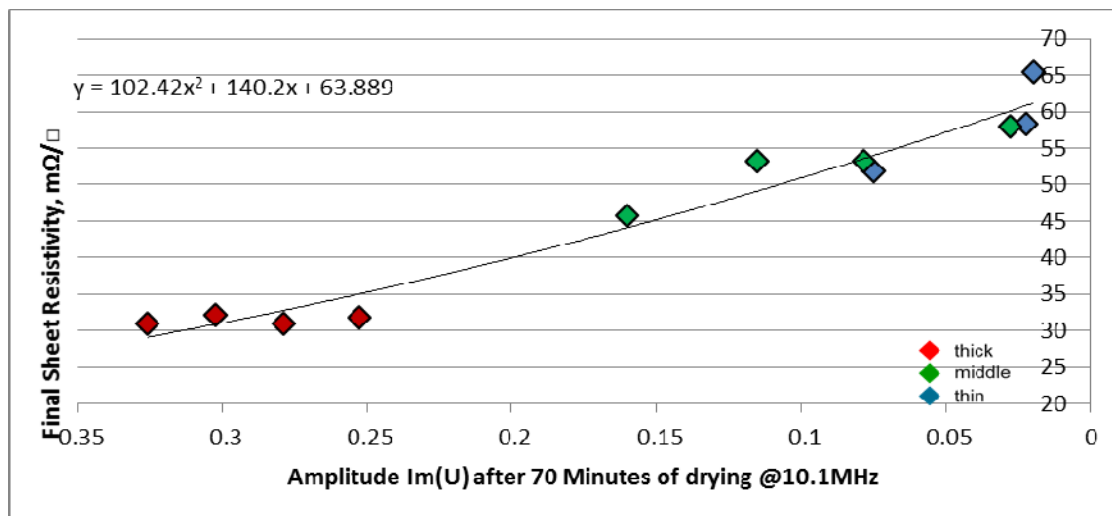


Figure 21. Final Sheet Resistivity as a Function of the Amplitude Im(U) of the Complex Voltage After 70 Minutes of Drying at a Frequency of 10.1MHz for High Conductive Coatings on Ceramic Substrate with a Coated Area of 2cm×2cm

4.4.4 Low Conductive Coatings with Coated Area of 4cm×4cm on Ceramic Substrate

- Correlation between the amplitude of the real part of the complex voltage (Figure 22) and/or the absolute value of the complex voltage (Figure 23) at a frequency of 5.7MHz and the final sheet resistivity.

❖ Absolute deviation, 37.07 mΩ/□

❖ Relative deviation, 12.49 %

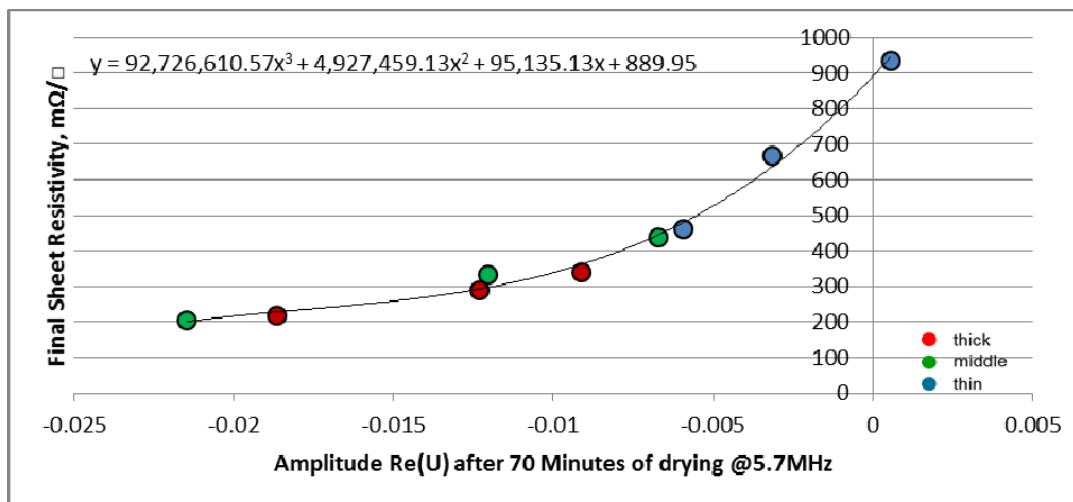


Figure 22. Final Sheet Resistivity as a Function of the Amplitude Re(U) of the Complex Voltage After 70 Minutes of Drying at a Frequency of 5.7MHz for Low Conductive Coatings on Ceramic Substrate with a Coated Area of 4cm×4cm

❖ Absolute deviation, 28.55 mΩ/□

❖ Relative deviation, 9.35 %

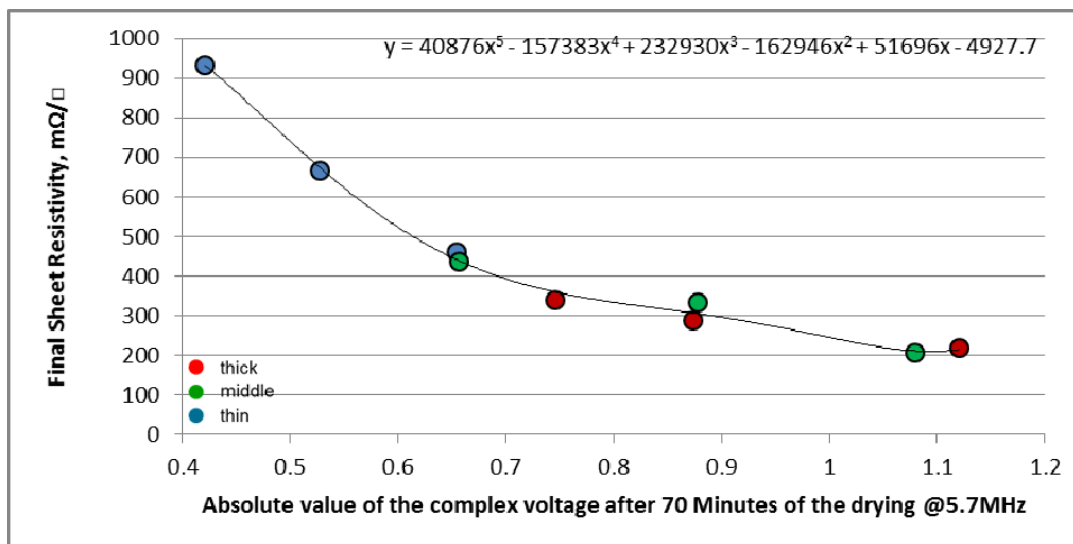


Figure 23. Final Sheet Resistivity as a Function of the Absolute Value of the Complex Voltage after 70 Minutes of Drying at A Frequency of 5.7MHz for Low Conductive Coatings on Ceramic Substrate with a Coated Area of 4cm×4cm

4.5 Characterization in State 3

4.5.1 Silver Coated Copper Based Layers with Coated Area of 4cm×4cm on Ceramic Substrate

- Correlation between the amplitude of the Absolute value of the Complex Voltage at a frequency of 12.9MHz and the final Sheet Resistivity (Figure 24).
- Correlation between the amplitude of the Imaginary Part of the Complex Voltage at a frequency of 10.1MHz and the final Sheet Resistivity (Figure 25).
- Correlation between the amplitude of the Real Part of the Complex Voltage at a frequency of 12.9MHz and the final Sheet Resistivity (Figure 26).

❖ Absolute deviation, 3.74 mΩ/□

❖ Relative deviation, 9.07 %

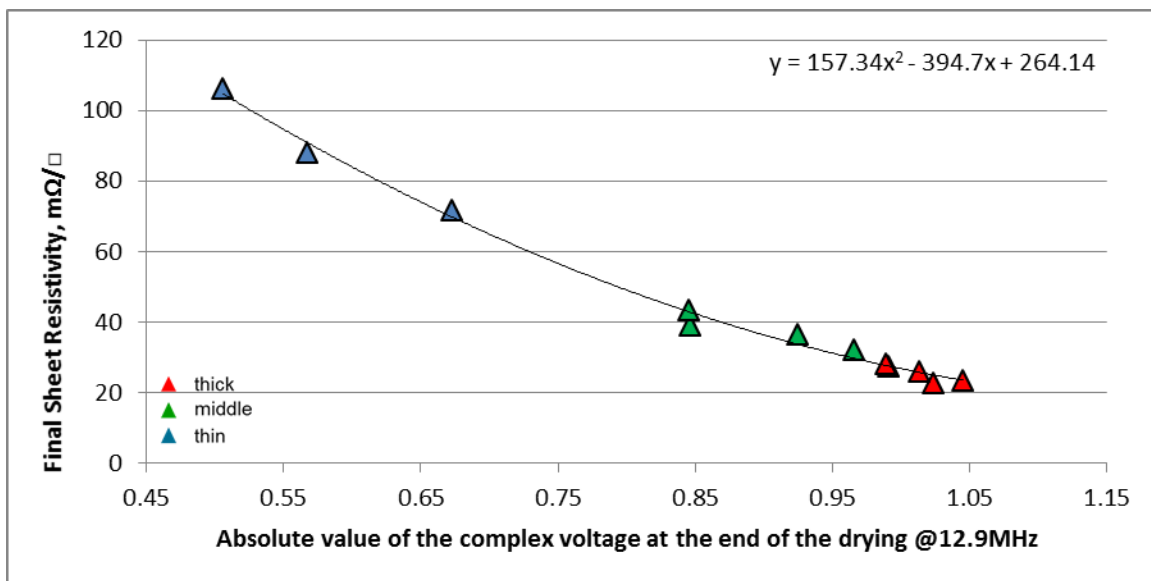


Figure 24. Final Sheet Resistivity as a Function of the Amplitude Re(U) of the Complex Voltage at the End of the Drying at a Frequency of 12.9MHz for High Conductive Coatings on Ceramic Substrate with a Coated Area of 4cm×4cm

- ❖ Absolute deviation, 3.96 mΩ/□
- ❖ Relative deviation, 7.74 %

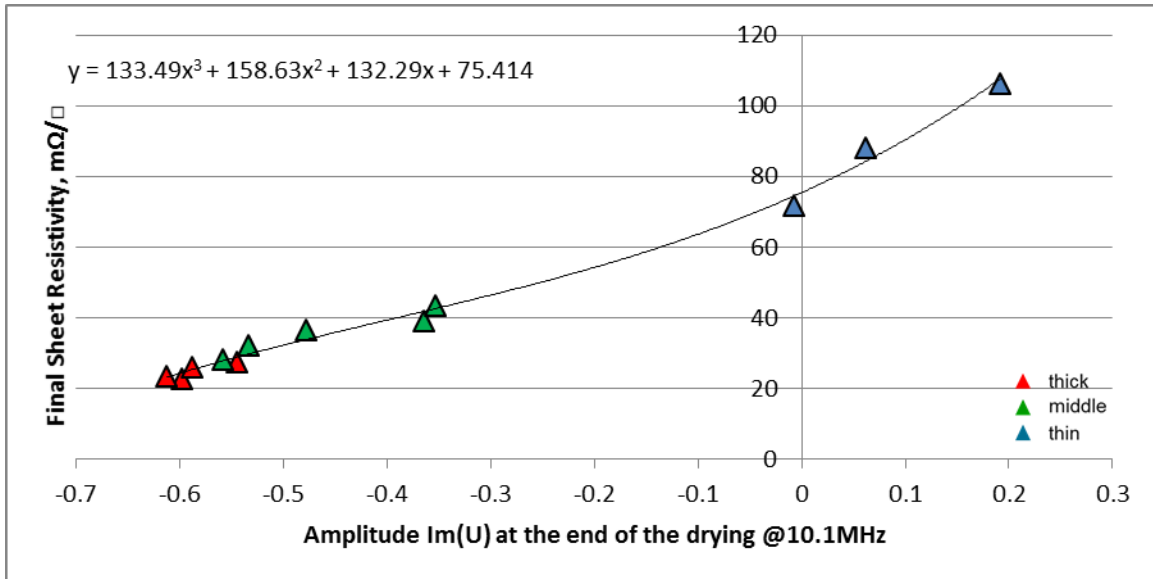


Figure 25. Final Sheet Resistivity as a Function of the Amplitude Im(U) of the Complex Voltage at the End of the Drying at a Frequency of 10.1MHz for High Conductive Coatings on Ceramic Substrate with a Coated Area of 4cm×4cm

- ❖ Absolute deviation, 3.38 mΩ/□
- ❖ Relative deviation, 11.83 %

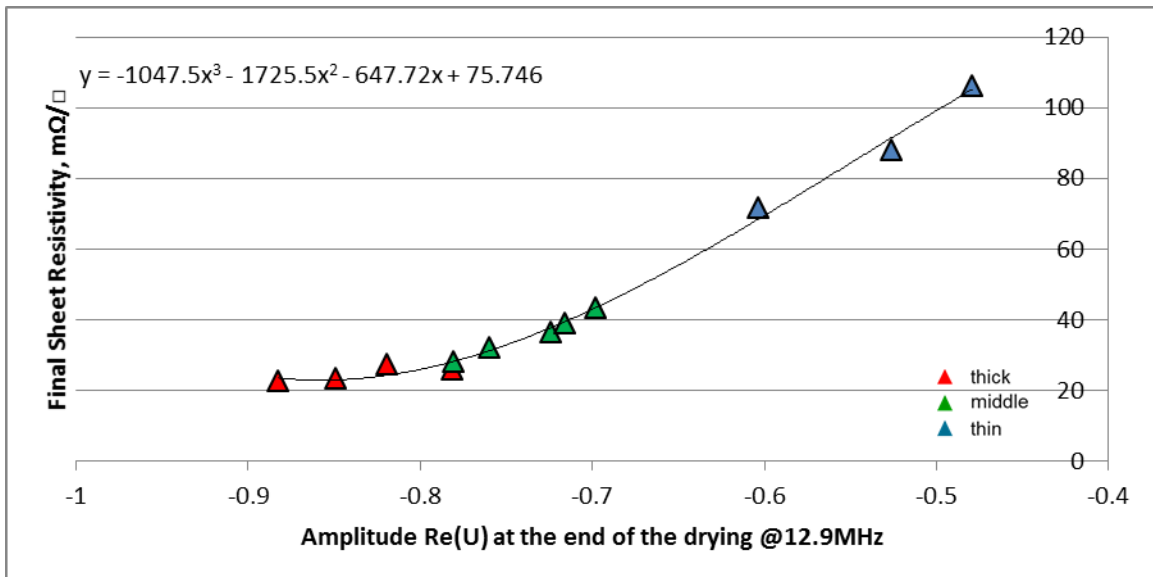


Figure 26. Final Sheet Resistivity as a Function of the Amplitude Re(U) of the Complex Voltage at the End of the Drying at a Frequency of 12.9MHz for High Conductive Coatings on Ceramic Substrate with a Coated Area of 4cm×4cm

4.5.2 Silver Coated Copper Based Layers with Coated Area of 4cm×4cm on CFRP Substrate

- Correlation between the amplitude of the Real Part of the Complex Voltage at a frequency of 12.9MHz and the final thickness (Figure 27).
- Correlation between the amplitude of the Real Part of the Complex Voltage at a frequency of 12.9MHz and the final Sheet Resistivity (Figure 28).
- Correlation between the amplitude of the Absolute value of the Complex Voltage at a frequency of 12.9MHz and the final Sheet Resistivity (Figure 29).

❖ Absolute deviation, 13.55 mΩ/□

❖ Relative deviation, 10.82 %

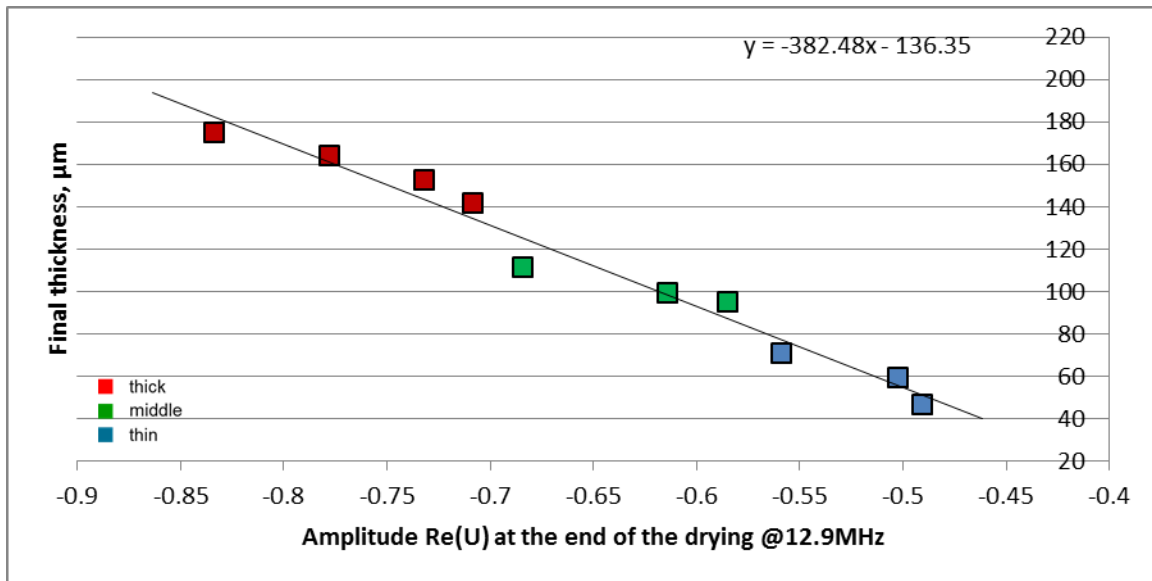


Figure 27. Final Thickness as a Function of the Amplitude Re(U) of the Complex Voltage at the End of the Drying at a Frequency of 12.9MHz for High Conductive Coatings on CFRP Substrate with a Coated Area of 4cm×4cm

- ❖ Absolute deviation, 5.96 mΩ/□
- ❖ Relative deviation, 15.47 %

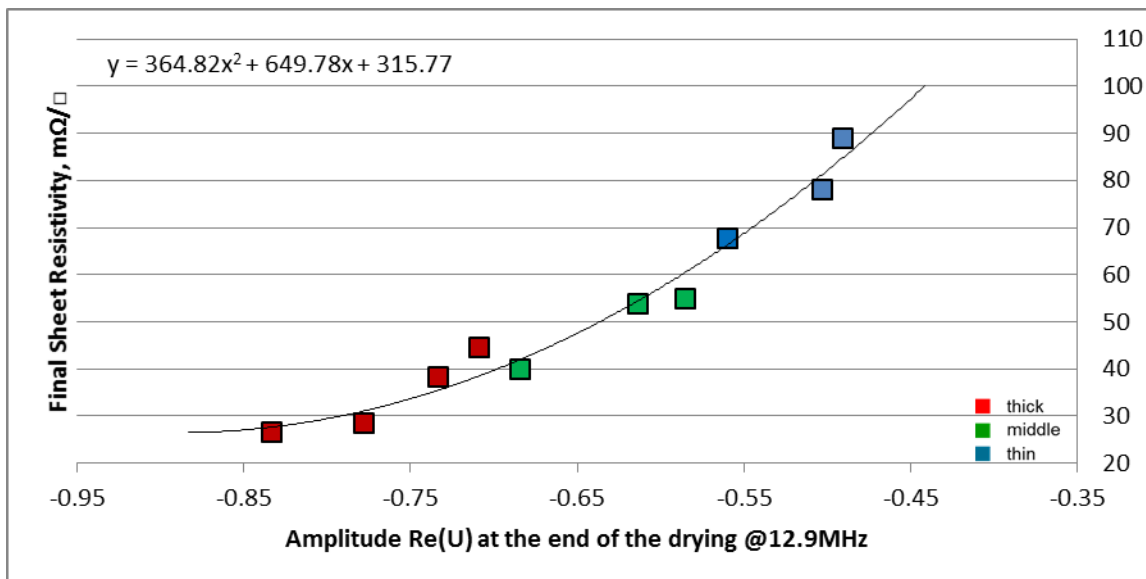


Figure 28. Final Sheet Resistivity as a Function of the Amplitude Re(U) of the Complex Voltage at the End of the Drying at a Frequency of 12.9MHz for High Conductive Coatings on CFRP Substrate with a Coated Area of 4cm×4cm

- ❖ Absolute deviation, 7.15 mΩ/□
- ❖ Relative deviation, 8.93 %

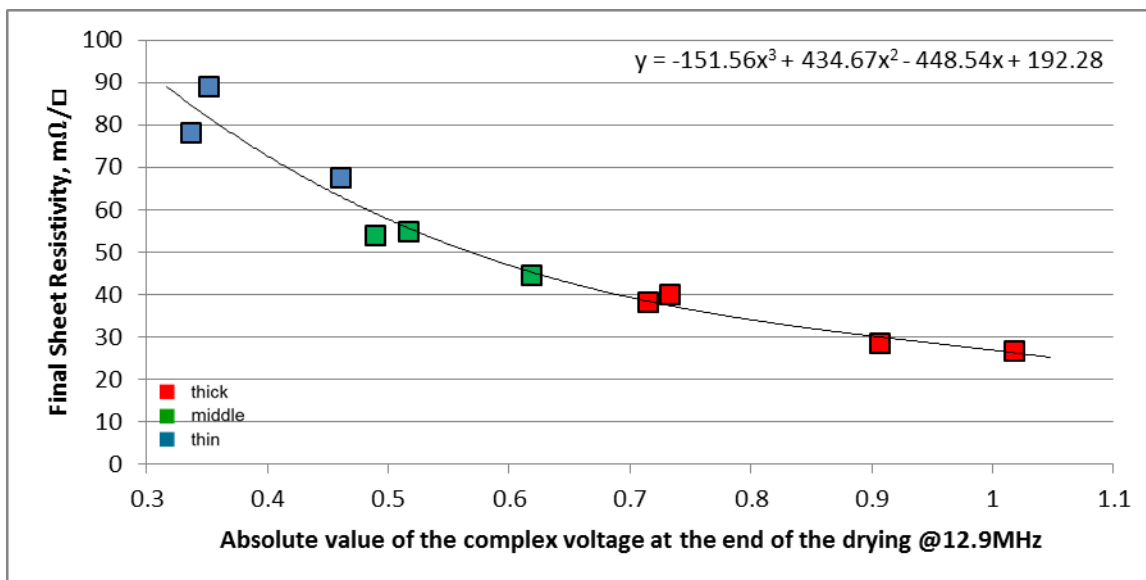


Figure 29. Final Sheet Resistivity as a Function of the Absolute Value of the Complex Voltage at the End of the Drying at a Frequency of 12.9MHz for High Conductive Coatings on CFRP Substrate with a Coated Area of 4cm×4cm

4.5.3 Silver Coated Copper Based Layers with Coated Area of 2×2cm on Ceramic Substrate

- Correlation between the amplitude of the Absolute value of the Complex Voltage at a frequency of 12.9MHz and the final Sheet Resistivity.
- Correlation between the amplitude of the Imaginary Part of the Complex Voltage at a frequency of 10.1MHz and the final Sheet Resistivity.

- ❖ Absolute deviation, 1.89 mΩ/□
- ❖ Relative deviation, 3.78 %

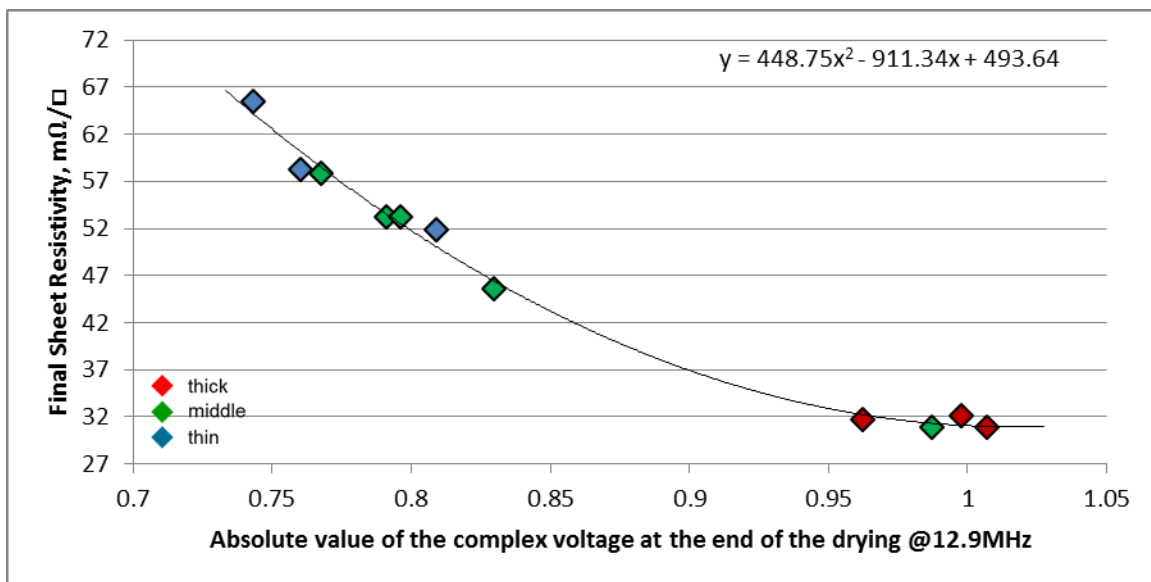


Figure 30. Final Sheet Resistivity as a Function of the Absolute Value of the Complex Voltage at the End of the Drying at a Frequency of 12.9MHz for High Conductive Coatings on Ceramic Substrate with a Coated Area of 2cm×2cm

- ❖ Absolute deviation, 2.11 mΩ/□
- ❖ Relative deviation, 3.5 %

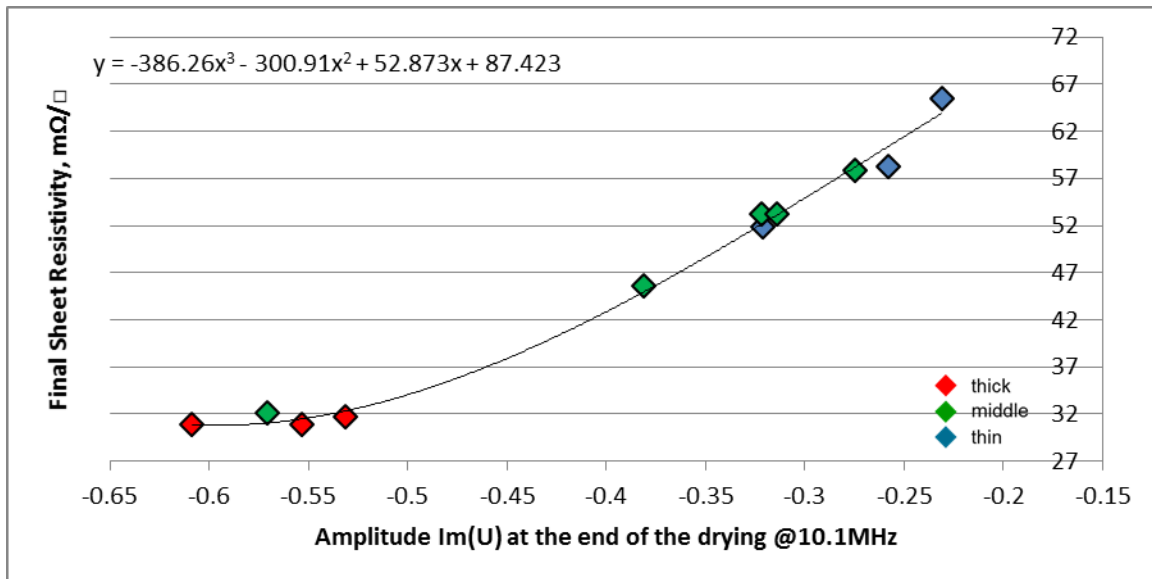


Figure 31. Final Sheet Resistivity as a Function of Amplitude Im(U) of the Complex Voltage at the End of the Drying at a Frequency of 10.1MHz for High Conductive Coatings on Ceramic Substrate with a Coated Area of 2cm×2cm

4.5.4 Silver Coated Glass Based Layers with Coated area of 4×4cm on Ceramic Substrate

- Correlation between the amplitude of the Real Part of the Complex Voltage at a frequency of 5.719MHz and the final Sheet Resistivity.
 - ❖ Absolute deviation, 29.21 mΩ/□
 - ❖ Relative deviation, 9.59 %

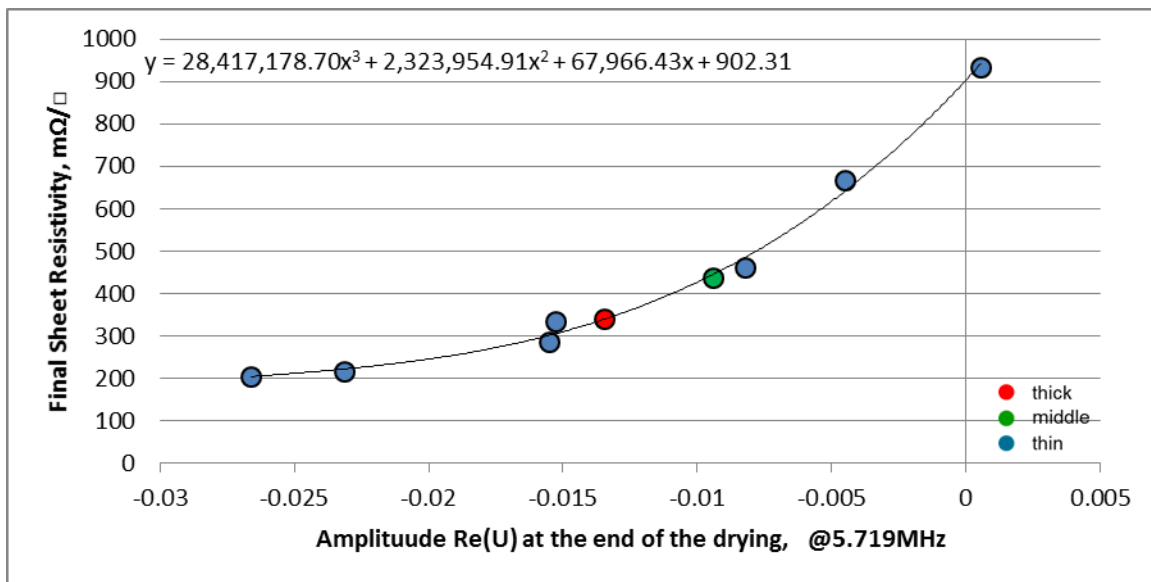


Figure 32. Final Sheet Resistivity as a Function of Amplitude Re(U) of the Complex Voltage at The End of the Drying at a Frequency of 5.719MHz for Low Conductive Coatings on Ceramic Substrate with a Coated Area of 4cm×4cm

4.6 Control Software including Algorithms

4.6.1 Control Software

Specific software “Eddy Wet” was developed to provide Eddy Current measurement software for the experiments to permit measurements in defined time steps over a longer time span. The following Figure 33 shows a screenshot of “Eddy Wet” software interface.

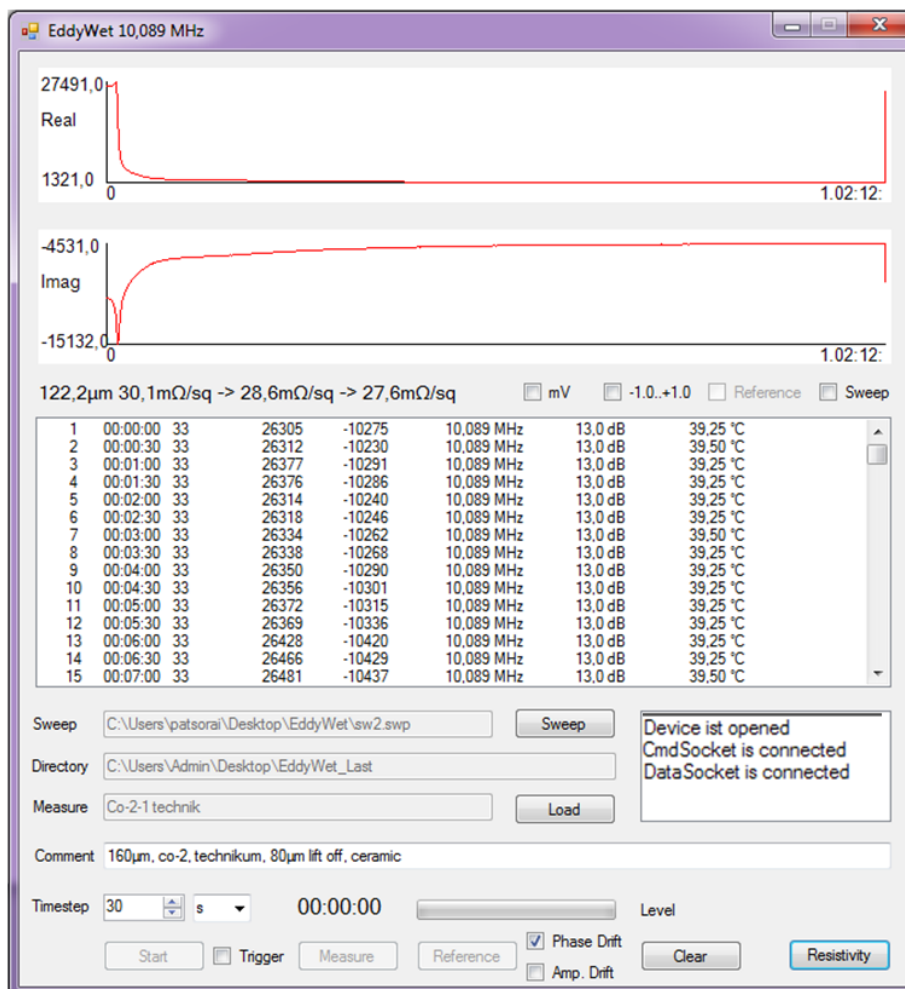


Figure 33. Screenshot of the Eddy Wet Main Menu

The Software has the following options:

- Create Frequency sweeps of a maximum of 245 frequencies.
- For each frequency, the matching gain is allocated.
- Time step of the measurements can be defined.
- When “Start” is pressed, Eddy Wet starts a sweep and saves the results and all parameters in a data file; Eddy Wet continues the sweep after each time step, and saves the data (results) in the same data file.
- Data recording stops, when “Stop” is pressed.

- Between single sweep measurements Eddy Current excitation will not run, to prevent heating the coil and the sample.
- In the event of a system failure during the total drying time, the system saves the data file automatically and all data until the failures are eliminated.
- Figure 34 shows two Eddy Current results in the Eddy Wet main menu (Real Part and Imaginary Part, or Magnitude and Phase of one Frequency) as a function of the time. Variations in the results can be obtained and visualized during the measurements.
- Using the ChooseDisplay menu, the frequency shown in the diagrams, can be selected and changed during measurements.
- The large text display under the diagrams in the main menu lists the obtained Eddy Current values.
- Resistivity (calibrations) for prediction of the final parameters of coatings (described below in detail).

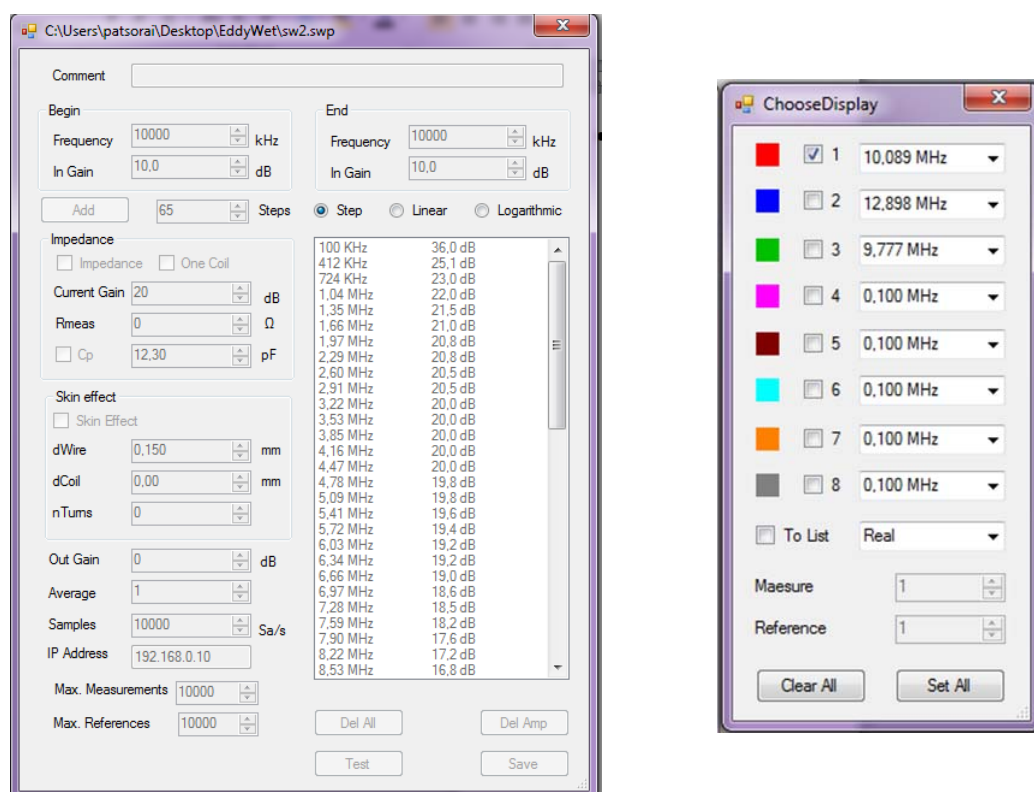


Figure 34. Screenshot of Eddy Wet: left Sweep Menu; right: ChooseDisplay Menu

4.6.2 Algorithm (Resistivity)

Based on the correlations described above an algorithm is developed. The principle of the algorithm can be described as follows. There are three dominating states during curing – liquid State “1”, wet State “2” and cured State “3” for next wet conductive coatings:

- (1) Silver coated copper based layers with coated area of 4cm×4cm on ceramic substrate;

- (2) Silver coated copper based layers with coated area of 4cm×4cm on CFRP substrate;
- (3) Silver coated copper based layers with coated area of 2cm×2cm on ceramic substrate;
- (4) Silver coated glass based layers with coated area of 4cm×4cm on ceramic substrate.

It was experimentally found and is seen from Figures 12 to 32 that each existing correlation has a trend line, which is defined by some polynomial functions and can be generalized in the form:

$$a_6x^6 + a_5x^5 + a_4x^4 + a_3x^3 + a_2x^2 + a_1x + a_0.$$

Using these coefficients, calibrations for new measurements can be created by using the “Resistivity” menu on the software panel.

	at Maximum	at Time	at End
a6			
a5			
a4		10323	
a3	3395535,95	-25568	
a2	-339567,89	23488	157,34
a1	4,3416	-9669,4	-394,7
a0	39,706	1589,6	264,14
Value		Magnitude	Magnitude
Scaled		12,898 MHz	12,898 MHz
Time		R=-6097 I=-7709 (R=-17984 I=-12118)	R=-3951 I=-4742 (R=-17984 I=-12118)
	0,0451	0,7545	0,9896
	00:19:00	01:10:00	23:59:30
	122,2 μm	28,6 mΩ/sq	27,6 mΩ/sq

Figure 35. Screenshot of the “Resistivity” Menu

In the “Resistivity” window as shown in Figure 35, calibration curves can be prescribed for State 1 (at Maximum), State 2 (at Time) and State 3 (at End) by changing the coefficients “a_n”. Because different coatings have different optimal frequencies for the prediction their final parameters, the interface of the “Resistivity” menu includes an option for selecting the frequency for each state as well as the EC parameter that should be analyzed (Real, Imaginary, etc.). After adding all coefficients, the file should be saved and can be used for following measurements.

For convenience, all coefficients for each existing correlation for measured coatings are given in Tables 7 to 10 below.

Table 7. Calibration Coefficients for High Conductive Coatings on Ceramic Substrate with a Coated Area of 4cm×4cm

Coating (1)		Frequency	EC Signal	Coefficients					
				a ₅	a ₄	a ₃	a ₂	a ₁	a ₀
State 1	R _f , mΩ/□	10.1MHz	$Re(U)$	0	0	3,395,535.95	-33,9567.89	11,446.57	-106.91
	d, μm		$Im(U)$	0	0	0	0	4.3416	39.706
State 2	R _f , mΩ/□	12.9MHz	$Re(U)$	0	0	0	537.79	855.16	360.4
			$ U $	0	10,323	-2.5568	23,488	-9,669.4	1,589.6
State 3	R _f , mΩ/□	10.1MHz	$Im(U)$	0	0	133.49	158.63	132.29	75.414
		12.9MHz	$Re(U)$	0	0	-1,047.5	-1,725.5	-647.72	75.746
			$ U $	0	0	0	157.34	-394.7	264.14

Table 8. Calibration Coefficients for High Conductive Coatings on CFRP Substrate with a Coated Area of 4cm×4cm

Coating (2)		Frequency	EC Signal	Coefficients					
				a ₅	a ₄	a ₃	a ₂	a ₁	a ₀
State 1	NO CORRELATIONS			0	0	0	0	0	0
				0	0	0	0	0	0
State 2	R _f , mΩ/□	12.9MHz	$Re(U)$	0	0	0	315.28	546.58	252.28
			$ U $	0	0	0	213.59	-440.67	239.27
State 3	d, μm	12.9MHz	$Re(U)$	0	0	0	0	-382.48	-136.35
	R _f , mΩ/□		$Re(U)$	0	0	0	364.82	649.78	315.77
			$ U $	0	0	-151.56	434.67	-448.54	192.28

Table 9. Calibration Coefficients for High Conductive Coatings on Ceramic Substrate with a Coated Area of 2cm×2cm

Coating (3)		Frequency	EC Signal	Coefficients					
				a ₅	a ₄	a ₃	a ₂	a ₁	a ₀
State 1	Rf, mΩ/□	9.8MHz	<i>time Re(U)</i>	0	0	-0.0133	0.7318	-13.376	112.22
	d, μm		<i>time Re(U)</i>	0	0	0	-0.1258	8.7411	42.294
State 2	Rf, mΩ/□	10.1MHz	<i>Im(U)</i>	0	0	0	102.42	140.2	63.889
			<i> U </i>	0	0	0	313.78	-621.96	321.77
State 3	Rf, mΩ/□	10.1MHz	<i>Im(U)</i>	0	0	-386.26	-300.91	52.873	87.423
		12.9MHz	<i> U </i>	0	0	0	448.75	-911.34	493.64

Table 10. Calibration Coefficients for Low Conductive Coatings on Ceramic Substrate with a Coated Area of 4cm×4cm

Coating (4)		Frequency	EC Signal	Coefficients					
				a ₅	a ₄	a ₃	a ₂	a ₁	a ₀
State 1	NO CORRELATIONS			0	0	0	0	0	0
				0	0	0	0	0	0
State 2	Rf, mΩ/□	5.7MHz	<i>Re(U)</i>	0	0	92,726,610.57	4,927,459.13	95,135.13	889.95
			<i> U </i>	40,876	-157,383	232,930	-162,946	51,696	-4,927.7
State 3	Rf, mΩ/□	5.7MHz	<i>Re(U)</i>	0	0	28,417,178.7	2,323,954.91	67,966.43	902.31

To create a file for calibration curves, an optimal EC Signal should be selected, the one that gives the highest accuracy, for example. For coating (1) (silver-coated copper-based coating with coated area of 4cm×4cm deposited on ceramic substrate) the time and the amplitude of the real part of the complex voltage at a frequency of 10.1MHz has to be selected at the characteristic point, after 70 minutes and at the end of the drying time the absolute value of the complex voltage at a frequency of 12.9MHz has to be analyzed from the viewpoint to get the highest accuracy of the measurements for these coatings. If the goal is to characterize coatings having different coated areas in the same plane, then another parameter and frequencies should be selected. After desired coefficients are selected, the saved file will appear as shown in Figure 36.

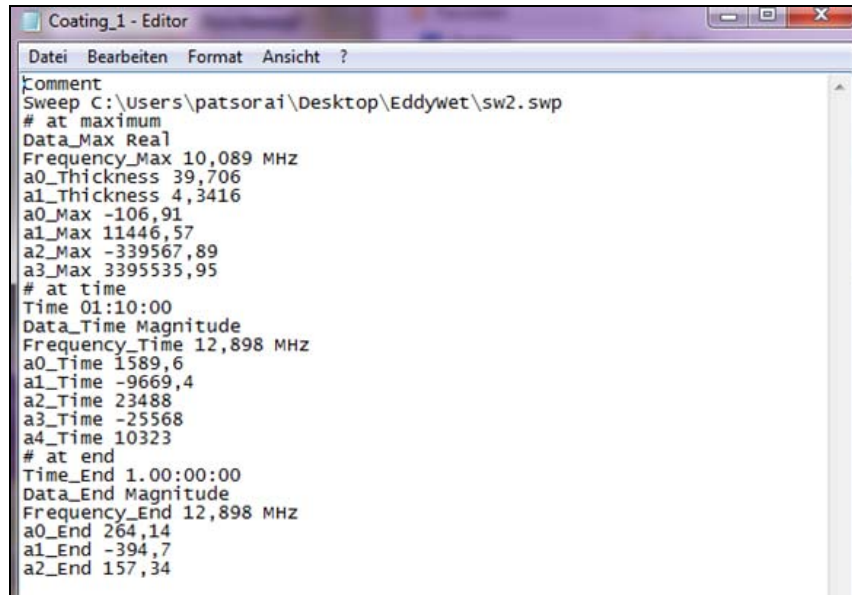


Figure 36. Calibration File for Prediction Final Parameters for Silver Coated Copper Based Layers on Ceramic Substrate with a Coated Area of 4cm×4cm

4.7 Development of the EC Testing System

A first High-Frequency Eddy Current for mobile application prototype is shown in Figure 37.

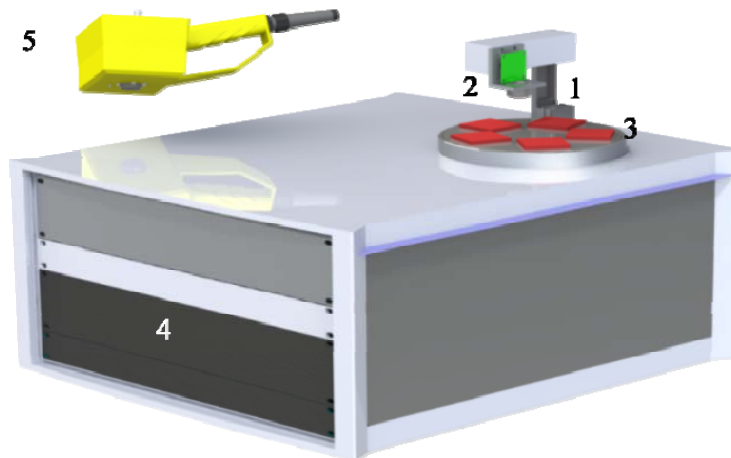


Figure 37. Drawing of the High-Frequency Eddy Current Based Testing System

The PC is located in a rack enclosure (4) and can be pulled out. The system includes two types of sensors. One of the two sensor systems for laboratory long time measurements is located at the top of the box. Figure 37 shows an Eddy-Current sensor (2), a sample holder (3) and a movable Z-axis for varying the distance between sensor and sample (1). This sensor can be used for measurements of wet coatings on flat samples under lab conditions; for example, to record data for calibration curves. The sample holder is easily changeable for different samples.

Another sensor, including a similar coil, is located in a handheld sensor (5), which can be manually applied. Figure 38 shows the drawing of the handheld-sensor.

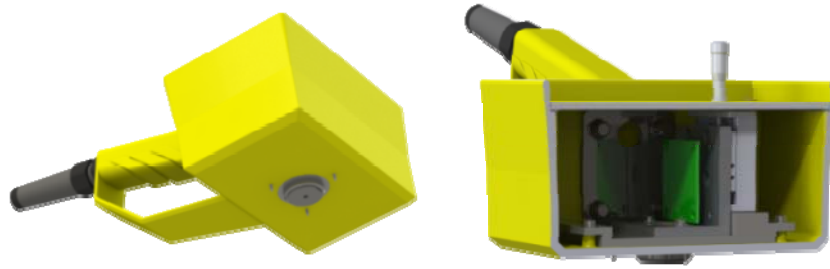


Figure 38. Drawing of the Concept of the Handheld-Sensor

This sensor consists of the same coil, and has 3 needles (stand-off) that contact the structure to achieve a constant distance between the coil and the sample. The length of the needles can be adjusted using a precision positioning plate, which is integrated in the sensor; the setup button is located on the bottom of the sensor. In addition, the sensor has a release on the handheld sensor.



Figure 39. Photo of the High-Frequency Eddy Current Based Testing System

A photo of the produced system is shown in Figure 39. A detailed description of the functionality and parameters of the EddyCus[®] System is a part of the user manual that will be delivered with the system.

5.0 CONCLUSIONS

High-Frequency EC measurements were performed on wet conductive coatings having different parameters. It was experimentally proven that there are three dominating states in wet conductive coatings during drying, and these states have both low and high conductive coatings deposited on ceramic and CFRP substrates. Drying behavior in State 1, called a percolation threshold, depends on the type of the particles used as a filling. Thus, silver-coated copper-based and silver-coated glass-based layers have different drying characteristics in State 1. Only silver-coated copper-based layers can be characterized in State 1, due to their characteristic point. Final sheet resistivity and drying behaviors in State 1 depend on the type of particles; the duration of each state depends on the type of polymer and thinner used. States 2 and 3 provide an opportunity for the characterization of all investigated coatings. Coatings based on same particles can be characterized at the same frequencies. There is an opportunity for separating coatings having different parameters, without knowing the time of deposition, by characterizing them in the complex voltage plane at a frequency of 10MHz. Based on results, an algorithm is developed and described in this report allowing the prediction of the final parameters of wet conductive coatings in a wet state. High-Frequency Eddy Current testing system for special issues in surface analysis is developed. The system comprises two special Eddy Current sensors, PC, all electronics and software based on multi-frequency algorithm.

6.0 PUBLICATIONS

1. ISSE 2013 (POSTER), 36th International Spring Seminar on Electronics Technology “Automotive Electronics”, 8 May - 12 May, Alba Iulia, Romania. *“Experimental setup for the characterization of the percolation behavior of wet conductive coatings by high frequency Eddy Current spectroscopy”*.
2. ASNT 2014 (ORAL PRESENTATION), ASNT Annual Conference 2014: 27 October - 30 October, Charleston, SC, USA. *“High-Frequency Eddy Current System for Analyzing Wet Conductive Coatings during Processing”*.
3. QNDE 2014 (ORAL PRESENTATION), 41st Annual Review of Progress in Quantitative Nondestructive Evaluation: Conference Boise Centre, Boise, Idaho, July 20-25, 2014. *“High-Frequency Eddy Current Based Impedance Spectroscopy for Characterization of the Percolation Process of Wet Conductive Coatings”*.
4. ECNDT 2014 (ORAL PRESENTATION), 11th European Conference on Non-Destructive Testing: October 6–10, 2014, Prague, Czech Republic. *“High-Frequency Eddy Current System for Analyzing Wet Conductive Coatings using Multi-Frequency Algorithm”*.
5. DGZfP 2014 (POSTER), DGZfP-Jahrestagung 2014 – Poster 27, 26 – 28 Mai, Potsdam. „Untersuchung des Trocknungsverhaltens von dünnen, elektrisch leitfähigen Lacken mit dem Wirbelstromverfahren“.

All papers are approved and submitted.

7.0 REFERENCES

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3. D. Lu, Q. K. Tong, C. P. Wong, “Conductivity Mechanisms of Isotropic Conductive Adhesives (ICA’s)”, *IEEE Transactions on electronics packaging manufacturing*, Vol. 22, No. 3, July 1999, pp. 223-227.
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5. S. Hillmann “*Evaluation eines zerstörungsfreien Prüfverfahrens zur Ermittlung des Flächenwiderstandes flüssiger, leitfähiger Schichten*”, Dresden International University, Studienrichtung, Zerstörungsfreie Prüfung“, Masterarbeit, 2013.
6. S. Hillmann, H. Heuer, J. G. Calzada, A. Cooney, B. C. Foos, N. Meyendorf, “Characterization of wet conductive coatings using eddy current techniques”, *AIP Conference Proceedings*, Vol. 1430, 2012, pp 441.
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LIST OF SYMBOLS, ABBREVIATIONS, AND ACRONYMS

<u>Acronym</u>	<u>Description</u>
AFRL	Air Force Research Laboratory
CFRP	Carbon Fiber-Reinforced Plastic
DOD	Department of Defense
DTIC	Defense Technical Information Center
EAR	Export Administration Regulation
EC	Eddy Current
ICA's	Isotropic Conductive Adhesives
ITAR	International Traffic in Arms Regulation
NDE	Non-destructive Evaluation
RX	Materials & Manufacturing Directorate
RXC	Structural Materials Division
RXCA	Materials State Awareness & Supportability Branch
RIMSS	Research Initiatives for Materials State Sensing
USAF	United States Air Force
WPAFB	Wright-Patterson Air Force Base