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## SURFACE TOPOGRAPHY CONTRIBUTION TO RFID'S TAGS EFFICIENCY , RELATED TO RESISTIVITY

by

Mario A. Cruz

A Thesis

Submitted to the Faculty of The Graduate College in partial fulfillment of the requirements for the Degree of Master of Science Department of Paper Engineering, Chemical Engineering, and Imaging

> Western Michigan University Kalamazoo, Michigan December 2006

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## SURFACE TOPOGRAPHY CONTRIBUTION TO RFID'S TAGS EFFICIENCY RELATED TO RESISTIVITY

#### Mario A. Cruz, M.S.

#### Western Michigan University, 2006

The Radio Frequency Identification (RFID) is one of the new technologies with more vision and acceptance oriented to the distribution and storage industries. The RFID is a technology that can be used to identify, track and store information about groups of products, individual items, or product components, using radio waves. The RFID device needs an antenna in order to receive a signal and transmit information. The stamping process applied today to produce the RFID antenna works with foil or copper. These etched metal RFID tags offer good benefits, but the production cost is too high to achieve a widespread implementation. However, with the use of conductive ink to print RFID tags, it is expected that a reduction in production cost to enable broader scale applications will be possible.

To obtain a suitable implementation of conductive inks in RFID technology it is imp[ortant to understand the ink/substrate interactions. For this work, the influence of SBS coated board properties on ink conductivity will be studied. With known ink and coated substrate properties, the present work will analyze the effects of coating applicator, coat weight and substrate finished on the electrical properties of ink.

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#### **CHAPTER I**

#### **INTRODUCTION**

#### **Radio Frequency Identification Overview**

A Radio frequency identification device (RFID), is a technology that can be used to identify, track, and store information about groups of products, individual items, or product components, using radio waves. An RFID consists of three important parts: a tag, reader (antenna) and transceiver with decoder<sup>1</sup>. The reader emits a radio signal that activates the tag and reads and writes data to it. The information encoded on the tag can be read and received by the reader, which is attached to a data collection system, consisting of computers running data processing software, which typically are networked with a larger information management system.

RFID technology is not new; it has been around since World War II (Identification of friend or foe airplanes) and then evolved to be used in the railroad industry, tracking the trains. In limited use, since 1970, RFID was applied in inventory management. RFID is now an established part of specific business processes in a variety of markets<sup>2</sup>.

#### Chain Management

Consumer Packaged Goods (CPG) depends upon barcodes to track their products through the supply chain. The degree of tracking of products varies greatly within the industry. The quality and type of electronic transactions between suppliers, distributors and retailers also varies greatly. Many different numbering systems are in use throughout the world. In the United States and Canada the Universal Product Code (UPC), Figure 1, is heavily used. In Europe and else where in the world EAN-8 and EAN-13 (European Article Numbering) codes are used. UPC, EAN-8 and EAN-13 have been fused in a Global Trade Item Number (GTIN).<sup>2</sup>



Figure 1: Universal Product Code

In the consumer business industry, large retailers (e.g. Wal-Mart, Target, Best-Buy and Albertson's) are requiring food and non-food suppliers to use RFID Electronic Product Codes (ePC) at the case and pallet level in early 2005<sup>1</sup>. In the industrial sector, the US Department of Defense (DOD) demands that suppliers use RFID tags in certain products looking to improve their supply chain management and reduce labor, errors and handling costs. Companies like Microsoft, IBM, and Philips Electronics, recently made product enhancement announcements in this area.

Wal-Mart and DOD announcements have accelerated the testing of pallet and case level RFID tagging in the supply chain<sup>3</sup>. Item level tagging will require much less expensive RFID tags than those currently being produced and tested<sup>4</sup>.

Agency	Application
Department of Defense	Logistic support and material tracking
Department of Health and Human Services	Drug authentication, chip implants
General Services Administration	Asset management and transportation
Department of Transportation	Freight and mass transport
Department of Homeland Security	Immigration, border patrol, search rescue
Department of Veterans Administration	Patient and supply chain tracking
Department of the Treasury	Records management
U.S Postal Service	Mail security and tracking
NASA	Hazardous materials management
Department of the Interior	Access cards
U.S. Department of Agriculture	E-Passports
Department of State	Animal tracking for disease control

**Table 1: U.S. Government RFID Applications** 

Many industries have accelerated the testing of pallet and case level RFID tagging in the supply chain. We will probably see this effort in a year's time. Item level tagging will require a much less expensive RFID tag than those currently being produced and tested. If item level ePC RFID occurs in the retail industry, the read range of the very inexpensive ePC tags will be very short by design.

#### **RFID Technology**

RFID systems allow manufacturers, retailers, and suppliers to improve collect, manage, distribute, store information (inventory), and security controls. The retailers can identify potential delays and shortages; the grocery stores can eliminate or reduce item spoilage; toll systems will identify and will collect auto tolls on roadways; and for the suppliers, to track shipments will allow receiving authorities to verify the security and authentication of shipped items. These varieties of uses are seen as the beginning, and RFID is dispersed across different sectors and services increasing

efficiency and visibility, several other applications and benefits may arise (Figure 2). Over the last three decades, RFID technologies have been getting more powerful, smaller and less expensive. As mentioned previously, the basic RFID system consists of three components:<sup>1</sup>

- A tag made up of a powered or non-powered microchip with an antenna.
- A reader with an antenna that communicates with the tag and receiving information.
- Transceiver that records and transmits the tag information to a hub point.



Figure 2: RFID System Components

**RFID Tags.** The tags are programmed with data that identify the item to which the tag is attached. Tags can be either read-only, volatile read/write, or write one/read many (WORM) and can be either active or passive<sup>2</sup>. In general, active tags use batteries to power the tag transmitter (radio) and receiver. These tags usually

contain a greater number of components than do passive tags. Therefore, active tags are usually larger in size and are more expensive than passive tags. In addition, the life of an active tag is directly related to battery life. Passive tags can be either battery or non-battery operated, as determined by the intended applications. Passive tags retransmit the RF signal transmitted to them from a reader or transceiver and add information by modulating the signal. A passive tag does not use a battery to boost the energy of the retransmited signal. A passive tag may use a battery to maintain memory in the tag or power the electronics that enable the tag to modulate the reflected signal. Table 1 lists the characteristics of RFID tags.

Туре	How they works	Pros	Cons	
Active Tags	Battery-run tags that constantly emit radio frequency signal.	Good for tracking large objects. Read range is 100+ feet. Not as impacted by metals/liquids. Require less power from reader.	May have greater range it may be more costly. The infrastructure cost may be lower than for a passive tag system.	
Passive Tags	Activated by electromagnetic waves of reader. Powered by reader energy.	Cost-effective for implementation. Smaller, no maintenance, lighter, less expensive.	Read range is currently 10 – 25 feet from the reader. Difficulty working through metal/liquid. Higher power.	
WORM Information written on the tag during manufacturing.		Good for one time. Cost- effective.	Information can never be changed.	
Read/Write Tags Users can add new or write over existing information when tag is near a reader.		Add information at any time.	Cost.	

 Table 2: Tag Types

**RFID Reader**. RFID readers can stimulate tags by sending signals, supplying power to passive tags, encoding the data signals going to the tag, and finally, decoding the data received from the tag. The power output and the radio frequency determine the range at which the tags can be read. RFID systems typically operate in frequency ranges between 30 KHz to 500 KHz (low frequency), 850 MHz to 950 MHz, and 2.4 GHz to 2.5 GHz (both considered ultra high frequency)<sup>5</sup>.

In an international RFID application the frequency of use becomes difficult, due to the regulations imposed by different countries. In many cases the frequency, power emission, and bandwidth of equipment used for RFID in the US cannot be used in other countries. Table 3 lists the frequencies used around the world, and their associated wavelength. Although some frequencies may be used in multiple regions, many exceptions exist and the allowable power emission and bandwidth use vary from country to country.

Frequency	Wavelength	Region	and the second second
125 KHz	7872 ft	1,2&3	2 4 1
13.56 MHz	72.6 ft	1,2&3	
868 MHz	1.13 ft	1	
915 MHz	1.08 ft	2	2
2.45 GHz	4.82 in	1,2&3	
5.8 GHz	2.03 in	Future Region	

Table 3: Frequencies Around the World<sup>3</sup>

**RFID Transceiver.** The third part of the system, the RFID transceiver consists of computer hardware and data processing software connected to main inventory or identification management systems. This part provides the operating system, data repository, and processing algorithms that convert multiples tag inputs into visible tracking or identification data.

RFID has some advantages over other automatic data capture technologies. The advantage of RFID systems is the non-contact, non-line-of-sight nature of the technology. Tags can be read through a variety of substances such as snow, fog, ice, paint, crusted grime, and other visually and environmentally challenging conditions, where barcodes or other optically read technologies would be useless. RFID tags can also be read in challenging circumstances at excellent speeds, in most cases responding in less than 100 milliseconds.<sup>6</sup>

RFID has some disadvantages. Radio frequency communication is subject to many forms of interference. Radio signals can fade in strength or become distorted due to the movement of nearby items, changing the atmospheric conditions, and adding a new source of radio frequency energy. Other disadvantages of RFID are the proliferation of incompatible RFID standards, very few frequencies are available on a global basis for RFID systems, and differences in regulations that govern the use of the frequencies with various countries<sup>7</sup>. The differences in regulation in other countries may also apply to power levels, interference, and frequency tolerances. Another problem area of RFID is with antenna design. For a radio antenna to efficiently radiate or project radio energy through the air it must be tuned with the radio frequency desired by adjusting its physical length relative to the wavelength of radiation.

There is growing misconception that RFID is a wonderful solution to all sorts of business problems. There are huge differences among the various RFID systems and products. The differences include frequency of operation, tag size, read and read-write capability, data capacity, read or writes range and speed, active or passive tags. The reality is that different applications will in all probability require very different RFID solutions and some possible applications may be a bit further over the horizon than others<sup>4</sup>.

RFID technologies are expected to grow quickly over the next few years as companies seek to improve their supply chain operations, and as the price of RFID drops. The application for RFID in the year 2000 reached most economic sectors although the most use was in industrial/manufacturing, transportation, distribution, and warehousing industries<sup>8</sup>. Smart label applications as well as high-speed processing will fuel growth in these sectors. Other growth sectors include health care, commercial, and retail services. Future revenue growth within these economic sectors will primarily be driven by the development of the smart label market, particularly to support growing applications such as baggage handling and high-speed processing (Figure 3). These two economic sectors are expected to account for the largest percentages of RFID hardware revenues in the near and long term; however, their annual growth will be slower in comparison to the annual growth of the emerging health care, commercial, and retail services sectors.<sup>7</sup>



Figure 3: Global Distribution of RFID in the Year 2000<sup>7</sup>

## RFID System Costs

The cost of acquiring, installing, and maintaining an RFID system will be a determining step in it's acceptance in the commercial sector. RFID system cost is composed of tags, readers, and processing and supporting information technology hardware and software<sup>9</sup>. Current tag costs range from 25 to 40 cents per tag (higher in some cases, depending upon the type of tag)<sup>7</sup>, making it expensive for low-end consumer items. The MIT Auto-ID lab expects tag prices to drop by 15 cents in 2006 for orders of 1 million units<sup>8</sup>. RFID reader costs are also relatively high, due to limited uptake of systems. The Auto-ID Lab also expects reader cost to come down from about \$100 in 2005 to about \$70 in 2006. Finally, RFID transceiver costs include computer hardware, software, data processing, data mining, personnel salaries, and personal training. A product company that ships 50 million cases per year could spend \$20 million for RFID implementation<sup>10</sup>.

For some companies, RFID system is extremely costly. However, the companies may also be able to reduce some cost due to the development of new technology applied to the current RFID system.

#### **CHAPTER II**

#### **CONDUCTIVE INK**

Conductive inks are made of metallic particles such as silver or copper flakes in a retaining matrix<sup>11</sup>, or carbon particles in a retaining matrix. Carbon is typically two times less conductive compared to metal, but is cheaper. Usually the matrix was a ceramic, such as glass frit, but now increasingly it is a polymer (polymer thick film, PTF), such as Polyaniline, Polypyrrole and polythiophene<sup>11,12,13</sup>. The retaining matrix is non conductive or weakly conductive. Once printed, the matrix needs to be reduced so that conductance through the material occurs by conductive particles in contact with each other, which is done by curing, which can be performed by temperature. Ceramic mixtures need high cure temperatures, such as 650<sup>o</sup>C for several minutes. PTF mixtures have lower cure requirements such as 150<sup>o</sup>C. However, cure temperatures depend on the ink formulation; some dry quickly at room temperature which is sufficient for some application, such as the RFID tag antennas at Ultra high frequency, UHF, (about 900 MHz)<sup>14</sup>.

Almost all the printing ink contains a high percentage of conducting material and also contains polymers as additives that perform many functions such as binding, vehicles, viscosity regulator etc. The use of conductive polymers in conductive inks may reduce the need for metal conductors, as well as external additives<sup>12</sup>.

The silver metal inks have been used to print test targets by lithography, gravure, flexography and letterpress. These inks generally incorporate silver flakes in resin binders. The use of carbon-conductive inks constitutes a low-cost method of applying conductors and resistors to various types of plastic polyester films, classic base material, ceramic and other materials, applied by the screen-printing process<sup>13</sup>.

Conductive inks are gaining a rapid commercial importance due to the development of the RFID technology. Carbon base inks have been used for EMI/RF (electromagnetic interference) shielding, such as screens and speakers. Metallic base

inks are used for membrane switches and circuits and now for RFID antennas. In the case of RFID antennas the conductivity of metal particle inks is more than sufficient so curing can be done at relatively low temperature for a few seconds.

These inks are used in a variety of applications, including RFID and smart labels. RFID labels are comprised of a silicon chip that carries identification data an antenna that transmits a radio frequency signal that can be detected at a distance<sup>15</sup>. The chip receives and transmits the data using the antenna. The radio signal is picked up by another source, such as a computer, which then reads the information that is being transmitted. Conductive ink can be used in place of traditional coils for the antenna<sup>13</sup>. Smart labels are active RFID labels using a conductive ink antenna can potentially gauge the temperature in shipment and alert a supplier to harmful shifts in temperature <sup>16</sup>. Antennas are printable with the use of conductive inks. Conductive inks are engineered for use in current printing configurations, but provide electrical qualities not commonly found in typical ink products. Inks need to have a fast drying time and the ability to adhere to a variety of substrates.

The printers that manage the UPC (Universal Product Codes) barcodes are now faced with a new challenge with EPC (Electronic Product Code). The traditional high price of RFID tags will not be possible for implementation in item-level identification<sup>17</sup>. In the same way UPC technology took years to evolve, item-level RFID technology will not likely happen overnight.

In order to produce conductive ink, additional conductive substances have to be integrated into the ink system. The concentration of these conducting substances must be such that a contact of the individual particles distance of > 10 nm, respectively, renders a current flow within the ink system possible<sup>18</sup>. The maximum concentration of the conducting particles is mainly dependent on their structure and the specific surface. Only so many conductive particle solids can be added to the lacquer as can be homogenously embedded by the polymer. Generally first-grade conductors such as a metal or precious metal powder and carbon powder only can be used as conducting matters. Compared to carbon powders, metal or precious metal

yield conductance higher by a factor of three times<sup>11</sup>. This is mainly due to the higher specific conductivity of metals, but also to the smaller specific surface of metallic powders so that higher concentrations in the lacquer are possible. On the other hand, metal or precious metal powders entail the problems of oxidation/corrosion and migration as in the case of the frequently used copper. Furthermore, the high material price of precious metal powders adversely influences the cost accounting.

#### CHAPTER III

#### **RFID PRINTED SUBSTRATES**

The ideal substrate properties for printed RFID devices: substrate must dissipate static build-up, be non-conductive, hydrolytically stable, abrasion resistant, and smooth enough to prevent distortion of the radio frequency signal at minimal ink thickness. In addition to these properties, the substrate flexibility plays an important role in the application of the RFID devices due to multiple shapes of products. Another desirable property is the product recyclability. A variety of substrates, such as paper, board, polyesters, polyethylene, polysulphone, cellulose and polyamide may be used for electronic printing<sup>18</sup>. The best results are achieved using smooth but absorbent materials, such as synthetic and filled papers. Substrate thicknesses of 0.050 to 0.30 mm can be printed on most offset and flexo presses<sup>19</sup>.

RFID devices are currently being printed onto a variety of substrates by a variety of different printing methods. Polyester and nylon fabric, polyethylene terephthalate (PET), polyvinylchloride (PVC), polyamide, silicon film, coated and uncoated paper and board, glassine and parchment paper are some that have been mentioned in the literature<sup>20</sup>.

#### Solid Bleached Sulfate (SBS) Board

Because of its versatility, durability, and relatively low cost, paperboard is used to package everything from carryout food to pharmaceutical, cereal and hardware. U.S paperboard production for folding cartons increased 30% during 1990's, to 6.8 millions tons annually (Figure 4). Three major types of paperboard are used for customer goods: coated recycled board (CRB), solid bleached sulfate (SBS), and coated unbleached kraft (CUK)<sup>20</sup>. CRB is made from 100% recovered paper and usually contain a minimum of 35% post consumer materials. In addition, several manufacturers produce board that combines outer layers of SBS with an inner layer of post consumer recycled materials.



Figure 4: Growth in U.S Paper Board Production for Folding Cartons<sup>21</sup>

SBS is a highest quality board produced. Due to its properties and cost, the prime area of application of SBS is high quality packaging, such as perfume boxes, and other high end products that require premium quality packaging to attract potential customer to pay a premium price. The requirements of the coated SBS are mostly brightness and gloss. In addition, the substrate must have surface properties that allow high quality print to be applied.

For nearly 20 years, the SBS segment of the paperboard industry was dominated by clay-based coating that used polyvinyl acetate as the primary synthetic binder and protein as a co-binder<sup>22</sup>.

#### **CHAPTER IV**

#### TOPOGRAPHY

Surface characteristics of paper play a vitally important role in controlling paper and printing quality. Today's paper products must be designed with optimum surface properties to meet increasingly strict performance criteria in a rapidly changing market.

The materials with atomically flat surface are great to scientist and industrials and for certain technological applications; however, the majority of materials have an irregular surface, made up of undulations and even perhaps steep gradients and pores. These constitute the topography of the surface, a property that is usually difficult to define but can have a considerable impact on a material's performance. Such importance reflects the surface specific nature of many properties: the ability to adhere to another material (coating, ink, etc), optical properties, etc.

In the paper and printing industries, it is essential to measure surface roughness, due to its effect on the achievable print quality. The standardized methods use the air leakage from a pressurized cylinder, held against the substrate surface, as a roughness measure<sup>23</sup>, but a more detailed characterization of the surface is desirable. Existing methods for detailed topography measurement are usually based on comparatively slow point scanning systems<sup>24</sup>.

The topography of a particular surface is measured by some kind of profilometry. This is a technique in which a probe is passed across a surface, following its contours, and the height of the probe at any particular point is recorded. The stylus instruments are quite adequate for hard, smooth surface, offering excellent accuracy. However, in the case of polymers, the stylus digs into the surface and the results do not truly represent the topography. "Other possible criticisms of stylus instruments for surface profiling include their sensitivity to microphonics and vibrations, particularly during long measurement times, uncertainly in point of

contact of stylus on rough surface, and the delicate nature of their stylus and mechanism"<sup>24</sup>.

The size of the probe will determine the size of the surface features that may be distinguished, and for smaller scale analysis, atomic force microscopy (AFM) may be used. AFM works by bringing a cantilever tip in contact with the surface to be imaged (figure 5). An ionic repulsive force from the surface applied to the tip bends the cantilever upwards. The amount of bending, measured by a laser spot reflected on to a split photo detector, can be used to calculate the force. By keeping the force constant while scanning the tip across the surface, the vertical movement of the tip follows the surface profile and is recorded as the surface topography by the AFM.



Figure 5: Atomic Force Microscopy<sup>24</sup>

#### **CHAPTER V**

## **COATING APPLICATION METHODS**

Many years ago, methods for applying coating on paper or board were limited. There were two or three process options, and for certain grades, one option was traditionally selected to apply the coating. However, in the past 15 years, a wide range of other options has been developed and are in large scale use. Many of these options have been around in other industries for many years.

From one coating process to another, a few key points are important to keep in mind when dealing with coat weight control, stresses on the paper, uniformity of the coating, and other operating problems such as scratches. Different coating processes use different methods to control the coat weight. In most cases, an excess of coating is applied to the base sheet, and the excess is removed with metering element.

#### **Blade Coaters**

The use of a blade is still the most common method for applying coating to the paper web. Application of the coating to the paper in front of the blade has undergone considerable change. In addition, methods to control blade angle and loading have improved in recent years<sup>19</sup>. Figure 6 is a schematic of a short dwell coater. In this case, the coating is pumped into a pond immediately upstream from the blade. Loading of the blade controls final coat weight. Excess coating is returned to the coating supply system.



Figure 6: Blade Coater

The disadvantage of any blade coating operation is the stress applied to the paper. Stress applied to the web can cause web breaks, which result in machine down time.

#### Air Knife Coaters

Air knife coating is similar to blade coating, except that a jet of air is used to meter the amount of coating onto the substrate (Figure 7). An excess of coating is applied to the web, and a jet of air is used to remove it. Air knife coating does not have the disadvantages associated with a blade in terms of scratches of high stresses to the web, but there is a limitation to how small coat weight can be obtained. Therefore, an air knife is often used for high coat weight board grades. The smoothness and uniformity of the coating is normally lower than that of blade coated products.

The angle at which the air strikes the wet coating is extremely important and is controlled by rotating the air knife assembly thereby adjusting the angle of attack.



Figure 7: Air Knife Coater

It is of paramount importance that the air knife can be cleaned without disturbing its setting and it is usual for the whole knife assembly and its supporting structure to pivot out so that the knife can be easily reached. Because air is used, there is a tendency for some of the mix to atomize (form very fine droplets like a mist).

The air knife can be operated in three ways:

- To brush or smooth the coating layer. In this case most of the metering has been carried out at the application stage and the air knife is left to level coating.
- The air curtain acts as a barrier and prevents the onward travel of any excess coating mix. This is employed when the very fluid mixes are in use. The excess mix is not removed as a spray but runs down the sheet into the applicator tray.
- The normal method of operation is a combination the first and second one. In this mode the air knife is one of the most versatile of all the coaters.

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#### **CHAPTER VI**

#### METHODOLOGY

The main objective of this investigation was the assessment of SBS board properties on conductive ink resistivity. The effect of coating applicator, coat weight and substrate finish, on electrical properties of printed SBS board was analyzed. To accomplish the objective of this research, the project was divided in three experimental parts: Silver flake ink analysis, coated substrate analysis, and inksubstrates interaction analysis. Before these analyses, the coating was prepared and applied to the SBS board using the air knife and blade application methods.

The process started with the coating preparation and application. For each application three different coat weights were applied; high, medium and low. The coated samples were then calendered at two different calendering pressures to obtain different levels of surface roughness (Table 4).

Air Knife		Blade		
Coat Weight (gr/m <sup>2</sup> )	Calendering (pli)	Coat Weight (gr/m <sup>2</sup> )	Calendering (pli)	
10.86	600	9.61	600	
14.42	1200	13.87	1200	
23.92	UnCal	22.54	UnCal	

**Table 4: Research Conditions** 

#### **Coating Application Methods**

The coating was applied by two different methods to the SBS Board.

- Blade Coater
- Air Knife Coater

Solvent base flexography silver ink, provided by Acheson Colloids Company, was applied to SBS coated and uncoated board at multiple conditions, using a 0.5 mil Byrd applicator, to obtain influence of the surface to the silver ink conductivity.

#### Silver-Flake Ink Analysis

**Rheology.** The rheological properties of the conductive ink were studied before the application on the substrates. The AR 2000 rheometer was used to define the rheological characteristics of the ink. The typical rheological properties that are measured with the rheometer are: viscosity profile, intersection of storage G' and loss G'' modulus, modulus profile with temperature or time (G', G'' vs. T or t), examining plateau modulus and measure the strain dependence of the materials (Length of linear viscoelastic region). A dynamic stress sweep was developed in order to define the linear viscoelastic region (LVR) for the ink used. The strain sweep experiment was carried out at a constant frequency (10 Hz); contrarily the amplitude of the oscillation was varied (Figure 7). As the amplitude is increased, more shear (energy) is applied to the sample. The sample is subjected to the shear: this energy can begin to break down the internal structure of the ink. At the beginning the storage modulus remains constant, as the amplitude is increased. This region is the LVR. When the sample is subjected to a stress in the LVR, it will be able to recover its structure once the stress is removed. Outside this region the deformation is not reversible. As the amplitude continues to increase, the structure of the ink begins to break down. The strain at which the material structure begins to break down is called the critical strain. The second type of experiment developed was the frequency sweep, where the amplitude of oscillation is fixed and the frequency is varied (Figure 8). The amplitude for the frequency sweep is chosen to be within the LVR as determined by the amplitude sweep measurement. "The frequency sweep is used to determine the time dependency of the sample's deformation".



Figure 8: Frequency Sweep (Top) and Strain Sweep (Bottom)

The steady flow is used to study the viscosity change under increasing shear stress. It also gives the idea of how viscous or elastic the material is under shear stress, giving two important properties, the low shear viscosity,  $\eta_o$ , and the high shear viscosity,  $\eta_{\infty}$ . The couette geometry was chosen, because it is appropriate for low viscosity samples and is available to resist high shear rate.

**Ink Film Thickness.** The film thickness was measured using the Emveco Profilometer, taking average of the 700 readings along the samples<sup>26,27</sup>. The ink film thickness was obtained to control the mileage, expressed as the number of square meters covered by a kilogram of ink, and used as a variable to find the ink resistivity.

### **Coated Substrate Analysis**

Each sample of SBS board was analyzed to obtain values of roughness, porosity and topography ranking using the Verity  $Topo^{TM}$  Software<sup>28</sup>. The tests were developed for uncoated and coated substrates to obtain a better understanding of the variables effects in the printed sample resistivity.

**Roughness.** The roughness values were obtained with the use of an Emveco Stylus Profilometer model 210. The profilometer uses a preloaded fine cone-shaped stylus with a radius of 0.001 inch. The test conditions were 500 reading per sample, 0.1 mm reading space, and 0.5 mm/sec of scanning speed. The roughness R was calculated using the equation:

$$R = \Sigma (Z_{i+1} - Z_i)/499; I = 1,2,3...,499$$

Where Z is the vertical position of the stylus.

**Permeability and Porosity**. The permeability of the substrate was calculated from its Parker Print Porosity value and its thickness using the following equation<sup>26,27</sup>.

#### K= 0.048838\*Q\*X

Where Q is the air flow rate (ml/min), X is the thickness of the sample (m) and K is the permeability in  $\mu$ m<sup>2</sup>. Parker Print-Surf Porosity (PPS) was used to measure the porosity of each sample. This equipment was designed to accurately measure the surface roughness of sheet materials under conditions similar to those experienced during the printing process. A clamping pressure of 1000 kPa and a soft baking was used.

**Verity Topo<sup>TM</sup>**. The Verity-Topo is a topography software system used to rank numerically the surface topography. The top surfaces of uncoated and coated samples at different conditions were measured using the system composed of a computer, a specially modified graphics quality scanner, and the Verity-topo<sup>TM</sup> analysis software (Figure 9)<sup>28</sup>.



Figure 9: Verity-Topo<sup>™</sup> Measurement Apparatus

The area scanned for each sample was 10 cm \* 10 cm, using a resolution of 600 ppi, the image was analyzed obtaining a number that represents the roughness of the entire surface. The substrates samples were scanned at the same points and area for the multiple conditions in two directions, machine (MD) and cross machine (CD).

Wetability. The wetability is defined as the contact angle between a droplet of a liquid in equilibrium on an horizontal surface (Figure 10). The contact angle of water with SBS coated board, for each condition, was determined using the First Ten Angstroms Dynamic Contact Angle Tensiometer<sup>29</sup>. The equipment works by adding a drop of water using a syringe onto a sample strip of the substrate. The camera will take many pictures of the drop in motion, and the computer software will analyze the images<sup>30</sup>. The wetting angle is given by the angle between the interface of the water droplet and the horizontal substrate sample.



Figure 10: Wetting Angle

## Ink-Substrate Interaction Analysis

**Conductivity/Resisitivity.** The silver conductive ink was printed on the different conditions using a 0.5 mil Byrd applicator. After printing, the resistivity of the samples was measured using a digital multimeter, a sample of the printed substrate and a voltage of 0.1 V to -0.1 V. Ink Resistivity was calculated using the following equation:

### s = L/(R\*A)

where L= Length of sample, A= Film Thickness \* sample width and R=potential difference/Electric current<sup>30</sup>.

#### **CHAPTER VII**

#### **RESULTS AND DISCUSSIONS**

#### **Rheology**

The silver flake conductive ink was studied to understand its behavior in terms of viscous and elastic modulus. Looking the Dynamic Stress Sweep (Figure 11), as the amplitude is increased, more energy is applied to the ink. This energy breaks down the internal structure of the sample. At the beginning the storage modulus remained constant as the amplitude increased. The name of this area is the linear viscoelastic region (LVR) (Figure 12). During the LVR the storage modulus values were greater than loss modulus and this prove the higher elastic behavior in comparison with the viscous. The stress point, at which the sample structure begins to break down, is known as critical stress.







Figure 12: Linear Viscoelastic Region

The first drop after the Critical Stress is the area when the structure created by the silver flakes starts its deformation. The second stability area observed in the dynamic stress sweep (figure 11) is due to the structure of the polymer particles in the ink. The structure deformation is shown graphically by the second drop.

The frequency sweep experiment was developed using a stress value of 0.1 Pa chosen from the stress sweep results within the linear viscoelastic region. The silver conductive ink sample shows a weakly associated particles so that the storage modulus has a crossing point with the loss modulus during the frequency of 4 Hz to 3 Hz. The ink maintains its strength before the frequency of 3 Hz, and then the sample deforms quickly (Figure 13).



**Figure 13: Frequency Sweep** 

Figure 14, steady state flow test was developed to obtain the low shear viscosity ( $\eta_o$ ) and the high shear viscosity ( $\eta_{\infty}$ ), using the cross model:

## $\eta = [(\eta_{\scriptscriptstyle 0} \text{-} \eta_{\scriptscriptstyle \infty})/(1 \text{+} {K_{_{Y}}}^m)] \text{+} \eta_{\scriptscriptstyle \infty}$

where K is known as dimensions of time<sup>m</sup> of the material, the greater the value the K, the further to the left the curve lies, and the greater the value of the index m, the greater of shear thinning.



Figure 14: Flow Curve Model



## Substrate Analysis

The SBS board was characterized using the variables shown in the Table 5.

Thickness (µm)		355.6
Basis Wt., gsm	(lb)	250 (155#)
Doughnoss (migron)	Avg.	4.89
Rouginess (micron)	StDev	0.28
Porosity (ml/min)	Avg.	271.4
	STD	19.4
	Avg. MD	3.27
Verity Number Arbitrary (AU)	StDev MD	0.301
	Avg. MD	3.464
	StDev CD	0.704

 Table 5: SBS Board Characterization

The conditions selected for this research were evaluated in terms of roughness porosity and topography. The results are showed ahead are grouped according to the Coat Weight.

#### Roughness

The paper roughness is the measurement of the small-scale variation in the height of a sample surface. The coated samples were calendered using two different pressures to obtain 3 different roughness levels.

## Air Knife Low Coat Weight

Roughness (µm)	Uncoated	Uncal	600 pli	1200 pli
Average	3.77	2.72	1.41	1.22
StDev	0.33	0.16	0.07	0.09

## Air Knife Medium Coat Weight

Roughness (µm)	Uncoated	Uncal	600 pli	1200 pli
Average	3.66	2.54	1.42	1.02
StDev	0.05	0.09	0.23	0.05

#### Air Knife High Coat Weight

Roughness (µm)	Uncoated	Uncal	600 pli	1200 pli
Average	3.85	2.63	1.31	1.02
StDev	0.34	0.12	0.19	0.08

#### Blade Low Coat Weight

Roughness (µm)	Uncoated	Uncal	600 pli	1200 pli
Average molte W	3.46	1.78	1.12	0.99
StDev	0.26	0.04	0.10	0.03

#### Blade Medium Coat Weight

Roughness (µm)	Uncoated	Uncal	600 pli	1200 pli
Average	3.93	1.65	1.17	1.05
StDev	0.43	0.05	0.08	0.03

#### **Blade High Coat Weight**

Roughness (µm)	Uncoated	Uncal	600 pli	1200 pli
Average	3.70	1.68	1.21	1.00
StDev	0.19	0.10	0.09	0.02

The roughness values decrease when the samples were coated and calendered, because one of the objectives of coating application and calendering is to improve printability and visual characteristic using the effect of the coating pigments. When the air knife coater was used, the smoothness and uniformity of the coating, was normally lower than the values of blade coated samples.

#### Porosity and Permeability

Porosity is a highly critical factor in printing papers and boards. It is an indicator of absorptivity or the ability of the sheet to accept ink or water. The permeability was calculated using the following equation<sup>26,27</sup> and the PPS porosity value: K=0.048838\*Q\*X

Where Q is the air flow rate measured as porosity (ml/min), X is the sample thickness (m) and K is the permeability in  $\mu m^2$ .

## Air Knife Low Coat Weight

PPS Porosity (ml/min)	Uncoated	Uncal	600 pli	1200 pli
Average	270.15	4.39	4.2	3.31
StDev	10.61	0.57	0.49	0.16
Permeability µm <sup>2</sup>	0.455*10 <sup>-3</sup>	7.63*10 <sup>-5</sup>	6.56*10 <sup>-5</sup>	4.56*10 <sup>-5</sup>

## Air Knife Medium Coat Weight

PPS Porosity (ml/min)	Uncoated	Uncal	600 pli	1200 pli
Average	270.26	3.90	3.00	2.18
StDev	10.01	0.13	0.37	0.05
Permeability µm <sup>2</sup>	0.455*10-3	5.14*10 <sup>-5</sup>	4.44*10 <sup>-5</sup>	3.98*10 <sup>-5</sup>

## Air Knife High Coat Weight

PPS Porosity (ml/min)	Uncoated	Uncal	600 pli	1200 pli
Average	273.51	2.90	2.05	1.13
StDev	11.94	0.11	0.25	0.05
Permeability µm <sup>2</sup>	0.46*10 <sup>-3</sup>	5.21*10-5	4.7*10 <sup>-5</sup>	3.87*10 <sup>-5</sup>

### Blade Low Coat Weight

PPS Porosity (ml/min)	Uncoated	Uncal	600 pli	1200 pli
Average	266.64	4.19	3.03	2.79
StDev	10.35	0.19	0.16	0.10
Permeability µ m <sup>2</sup>	$0.462*10^{-3}$	6.19*10 <sup>-5</sup>	5.65*10 <sup>-5</sup>	4.12*10 <sup>-5</sup>

## Blade Medium Coat Weight

PPS Porosity (ml/min)	Uncoated	Uncal	600 pli	1200 pli
Average	270.46	3.77	2.91	2.58
StDev	11.38	0.09	0.06	0.03
Permeability µm <sup>2</sup>	0.451*10 <sup>-3</sup>	5.98*10 <sup>-5</sup>	5.03*10 <sup>-5</sup>	3.5*10 <sup>-5</sup>

### Blade High Coat Weight

PPS Porosity (ml/min)	Uncoated	Uncal	600 pli	1200 pli
Average	269.78	3.39	2.63	2.54
StDev	10.92	0.47	0.06	0.27
Permeability µm <sup>2</sup>	0.44*10 <sup>-3</sup>	5.54*10 <sup>-5</sup>	4.42*10 <sup>-5</sup>	3.03*10 <sup>-5</sup>

According to the Parker Print-Surf results, the Air knife High Coat weight sample at 1200 pli calendering has the lowest PPS porosity value, and the uncalendered and uncoated samples have the highest values for Air knife and blade in all the conditions. The other PPS porosity values fluctuated between values of 4.19 and 2.18 ml/min. The calendering effect exhibited a values diminution in comparison with the uncalendered samples. The substrate coated with blade (high coat weight) and calendered with 1200 pli pressure exhibited the lowest permeability.

## Verity Topo<sup>™</sup> Software

#### Air Knife Low Coat Weight

Verity (AU)	Uncoated	Uncal	600 pli	1200 pli
Machine Direction				
Average	3.23	0.93	0.50	0.32
StDev Marine	0.29	0.12	0.29	0.02
Cross Direction				
Average	3.33	1.05	0.74	0.47
StDev	0.70	0.23	0.56	0.09

## Air Knife Medium Coat Weight

Verity (AU)	Uncoated	Uncal	600 pli	1200 pli
Machine Direction				
Average	3.29	0.98	0.76	0.41
StDev	0.34	0.70	0.30	0.09
Cross Direction				
Average	3.3	0.89	0.69	0.56
StDev	0.30	0.92	0.06	0.16

#### Air Knife High Coat Weight

Verity (AU)	Uncoated	Uncal	600 pli	1200 pli
Machine Direction			_	
Average	3.53	0.68	0.63	0.55
StDev	0.51	0.10	0.06	0.14
Cross Direction				
Average	4.12	0.64	0.47	0.41
StDev	1.68	0.08	0.06	0.04

#### Blade Low Coat Weight

Verity (AU)	Uncoated	Uncal	600 pli	1200 pli
Machine Direction				
Average	2.88	0.73	0.42	0.21
StDev	0.27	0.20	0.04	0.01
Cross Direction				
Average	2.88	0.77	0.58	0.23
StDev	0.41	0.10	0.04	0.04

### Blade Medium Coat Weight

Verity (AU)	Uncoated	Uncal	600 psi	1200 psi
Machine		_		
Direction				
Average	3.08	0.74	0.43	0.19
StDev	0.22	0.07	0.04	0.03
Cross	1.1			
Direction				
Average	3.16	0.96	0.58	0.32
StDev	0.38	0.21	0.04	0.24

### Blade High Coat Weight

Verity (AU)	Uncoated	Uncal	600 pli	1200 pli
Machine Direction				
Average	3.35	0.70	0.39	0.23
StDev	0.27	0.07	0.07	0.02
Cross Direction				
Average	3.13	0.64	0.53	0.50
StDev	0.37	0.05	0.12	0.35

The values for the Verity software showed a very similar trend for the machine direction and cross direction, the uncoated samples have a higher Verity number (lower topography) and the 1200 pli calendered samples have the lower, for Blade and Air Knife.

The substrates coated with the Blade had the lower Verity number in comparison with those coated with Air Knife.

#### **Ink-Substrates Interaction Analysis**

**Conductivity/Resistivity.** Silver flake ink was printed to the SBS board conditions using a 0.5 mil rod ink applicator. Each board was printed using the same technique, the ink film thickness varied from condition to condition, this effect could be attributed to differences in ink holdout. The ink film thickness was measured using Emveco Stylus Profilometer, taking samples with unprinted and printed areas, 700 readings were measured, 200 for the unprinted and 500 for the printed area. The data were plotted. In the graphic obtained were observed 2 regions, the first one represents the height of the unprinted area and the second the height of the printed area, both in relation of a parameter pre-established for the software.

The values were taken in consideration during the conductivity measurements. The conductivity was measured using a digital multimeter in samples of 2" length, wide between 0.05 to 0.1", and a film thickness between 5 to 9  $\mu$ m.

**Emveco Profilometer.** The effect of roughness on resistivity is shown in the graphics 15, 16 and 17. The best resistivity is represented by the sample coated with high coat weight using the blade applicator and a calender finish of 1200 pli of pressure.



Emerco Profilometer Bucium Coat Weight

Figure 15: Emveco High Coat Weight

Figure 16: Emveco Medium Coat Weight



Figure 17: Emveco Low Coat Weight

Verity-Topo<sup>™</sup>. The results obtained in the graphics above for Verity-Topo show a similar trend that the one observed for the previous figure (Emveco Profilometer). The sample coated with high coat weight using the blade applicator and a calender finish of 1200 pli of pressure, gave the best resistivity. The resistivity of the Blade coated samples were below the Air Knife coated.



Figure 18: Verity High Coat Weight

Figure 19: Verity Medium Coat Weight



Figure 20: Verity Low Coat Weight

Wetability. The contact angle of the substrates was measured using the method 3 Girifalco-Good-Fowkes-Young (GGFY) and water with a surface tension of 72.8 dynes/cm<sup>31</sup>. The high contact angle value correlates with its low permeability, the samples coated with blade and calendered at 1200 pli, had the lowest contact angle and resistivity.



Figure 21: Wetability High Coat Weight

#### Substrate Finish

The effect of Coat Weight and substrate finished is shown in the figures 22, 23 and 24. The blade-coated values are lower than those for air knife coater. As the coat weight increases the resistivity values decrease. The samples calendered with 1200 pli pressure got lower resistivity values than the calendered with 600 pli and the uncalendered.



Figure 22: A.K and Blade 600 pli

Figure 23: A.K and Blade 1200 pli



Figure 24: A.K and Blade Uncalender

**Topography.** The printability of paper is extremely dependent on the topography properties of the substrate. The top surfaces of uncoated and coated samples at different conditions were measured to understand the effect of coating applicator, coat weight and substrate finished on substrate topography. The unprinted and printed samples topography values were measured with three different devices, Verity, Emveco And Parker Print Surf. The correlation between each device was developed.

#### Unprinted Samples

The topography results obtained from the unprinted samples have a similar trend for the multiples coat weights, the uncoated samples show the higher verity number, decreasing the values in succession with uncalendered and calendered samples. The calendered substrate with 1200 pli pressure and coated with blade, represents the best topography number. A substrate with good topography can be a product with good printability, due to the dependency between the two variables.

The coat weight play an important role in coated-uncalendered samples, the low coat weight substrates show a higher topography values than the samples with medium and low coat weight.

The samples coated with blade coater shows a smaller verity number than the samples coated with air knife. These results are due to the smoothest surface obtained by the blade coater.



Figure 25: Verity Topography Unprinted Air Knife



Figure 26: Verity Topography Unprinted Blade



Figure 27: Verity Topography Printed Air Knife



Figure 28: Verity Topography Printed Blade

The printed samples yield an interesting phenomenon in relation with topography. The verity numbers were extremely high in comparison with the unprinted samples. This phenomenon is due to the effect of the silver flakes particle size. The samples coated with higher coat weight represent the best topography result, for the printed and unprinted samples.

#### **Enveco Roughness**

The values obtained using the Emveco technique show a higher results for the printed, coated and calendered samples. The silver particle size effect was presented in the Emveco measurements; the results show a surface with higher roughness. The effect of the coating coater was present in the printed samples, the ink absorption for the samples printed with blade was higher than the ones coated with air knife. The film thickness of the air knife was higher than the blade. Air Knife film thickness was 8.89 µm with a standard deviation of 0.9, and for air knife the film thickness was 6.85 µm with a standard deviation of 1.1.



Figure 29: Emveco Topography Unprinted Air Knife



Figure 30: Emveco Topography Unprinted Blade



Figure 31: Emveco Topography Printed Air Knife

### Verity, PPS and Emveco

The comparison of the three topography devices shows the lower values for the Verity Topo at all conditions and Emveco got the higher values at the condition 3 and 4. Parker Print Surf results show values between the Emveco and Verity for condition 3 and 4 and the higher for condition 2.



Figure 32: Topography High Coat Weight Blade



Figure 33: Topography High Coat Weight Air Knife



Figure 34: Topography Medium Coat Weight Blade



Figure 35: Topography Low Coat Weight Blade



Figure 36: Topography Low Coat Weight Air Knife

**Correlation.** The comparison of the three topography testing methods are presented in the Figures 38 and 39. A good correlation was obtained between Verity and PPS (r=0.869). A lower correlation was obtained between Verity and PPS (r=0.747). It is important to note that the Emveco values were 1 order of magnitude larger than the PPS.



Figure 37: Correlation PPS vs. Verity



Figure 38: Correlation Emveco vs. Verity

#### **CHAPTER VIII**

#### CONCLUSION

In this project, SBS board was coated using two different coating devices, three coat weights, and three calendering pressures. The uncoated and coated samples were printed using a 0.5 mil Byrd applicator. The printed and unprinted samples were evaluated to obtain the correlation between the coating method, coat weights, substrate finish and ink resistivity using three topography devices (Emveco, Verity and PPS)

It was observed that decreased roughness caused a decrease in the resistivity of the printed samples. The samples calendered with higher pressure, showed lower resistivity. Also Air Knife coater applicator showed higher resistivity values than Blade, the difference is due to the smoother surface obtained after coating application with Blade and calendered at 1200 pli pressure.

Other interesting phenomena observed in this research was the effect of coat weight on ink resistivity; increased coat weight caused a decrease in resistivity of printed samples, these effects are also due by the relation between coat weight and roughness. In this case at higher coat weigh the substrate roughness decreased. The substrate contact angle showed an effect in ink resistivity; a substrate coated with blade and calendered with 1200 pli exhibited high contact angle, low permeability and resulted in low resistivity.

It was observed that decreased coat weight caused a decrease in the topography number. For printed and unprinted samples. Blade samples showed a better topography than Air knife, the effect of coating coater and surface smoothness was very clear.

The ink particle size plays an important role in the topography of printed samples, affecting the surface roughness.

Paper as a substrate in the printed electronic industry play an important role, the characteristics of it can increase or decrease the quality of the final product. To obtain a suitable implementation of paper as a substrate it is necessary to understand each of the variables that affect the conductivity of the ink. This research can provide interesting conclusions to lead future projects to produce the substrate with special characteristic for the electronic industry.

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