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EFFECT OF CALENDERING ON FLEXOGRAPHICALLY  
PRINTED SILVER FLAKE INK

by

Sujay Pandkar

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  
August 2008

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2008

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Sujay Pandkar

# EFFECT OF CALENDERING ON FLEXOGRAPHICALLY PRINTED SILVER FLAKE INK

Sujay Pandkar, M.S.

Western Michigan University, 2008

Interest in the printed electronics is increasing on a day-to-day basis. To utilize the concept of printed electronics on a wider range, printed circuit needs to perform more and more efficiently. The simplest printed circuit components include conductive traces, which can function as interconnects or contact electrodes. There are still some limitations to the conductivity of printed traces. Previous results showed an improvement in the conductivity of printed traces with a post cure treatment of 4 hours at 105°C. The present research explores a different approach towards improving conductivity using a post calendering treatment on-line.

The effects of calendering temperature, pressure and the combination of both were observed to improve the conductivity of traces. The results from this study suggest that conductivity can be improved in-line and thus eliminates the need for further post-treatment. The effects of temperature and pressure during calendering were studied and statistical methods were used to determine the effect of calendering on various properties, such as ink conductivity, roughness, ink film thickness and RFID antenna performance.

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## LIST OF ABBREVIATIONS

SUB\_1 – Substrate 1

SUB\_2 – Substrate 2

SUB\_3 – Substrate 3

WB – Water Based Ink

SB – Solvent Based Ink

MD – Machine Direction

CD – Cross Direction

IFT – Ink Film Thickness

T – Temperature (°C)

P – Pressure (PLI)

N – Number for Nips

BCM – Billion Cubic Microns

LPI – Lines per Inch

UHF – Ultra High Frequency

RFID – Radio Frequency Identification

DOE – Design of Experiments

RH – Relative Humidity

## CHAPTER I

### INTRODUCTION

Printed electronics is an inventive and fast growing concept for the printing industry. For the past 3-4 years, there have been many breakthroughs in the field of printed electronics. One popular example is printable RFID (Radio Frequency Identification) tags<sup>1, 2, 3</sup>. Research and development in printed electronics is providing the print industry an opportunity to expand business beyond the traditional applications of publishing and packaging.

The printing of electronic devices all started with the screen printing of conductive, resistive and dielectric inks for thick film applications<sup>4</sup>. The trend continued for a while but, due to the high demand for inexpensive, flexible and large area electronics, faster roll to roll printing processes such as gravure and flexography were introduced<sup>5, 6</sup>. One of the biggest advantages of any printing process is that semiconductive and conductive layers can be printed on many different kinds of flexible substrates, such as paper/paperboard and plastics<sup>7</sup>. However, bearing in mind that conventional printing processes still have some limitations in printing electronic circuits, researchers are now moving towards improving the performance of these circuits by increasing print resolution and



overlay accuracy, as well as, the conductivity of the conductors, mobility of the semiconductors and insulating properties of the dielectrics.

According to IDTechEx, the printed electronics industry will grow at a rate of 450 times between 2006 and 2016<sup>8</sup>. With this huge increase in market demand, the need for advancements in printed electronic circuits cannot be overlooked. Researchers are currently seeking rollable, edible, and biodegradable circuits, eventually leading towards the development of invisible electronics<sup>9</sup>. But to accomplish and produce these prodigious structures on a large scale, with appropriate and consistent performance, is a difficult task.

Considering the present and future demand for good quality and inexpensive printed electronics, it can be said that conductivity will have a significant effect on the performance of printed circuits. The accuracy of the circuit construction is also an important factor<sup>10</sup> that can be defined in terms of position and material deposition, i.e., thickness, uniformity, precise overprinting and resolution<sup>10</sup>.

In this work, conductivity of traces printed with silver conductive inks was investigated. For conductive traces, it is important that the traces are printed continuously and uniformly, because any discontinuities in the conductive path will hamper the conductivity of the printed traces<sup>11</sup>. In addition, the conductivity

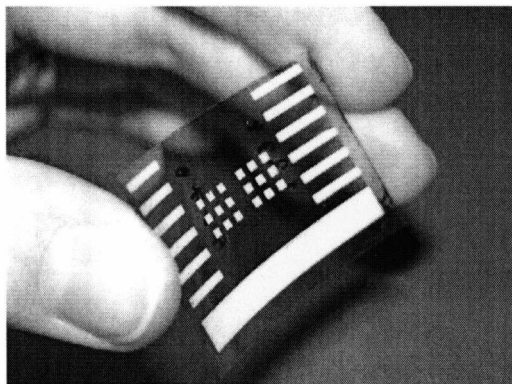
of the printed traces depends upon how the silver particles make contact with each other<sup>12</sup>. To overcome the limitations in the flow of charged carriers within printed conductive traces on paper-based substrates, calendering after printing was investigated.

## CHAPTER II

### THEORETICAL BACKGROUND

#### Printed Flexible Electronics

Printed flexible electronics is defined as the printing of electronic circuits on paper, board or film materials, with the help of functional inks (Figure 1). An electronic circuit is a collection of objects called circuit elements, joined together by wires through which current can flow<sup>13</sup>. Different kinds of electronic components, such as resistors, diodes, transistors and interconnects and other circuit components could be printed by conventional processes. Current processes for making solid state circuits<sup>14</sup> are being augmented with less time consuming and more environmentally friendly processes of conventional printing<sup>15</sup>. It now appears that printing is a more economical and faster way to produce electronic circuits<sup>10</sup>.



**Figure 1** Example of printed electronics printed on plastic film<sup>16</sup>.

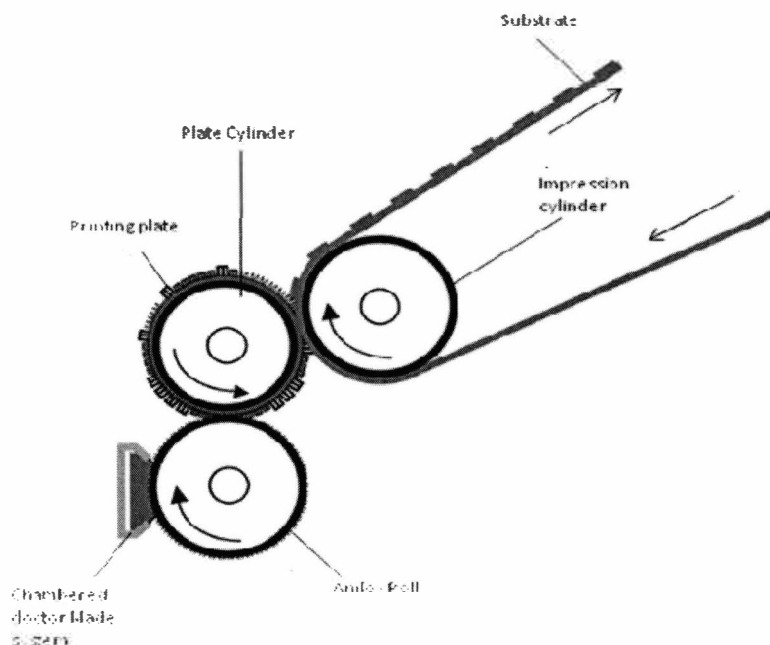
## Printing Process

There are different kinds of electronic devices that can be printed. Printing processes have the following advantages<sup>17</sup> over conventional technologies used for manufacturing electronics:

1. Ease of use
2. Low cost
3. Portability
4. Universal acceptance
5. Versatility
6. Durability
7. Practicality
8. Environmentally sustainable

In this study, flexography was used to print conductive traces for applications in electronics devices. As flexography is a direct rotary relief printing process, it requires a resilient raised image carrier made of rubber or photopolymer material<sup>18</sup>. The plates are fixed to a plate cylinder, which is inked by a cell structured ink-metering roll, called an anilox roll. Fast drying fluid inks can be used to print onto virtually any substrate, absorbent or nonabsorbent<sup>19</sup>. For each revolution of the plate cylinder, an image is produced (Figure 2). The

flexography process is primarily used to print packaging substrates – board, paper, foil and film in large quantities.



**Figure 2** Basic principle of flexography printing process.

In the case of flexography, the dot size and shape depends upon the resolution of the plate. Print quality also depends on resolution and type of anilox roll used. The amount of ink transferred from the raised areas of the plate determines the effective resolution of the image. Usually, the finer the resolution, the less ink transferred. Tones are reproduced by translating the density into dot sizes<sup>20</sup>. The flexographic printing process can accommodate solvent based, water based or UV curable inks. Ink viscosities can vary from 0.05 to 0.2 Pa.s. Printed ink film thicknesses can range from 0.8 to 15 microns<sup>21</sup>.

## **Conductive Inks**

Silver flake and carbon based inks are widely used as fillers in conductive inks. Nowadays, some inks use nano sized particles of silver and carbon to reduce the temperature required to sinter the silver particles and improve conductivity. The concentration and distribution of conductive particles in the ink films determines the conductivity of the printed traces<sup>22</sup>.

## **Conductivity**

An electric charge may be conveyed from one point to another by means of what is called a conductor and the ability of material to conduct electric current is called conductivity<sup>23</sup>. All materials that transfer electric current also offer a resistance to the transfer of electrons. Resistance can be defined as a parameter, which opposes the flow of current and causes a non-reversible transformation of electrical energy to heat energy within the conductor<sup>24</sup>. The amount of resistance offered by a conductor depends upon the material it is made of and conductor dimensions (length, shape and its cross sectional area)<sup>25</sup>. In the case of solid metals, resistance depends upon its temperature and it increases as the temperature increases due to thermal expansion. In contrast,

resistance decreases as the temperature increases in carbon containing materials, various insulators and electrolytic solutions<sup>24</sup>.

Resistance is determined from the voltage (V) and current values (I) according to Ohm's law, published in 1827<sup>26</sup>.

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

The R stands for resistance it is measured in the units of Ohms. The values of resistance are determined mainly by the physical dimensions and the resistivity ( $\rho$ ) of the material, of which the resistor is composed. For a bar of resistive material of length  $l$  and cross-sectional area  $A$ , the resistance is given by<sup>23</sup>,

$$\rho \frac{l}{A} = R \quad (2)$$

Where  $\rho$  is the resistivity of the material in ohm-meters ( $\Omega\cdot m$ )

For alternating current (AC) measurements, the term electrical impedance is used and it expresses the resistance at frequency  $\omega \neq 0$  Hz, describing not only the relative amplitudes of the voltage and current, but also the relative phases. Impedance is designated by  $Z$ <sup>23</sup>. Impedance is not a phasor, since it does not rotate on its complex axes but remains stationary<sup>26</sup>.

In general, impedance is a complex number and can be decomposed into its real and imaginary parts. It is customary to write

$$Z = R + jX \quad (3)$$

Here  $R$  is called the resistive part of the impedance, and  $X$  is called the reactive part. Resistance,  $R$ , is the real part of impedance; a device with purely resistive impedance exhibits no phase shift between the voltage and current. Reactance  $X$  is the imaginary part of the impedance. A component with a finite reactance induces a phase shift,  $\theta$ , between the applied voltage and measured current.

## Calendering

In printed electronics, smoothness of printed layers is crucial for device performance. It was thought that the use of calendering could improve the smoothness of printed conductive traces and in addition, increase the conductivity of printed ink films. This is believed possible, due to the temperature and pressure of calendering, which can sufficiently soften the ink resins to enable the alignment and compaction of the conductive particles. Furthermore, elevated temperature can help in particle sintering, thus increasing



conductivity. The basic principles and characteristics of calendering will now be discussed.

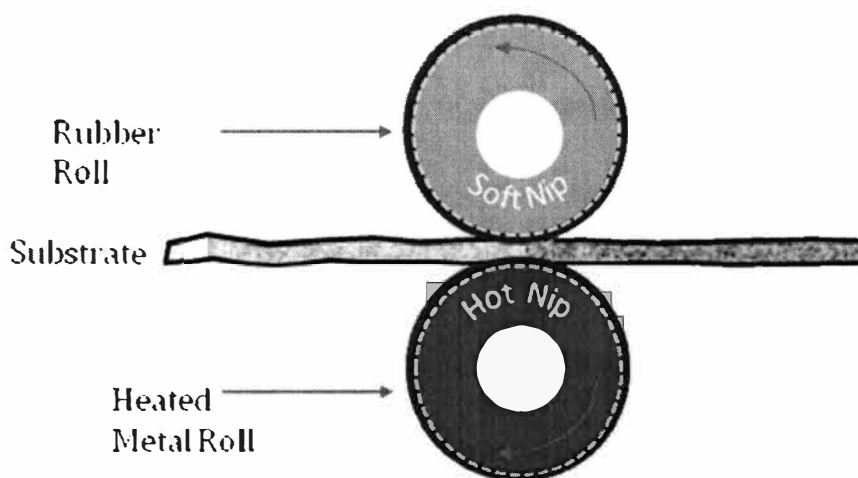
Calendering is a vital operation in traditional paper making. Calendering is a process where a web passes through a nip composed of two rollers under pressure, which results in the compression of the web, thereby changing the surface and interior properties of the sheet<sup>27, 28</sup>. The purpose of calendering is to press the paper against a polished, metal cylinder, with enough force to replicate the surface of the polished roll through plastic deformation<sup>29</sup>. Other than improving paper smoothness, calendering also helps to correct irregularities in the sheet by compacting and densifying the fibers<sup>27</sup>. During calendering, the paper is subjected to a pressure pulse in the Z-direction and heat usually flows from at least one roll into the sheet<sup>28</sup>. Although compression in the nip is partly elastic, the plastic and viscoelastic deformations within the fibers cause changes in sheet thickness, roughness, density, porosity, gloss, opacity and strength. An increase in density corresponds to a reduction in spacing between fibers.

A calender consists of a number of rolls arranged to form multiple nips depending on desired smoothness. The rolls can be of different hardness and may, or may not, be heated. By controlling linear pressure, calendering speed, hot roll surface temperature, the surface finish on the hard rolls, soft roll cover

material, sheet moisture, soft roll position (against top or bottom side of the web)<sup>30</sup>, roll size and the number of nips, a papermaker is able to control surface finish and amount of sheet compaction (density) without adversely affecting the strength properties of the paper. Heating the rolls enables a desired surface finish to be obtained at lower pressures and with fewer nips, because it softens the fibers, making them more pliable.

Most calendering done today is performed on-line, using a combination of heated metal and soft covered rolls, which is known as hot-soft nip calendering<sup>29</sup>. Two roll calenders are primarily used for calendering webs that do not need heavy calendering<sup>27</sup>. Two types of common calenders preferred in industry are hard nip and soft nip calenders. The major difference in the nip behavior between hard and soft nip calenders is that both the web and the roll cover are compressing for soft nip calendering, which results in a significantly lower actual pressure in the nip. The nip is wider than that for hard nip calenders allowing better heat transfer and subsequent deformation of the calendering web. Another significant difference is that the compression of the web in both high and low spots is more evenly distributed. The deformation of the soft cover reduces the maximum local nip pressure resulting in more uniform calendering.

## Hot-soft nip calender



**Figure 3** Hot-soft nip calender.

The main components of a two roll soft calender (Figure 3) are the soft covered roll and the heated roll with a smooth, polished surface. Hard rolls are usually made up of cast iron. The linear pressure of a soft calender ranges from approximately  $10\text{-}350\text{ Nmm}^{-1}$  and the surface temperature can go up to  $230^{\circ}\text{C}$ <sup>31</sup>. These rollers can be heated through circulating hydraulic oil, which also helps to maintain uniform surface temperature. Soft rolls are covered with a 10-20 mm thick elastomeric material on steel or cast iron shells. After calendering, paper dimension in the MD and CD change by fractions of 1%<sup>27</sup>.

## CHAPTER III

### PROBLEM STATEMENT

The printed electronics industry is growing rapidly<sup>32</sup>. To utilize the concept of printed electronics over a wider range of products, better performing printed circuits are needed.

Different techniques have already been employed to improve the conductivity of silver flake printed traces and antennae<sup>33,34</sup>. Heat curing of the printed samples after printing is called post curing. Previous results using the post cure treatment showed an improvement in the conductivity of printed traces with a treatment of 4 hours at 105°C<sup>33,34,35</sup>. On a commercial level, the viability of the time and amount of heat required are in question. Additional inline dryers or heaters after printing are possible solutions, but are not ideal. Additional dryers will change the dimensions of existing presses and to install them would require a costly investment. The installation of extra dryers only provides heat and not pressure. In calendering, the physical contact between heated roll and printed conductive traces increases the density of ink film and particle alignment, thus increasing conductivity.

## CHAPTER IV

### RESEARCH OBJECTIVES

This research explores a different approach to improving the conductivity of the silver printed traces. An off-line post calendering treatment is proposed. The objective of this study was to determine the effects of hot-soft nip calendering on the conductivity of printed traces. This was done to assess the applicability and effectiveness of using calendering to increase conductivity off-line, thus eliminating the need for a post cure treatment.

It was hoped that calendering would offer significant improvement in the conductivity of printed traces by increasing density of conductive ink film (reducing the distance between conductive particles). The effects of temperature, pressure, and the combination of both, on the conductivity of printed traces were studied. In this study, the conductivity of printed silver traces before and after calendering are compared. The performance of UHF RFID tag printed antennae before and after calendering were also tested. Correlations between antenna performance and ink film thickness (IFT) are already stated<sup>33, 34</sup>.

The effects of temperature and pressure during calendering were studied and statistical methods used to determine the effect of calendering on ink

conductivity, roughness, IFT and UHF (Ultra High Frequency) RFID tag antenna performance.

## CHAPTER V

### MATERIALS, EXPERIMENTAL DESIGN AND PROCEDURES

#### Materials

For this research different grades of label stock papers were used.

Three different kinds of papers were used. The papers were coded as,

- SUB\_1
- SUB\_2
- SUB\_3

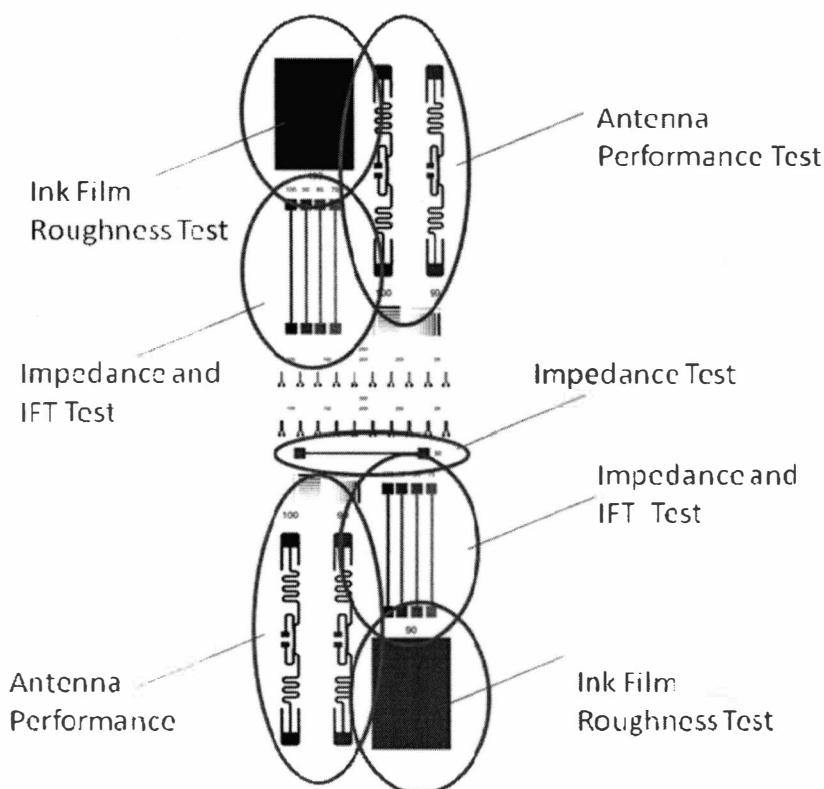
Two types of inks were used; water based (WB) and solvent based (SB), both inks are silver-flake conductive inks. **Error! Reference source not found.**1 shows the basic components and properties of the inks used for printing the conductive traces.

**Table 1** Basic components and properties of inks used for printing conductive traces (Source – Manufacturer).

| Ink ID | Solvent    | Ideal Resistance<br>(mOhms/square/mil) | Solids<br>(wt. %) | Density<br>(kg/l) | Particle Size<br>( $\mu\text{m}$ ) |
|--------|------------|--|-------------------|-------------------|------------------------------------|
| WB     | Water      | 20                                     | 85                | 3.2               | < 3                                |
| SB     | PM acetate | 6                                      | 64                | 2.1               | < 7                                |

## Printing

Conductive silver inks were printed on WMU's Comco Commander narrow-web flexographic press. The design (Figure 4) included lines at 4 different tones 70, 80, 90 and 100 %. A 90 % tone trace was printed in both the machine and cross directions. To ensure sufficient drying, three dryers were used during the print trial. All dryers were set to 225 °F (107°). A 12 BCM and 200 lpi anilox roll was used.



**Figure 4** Design used for printing of conductive traces.



## Design of Experiments

Calendering experiments followed the multilevel full factorial DOE (design of experiments). Four factors were used, including the substrate, ink type, calendering temperature and calendering pressure. The substrates were printed with 2 different inks (SB and WB) then calendered at four different pressures and four different temperatures. A design of experiments was created resulting in 80 runs including 16 different combinations based on four levels of each factor and each calendering condition, having 5 replicates for better sampling. The levels for temperature and pressure are outlined in the Table 2.

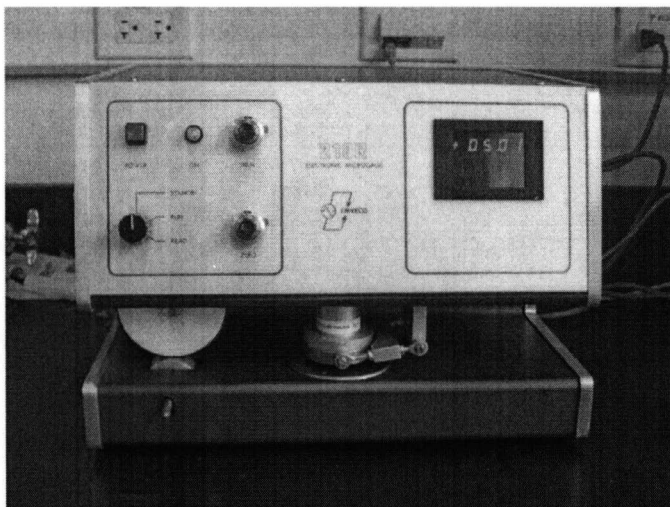
An initial study found the samples to stick to the hot metal calendering roll at a roll temperature of 80°C, so the DOE was limited to a maximum temperature of 75°C. Multiple passes (2, 3 and 4) through the calender were performed for the SUB\_1 samples printed with WB and SB inks after which impedance was measured. For samples receiving no pressure (0 PLI), the samples were kept in an oven for 4 hrs at the desired temperature (23, 50, 65 and 75°C) and impedance measurements were made after cooling the samples for 1 hr in a controlled room according to TAPPI standard conditions of 23°C and 50% RH (ASTM D 685).

**Table 2** Temperature and pressure levels used in calendering study

| Temperature<br>(°C) | Pressure<br>(PLI) |
|---------------------|-------------------|
| 23                  | 0                 |
| 50                  | 375               |
| 65                  | 950               |
| 75                  | 1500              |

### **Ink Film Roughness**

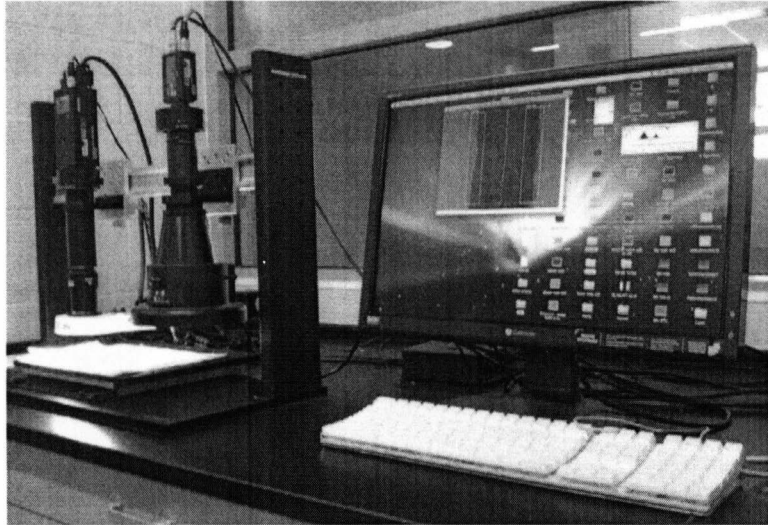
An Emveco 210R Electronic Microgage stylus profilometer (Figure 5) was used to measure the roughness of the printed ink films before and after calendering. This method is a direct measurement of surface roughness and uses a preloaded fine cone-shaped stylus (0.001 mil) to mechanically trace the substrate surface<sup>36</sup>. TAPPI 'T575 om-07' standard was followed to set the parameters on the testing instrument. For precise readings, measurements were carried out at 3 areas in both, machine direction and cross direction on the printed surface for each sample. For further analysis, an average of 3 measured values was calculated. The values were measured in 0.001 inch and converted to microns.



**Figure 5** Emveco 210R electronic microgauge.

### **Image Analysis**

An ImageXpert (KDY Inc.) image analysis system (Figure 6) was employed to measure line length, width and raggedness. It is a camera based system that uses a calibrated high precision optical measurement system to provide a full suite of image analysis algorithms to characterize the quality of printed output. The ImageXpert (IX) image quality measurement software (IX 10.0b63) processes the data and provides quantitative, objective image quality measurements that include dot quality, line quality, text quality, edge raggedness, halftone quality, resolution etc<sup>37</sup>.

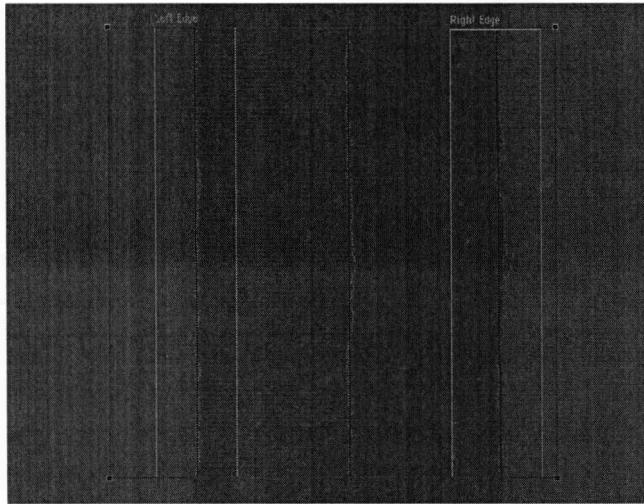


**Figure 6** ImageXpert (KDY Inc.) with MacRail software for image analysis.

Printed lines and the quality of line edges (raggedness), Figure 6, were the basic image elements that were examined. This system uses a binary threshold method for image analysis. The binary threshold separates dark pixels from light pixels, all pixels below the threshold are dark pixels; all pixels above the threshold are light pixels<sup>38</sup>. The reason behind adjusting the binary threshold is to mark which regions are the part of an image and which regions are the background. Different Regions of Interest (ROIs) were adjusted to measure line width, right edge and left edge mean deviation as shown in Figure 7.

The line width and raggedness for each printed trace was measured at 5 different places and the average is reported. Line length was measured

twice and the average compared to the nominal length of 50 mm to determine the line length gain. The line length and line width values were then used to calculate sheet resistivity ( $R_{SH}$ ) (Equation 4).



**Figure 7** Image seen under ImageXpert of SUB\_3/WB at 90% tone line trace.

### Electrical Resistance Measurements

The electrical properties of the printed traces were measured before and after calendering in terms of resistance ( $R$ ) and reactance ( $X$ ) using an Agilent 4338B milliohmmeter (Figure 8), which operated at a frequency of 1 kHz. These values were then used to calculate AC impedance of the traces (Equation 3). Finally, the sheet resistivity was calculated using the following equation<sup>4</sup>:

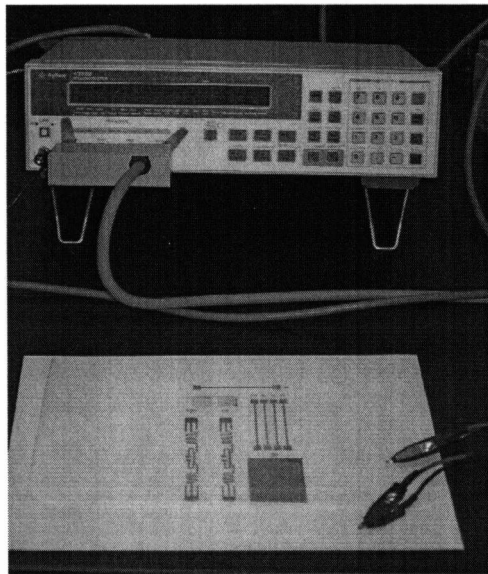
$$R_{SH} = R \frac{w}{l} \quad (4)$$

where:  $R_{SH}$  is sheet resistivity in  $\odot \text{sq}^{-1}$ ,

$R$  is the measure line resistance in  $\Omega$ ,

$w$  is the measured line width in mm,

$l$  is measured line length in mm.



**Figure 8** Agilent 4338B milliohm meter used to measure impedance.

### **Ink Film Thickness**

Line tones of 90 and 100% were used for this measurement. Initially, samples were embedded in an epoxy resin and allowed to solidify for at least 18 hrs. These samples were then ground and polished by using a grinder/polisher from Leco Corp. to achieve a highly polished surface. An

Olympus microscope in combination with a CCD camera, Figure 9, providing a total magnification of 1000x was used with Pax-it software to measure IFT (see Figures 10 and 11), from which the bulk resistivity was calculated (Equation 4). Two samples printed at 90 and 100% tone were selected for each calendering condition.

The IFT with resistance values were used to calculate bulk resistivity according to:

$$\rho_{DC} = R \frac{w}{l} t \quad (5)$$

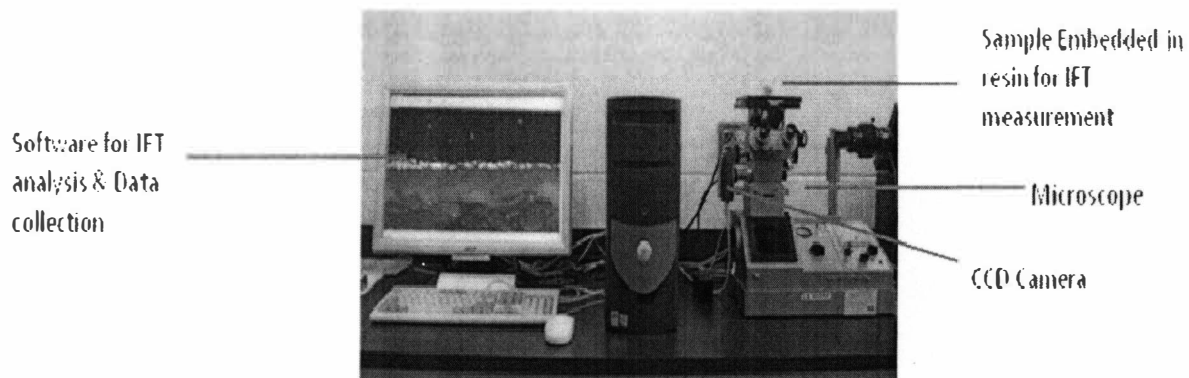
where:  $\rho_{DC}$  is bulk resistivity in  $\Omega$  mil,

R is line resistance in  $\Omega$ ,

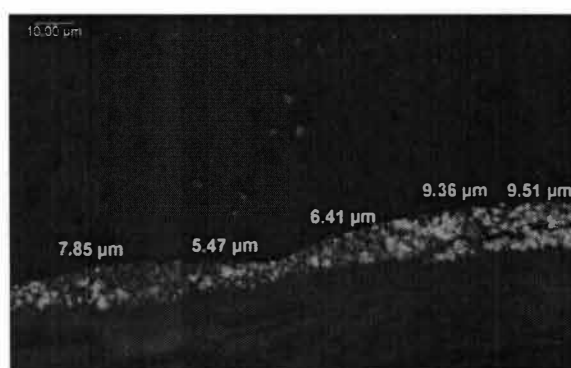
w is line width in mm,

l is line length in mm,

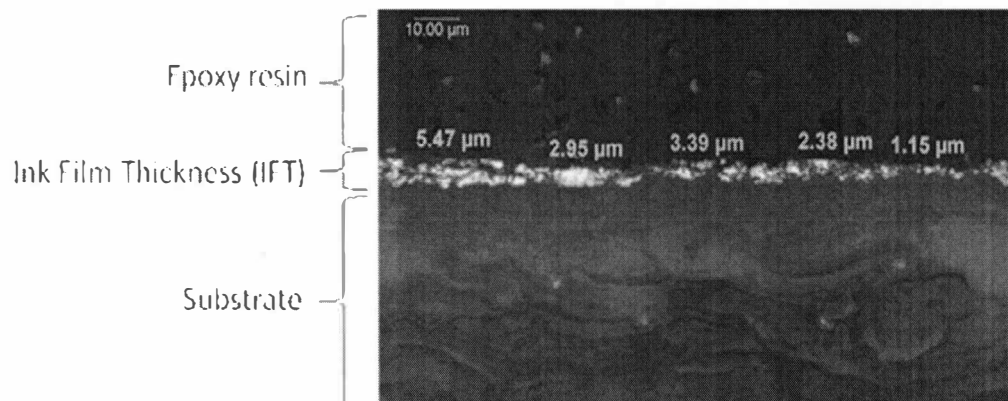
t is line thickness in mil ( $10^{-3}$  in).



**Figure 9** Olympus microscope with Pax-It software showing IFT on screen.



**Figure 10** IFT seen under Olympus microscope of SUB\_1/WB at 100% tone.



**Figure 11** IFT seen under Olympus microscope of SUB\_1/SB at 100% tone.

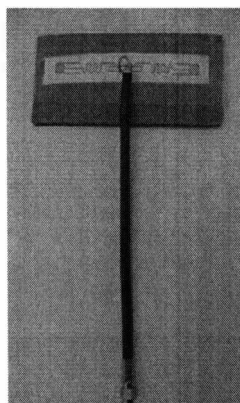


## UHF RFID Tag Antenna Performance

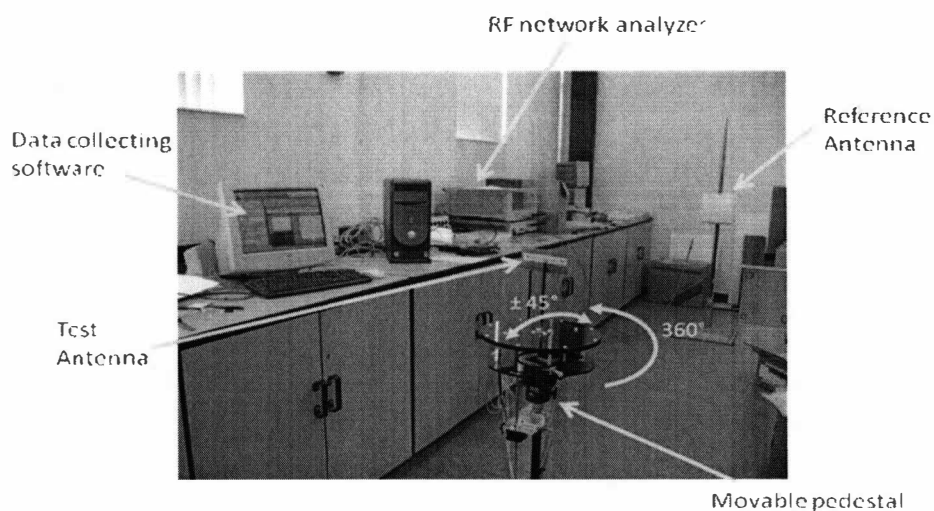
An Alien Technology UHF RFID tag “squiggle” (2nd generation) antenna design was printed and the effect of calendering conditions on its performance was studied. RFID antenna testing was conducted for each calendering condition. Initially, two samples for each condition were prepared by attaching a testing cable to the printed antenna with the help of a silver epoxy adhesive (Figure 12). Only the 100% tones were tested for antenna performance.

The antenna range consists of a fixed reference antenna at a defined distance to a test antenna placed on a movable pedestal, Figure 13. An RF (radio frequency) network analyzer (Agilent 4396B) was used to source a continuous wave RF signal at a known frequency and phase to the reference antenna. The antenna transmits the RF wave to the test antenna, where it is received as an input to the RF network analyzer. The network analyzer determines the incoming signals magnitude and relative phase as compared to the transmitted signal. The results are captured over an HP-IB network connection to a personal computer (PC), where operational software controls the test and data collection. The software and PC are also in control of the movable pedestal mounted on top of a tripod. The pedestal is programmed to

rotate 360 degrees in the horizontal plane shown bellow (Figure 12) and it can tilt the plane by  $\pm 45$  degrees. This allows a three-dimensional pattern of the antenna performance to be collected for viewing and analysis.



**Figure 12** Sample prepared for antenna performance test.



**Figure 13** Instrumental setup for antenna performance test.

## Data Analysis

The effects of substrate and ink type, line tone, calendering temperature and pressure were studied in terms of electrical impedance, ink film roughness, UHF RFID tag antenna performance, ink film thickness, sheet resistivity and bulk resistivity. Readings obtained from calendering experiments for each substrate were compared to each ink system. Each calendering parameter was first plotted for each condition with standard deviations. All calculations were done using Microsoft Excel 2007. This was first done to determine which particular measurements responded to the calendering conditions. MINITAB 15 was further used to analyze the data. An ANOVA analysis (analysis of variance) was performed to calculate P-values, and the main effects and interactions between the tested factors were plotted against each response.

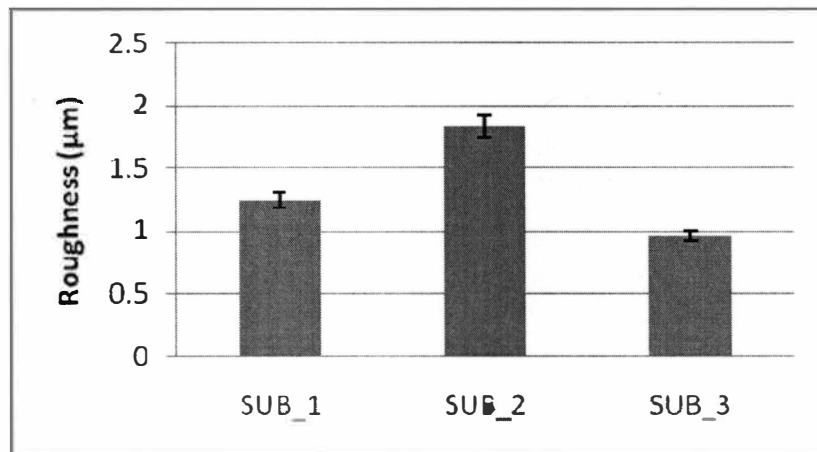
## CHAPTER VI

### RESULTS

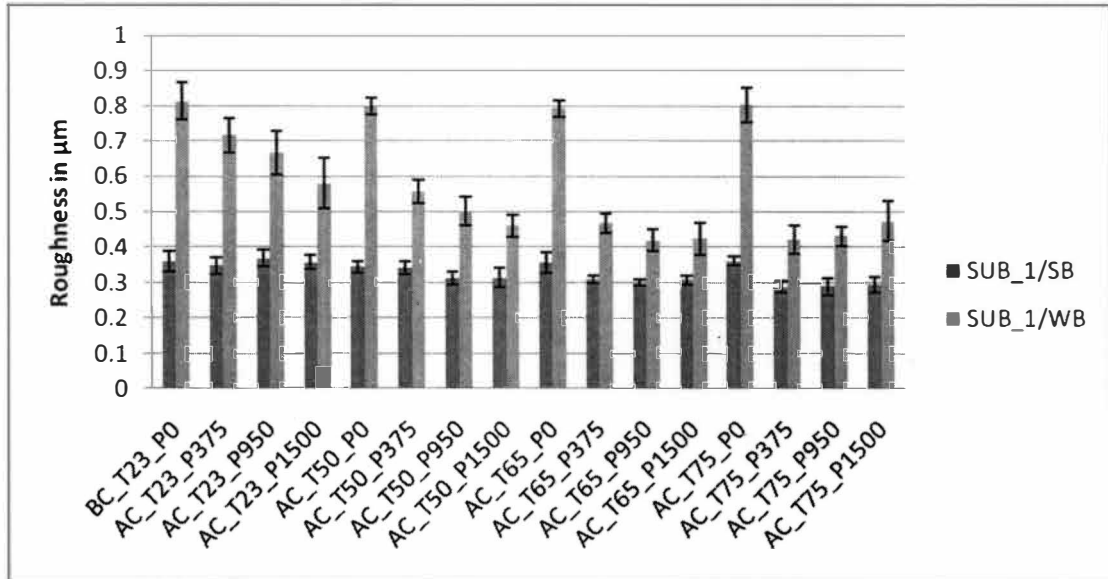
#### Ink Film Roughness

It is important to understand the physical differences between different samples in terms of fundamental properties. Calendering pressure and temperature could have an effect on ink film roughness and thickness properties. Substrate and ink film roughnesses were measured. The 90 and 100% line tones in the MD (machine direction) and 90% line tone in the CD (cross-machine direction) were measured and compared for each calendering condition.

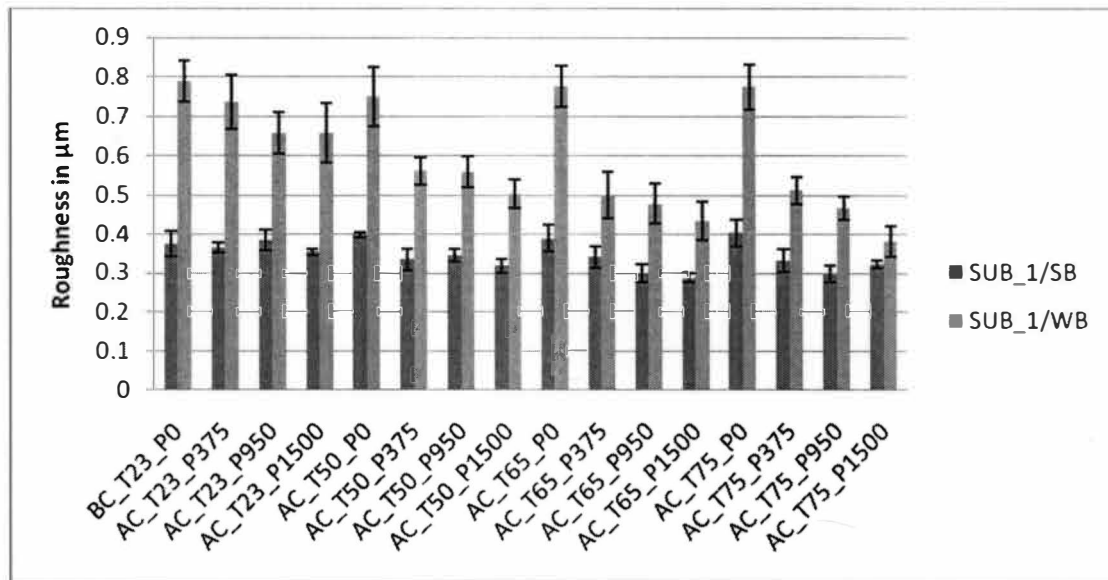
Figure 14 shows the Emveco roughness values in the machine direction for the uncalendered substrates. As SUB\_3 was a thermal coated paper it was found least rough ( $0.96 \mu\text{m}$ ).



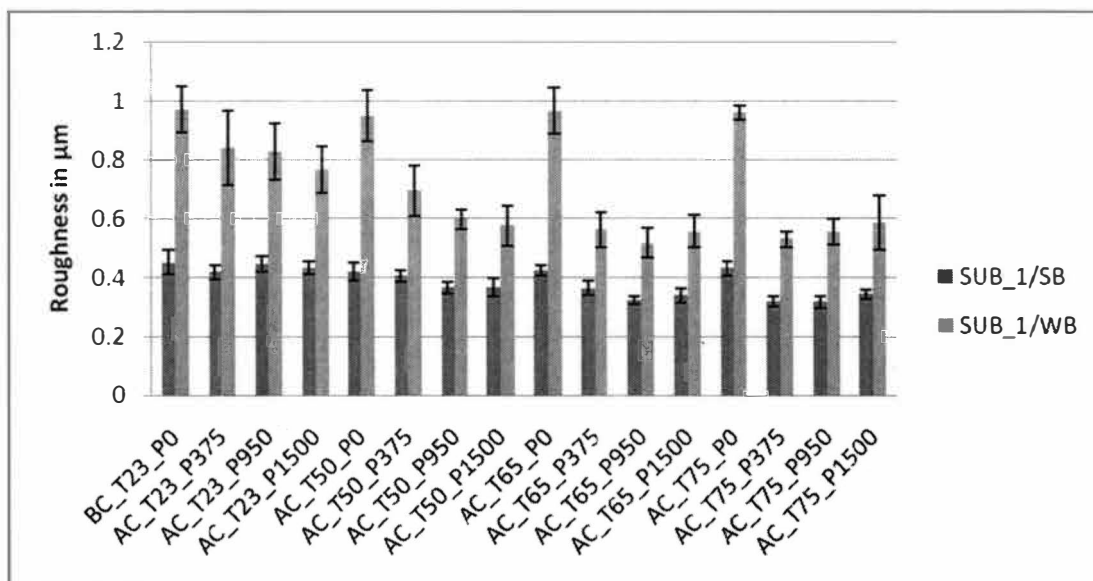
**Figure 14** Emveco roughness before calendering.



**Figure 15** Emveco roughness for SUB\_1 printed with WB & SB ink at 90% tone in MD.

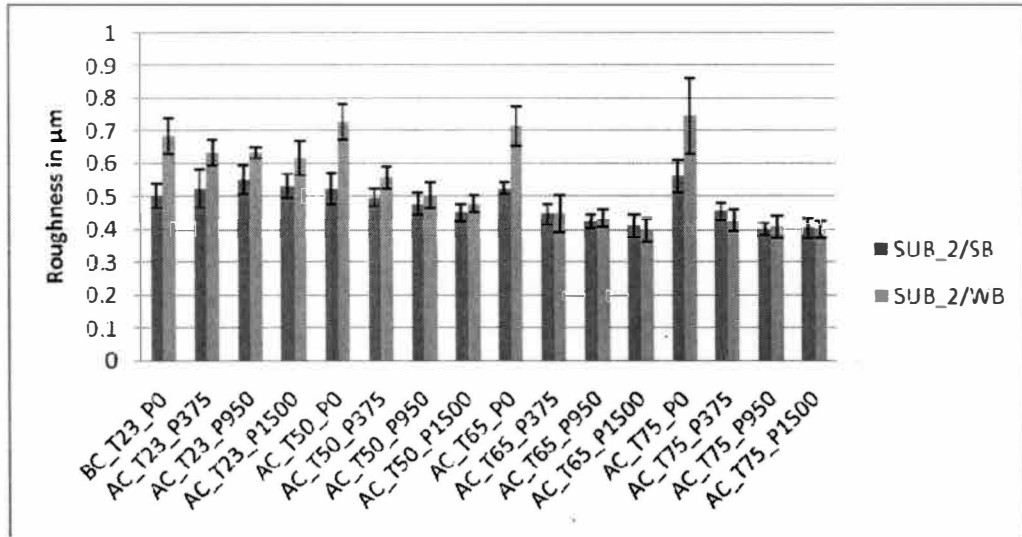


**Figure 16** Emveco roughness for SUB\_1 printed with WB & SB ink at 100% tone in MD.



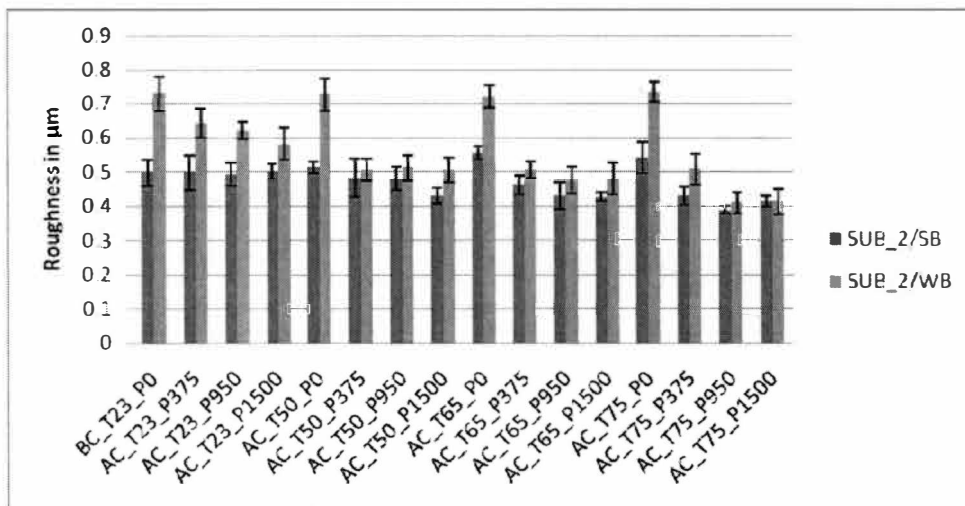
**Figure 17** Emveco roughness for SUB\_1 printed with WB & SB ink at 90% tone in CD.

Figures 15, 16 and 17 show the effect of calendering on ink film roughness printed with WB and SB inks for 90, 100 and CD 90% tones. At conditions where no calendering and at variable temperatures (23, 50, 65, 75°C) it is seen that calendering pressure has no significance influence on ink film roughness. It can be said that only calendering temperature is not making any significant effect on ink film roughness. When calendering pressure and temperature is acting together it is seen significant difference in the improvement of ink film roughness.

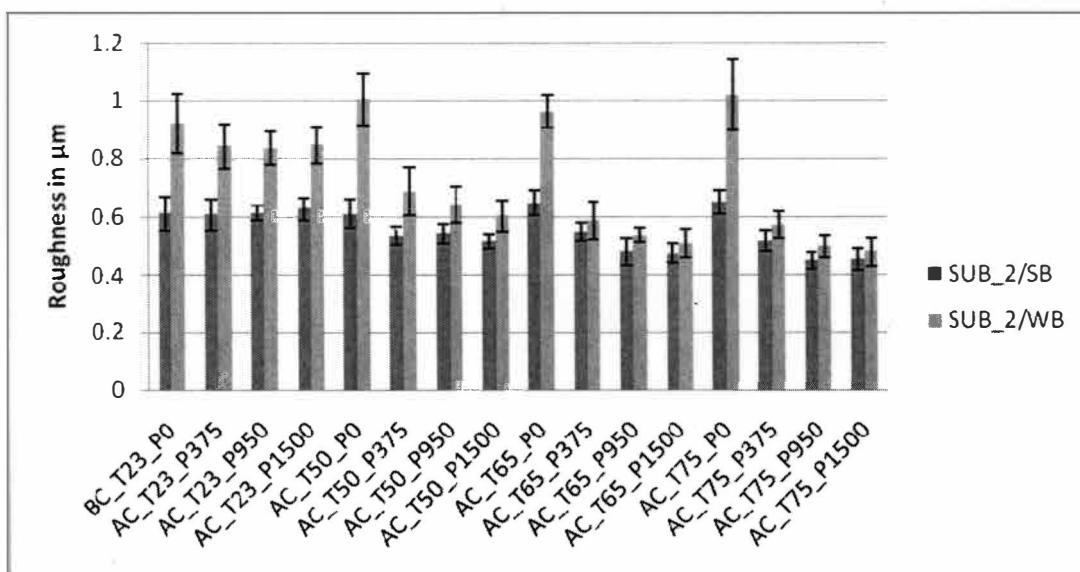


**Figure 18** Emveco roughness for SUB\_2 printed with WB & SB ink at 90% tone in MD.

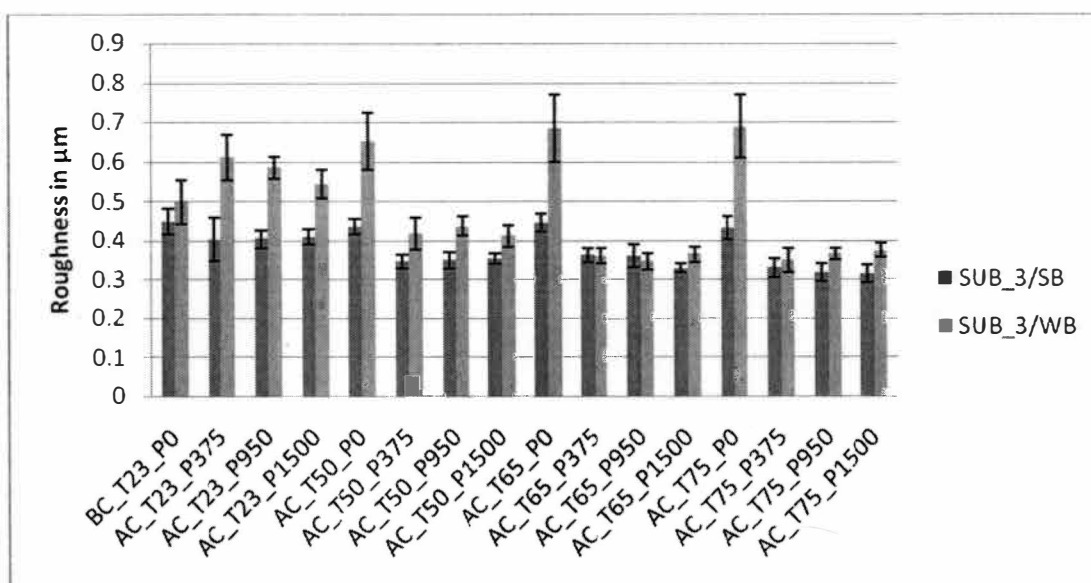
Figures 18 -23, show the ink film roughness values for SUB\_2's and SUB\_3's 90, 100 and CD 90% tone values. The same effect was observed as in the case for SUB\_1.



**Figure 19** Emveco roughness for SUB\_2 printed with WB & SB ink at 100% tone in MD.

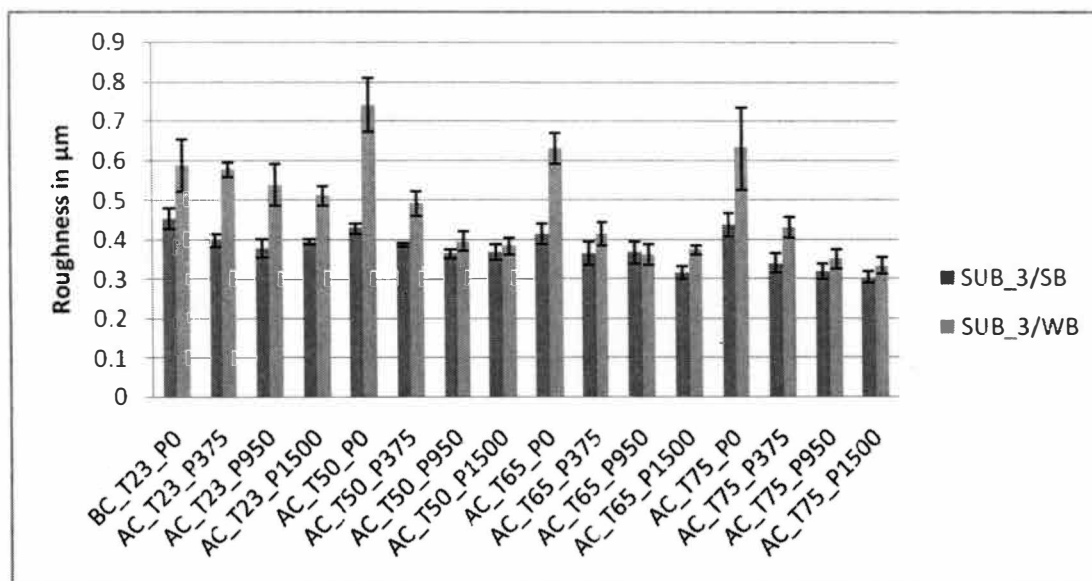


**Figure 20** Emveco roughness for SUB\_2 printed with WB & SB ink at 90% tone in CD.

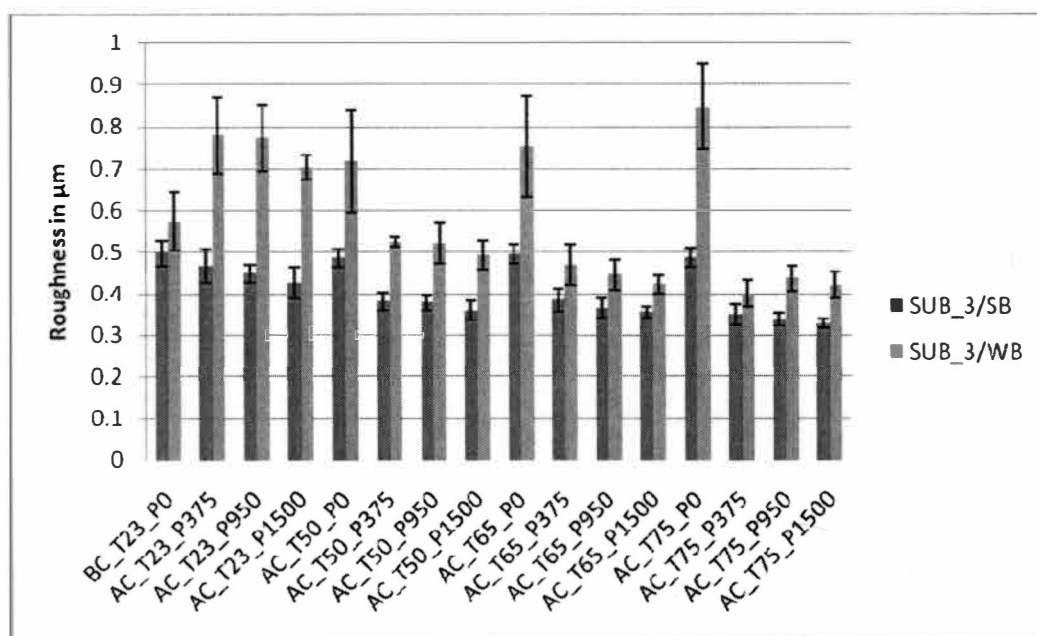


**Figure 21** Emveco roughness for SUB\_3 printed with WB & SB ink at 90% tone in MD.





**Figure 22** Emveco roughness for SUB\_3 printed with WB & SB ink at 100% tone in MD.



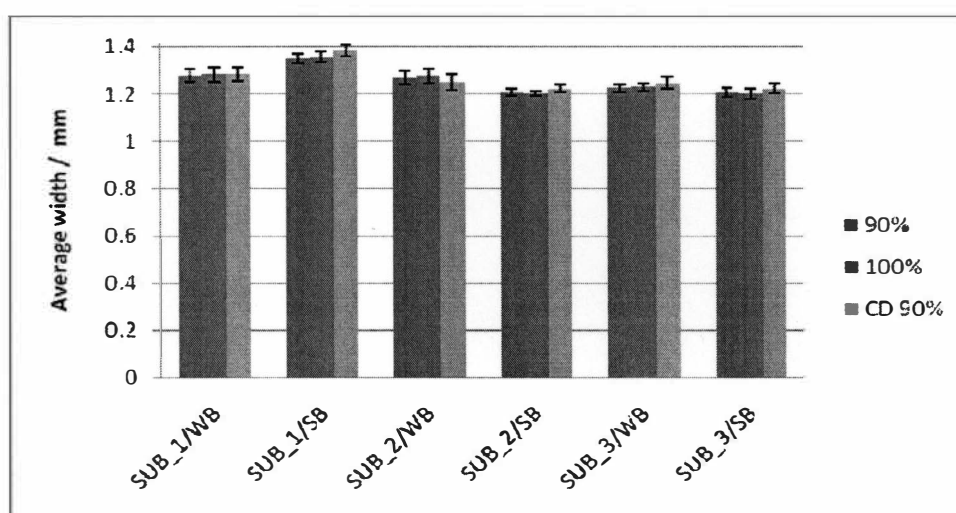
**Figure 23** Emveco roughness for SUB\_3 printed with WB & SB ink at 90% tone in CD.

All the substrates show lower ink film roughness values for SB printed samples. From the results of ink film roughness, it is seen that, as calendering pressure and temperature increased, the smoothness of the printed WB and SB inks improved. A more pronounced decrease in ink film roughness was observed for the WB ink. It is believed that both the softening and compression of the resins in the ink film and the replication of the metal roll surface resulted in the increased smoothness of the printed image. For all substrates, the WB ink showed a higher decrease in roughness than those printed with SB ink. This is most likely due to the differences in ink film thickness. The ink film thicknesses of the WB ink were higher than the SB ink, so these films could be compressed more. It was also found that temperature alone did not affect the roughness of the printed image.

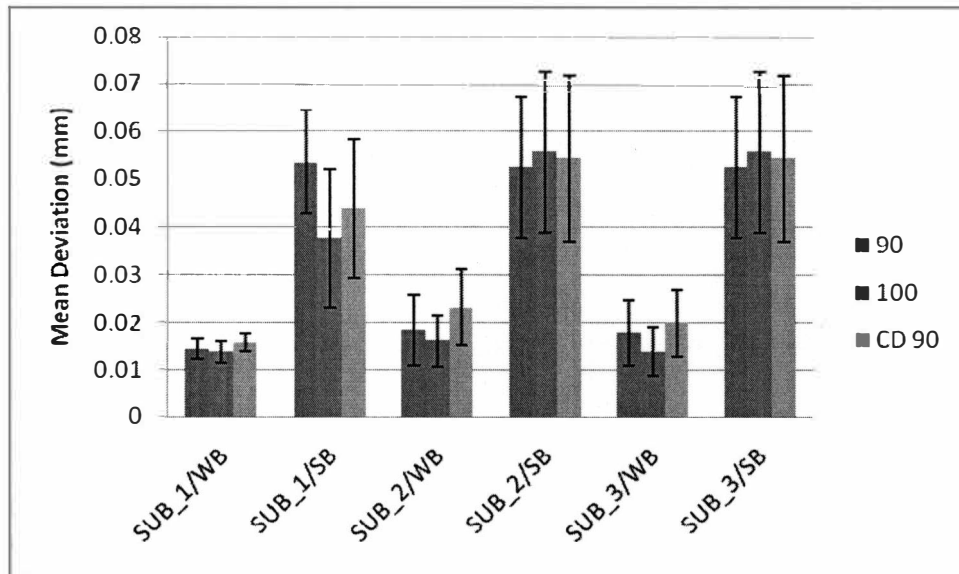
### **Line Width and Line Length**

Print accuracy for all printed electronic devices is very important for performance. Line widths and lengths were measured to determine the fidelity of the printed traces. These values were also used to calculate sheet resistivity. The following graphs (Figures 24, 25 and 26) show the average line widths, raggedness, and lengths for the MD 90 and 100%, and CD 90% line tones printed

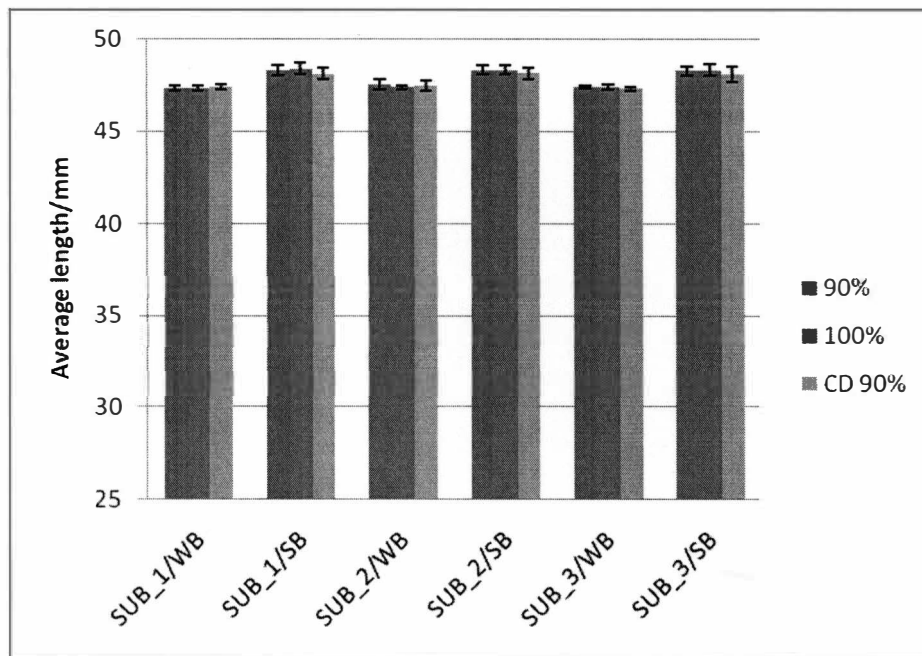
on various substrates. The nominal line width for the measured line is 1mm and the length is 50 mm. The average line widths of the measured samples ranged from 1.20mm ( $\pm 0.01$ mm) to 1.38mm ( $\pm 0.02$ mm). Increase in the line width was observed after printing compared to original 1 mm. The average line lengths ranged from 47.27mm ( $\pm 0.09$ mm) to 48.36mm ( $\pm 0.32$ mm). Decrease in the line length was observed after printing compared to original 50 mm.



**Figure 24** Average line width for WB and SB ink.



**Figure 25** Average line raggedness for the WB and SB ink.

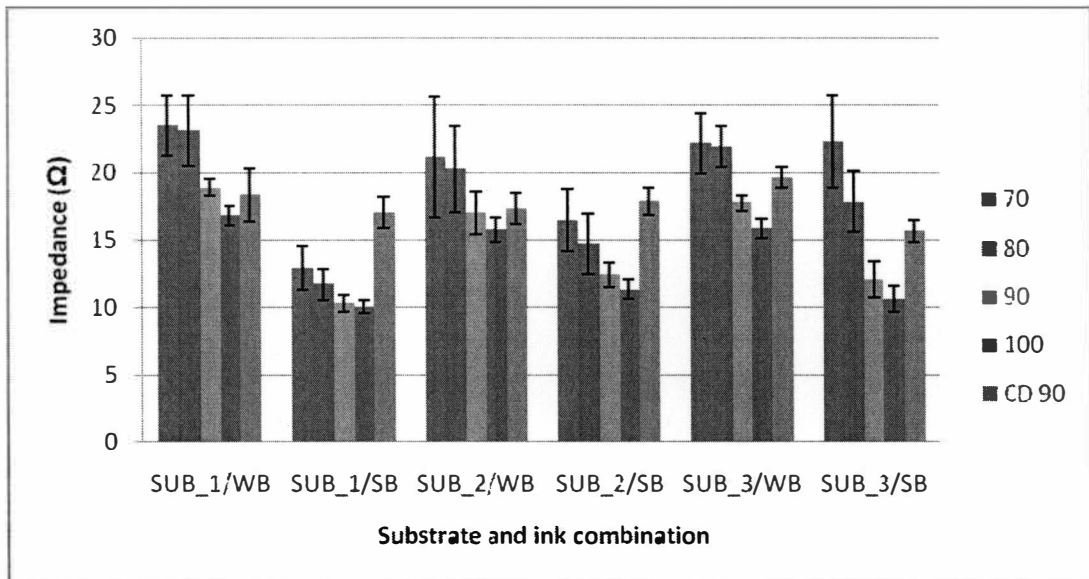


**Figure 26** Average line length for the WB and SB ink.

The results of image analysis (Figure 24, 25 and 26) showed increased in line width and decreased in line length. The reason behind this can be line length is printed in cylinder rotation direction and widths are printed across direction. When printing pressure is applied, it has a more pronounced effect on line edges than line length. It was also seen that WB ink has less line edge deviation compared to SB inks. Samples were checked for after calendering conditions. It was found there is no significant difference between the line length and line width values. Therefore results of after calendering are omitted for brevity.

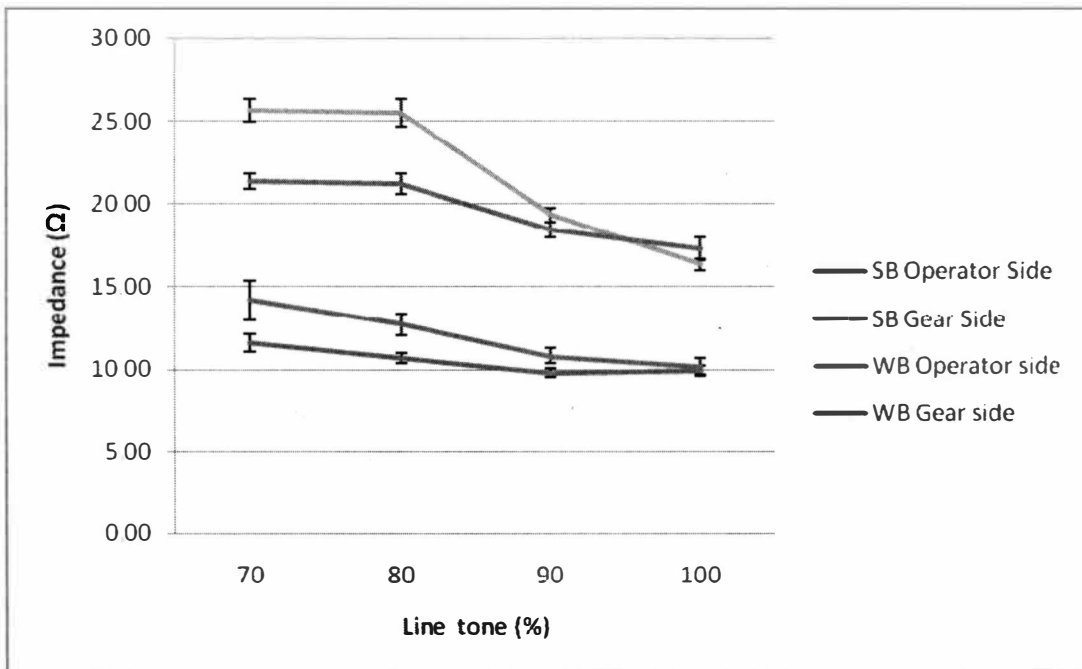
### **Electrical Impedance**

Before calendering, the AC impedance of each sample was measured. These values are shown in Figure 27. The lowest impedance values were found for the 100% line tone, which had the highest ink coverage.



**Figure 27** Comparisons of uncalendered impedance values.

During measurement, it was noticed that the impedance of the printed traces showed differences between the gear side and operator side of the press. It is believed that the differences were due to an uneven impression pressure being applied resulting in variations in ink transfer from side-to-side.



**Figure 28** Impedance comparisons between two sides for WB and SB ink in relation to tone step value for SUB\_1.

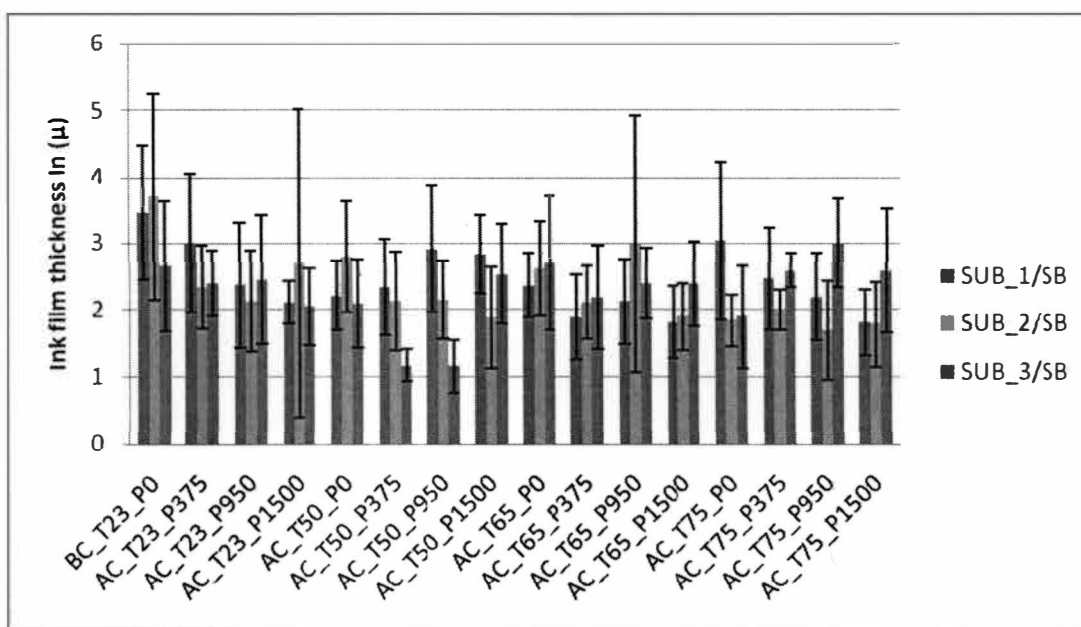
This was observed for all substrates, for both ink systems, so for brevity, only the results for SUB\_1 are reported (Figure 27). The 70 and 80 % tones were most affected by the “gear-sidedness” of the press, since their ink coverage suffered most by the differences in impression pressure. For the 90 and 100 % tones, the values were within one standard deviation.

### Ink Film Thickness

Ink film thickness was measured for the 90 and 100% line tones. Two samples for each calendering condition were tested. Images were captured for

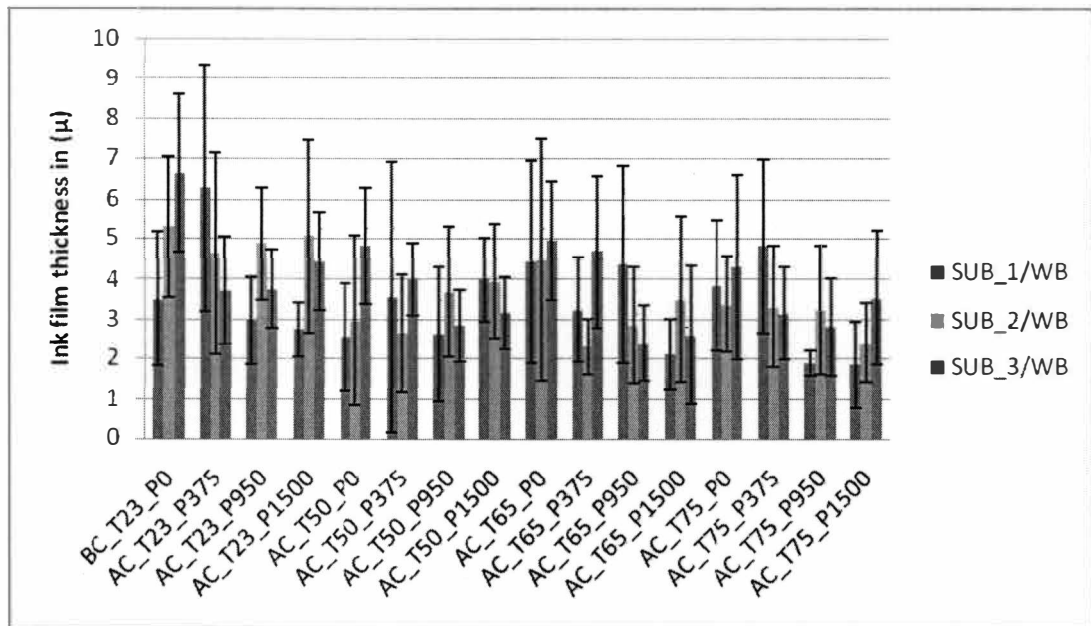
each line tone at 3 different positions along the line. At each of the 3 positions, 5 measurements were taken. The measured values are shown in Figures 29 - 32.

The thicknesses of the WB ink films were higher than the SB ink. The variation in IFT was also found to be lower for the SB ink than the WB ink. The standard deviations for the 90 and 100% line tones, for both inks, for all substrates, were large.

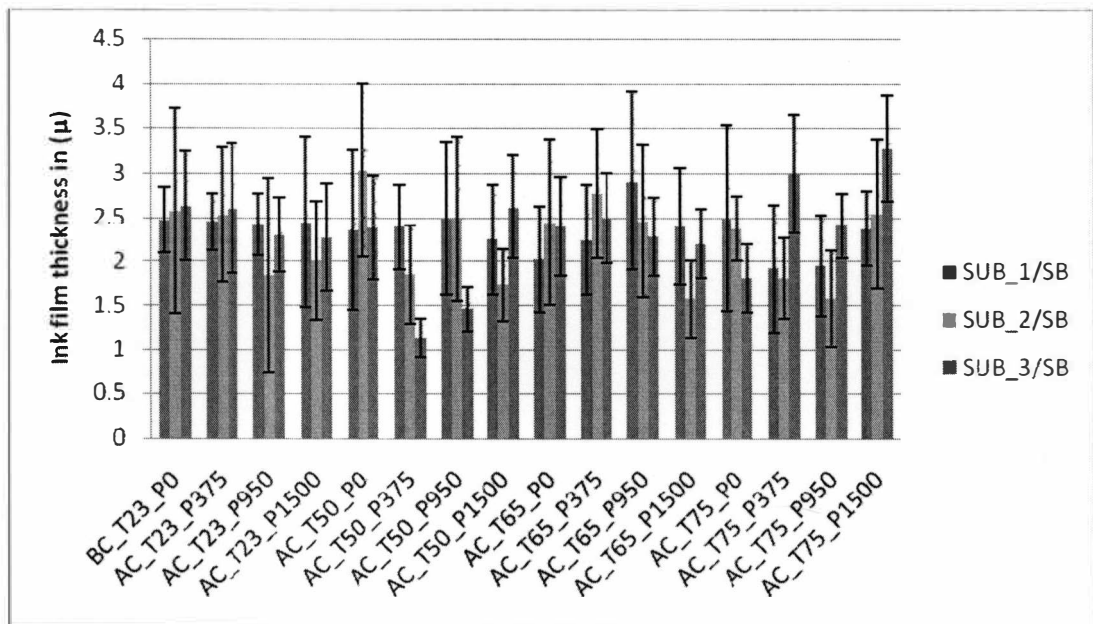


**Figure 29** Ink film thickness for SB ink at 90% tone.

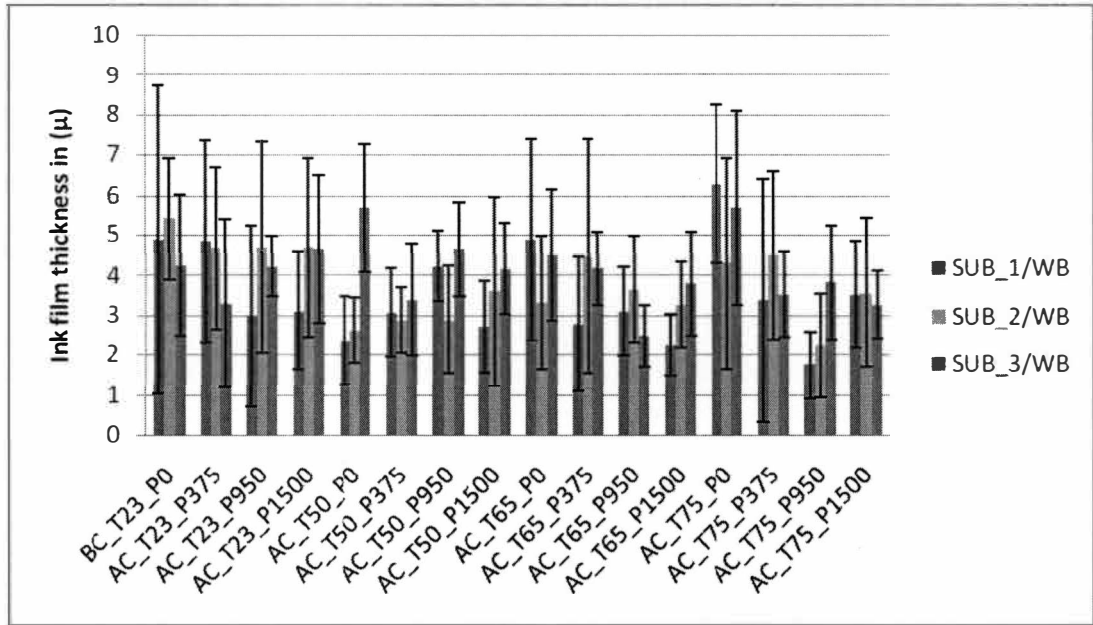




**Figure 30** Ink film thickness for WB ink at 90% tone.



**Figure 31** Ink film thickness for SB ink at 100% tone.



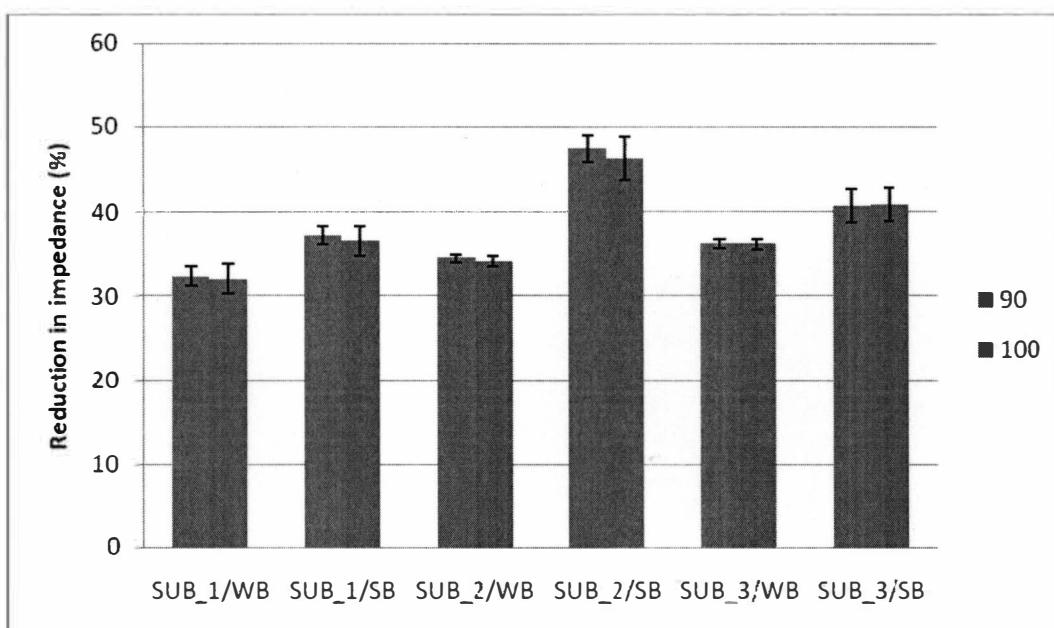
**Figure 32** Ink film thickness for WB ink at 100% tone.

The results of IFT showed lower variation in IFT for the SB ink indicates that the silver particles may be better aligned. However, SEM pictures should be taken to confirm this. A smoother ink film is capable of more readily transferring electrons. This would reduce the impedance of the printed trace and is most likely why the SB ink impedance is lower than the WB ink. As seen from the figures, there is no visible trend in reduction of IFT after calendering.

### Effectiveness of Calendering in Reducing Electrical Resistance

For a better comparison, the impedance values for all calendering conditions were converted in % reduction in impedance. The 0% reduction for the impedance values are given for the before calendering conditions, and all

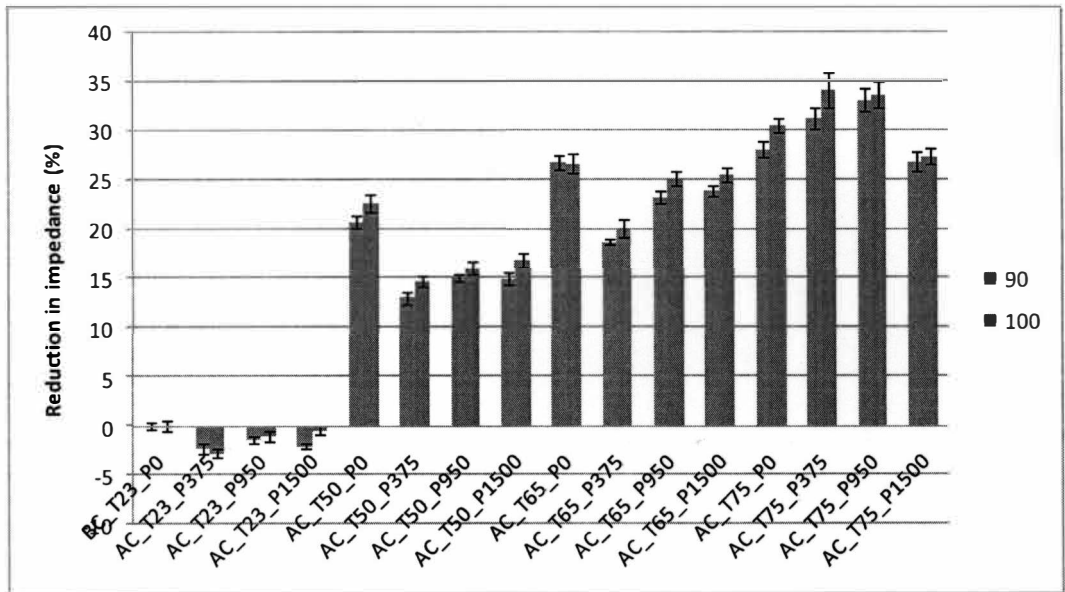
other conditions are compared to that. To compare the efficiency of calendering with post-heat treatment, reduction in impedance was also measured for samples heated in the oven at 105°C for 4 hrs and the results are plotted in Figure 33. It was found that SB ink responded more readily to the heat treatment compared to WB ink, regardless of the substrate used. This is observed because resins used to formulate WB and SB are different, they must have had reacted differently to temperature.



**Figure 33** Reduction in impedance for all substrates heat treated in an oven at 105°C for 4 hrs.

Figure 34 shows, percentage reduction in impedance after calendering for SUB\_1 with the water based ink system. No reduction in impedance was observed at constant calendering temperature of 23 °C and varying pressures.

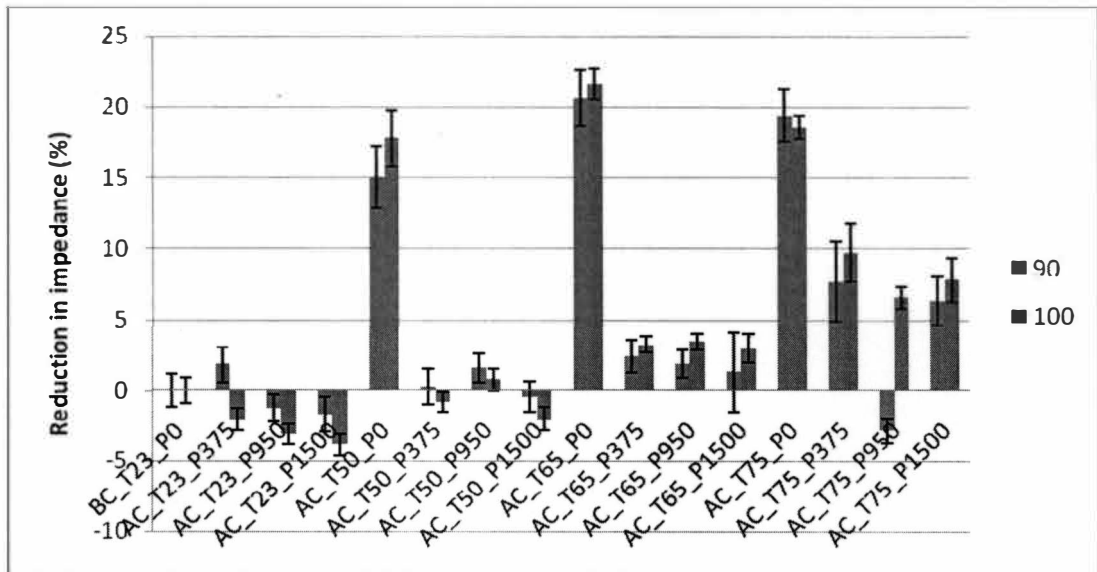
But after increasing the calendering temperature, a significant improvement was observed. Increasing the calendering pressure caused a greater reduction in impedance for the 50 and 65 °C temperatures, than at 75 °C. At 75 °C, increasing the pressure resulted in a greater reduction in impedance. As much as a 34% reduction in impedance was achieved by calendering. This large reduction indicates that calendering is a useful method for improving the conductivity of the silver flaked inks without the need for post cure.



**Figure 34** Reduction in impedance for SUB\_1/WB after calendering at various temperatures and pressures.

Figure 35 shows the percent reduction in impedance for the SUB\_1 substrate printed with the SB ink. Similar to the WB ink, no reduction in impedance was observed at 23 °C with pressure. For the 90% and 100% line tones

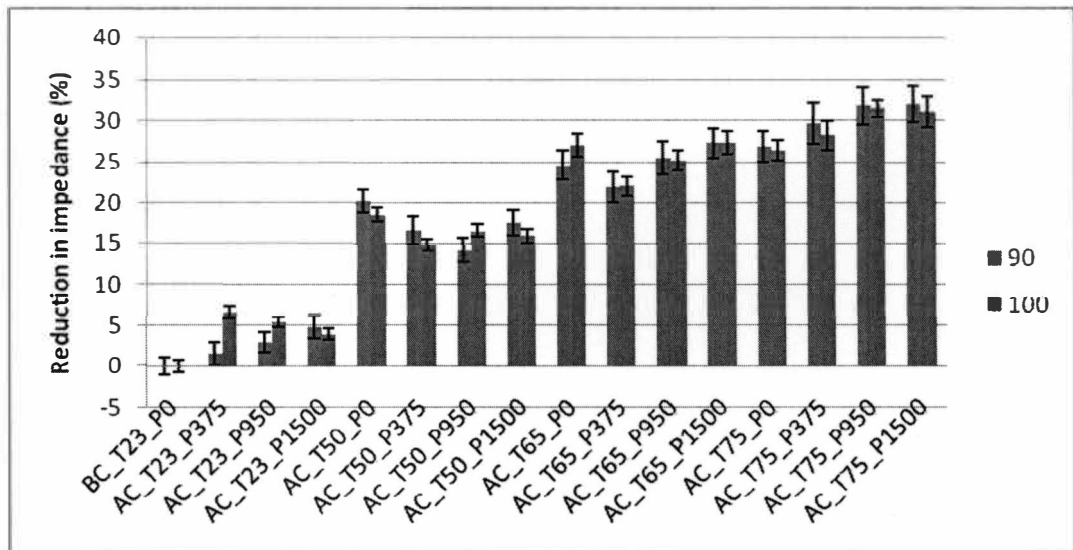
at the AC\_T65\_P0 condition, a maximum reduction in impedance 20.65% and 21.61%, respectively was obtained. At conditions 23°C temperature and varying pressures, negative values observed for impedance reduction as pressure increases. This is due to the pressure is making SB inks rough with application of pressure, and the alignment of silver particles get disturb. As it is mention before (Table 1) that SB inks have larger particle size than WB inks, so for only oven heated samples it has been seen a reduction in impedance values for SB inks.



**Figure 35** Reduction in impedance for SUB\_1/SB after calendering at various temperatures and pressures.

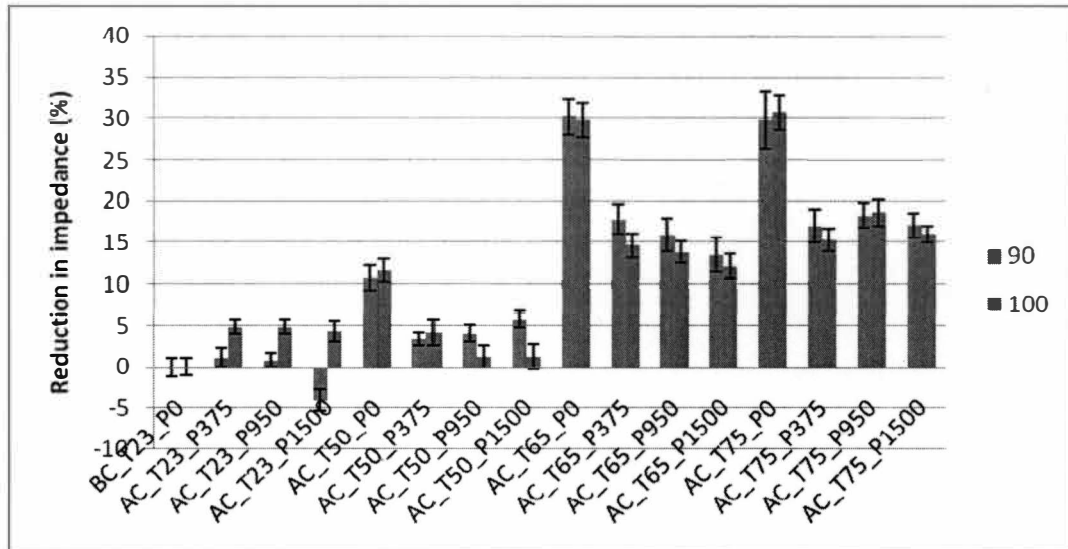
Figure 36 shows the percentage reduction in impedance obtained for the SUB\_2 and WB ink sample. The highest reduction in impedance, 32.0% ( $\pm 2.5$ ), was obtained for the 90% line tone at the AC\_T75\_P1500 condition. The 100%

line tone showed a 31.4% ( $\pm 1.0$ ) reduction at the AC\_T75\_P950 condition. Here similar kinds of effects are seen as of SUB\_1 with WB inks



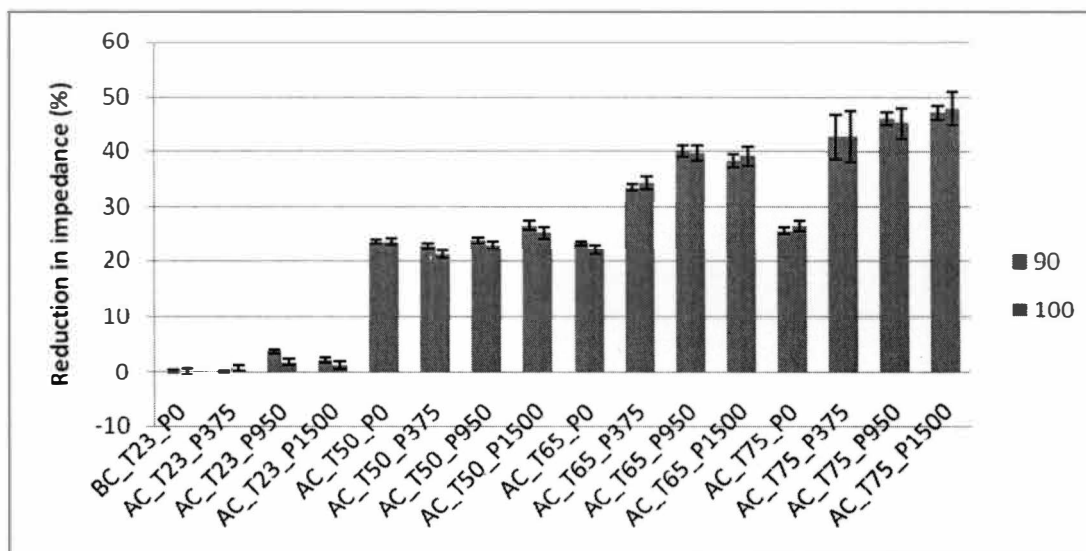
**Figure 36** Reduction in impedance for SUB\_2/WB after calendering at various temperatures and pressures.

An increase in the impedance was observed for the SUB\_2 with SB ink for the 90% line tone at the AC\_T65\_P0 condition and 100% line tone at AC\_T75\_P0, Figure 37. Calendering temperature having more significant effect on the reduction of impedance for SB inks than pressure.



**Figure 37** Reduction in impedance for SUB\_2/SB after calendering at various temperatures and pressures.

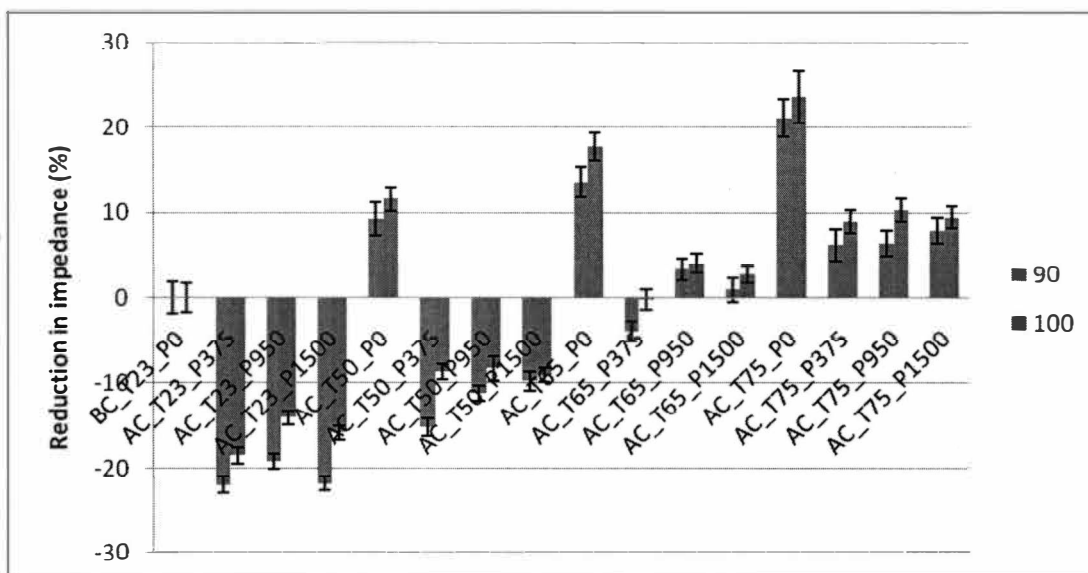
Figure 38 shows, the %reduction in the impedance after calendering for the SUB\_3 with the WB ink system. No major reduction in the impedance was observed at constant calendering temperature of 23 °C and varying pressures. But after increasing in the calendering temperature, a significant reduction was observed. For the 90% and 100% line tone at the AC\_T75\_P1500 condition, a maximum reduction in the impedance was observed- 46.96% and 47.77% respectively. As the ink film thickness for SB in is lower they get affected easily is higher temperature and pressure combinations. Which in turns into increase in the impedance values.



**Figure 38** Reduction in impedance for SUB\_3/WB after calendering at various temperatures and pressures.

Figure 39 shows, the % reduction in the impedance after calendering for the SUB\_3 with the SB ink system. For the 90% line tone at the AC\_T75\_P0 condition, the maximum impedance was 21.10% and for the 100% line tone, the maximum reduction (23.59%) occurred at AC\_T75\_P375. As it is seen that surface of this substrate is smoother compared to others. Calendering pressure affects in disturbing in the ink film thickness, and that is the reason maximum impedance values were observed for this combination.

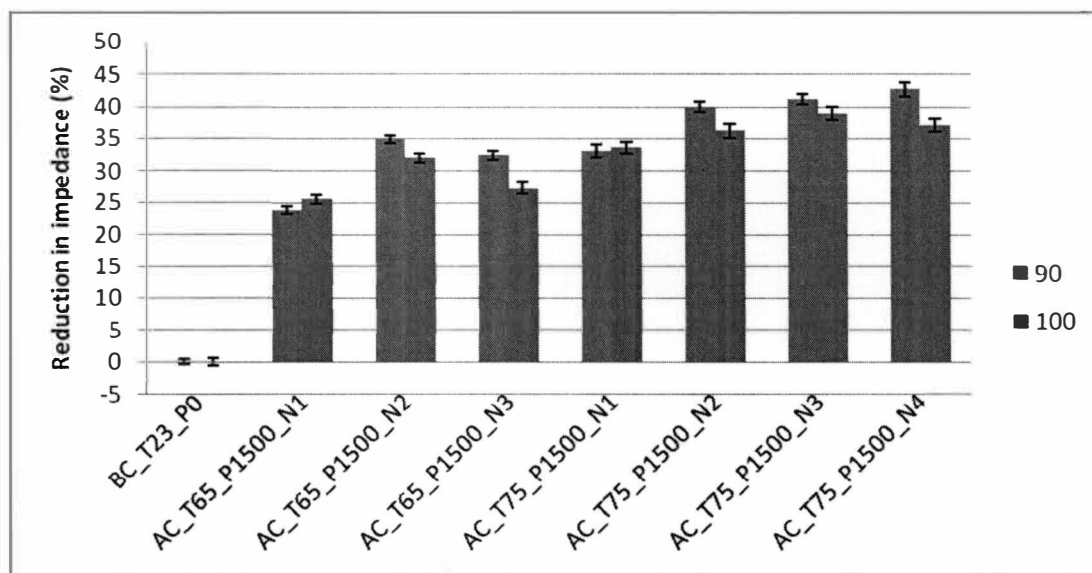




**Figure 39** Reduction in impedance for SUB\_3/SB after calendering at various temperatures and pressures.

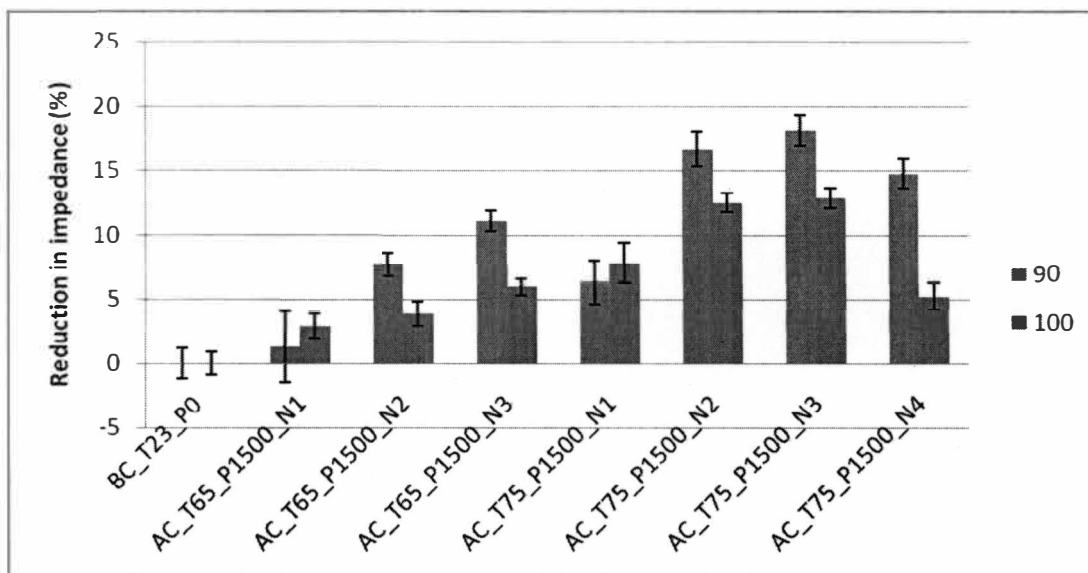
Additional studies were performed to determine if impedance could be further reduced by calendering the substrates through more than one nip. For this study, SUB\_1 printed with the WB and SB inks were passed through the calender 2, 3, and 4 times. The changes in impedance were then compared to the single nip findings (Figure 40). It was found that an additional reduction in impedance could be obtained by calendering the samples through 2 nips. As samples are passed more than one time through calendering nip and this is done within a quick time, resins didn't get a chance to dry and therefore they become softer than for one pass nips. At the same time application of pressure arrange silver particles closer. Therefore it has been observed that higher calendering

pressures and temperatures with maximum nip has maximum reduction in impedance.



**Figure 40** Reduction in impedance for SUB\_1/WB at 2, 3 and 4 passes through the calender nip.

Comparisons between the values of 2, 3 and 4 passes through the calendering nip for SUB\_1 printed with SB ink are plotted in the Figure 41. At the AC\_T75\_P1500\_N2 condition we found significant difference and after that there is no significant reduction in impedance was seen for 3<sup>rd</sup> and 4<sup>th</sup> pass. As it is seen before pressure is affecting SB inks, therefore these inks are not reacting as the same way as WB inks.

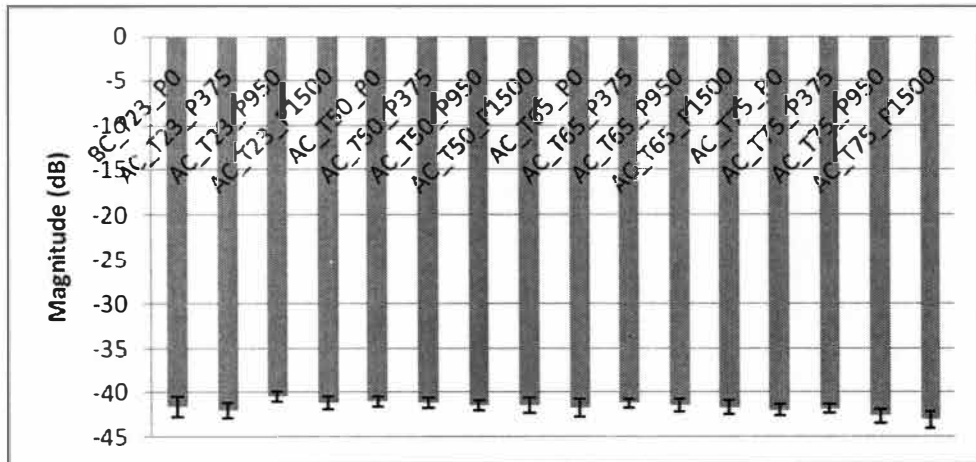


**Figure 41** Reduction in impedance for SUB\_1/SB at 2, 3 and 4 passes through the calender nip.

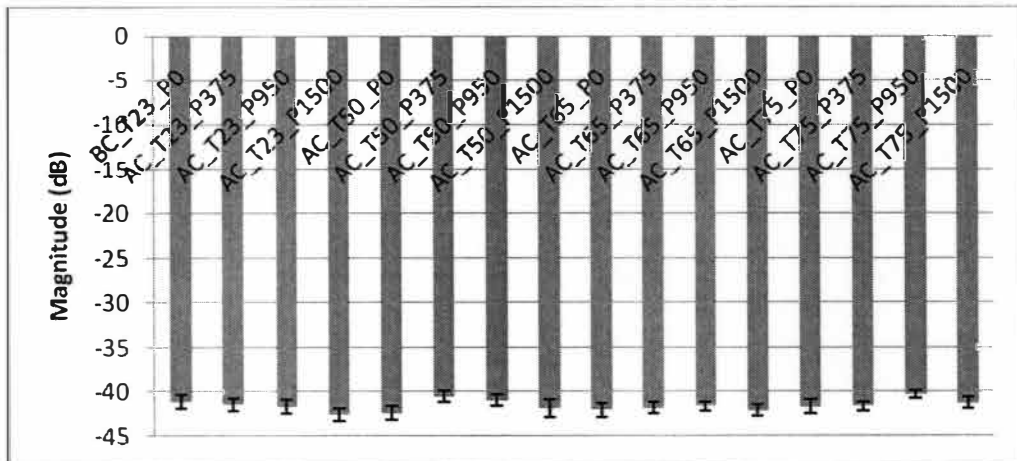
Results of reduction in conductivity showed that maximum reduction in impedance values was observed for WB inks. Even it was found better when samples pass at multiple times through calendering nip.

### UHF RFID Antenna Performance

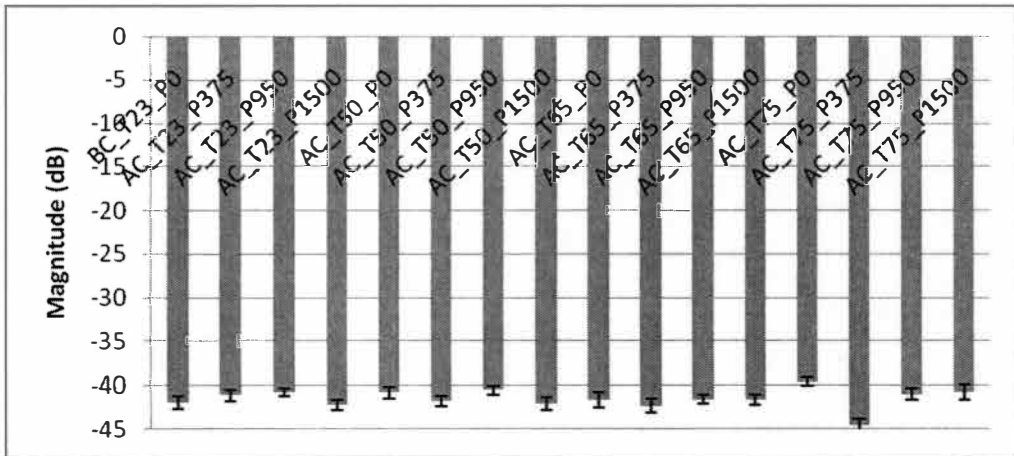
For each condition, 2 samples were tested. As seen from Figure 27, the 100% line tone performed the best. Based on these results, the 100% line tone was selected for antenna testing. Figures 42 – 47 show the antenna performance for each ink with each substrate.



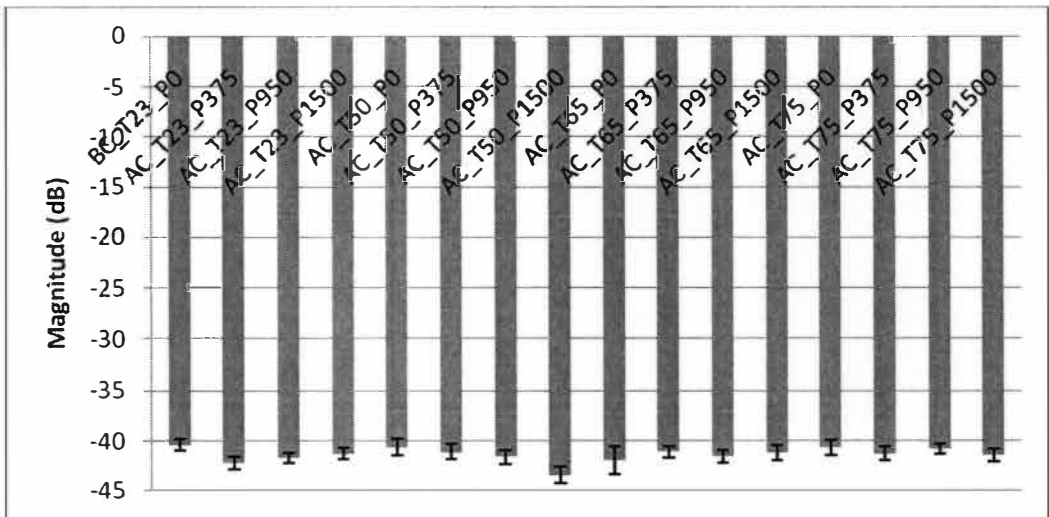
**Figure 42** Antenna performance results for SUB\_1/WB at 100% tone.



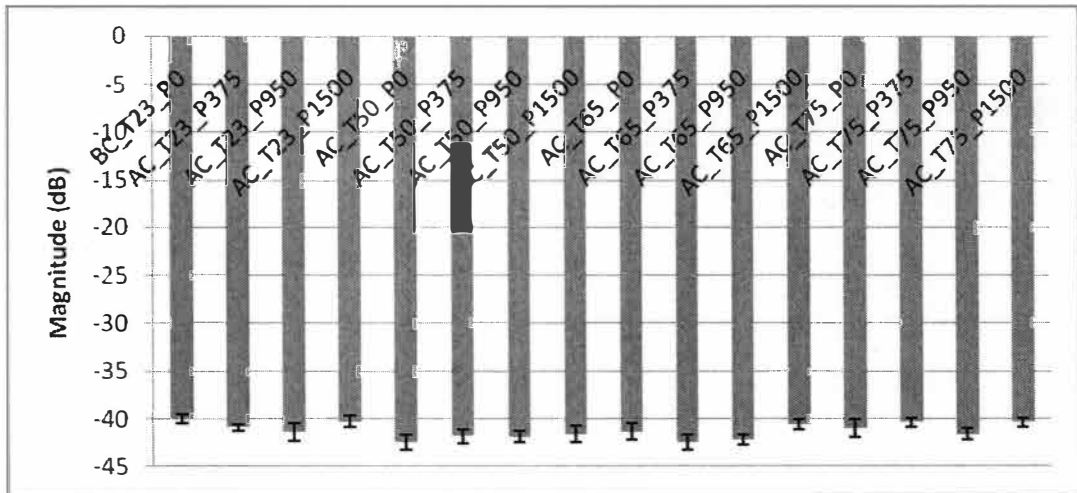
**Figure 43** Antenna performance results for SUB\_1/SB at 100% tone.



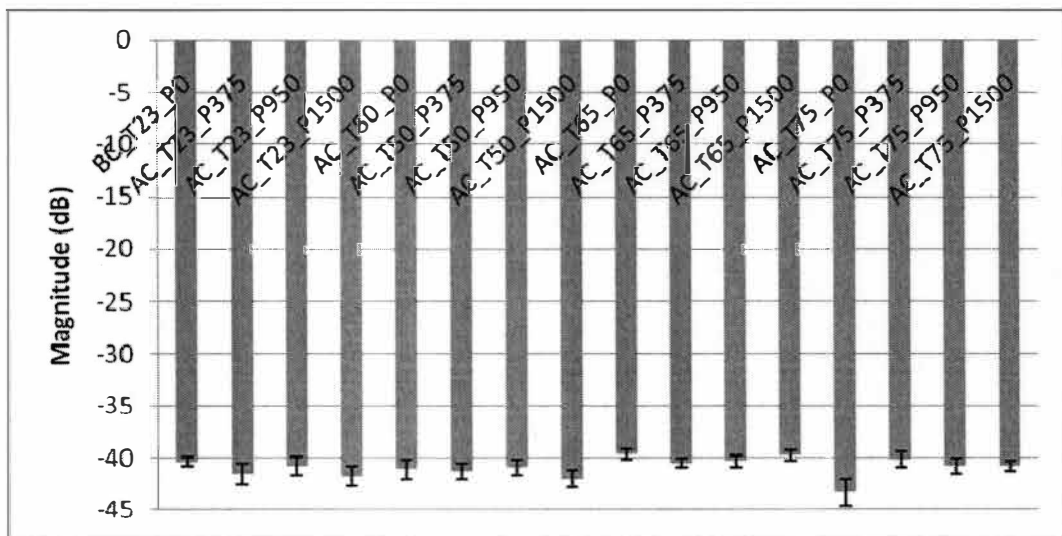
**Figure 44** Antenna performance results for SUB\_2/WB at 100% tone.



**Figure 45** Antenna performance results for SUB\_2/SB at 100% tone.



**Figure 46** Antenna performance results for SUB\_3/WB at 100% tone.

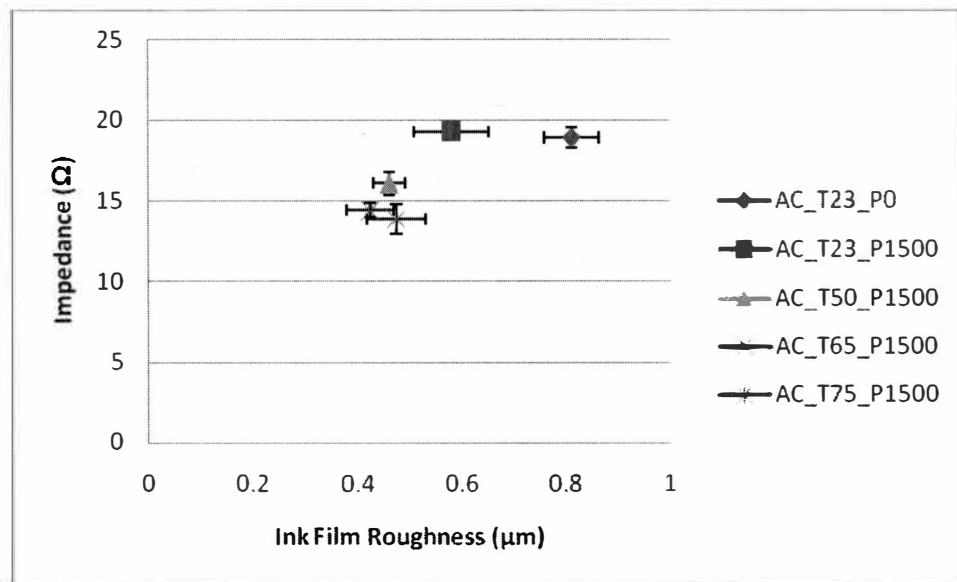


**Figure 47** Antenna performance results for SUB\_3/SB at 100% tone.

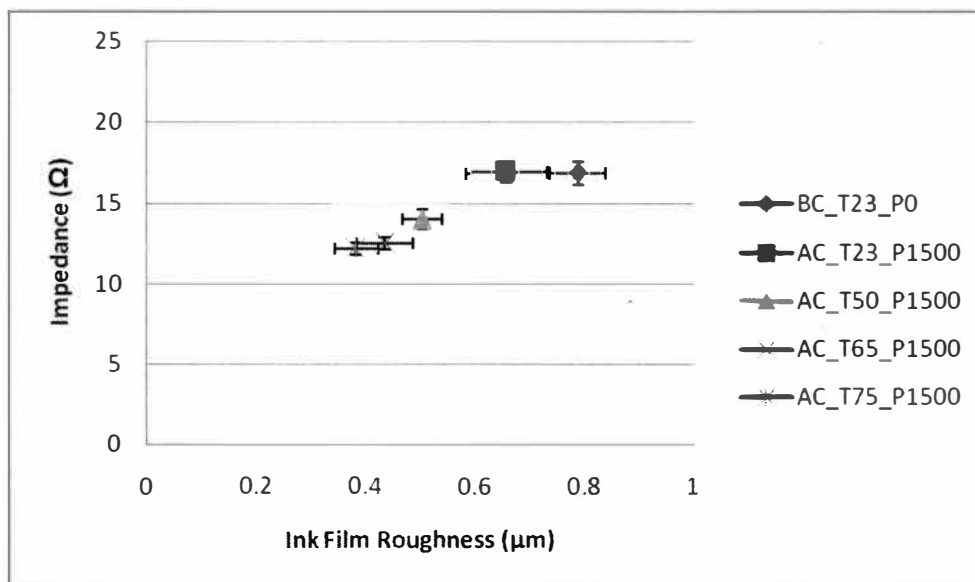
For antenna performance results no particular trend is observed as increase in calendering temperature and pressure. All the tested antennae performed well, with the loss in magnitude being within an acceptable range (above -46.4 dB).

## Resistance and Ink Film Roughness

A comparison was made to check the results of the effects of calendering on resistance and ink film roughness. As seen before, there was a maximum reduction in impedance and improvement in ink film roughness at the higher calendering temperature and pressure conditions. Therefore, attempts to correlate the relationship between resistance/ink film roughness for only these samples were made (Figures 48 and 49).



**Figure 48** Impedance versus ink film roughness plots for SUB\_1/WB printed at 90% line tone.

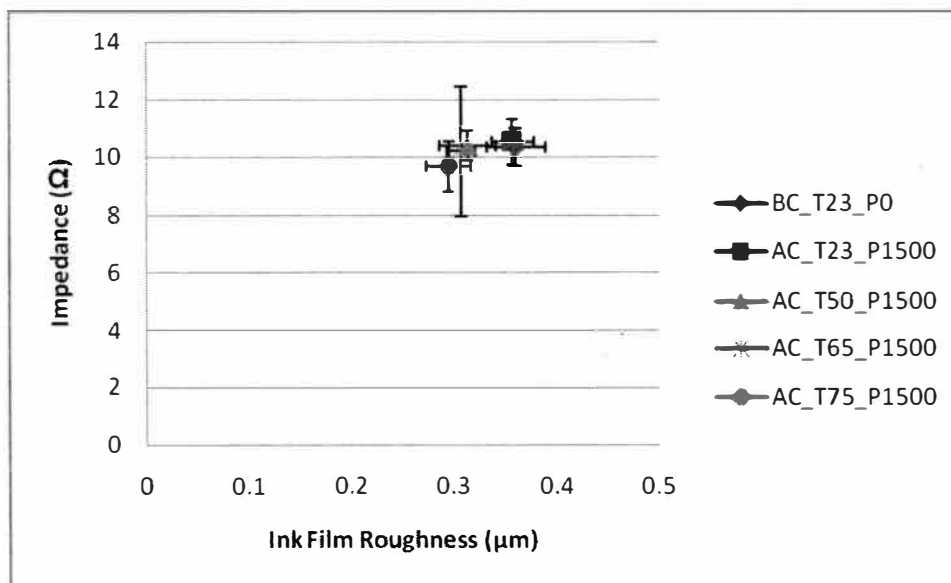


**Figure 49** Impedance versus ink film roughness plots for SUB\_1/WB printed at 100% line tone.

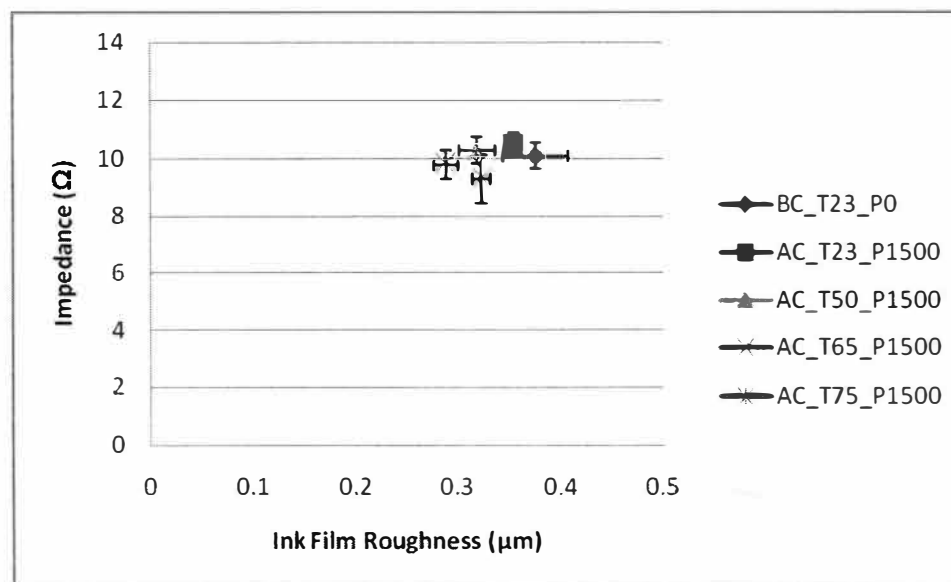
From Figures 48 and 49, it is evident that at calendering temperatures of 65 and 75°C along with highest pressure shows the maximum reduction in impedance and smoothness in ink film thickness. From both the graphs it was observed that when smoothness of the ink film increases there is reduction in impedance values.

From Figure 50 and 51, it seen that for SB inks, as improvement in the roughness reduction in the impedance values. But it is not seen as big as for the WB inks.





**Figure 50** Impedance versus ink film roughness plots for SUB\_1/SB printed at 90% line tone.



**Figure 51** Impedance versus ink film roughness plots for SUB\_1/SB printed at 100% line tone.

A statistical analysis was done to find out the correlation between ink film roughness and impedance. Regression fit indicates the strength of a linear relationship between two variables. It was found regression coefficient of Impedance ( $\Omega$ ) and Roughness (microns) = 11.637, P-Value = 0.000. Since the coefficient is 11.637 it can be said that the corresponding variables closely vary together in the same direction. A scatter-plot for impedance and roughness (refer Appendix A) shows that there is a positive relationship between the two variables. When roughness is decreasing impedance also decreases. But at certain stage there is stability in decrease in the impedance values in spite of the reduction in the roughness. This indicates over a certain obtained roughness value the impedance will remain constant. It is because at higher temperature and pressure conditions when It can be better described through a mathematical equation as follows,

$$\text{Impedance } (\Omega) = 6.58 + 11.6 \text{ Roughness } (\mu\text{m})$$

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#### Regression Analysis: Impedance ( $\Omega$ ) versus Roughness (microns)

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The regression equation is

$$\text{Impedance } (\Omega) = 6.58 + 11.6 \text{ Roughness (microns)}$$

| Predictor           | Coef   | SE Coef | T     | P     |
|---------------------|--------|---------|-------|-------|
| Constant            | 6.5786 | 0.5495  | 11.97 | 0.000 |
| Roughness (microns) | 11.637 | 1.127   | 10.33 | 0.000 |

$$S = 2.04018 \quad R\text{-Sq} = 36.0\% \quad R\text{-Sq(adj)} = 35.6\%$$

PRESS = 809.609 R-Sq(pred) = 34.44%

#### Analysis of Variance

| Source         | DF  | SS      | MS     | F      | P     |
|----------------|-----|---------|--------|--------|-------|
| Regression     | 1   | 444.14  | 444.14 | 106.70 | 0.000 |
| Residual Error | 190 | 790.84  | 4.16   |        |       |
| Total          | 191 | 1234.98 |        |        |       |

#### Statistical Analysis

To support the above findings statistical analysis was performed for all measurements. ANOVA (analysis of variance) analysis method was used. In ANOVA analysis, a factor is considered statistically significant if its p-value is lower than the chosen level of significance ( $\alpha$ ), in all condition it was kept 5% or 0.05. For detailing main effects and interactions plots were studied. The results acquired from the statistical analysis can be found in Appendix A.

In case of roughness p-values found for each factor are below 0.05 except for the interaction between substrate and temperature and between substrate and pressure, therefore it can be concluded that all of the factors significantly affect roughness. The statistical analysis for roughness supported above results, as roughness decrease with temperature and pressure. The interaction plots showed an opposite trend in roughness for SUB\_1 printed with the SB than with the WB

ink. For each pressure condition, roughness decreased more at elevated temperatures. The ink and pressure interaction plots showed a significant decrease in roughness at the 375 PLI pressure for the WB ink and an increase in roughness above this pressure. For the SB ink, a linear decrease in roughness is observed.

Based on the findings in Figures 27 and 28, the 90% and 100% tones were selected for sheet resistivity analysis. The same way as stated above ANOVA was run for sheet resistivity. It can be seen in case of main effects plot and interactions plot (refer Appendix A) for sheet resistivity that the sheet resistivity increased in the following order SUB\_2, SUB\_3 and SUB\_1. For the CD 90% line tone was found to have the highest impedance, while the impedance of the 100% line tone was the lowest. As expected, an increase in temperature helped to decrease the sheet resistivity. From the ANOVA table it is observed that the P-value for pressure is 0.005. This means that pressure had a significant effect on impedance. But from the main effects plot it is determined that the effect is not as strong as temperature, ink, substrate or tone. Interaction between temperature and pressure causes impedance to decrease. The changes are more pronounced for calendering temperature than for pressure.

The ANOVA table for the IFT and the effects of the tested factors and their interactions are reported in Appendix A. The  $R^2$  (correlation) and  $R^2_{adj}$  are very low (68.84% and 58.38%, respectively), indicating a poor fit to the tested model. This may be due to other factors influencing the tested responses. Further study is needed to better understand these findings. The calculated p-values for each factor, except substrate and line tone, were below 0.05, indicating that all other factors significantly affected the IFT. Considering main effects and interaction plots for IFT it supports above findings, a lower IFT was measured for the SB ink than for the WB ink. In the case of the temperature plot, stable values were observed after 50°C. The IFT decreased up to 950 PLI and did not decrease further at 1500 PLI. For all substrates, the thickness values were in a comparable range. No particular trend was observed for a reduction in IFT with pressure and temperature. The ink film of the WB compressed more at higher pressures than the SB ink.

Bulk resistivity was calculated using the IFT and sheet resistivity data (Equation 5). The ANOVA results showed (Appendix A) higher  $R^2$  and  $R^2_{adj}$  values than for IFT studies, indicating that more of the error in the tested model can be contributed to the deviations in the bulk resistivity measurements. Results of bulk resistivity shows that lower bulk resistivity was measured for SB ink than

for WB ink. At higher pressures, a decrease in bulk resistivity was observed. This is most likely due to the softening of the ink resins at elevated temperature as well as particle sintering and alignment and compaction of the silver particles with pressure. An interaction plot it is seen that increasing the temperature gradually reduces bulk resistivity for SB ink. But for WB ink, resistivity reduces at 50°C, does not significantly change for 65°C and then slightly increases for 75°C. In the case of ink and pressure interaction plot, a rather significant gradual decrease in bulk resistivity was observed for WB ink with increasing pressure. However, the opposite effect is seen in the case of SB ink, where at increasing pressure, a slight increase in resistivity is detected. For both inks, the combination of pressure and temperature reduced the resistivity of the samples.

An ANOVA analysis was used to determine how other factors affect the performance of antennae (see Appendix A). No significant factors were found. The p-values for all factors and combinations were above 0.05. For antenna performance results no particular trend is observed as increase in calendaring temperature and pressure. All the tested antennae performed well, with the loss in magnitude being within an acceptable range (above -46.4 dB). Statistical results showed no significant results for any factors.

## CHAPTER VII

### CONCLUSIONS

Two commercially available conductive inks were printed on label stock and calendered at 16 different conditions. Line tone of 90, 100 and CD 90% were tested in terms of their ink film roughness and electrical conductivity. The UHF RFID antenna performance was also measured.

The application of calendering pressure and temperature were found to improve the smoothness of the ink films and reduce the thickness of the ink films. Calendering pressure had a more pronounced effect on roughness than temperature. Lower roughness values were obtained at higher pressures. The IFT of the WB ink were higher than the SB ink. Calendering slightly reduced the IFT of the inks, however, a clear trend with temperature and pressure were not found. The combination of higher calendering temperature and pressure decreased the standard deviation in IFT values.

Image analysis data showed no significant difference in image quality after calendering; no significant differences were observed between line width, raggedness and length before and after calendering.

The resistance of the 100% line tone was the lowest of all samples tested. The 90% tone printed in the CD contained some uncovered areas which resulted in higher impedance values.

Higher pressure and more nips resulted in a significant reduction in impedance. Calendering at 1500 PLI and 75 °C yielded comparable impedance values to the 105 °C /4 hours post cure treated samples. This indicates that off-line calendering may be able to eliminate the need for a post cure treatment of the silver based inks.

The antenna performance values observed for all calendering conditions were within the acceptable magnitude range i.e. above -46.4 dB, so no significant effect in antenna performance was found after calendering.



## CHAPTER VIII

### DISCUSSION

For this research samples were calendered off-line. On-line measurements would have been preferred, but such a press does not currently exist. The ability to calender on-line offers the additional advantage of less handling and lower calendering temperatures as the heat from the driers may be sufficient to soften the ink resins.

Smooth functional layers are necessary for building multilayered electronics devices. For integrated circuits, the low smoothness of an underlying layers can have detrimental effect on device performance.

For the conductive layers of an electronic device, nano silver inks are being used. These inks are more expensive than silver flake inks. As it was shown that calendering is an effective method to significantly improve the smoothness of silver flake inks, it may also work to improve the smoothness of nano silver inks. The ability to improve conductivity through calendering could offer costs savings by reducing ink mileage. For now it has been shown to eliminate the need for an off-line post cure treatment.

## Appendix

### Statistical Analysis

**Table 3** ANOVA results for Ink Film Roughness ( $\mu\text{m}$ ) versus Substrate, Ink, Line tone, Temperature ( $^{\circ}\text{C}$ ) and Pressure (PLI).

| <b>Analysis of Variance for Roughness (microns)</b> |    |         |         |         |       |
|---|----|---------|---------|---------|-------|
| Source  | DF | SS      | MS      | F       | P     |
| Ink   | 1  | 2.12218 | 2.12218 | 1469.05 | 0.000 |
| Substrate   | 2  | 0.54324 | 0.27162 | 188.03  | 0.000 |
| Line Tone   | 2  | 0.52578 | 0.26289 | 181.98  | 0.000 |
| Temperature ( $^{\circ}\text{C}$ )                  | 3  | 0.56834 | 0.18945 | 131.14  | 0.000 |
| Pressure (PLI)                                      | 3  | 1.44850 | 0.48283 | 334.24  | 0.000 |
| Ink*Substrate                                       | 2  | 0.40467 | 0.20234 | 140.06  | 0.000 |
| Ink*Line Tone                                       | 2  | 0.09252 | 0.04626 | 32.02   | 0.000 |
| Ink*Temperature ( $^{\circ}\text{C}$ )              | 3  | 0.11569 | 0.03856 | 26.70   | 0.000 |
| Ink*Pressure (PLI)                                  | 3  | 0.42085 | 0.14028 | 97.11   | 0.000 |
| Substrate*Line Tone                                 | 4  | 0.03516 | 0.00879 | 6.08    | 0.000 |
| Substrate*Temperature ( $^{\circ}\text{C}$ )        | 6  | 0.00462 | 0.00077 | 0.53    | 0.783 |
| Substrate*Pressure (PLI)                            | 6  | 0.00714 | 0.00119 | 0.82    | 0.553 |
| Line Tone*Temperature ( $^{\circ}\text{C}$ )        | 6  | 0.02545 | 0.00424 | 2.94    | 0.009 |
| Line Tone*Pressure (PLI)                            | 6  | 0.02256 | 0.00376 | 2.60    | 0.018 |

Table 3 – Continued

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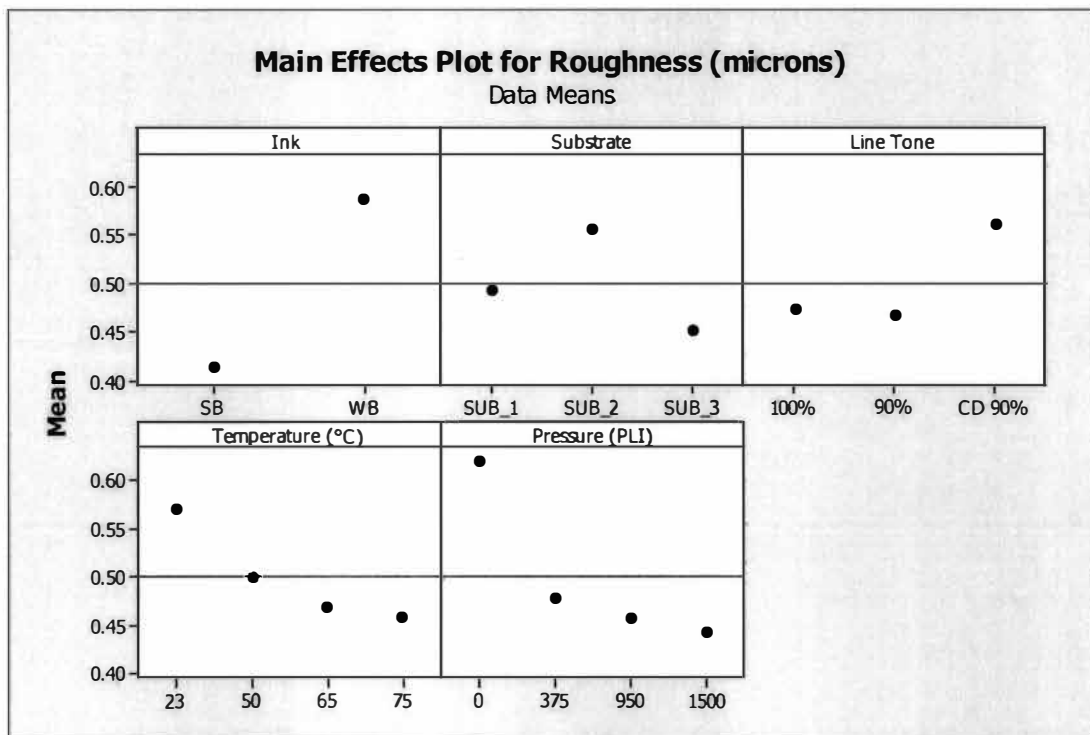
Temperature (°C)\*Pressure (PLI) 9 0.33622 0.03736 25.86 0.000

Error 229 0.33081 0.00144

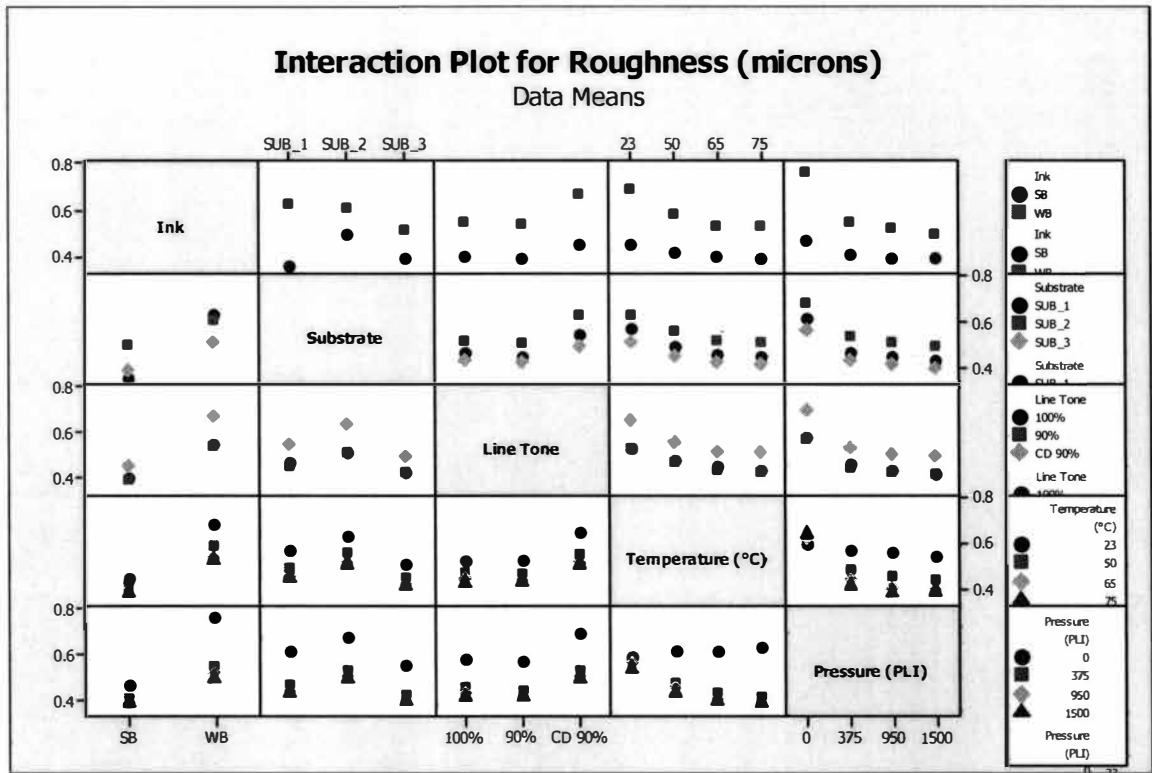
Total 287 7.00374

S = 0.0380078 R<sup>2</sup> = 95.28% R<sup>2</sup>a = 94.08%

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**Figure 52** Main effects plot for ink film roughness ( $\mu\text{m}$ ).



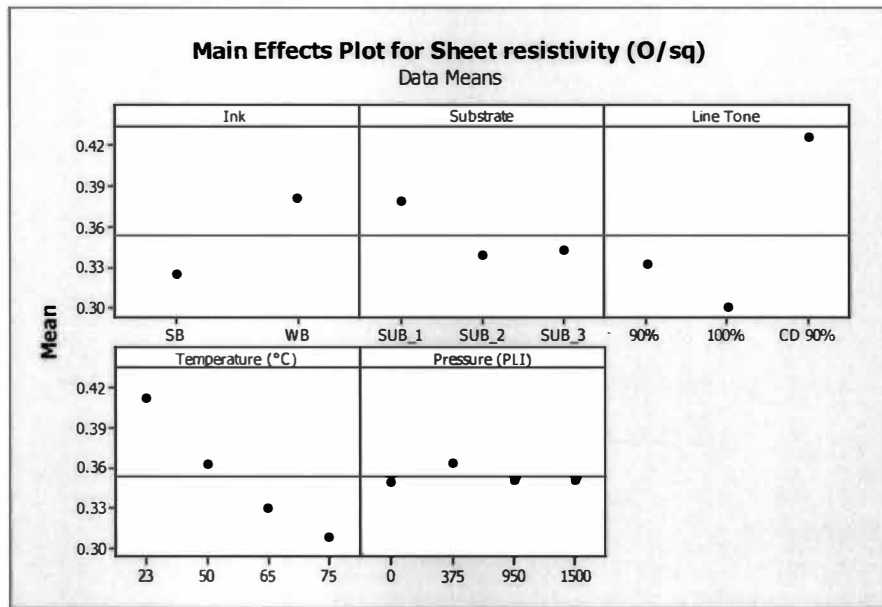
**Figure 53** Interactions plot for ink film roughness ( $\mu\text{m}$ ).

**Table 4** ANOVA results for Sheet resistivity ( $\Omega/\text{sq}$ ) versus Substrate, Ink, Line tone, Temperature ( $^{\circ}\text{C}$ ) and Pressure (PLI).

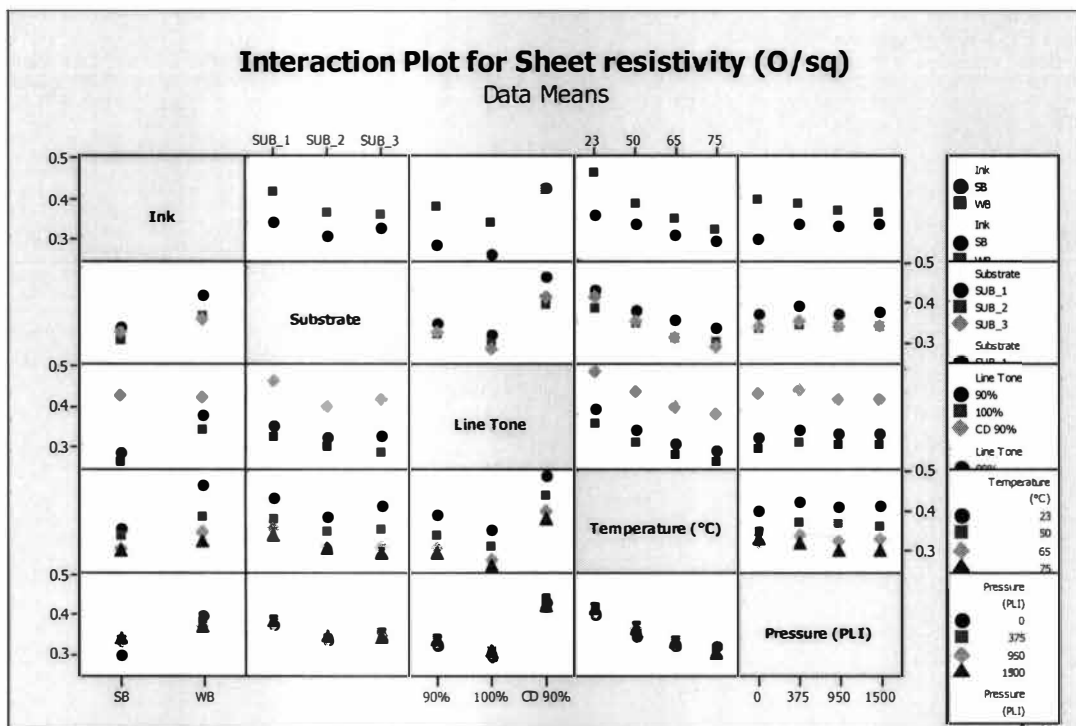
| Analysis of Variance for Sheet Resistivity ( $\Omega/\text{sq}$ ) |    |          |          |        |       |
|---|----|----------|----------|--------|-------|
| Source  | DF | SS       | MS       | F      | P     |
| Ink   | 1  | 0.230638 | 0.230638 | 328.53 | 0.000 |
| Substrate   | 2  | 0.094300 | 0.047150 | 67.16  | 0.000 |
| Line Tone   | 2  | 0.825764 | 0.412882 | 588.13 | 0.000 |

Table 4 – Continued

|  |     |          |          |        |       |
|--|-----|----------|----------|--------|-------|
| Temperature (°C)   | 3   | 0.447112 | 0.149037 | 212.30 | 0.000 |
| Pressure (PLI)   | 3   | 0.009146 | 0.003049 | 4.34   | 0.005 |
| Ink*Substrate  | 2   | 0.026439 | 0.013219 | 18.83  | 0.000 |
| Ink*Line Tone  | 2   | 0.143487 | 0.071743 | 102.19 | 0.000 |
| Ink*Temperature (°C)   | 3   | 0.062618 | 0.020873 | 29.73  | 0.000 |
| Ink*Pressure (PLI)   | 3   | 0.052459 | 0.017486 | 24.91  | 0.000 |
| Substrate*Line Tone  | 4   | 0.023009 | 0.005752 | 8.19   | 0.000 |
| Substrate*Temperature (°C)   | 6   | 0.014219 | 0.002370 | 3.38   | 0.003 |
| Substrate*Pressure (PLI)   | 6   | 0.001522 | 0.000254 | 0.36   | 0.903 |
| Line Tone*Temperature (°C)   | 6   | 0.002498 | 0.000416 | 0.59   | 0.736 |
| Line Tone*Pressure (PLI)   | 6   | 0.008632 | 0.001439 | 2.05   | 0.060 |
| Temperature (°C)*Pressure (PLI)  | 9   | 0.013681 | 0.001520 | 2.17   | 0.025 |
| Error  | 229 | 0.160764 | 0.000702 |        |       |
| Total  | 287 | 2.116287 |          |        |       |
| S = 0.0264958   R <sup>2</sup> = 92.40%   R <sup>2</sup> <sub>a</sub> = 90.48% |     |          |          |        |       |



**Figure 54** Main effects plot for Sheet resistivity ( $\Omega/\text{sq}$ ).



**Figure 55** Interactions plot for Sheet resistivity ( $\Omega/\text{sq}$ ).

**Table 5** ANOVA results for IFT ( $\mu$ ) versus Substrate, Ink, Line tone, Temperature ( $^{\circ}\text{C}$ ) and Pressure (PLI).

| Analysis of Variance for IFT (microns)            |    |         |         |        |       |
|---|----|---------|---------|--------|-------|
| Source  | DF | SS      | MS      | F      | P     |
| Ink   | 1  | 94.2932 | 94.2932 | 198.87 | 0.000 |
| Substrate   | 2  | 1.4871  | 0.7436  | 1.57   | 0.212 |
| Line Tone   | 1  | 0.1944  | 0.1944  | 0.41   | 0.523 |
| Temperature ( $^{\circ}\text{C}$ )                | 3  | 12.0393 | 4.0131  | 8.46   | 0.000 |
| Pressure (PLI)                                    | 3  | 13.5820 | 4.5273  | 9.55   | 0.000 |
| Ink*Substrate                                     | 2  | 3.0493  | 1.5247  | 3.22   | 0.043 |
| Ink*Line Tone                                     | 1  | 0.4078  | 0.4078  | 0.86   | 0.355 |
| Ink*Temperature ( $^{\circ}\text{C}$ )            | 3  | 3.9695  | 1.3232  | 2.79   | 0.043 |
| Ink*Pressure (PLI)                                | 3  | 5.3367  | 1.7789  | 3.75   | 0.012 |
| Substrate*Line Tone                               | 2  | 0.1999  | 0.1000  | 0.21   | 0.810 |
| Substrate*Temperature ( $^{\circ}\text{C}$ )      | 6  | 3.2353  | 0.5392  | 1.14   | 0.344 |
| Substrate*Pressure (PLI)                          | 6  | 3.5342  | 0.5890  | 1.24   | 0.288 |
| Line Tone*Temperature ( $^{\circ}\text{C}$ )      | 3  | 1.7652  | 0.5884  | 1.24   | 0.297 |
| Line Tone*Pressure (PLI)                          | 3  | 0.3197  | 0.1066  | 0.22   | 0.879 |
| Temperature ( $^{\circ}\text{C}$ )*Pressure (PLI) | 9  | 6.3650  | 0.7072  | 1.49   | 0.156 |



Table 5 – Continued

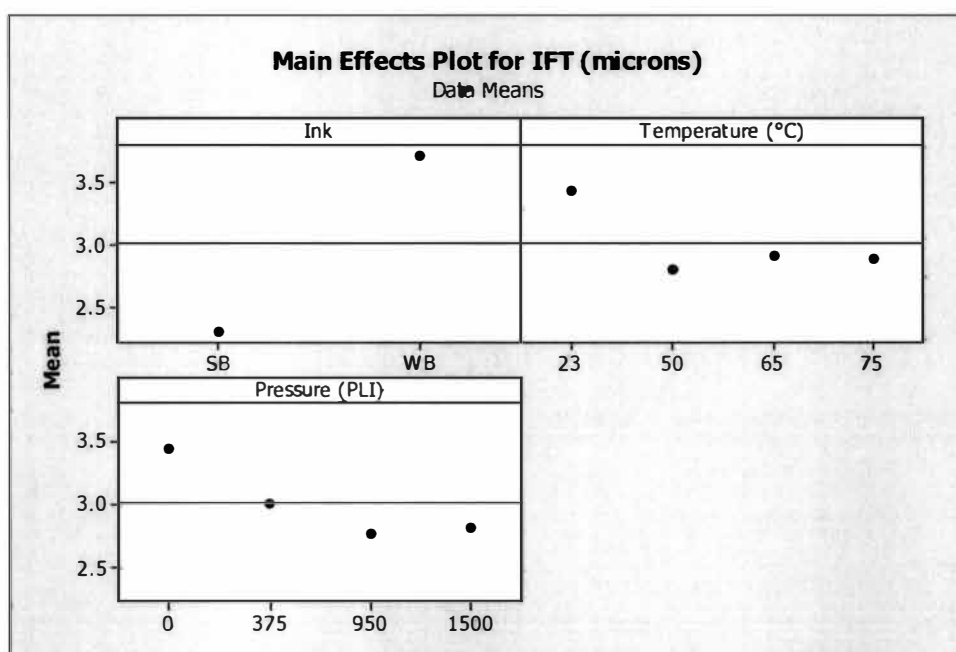
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|       |     |         |        |
|-------|-----|---------|--------|
| Error | 143 | 67.8022 | 0.4741 |
|-------|-----|---------|--------|

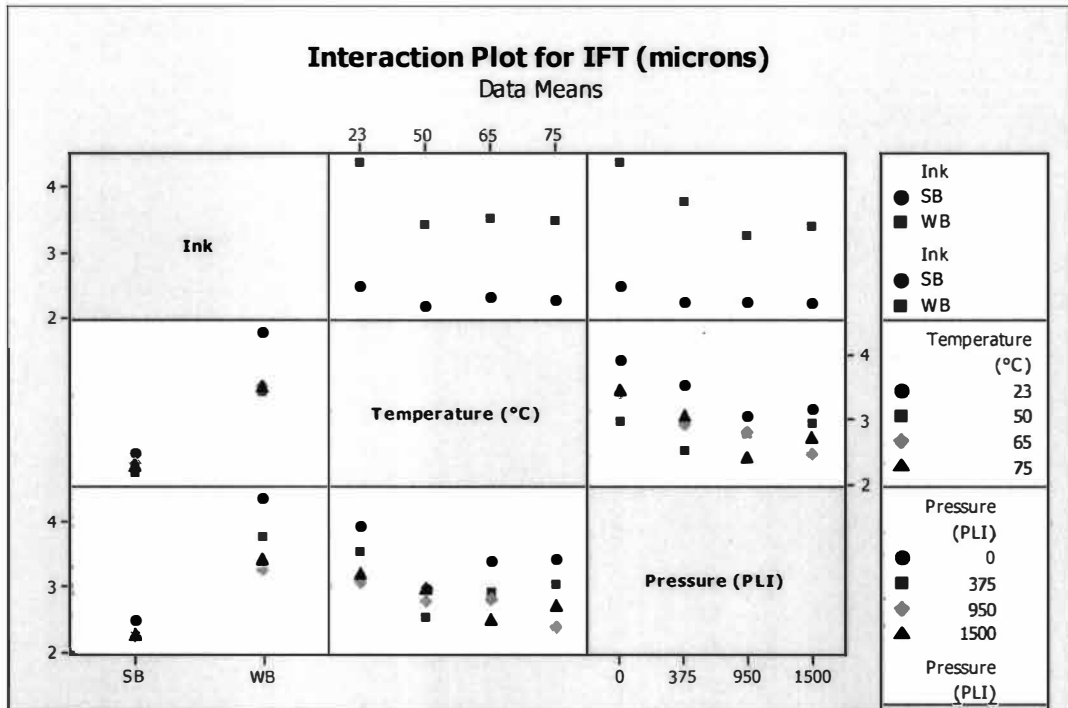
|       |     |          |  |
|-------|-----|----------|--|
| Total | 191 | 217.5809 |  |
|-------|-----|----------|--|

S = 0.688579 R-Sq = 68.84% R-Sq(adj) = 58.38%

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**Figure 56** Main effects plot for ink film thickness ( $\mu\text{m}$ ).



**Figure 57** Interactions plot for Ink film thickness ( $\mu\text{m}$ ).

**Table 6** ANOVA results for bulk resistivity versus Substrate, Ink, Line tone, Temperature ( $^{\circ}\text{C}$ ) and Pressure (PLI).

| Analysis of Variance for Bulk resistivity ( $\Omega/\text{sq}/\text{mil}$ ) |    |           |           |        |       |  |
|---|----|-----------|-----------|--------|-------|--|
| Source  | DF | SS        | MS        | F      | P     |  |
| Ink   | 1  | 0.0393112 | 0.0393112 | 380.95 | 0.000 |  |
| Substrate   | 2  | 0.0001114 | 0.0000557 | 0.54   | 0.584 |  |
| Line Tone   | 1  | 0.0004991 | 0.0004991 | 4.84   | 0.029 |  |
| Temperature ( $^{\circ}\text{C}$ )  | 3  | 0.0134725 | 0.0044908 | 43.52  | 0.000 |  |
| Pressure (PLI)  | 3  | 0.0022111 | 0.0007370 | 7.14   | 0.000 |  |

Table 6 – Continued

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|  |     |           |           |       |       |
|--|-----|-----------|-----------|-------|-------|
| Ink*Substrate                                  | 2   | 0.0000959 | 0.0000480 | 0.46  | 0.629 |
| Ink*Line Tone                                  | 1   | 0.0000148 | 0.0000148 | 0.14  | 0.705 |
| Ink*Temperature (°C)                           | 3   | 0.0059785 | 0.0019928 | 19.31 | 0.000 |
| Ink*Pressure (PLI)                             | 3   | 0.0024116 | 0.0008039 | 7.79  | 0.000 |
| Substrate*Line Tone                            | 2   | 0.0000065 | 0.0000032 | 0.03  | 0.969 |
| Substrate*Temperature (°C)                     | 6   | 0.0003301 | 0.0000550 | 0.53  | 0.782 |
| Substrate*Pressure (PLI)                       | 6   | 0.0011631 | 0.0001939 | 1.88  | 0.088 |
| Line Tone*Temperature (°C)                     | 3   | 0.0004913 | 0.0001638 | 1.59  | 0.195 |
| Line Tone*Pressure (PLI)                       | 3   | 0.0000903 | 0.0000301 | 0.29  | 0.831 |
| Temperature (°C)*Pressure (PLI)                | 9   | 0.0016118 | 0.0001791 | 1.74  | 0.086 |
| Error  | 143 | 0.0147566 | 0.0001032 |       |       |
| Total  | 191 | 0.0825560 |           |       |       |
| S = 0.0101584 R-Sq = 82.13% R-Sq(adj) = 76.13% |     |           |           |       |       |

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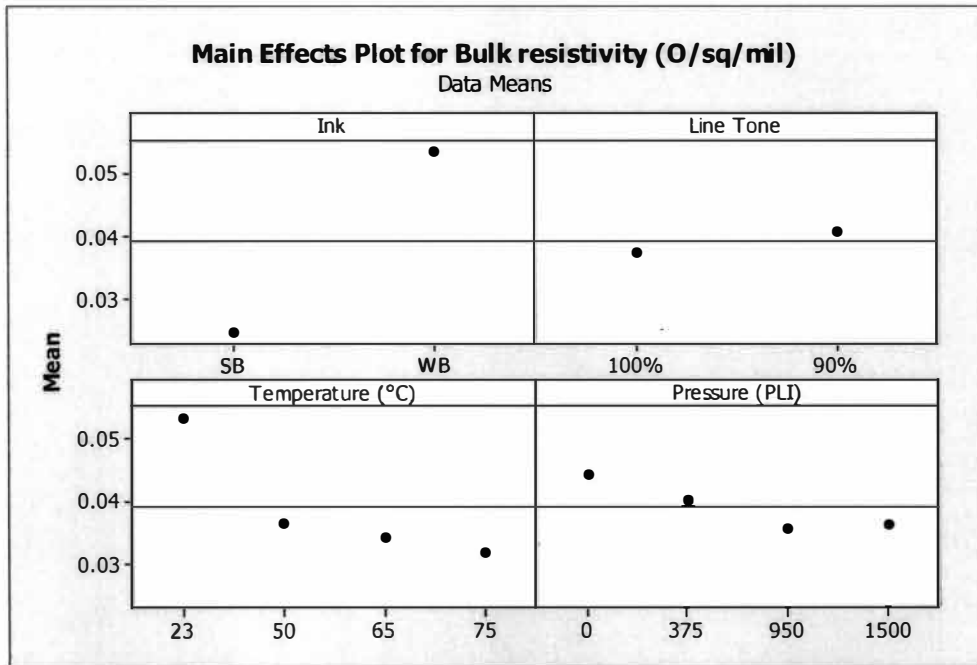


Figure 58 Main effects plot for bulk resistivity ( $\Omega/\text{sq}/\text{mil}$ ).

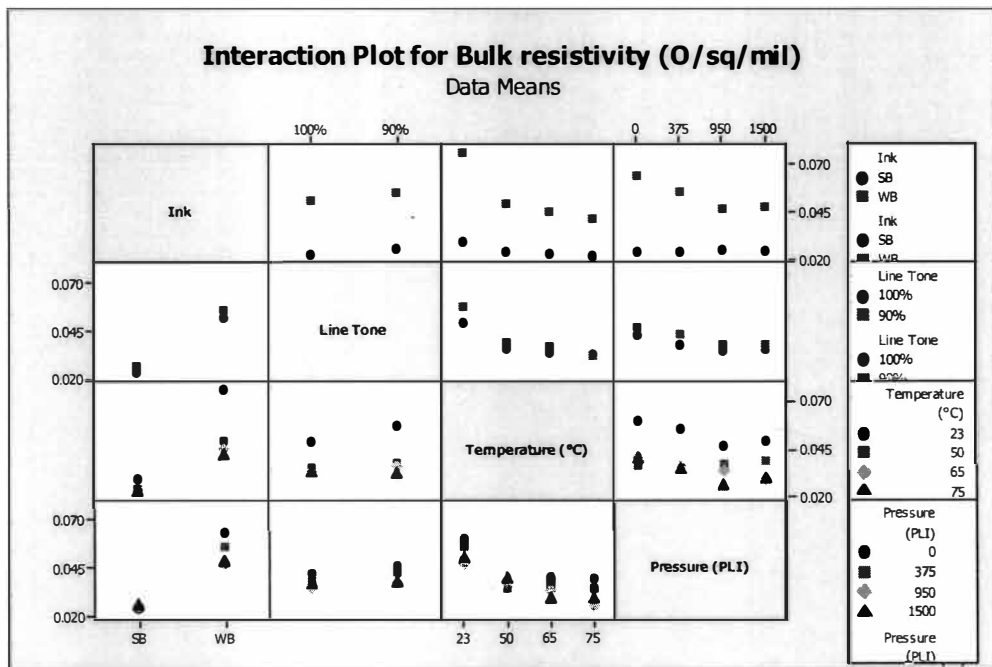
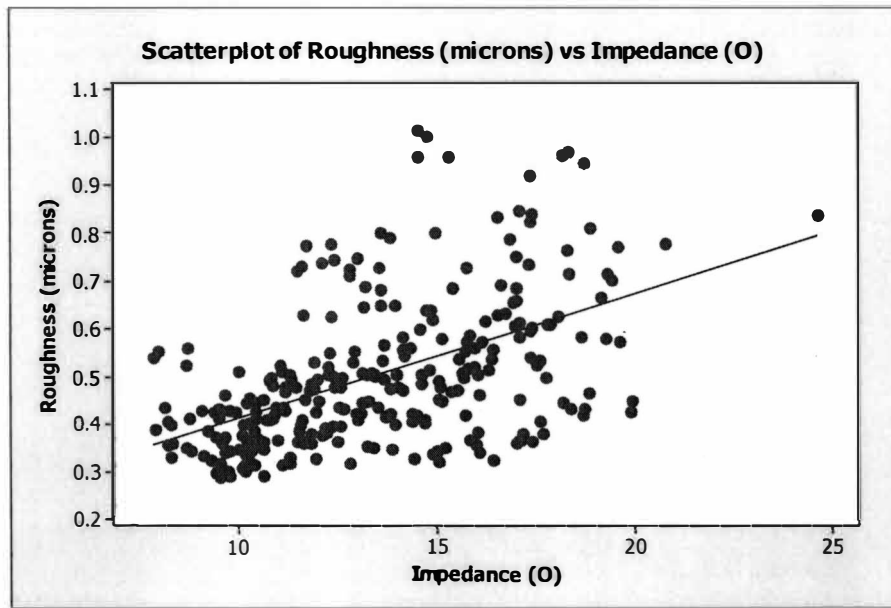


Figure 59 Interactions plot for bulk resistivity ( $\Omega/\text{sq}/\text{mil}$ ).

**Table 7** ANOVA results for Antenna performance (dB) versus Substrate, Ink, Line tone, Temperature (°C) and Pressure (PLI).

| <b>Analysis of Variance for Antenna Performance (dB)</b> |    |         |        |      |       |
|--|----|---------|--------|------|-------|
| Source   | DF | SS      | MS     | F    | P     |
| Ink  | 1  | 0.5620  | 0.5620 | 0.81 | 0.372 |
| Substrate  | 2  | 3.9509  | 1.9755 | 2.84 | 0.067 |
| Temperature (°C)   | 3  | 0.6288  | 0.2096 | 0.30 | 0.824 |
| Pressure (PLI)   | 3  | 1.5708  | 0.5236 | 0.75 | 0.525 |
| Ink*Substrate  | 2  | 0.2710  | 0.1355 | 0.20 | 0.823 |
| Ink*Temperature (°C)                                     | 3  | 2.3404  | 0.7801 | 1.12 | 0.347 |
| Ink*Pressure (PLI)                                       | 3  | 2.4079  | 0.8026 | 1.16 | 0.335 |
| Substrate*Temperature (°C)                               | 6  | 3.9900  | 0.6650 | 0.96 | 0.462 |
| Substrate*Pressure (PLI)                                 | 6  | 5.7667  | 0.9611 | 1.38 | 0.237 |
| Temperature (°C)*Pressure (PLI)                          | 9  | 4.4701  | 0.4967 | 0.71 | 0.693 |
| Error  | 57 | 39.6001 | 0.6947 |      |       |
| Total  | 95 | 65.5590 |        |      |       |
| S = 0.833510 R-Sq = 39.60% R-Sq(adj) = 0.00%             |    |         |        |      |       |



**Figure 60** Correlation plot between roughness vs. impedance.

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