Evaluation of Novel Thermosetting Stretchable Conductive Inks and Substrates for "Stretchtronics" Applications

Jie Lian, Ph.D., Weifeng Liu, Ph. D., William Uy, Dennis Willie, Anwar Mohammed, Ph.D., Michael Doiron
FLEX International, Milpitas, CA

Andy Behr, Tomoaki Sawada, Takatoshi Abe, Fukao Tomohiro
Panasonic Electronic Materials Division, Cupertino, CA

Abstract
Stretchable electronic constructions (or stretchtronics) are of increasing interest for many wearable applications, particularly in the realm of electronic textiles. Stretchable substrates and conductive inks are crucial elements because they form the basis for the elastic and conformable circuitry that enables these devices to function. New form factors, functionality and durability requirements are pushing the limits of commercially available thermoplastic stretchable films and conductive inks. While silicone materials offer improved durability and temperature resistance, they are limited by adhesion and material compatibility issues. To address these challenges, conductive inks and substrates based on a new stretchable non-silicone thermosetting polymer technology are being developed. Previous studies show that stretchable circuit traces exhibit an increase in resistance when subjected to elongation stresses. A variety of factors may influence the conductivity versus elongation performance of stretchable circuits, such as ink composition, substrate properties, substrate/ink interfacial adhesion strength, substrate thickness, methods of encapsulation, etc. These factors need to be fully evaluated and understood. This paper presents an evaluation of stretchability testing under various constructions of test traces made from these thermosetting inks and substrates and sheds light on how different construction and environmental conditions affect the performance of stretchable conductive circuits.

Key words: Conductive Ink, Film, Substrate, Printed Electronics, Stretchability, Cyclic Stretch, Thermosetting Polymer

Introduction
Polymeric materials used in the manufacturing of stretchtronics devices should exhibit not only a low elastic modulus and high elongation, but also the ability to recover following bending, stretching, twisting and any number of other deformation stresses. To be usable in commercial devices, these materials need to be durable, tolerate elevated temperatures and exhibit chemical resistance. Preferably, they will also be compatible with the other standard materials and processes typically used in high volume electronics manufacturing. A variety of thermoplastic materials, such as thermoplastic polyurethane (TPU) have been evaluated and used for stretchtronic device constructions. However, these materials have some performance drawbacks, particularly in terms of permanent deformation (hysteresis), mechanical durability and temperature and humidity resistance [1].

Thermosetting resins are widely used in electronics manufacturing due to their broad range of desirable properties like heat tolerance, chemical resistance and electrical insulation. While many thermosetting resins have a degree of flexibility, particularly as an inverse function of film thickness, considerably fewer exhibit a significant level of elasticity or stretchability. The thermosetting substrate film and conductive ink supplied by the author electronics material company for this evaluation utilize a novel thermosetting resin system. This resin system is currently being used for a number of developmental embodiments specifically designed for printed electronic and stretchtronics applications.

Substrate Film

The liquid form of the resin (the A-stage) can be cast into stretchable films using standard film coating and drying equipment. Films made from these resins exhibit unique and attractive properties. The pilot coating equipment, on which these developmental materials were formed can make films from 25 to 150 microns thick. The stretchable polymer film is made on a 75-micron thick high temperature polyester carrier, which provides mechanical stability, allowing the film to be handled. Because of this construction, the film is compatible with many "down-stream" assembly processes such as lamination, slitting and die cutting. The polymer is fully reacted during the manufacturing process, meaning that the film is stable at room temperature and does not require refrigeration. Film thicknesses of 50 and 100 microns were supplied for this evaluation.

This film has good elongation and interesting stress relaxation properties. The mechanical properties vary with the film thickness. For example, the stress relaxation and modulus increase significantly between the 50-micron and 100-micron films. This stress relaxation property may be attractive for epidermal devices where skin comfort is a design priority. The film exhibits low hysteresis, which means that it incurs limited permanent deformation after elongation. The film has a high surface energy which allows it to be bonded or encapsulated without priming or surface treatment. Both thermoplastic and thermosetting resin systems have been successfully tested for bonding and encapsulating the film surface. This material also has a high temperature resistance. The film does not degrade after floating in liquidous solder at 260°C. Polymer decomposition occurs around 325°C. The film properties are presented and compared to a representative thermoplastic polyurethane (TPU) film in Table 1.

Conductive Ink

Interfacial adhesion between conductive elements and dielectric substrates is important for virtually all types of electronic circuitry, but perhaps nowhere quite as much as in the creation of stretchable circuitry. The use of compatible resin systems is a well-known approach to increase circuitry and substrate compatibility.

An experimental conductive ink based on the stretchable thermoset resin chemistry noted previously was used in this evaluation. The formulation consisted of silver particles, resin, solvent and additives blended together to form a thixotropic ink designed for deposition via screen printing. The viscosity of this material is 20 Pa・s. Ink viscosity is well known to have a great influence on printing quality, and this is an appropriate value for screen printing. Volume resistivity is about 10-5 Ω・m. When printing onto the thermosetting film substrate based on the same resin system, no surface preparation, like abrasion or plasma cleaning, is required. Because the inks contain unreacted polymer, storage conditions of ≤ 5°C are recommended. After printing, curing conditions of 170°C for 60 minutes is recommended. The properties of the conductive ink are presented in Table 2.

Table 1. Thermoset Stretchable Film Property Comparison

Substrate	Thermoset Film		TPU
Thickness [microns]	50	100	100
Elongation ratio at break [%]	201.7	212.6	>1,000
Elastic modulus [Mpa]	6.8	21.5	7.2
Stress relaxation value [%]	37.3	60.4	25.3
Hysteresis [%]	0.1	1.7	5.9

Table 2. Properties of Thermoset Stretchable Conductive Ink A

Silver Ink		Condition
Viscosity [Pa・s]	20	Paste
Elongation ratio at break [%]	71.7	Cured
Elastic modulus [MPa]	20.1	Cured
Volume resistivity [Ω・m]	0.3×10^{-5}	Cured

Experimental Procedure

For this study, test vehicles were created by screen printing thermosetting stretchable conductive ink onto the thermosetting stretchable film substrate. The print pattern consisted of parallel conductive traces (visible in Figure 1). Two different screen printing methods were used to create the parts to be tested. One method used standard screen printing mesh for printing (designated Print #1) and the other method used 3D screen printing mesh for printing (designated Print #2). Primarily, these two printing methods resulted in different amounts of conductive ink deposition. Three-dimensional (3D) microscopy was used to measure the profile, including width and thickness, of the printed and cured inks. To a gain better understanding of the microstructure of the stretchable conductive inks at rest and under strain, scanning electron microscopy imaging was applied at 0%, 50% and 100% of stretching of film and ink structure.

Stretchability tests were performed under three conditions, i.e. testing at room temperature (RT), testing at RT after pre-conditioning the sample in a temperature humidity chamber at 85˚C/ 85% relative humidity (RH) for 96 hours, and testing inside of a temperature chamber at 85 ˚C. Figure 1 shows the universal flexibility tester used for the uniaxial tensile tests at room temperature and at 85 ˚C. A constant speed of 1mm/s was used for all tests, which is equivalent to a strain rate of 0.01 s^{-1}. The test parts were subjected to 20% elongation, with up to 2000 cycles of stretching. Two probes were attached to two ends of the stretchable conductive ink trace lines, which were directly connected to a multimeter. Resistance was measured in-situ during the stretchability testing. The relationship between the resistance change with number of stretch cycles was recorded.

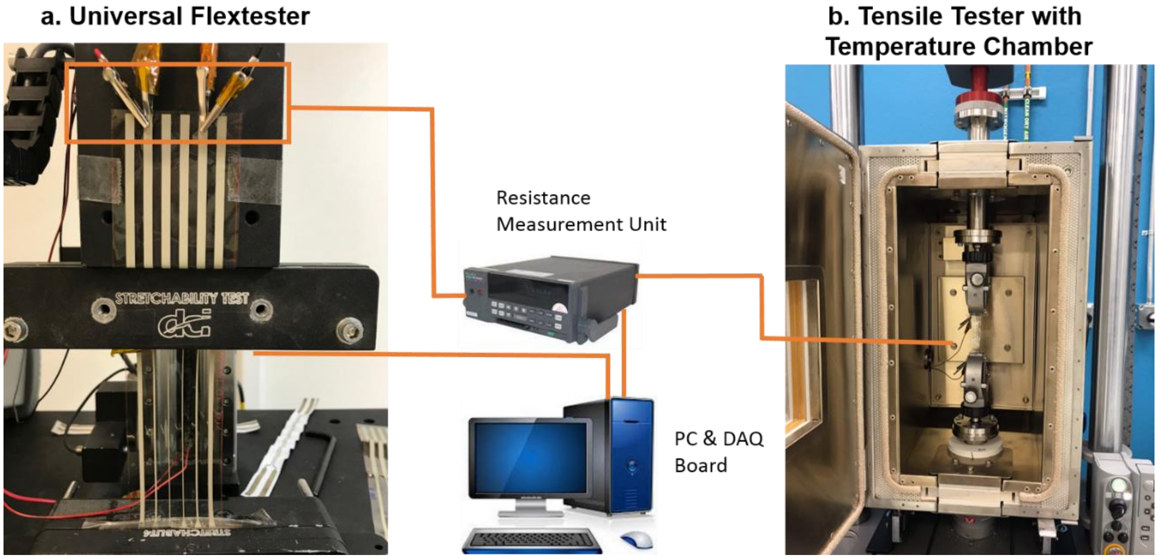


Figure 1 (a). Stretch Test at Room Temperature; (b). Stretch Test at temperature of 85˚C

Results

Figure 2 and Figure 3 show the 3D microscopy images with z height profile for Print #1 and Print #2. Thickness of the conductive lines were measured to be 15 μm for Print #1 and 22 μm for Print #2. To compare the microstructure, scanning electron microscopy images were taken for both inks at 0% stretch, as shown in Figure 4. At a magnification of 50X, the screen mesh pattern and holes in the ink were quite visible for Print #1. Also, a path of missing ink was observed along one edge side of the trace. However, this effect was not observed in SEM images of traces made with Print #2. The separated ink islands are believed to be artifacts from the screen printing process. At a magnification of 2000X, the surface of Print #1 and Print #2 exhibited similar microstructures, with silver particle size ranged from 0.5-5 μm. In summary, traces printed with Print #2 are thicker than those printed with Print #1. Additionally, there were no printing artifacts observed with Print #2.

To understand the microstructural changes of the silver inks under strain, several test parts made with inks were elongated and held at 50% and 100% inside the SEM. Figure 5 and Figure 6 show the SEM images of both materials under 0%, 50% and 100% stretch at magnifications of 50X, 1000X and 3500X. At a magnification of 50X, traces created with both inks show reduced line width. In addition, for Print #1, breakage of the long path of silver islands noted above was observed upon stretch. Little change was observed on the traces made with Print #2. At higher magnifications of 1000X and 3500X, multiple vertically aligned cracks were observed in both inks, and are indicated with red arrows. Higher elongations correlated with wider cracks. Compared to ink cracks observed for different silver inks from a previous study [2], the crack size from these inks are significantly smaller.

Figure 7 and Figure 8 show the change in the ratio of final resistance Rf over original resistance R0 with stretch percentage for the first stretch cycle at three different environmental conditions, i.e. at room temperature, at room temperature with preconditioning, and at 85ºC. There is no clear trend showing which condition or on which film thickness the ink has a better performance in terms of the resistance change. The variation of results among the different testing conditions are thought to originate from small differences in testing setup, ink thickness and microstructure. The ink resistance variation does not show significant change upon preconditioning and at a temperature of 85 ºC for one cycle stretch of 20%, indicating that preconditioning and elevated operating temperatures do not have a significant impact on the performance of the inks under these conditions.

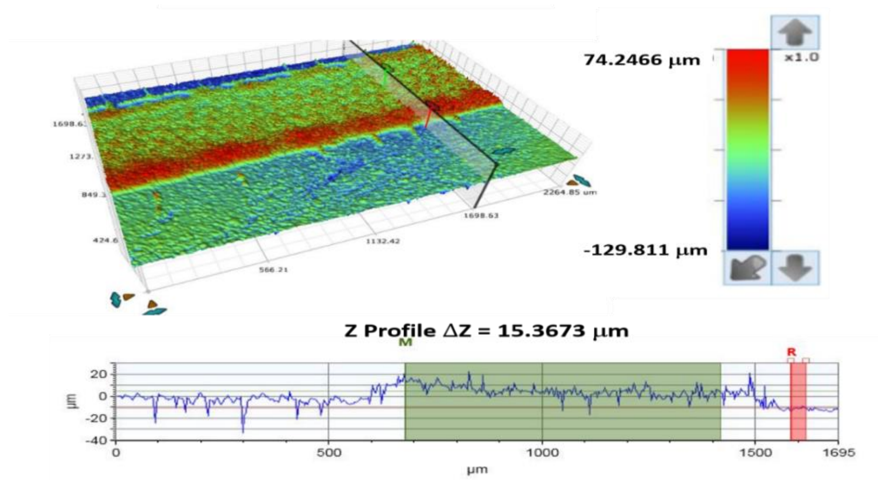


Figure 2. 3D microscopy images of ink printed with standard screen printing mesh (Print #1) at 0% stretch

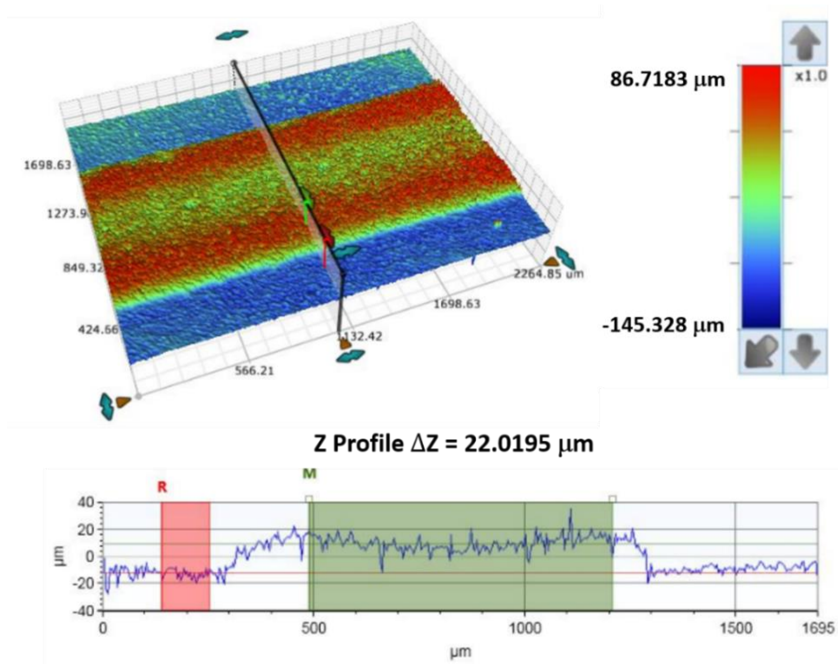


Figure 3. 3D microscopy images of ink printed with 3D screen printing mesh (Print #2) at 0% stretch

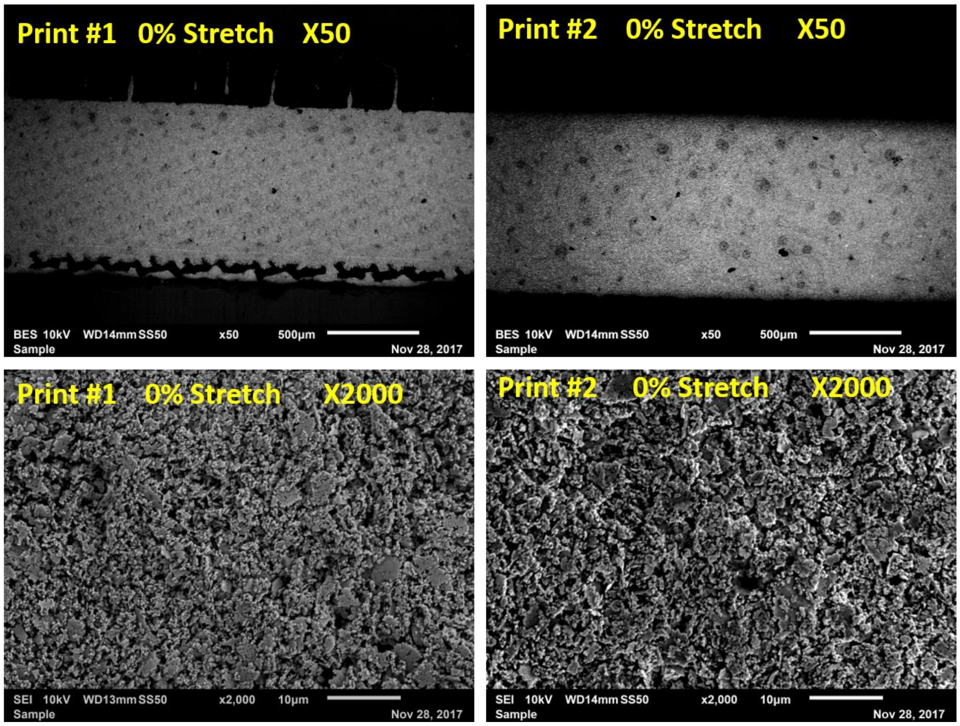


Figure 4. Comparison of SEM images of Print #1 and Print #2 at 0% stretch

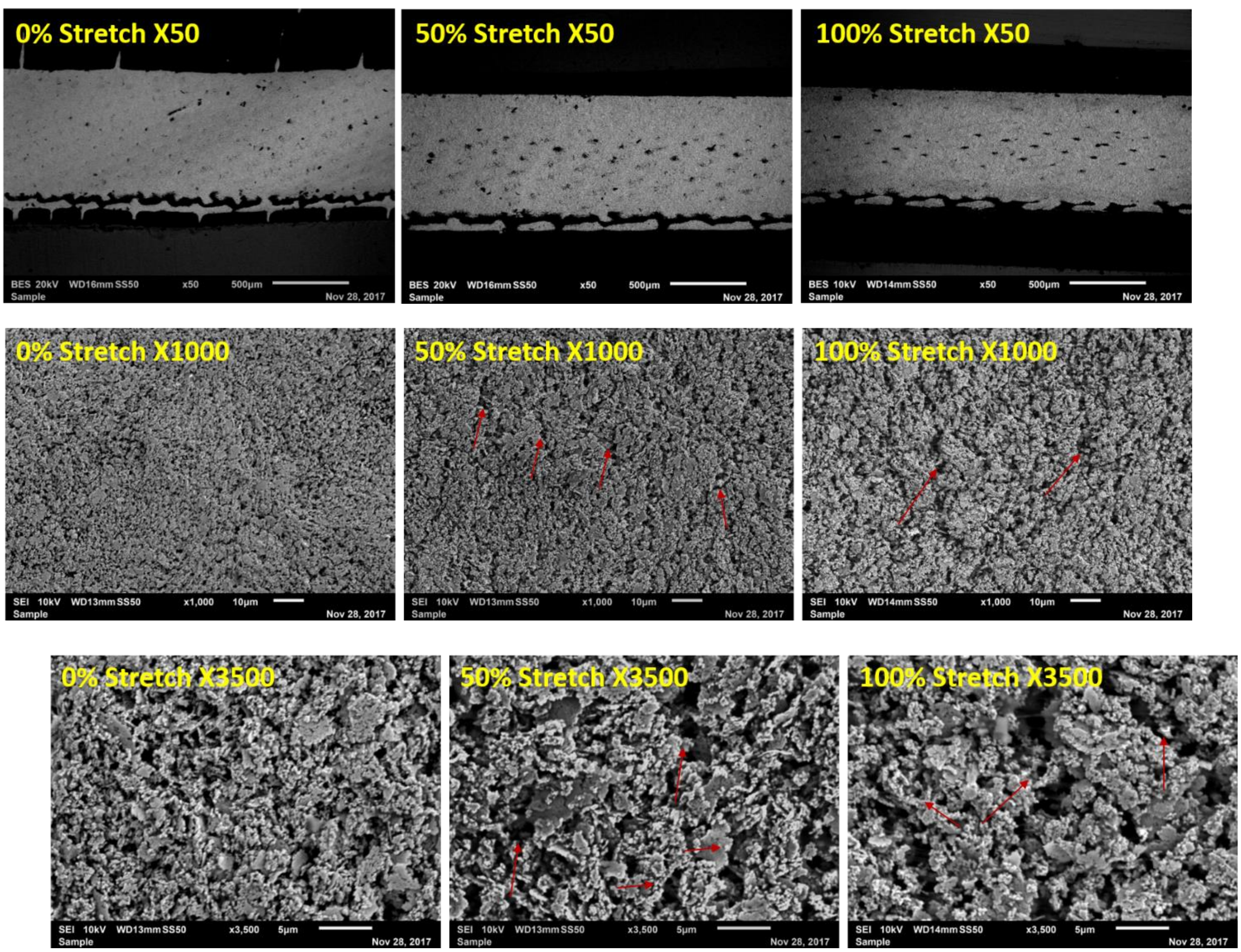


Figure 5. SEM images of silver ink with Print #1 at 0%, 50% and 100% stretch

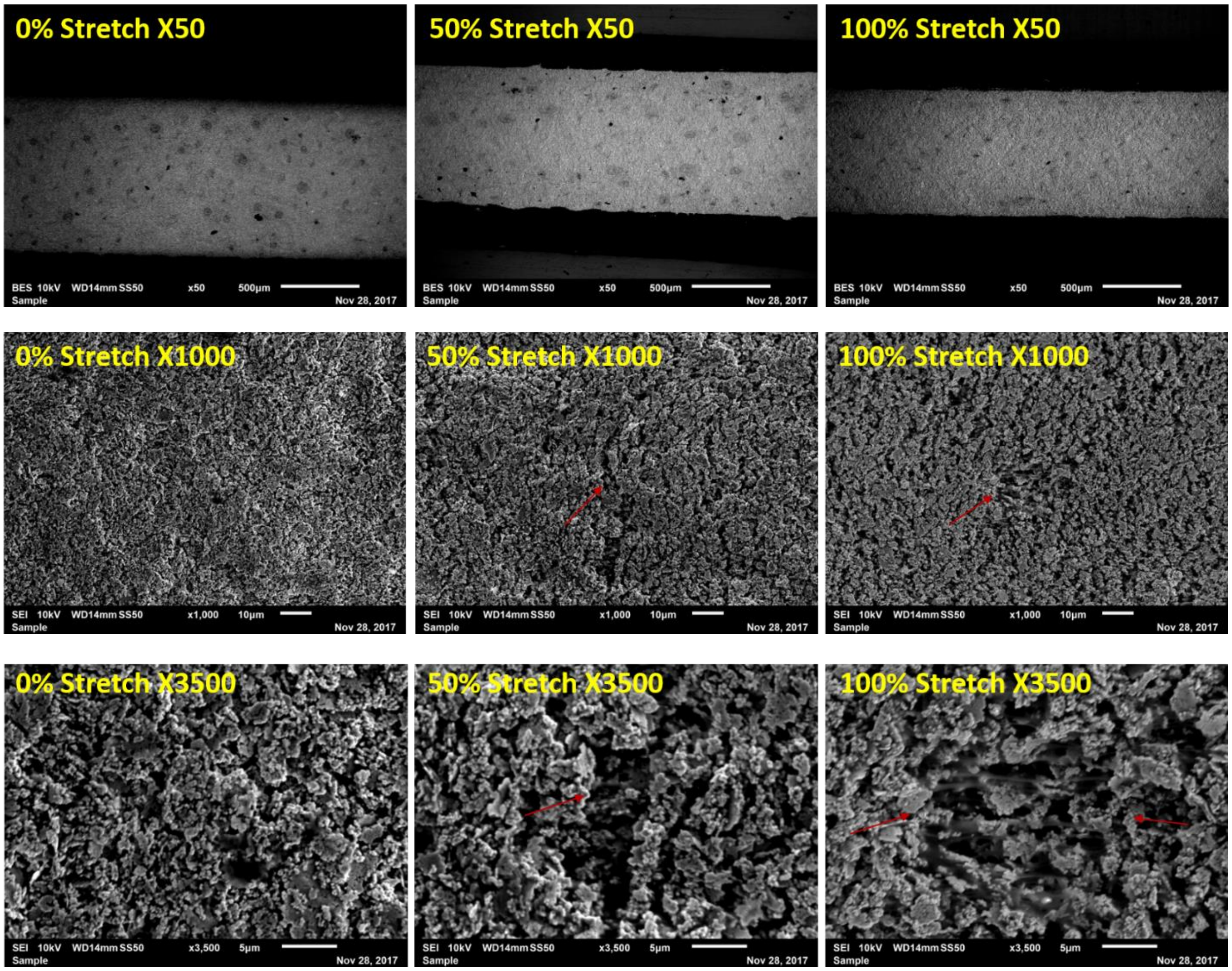


Figure 6. SEM images of silver ink with Print #2 at 0%, 50% and 100% stretch

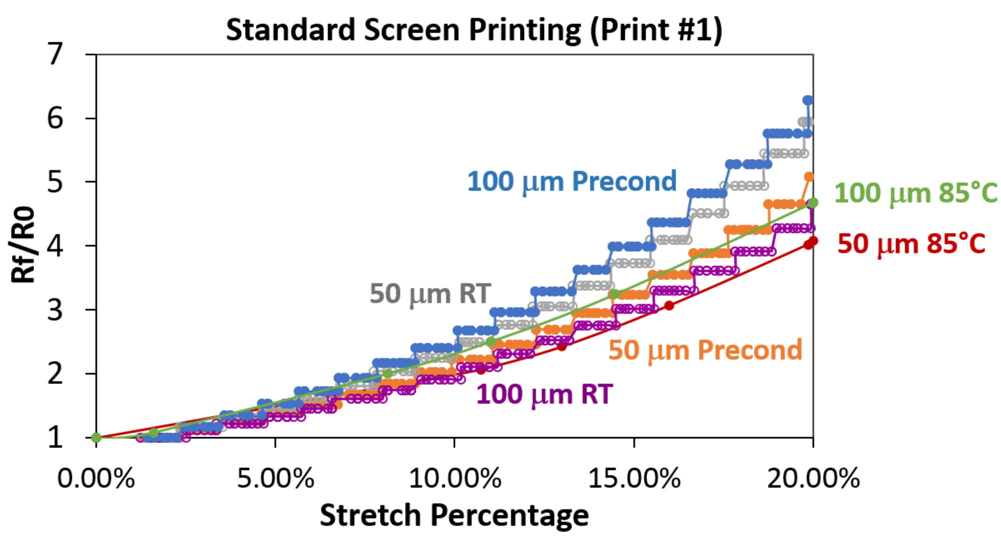


Figure 7. Resistance Rf/R0 change upon stretch for Print #1 at various testing conditions

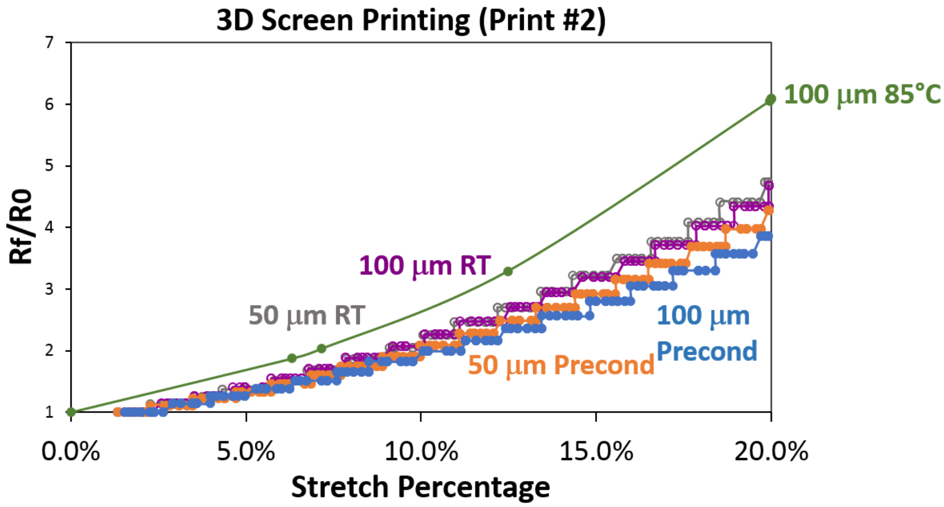


Figure 8. Resistance Rf/R0 change upon stretch for Print #2 at different testing conditions

Figures 9-11 show the Rf/R0 change of cyclic stretch tests at room temperature, at room temperature with preconditioning, and at 85ºC. At room temperature (Figure 9), it can be observed that the test parts made with Print #2 had a better conductivity performance than Print #1, which is expected given the difference in deposition thickness. For both inks, a thicker film of 100 μm shows better conductivity performance than thinner film of 50 μm. The Rf/R0 ratio for Print #2 printed on 100 μm film is only 9.4 after 2000 cycles of stretch, and it also shows the smallest variation between the maximum and minimum resistance in each cycle of stretch.

Similar trends were found in the parts that were pre-conditioned at 85ºC and 85%RH and tested at room temperature (Figure 10). Print #2 printed on 100 μm film also shows the best conductivity performance, but the part broke after 176 cycles. It is not clear why this part broke after 176 cycles because the same ink and substrate combination survived 2,000 cycles with no pre-conditioning (Figure 9) and the 100 μm film with Print #1 went 2,000 cycles with pre-conditioning (Figure 10).

For in-situ testing at 85 ºC (Figure 11), resistance spikes form during early cycles of stretching. The arrangement for stretch tests in the temperature chamber could potentially be the cause the high amplitude of the resistance spikes. When the temperature chamber is operating, air flow was kept running, which moved the probe contacting part of the trace. Due to the limited space inside the chamber, there was less fixtures used to fix the probe touching parts, thus raised the potential of instability of ink conductivity. A better design of sample geometry could be done for future studies.

Since the electrical resistance of the metallic conductor is dependent upon an internal collisional process, the resistance could be expected to increase with temperature since there will be more collisions. A fractional change in resistance which is proportional to the temperature change:

$$\frac{R-R_0}{R_0} = \alpha \Delta T$$

where R is the resistance at a certain temperature, R_0 is the resistance at room temperature, α is the temperature coefficient of resistance (a material property), and ΔT is the temperature change. This could explain why the starting resistance at 85ºC was much higher than that at room temperature.

Besides comparisons of the stretchability of different ink printing methods at the same testing conditions, another comparison was also made for the same ink with different testing conditions. Figures 12 and 13 show the Rf/R0 correlation with stretch cycles at room temperature, with pre-conditioning, and at 85ºC for Print #1 and Print #2 respectively. Tests at room temperature and with pre-conditioning show similar Rf/R0 trends before 200 cycles for both Print #1 and Print #2. As observed previously, for stretch testing at 85ºC, the Rf/R0 increases much faster than the other two testing conditions. Since from Figure 7 and Figure 8, we know the temperature does not have a big impact on the resistance change for one cycle stretch, it is thought that wear from the moving probe contacting trace might be an important contributing factor. Furthermore, with longer time of exposure to high temperature and cyclic loading, there could be thermo-mechanical induced fatigue inside the polymeric materials. High temperature might accelerate the oxidation of silver ink. Although silver oxide is conductive, this effect might lead to weakening of the particle surface resulting in surface flaws and increased crack propagation. As the crack propagates, the newly exposed crack surface then oxidizes, weakening the material further and enabling the crack to extend. Thus, the resistance changes at 85ºC show a significantly higher variation than testing at room temperature.

Figure 14 summarizes Rf/R0 ratio at 1, 100 and 300 cycles for Print #1 and Print #2 at room temperature and with pre-conditioning. The Rf/R0 ratios are quite similar during the first stretch cycle in all conditions, and Print #2 shows less resistance change over more stretch cycles. In test conditions of RT and Pre-conditioning, the Rf/R0 ratios were less than 45 for samples with 50 μm substrate, and all less than 20 for samples with 100 μm substrate. This is much lower than what we have previously measured with other conductive inks that printed on 100 μm thermoplastic urethane (TPU) substrates, with Rf/R0 over 200 after at 500 cycles of 20% stretch. [2] To further understand the mechanisms that cause this difference, percolation theory was applied for this silver flakes polymer composite. From percolation theory, it is known that resistance change was correlated to the volume fraction change of silver flakes. The relation between the resistance and the stretch strain can also be predicted as follows:

$$\sigma = \sigma_{silver}(V_f - V_c)^S \qquad [3]$$

where σ is the electrical conductivity of the composite, σ_{silver} is the electrical conductivity of the silver flakes, V_f is the volumetric fraction of the silver flakes, V_c is the volumetric fraction at percolation threshold (a constant), and S is the fitting exponent. From previous studies on other ink materials printed on 100 μm TPU substrate [2], it was known that the elastic modulus of the silver ink is on the order of 1000 MPa, which is significantly higher than the inks studied here. When silver ink has a higher modulus than the substrate, the ink experienced a higher level of stress. This is because the stress increases linearly with increment of the elastic modulus at a fixed strain. Thus, more cracks could be formed at a higher stress field in the silver ink, which can reduce the volume fraction of the silver flakes. This could explain why better conductivity was found in this silver ink upon the same level of stretch compared to our previous study. In addition, Table 1 shows that the thermoset film substrate in this study has much less hysteresis percentage than TPU film, which means in the unloading process the thermoset film experiences less strain than TPU film. This could also explain why Rf/R0 is much less than the previous study.

Considering silver inks with 50 μm and 100 μm thermoset film substrates, from Table 1 and Table 2, the 50 μm substrate has a modulus of 6.8 MPa, and 100 μm substrate has a modulus of 21.5 MPa, and the elastic modulus of the silver ink is 20.1 MPa. The 100 μm substrate is more rigid to hold the silver flakes in place during stretching, while a less rigid 50 μm substrate might have more microcracks formed. This could explain why the 100 μm thermoset film substrate usually shows better conductivity variation upon stretch than the 50 μm substrate.

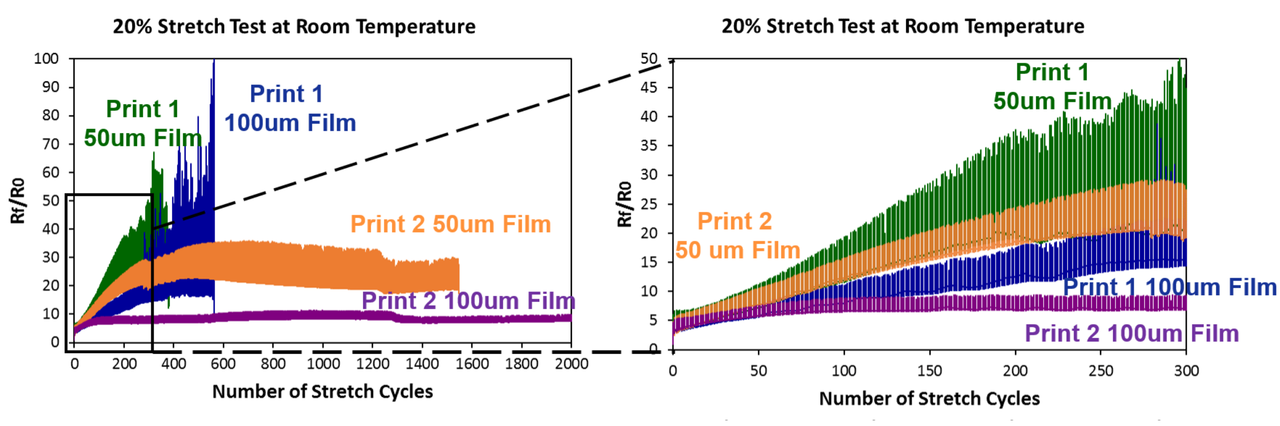


Figure 9. Resistance Rf/R0 change upon cyclic stretch at room temperature.

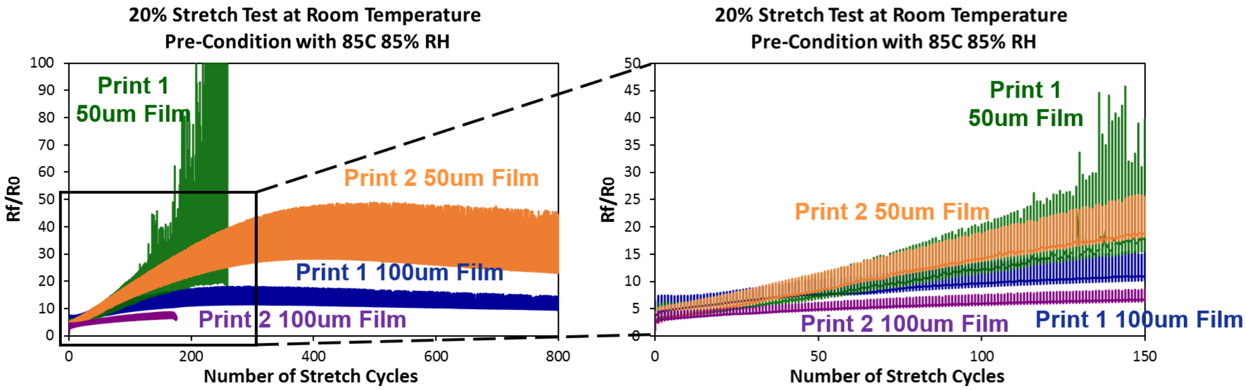


Figure 10. Resistance Rf/R0 change upon cyclic stretch at room temperature with pre-condition of 85ºC /85%RH

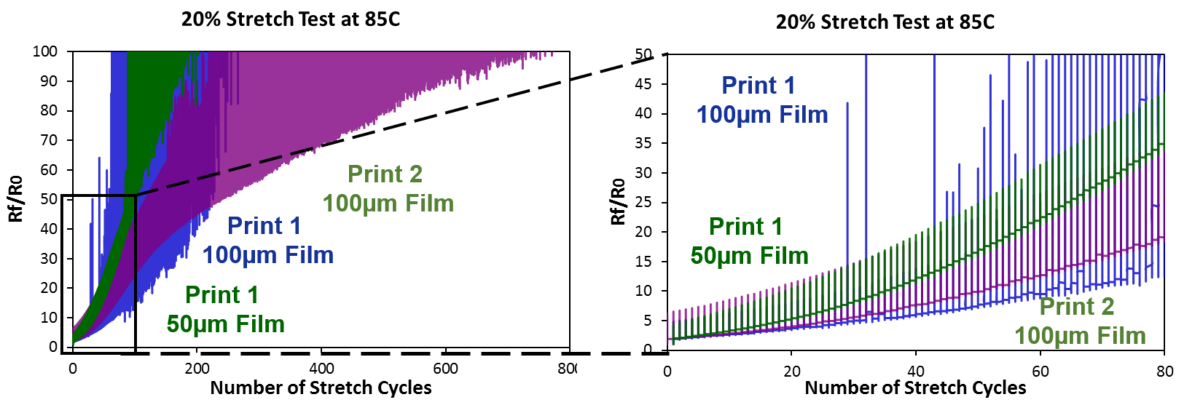


Figure 11. Resistance Rf/R0 change upon cyclic stretch at 85ºC

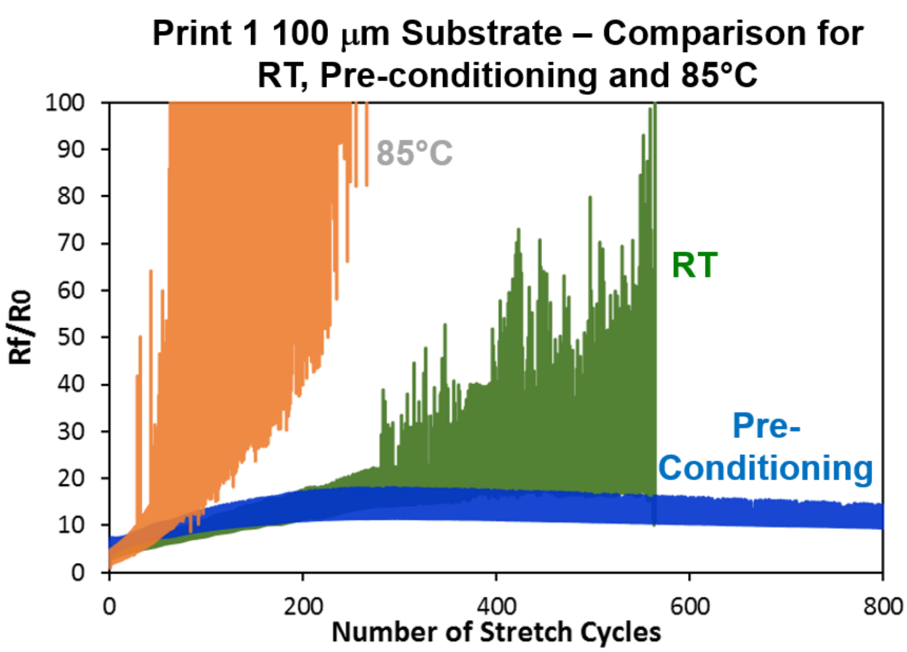


Figure 12. Resistance Rf/R0 change of Print #1 upon cyclic stretch at RT, with pre-conditioning, and at 85ºC

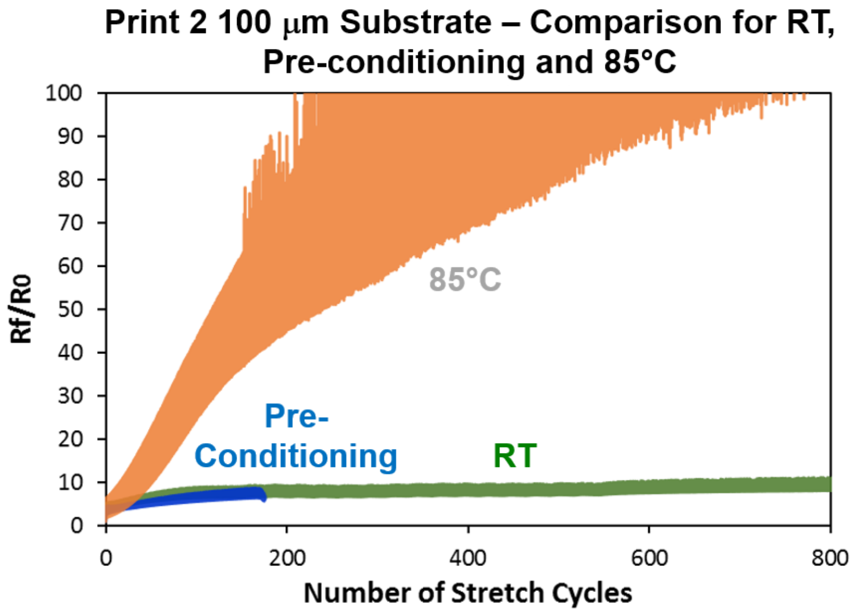


Figure 13. Resistance Rf/R0 change of Print #2 upon cyclic stretch at RT, with pre-conditioning, and at 85ºC

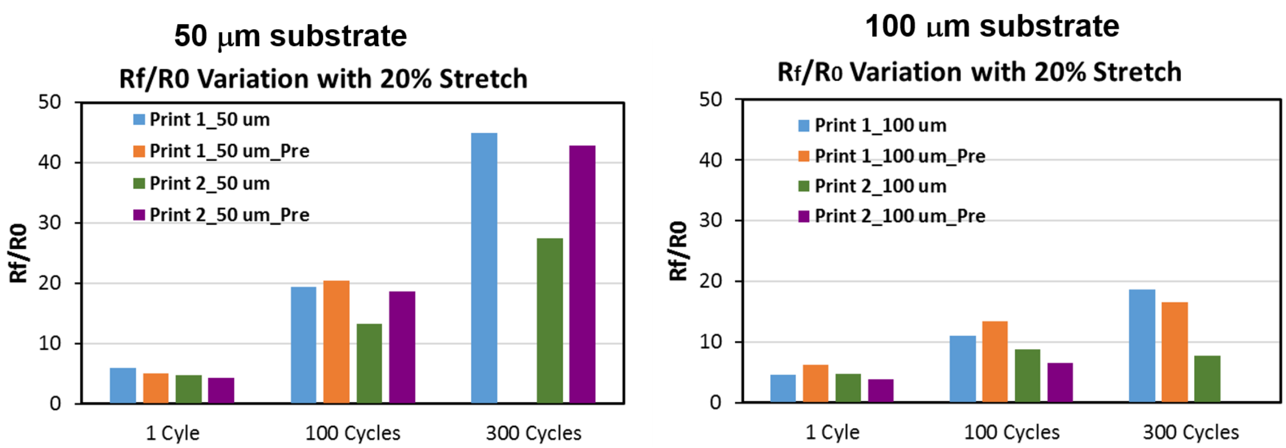


Figure 14 Resistance Rf/R0 change at 1, 100 and 300 cycles for Print #1 and Print #2 at room temperature and with pre-conditioning with substrate of (a) 50 μm and (b) 100 μm.

Conclusions

In this study, the stretchability of thermosetting silver inks printed on thermosetting film substrates were evaluated at three different conditions, i.e. at room temperature, at room temperature with pre-conditioning of 85°C and 85% RH for 96 hours, and at a high temperature of 85°C. In all test conditions, the resistance shows an increment of less than 100 times for up to 500 cycles of stretch. For test parts made with a thicker ink print (Print #2 at 22 μm thickness) on a thicker substrate of 100 μm, the resistance only increased 9.4 times at 2000 cycles of stretch at room temperature, while the resistance increased only 6.5 at 176 cycles of stretch at room temperature with pre-conditioning. In addition, inks that were pre-conditioned at 85°C and 85%RH did not show significant change in Rf/R0, which indicates that these materials are resistant to temperature and humidity. For in-situ testing at a high temperature of 85°C, Rf/R0 is significantly higher than testing at room temperature and with pre-conditioning, however, the wear out of the probe contacting the test parts was thought to be the main contributing factor.

Acknowledgements

The authors would like to thank the materials testing lab at Multek for testing with their tensile tester and temperature chamber. Brendan Nagle provided great help and valuable discussions for this project. We are also thankful to Michael Glickman for his timely support. Many thanks to the Reliability and Failure Analysis Lab at Flex for all their support in the Advanced Engineering Group.

References

1. Qi, H.J., Boyce, M.C., “Stress–strain behavior of thermoplastic polyurethanes”, Mechanics of Materials 37, 817-839, 2005
2. Liu, W., Lian, J. Uy, W., Feng, Z. Mohammed, A., Kurwa, M., “Evaluation of Stretchable Conductive Ink”, Proceedings, IPC Conference, Las Vegas, 2016
3. Taya, M., Kim, W.J., Ono, K., “Piezoresistivity of a short fiber elastomer matrix composite”, Mechanics of Materials 28, 53–59, 2008

Evaluation of Novel Thermosetting Materials for "Stretchtronics" Applications

Jie Lian, Ph.D., Weifeng Liu, Ph. D., William Uy, Dennis Willie, Anwar Mohammed, Ph.D., Michael Doiron

FLEX International, Milpitas, CA

Andy Behr, Tomoaki Sawada, Takatoshi Abe, Tomohiro Fukao

Panasonic Electronic Materials Division, Cupertino CA

Outline

- Study Objective
- Introduction
  - *Conductive Ink and Substrate Film Technology*
- Experiment
  - *Microstructural Analysis*
  - *Stretch Test Set up*
- Results
  - *Microstructure at 0%, 50% and 100% stretch*
  - *Stretch cycling resistance curves at room temperature (with and without pre-conditioning) and elevated temperature*
  - *Comparison to previous study results*
- Discussion & Conclusions

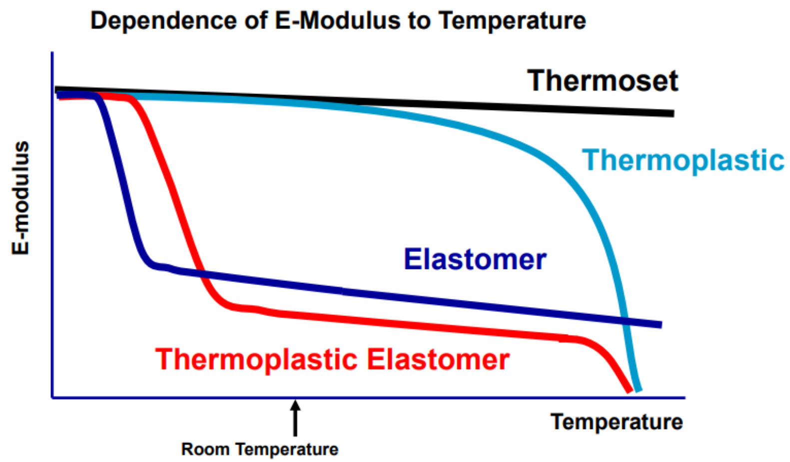
Study Objectives

Conduct a preliminary evaluation on these new materials to:

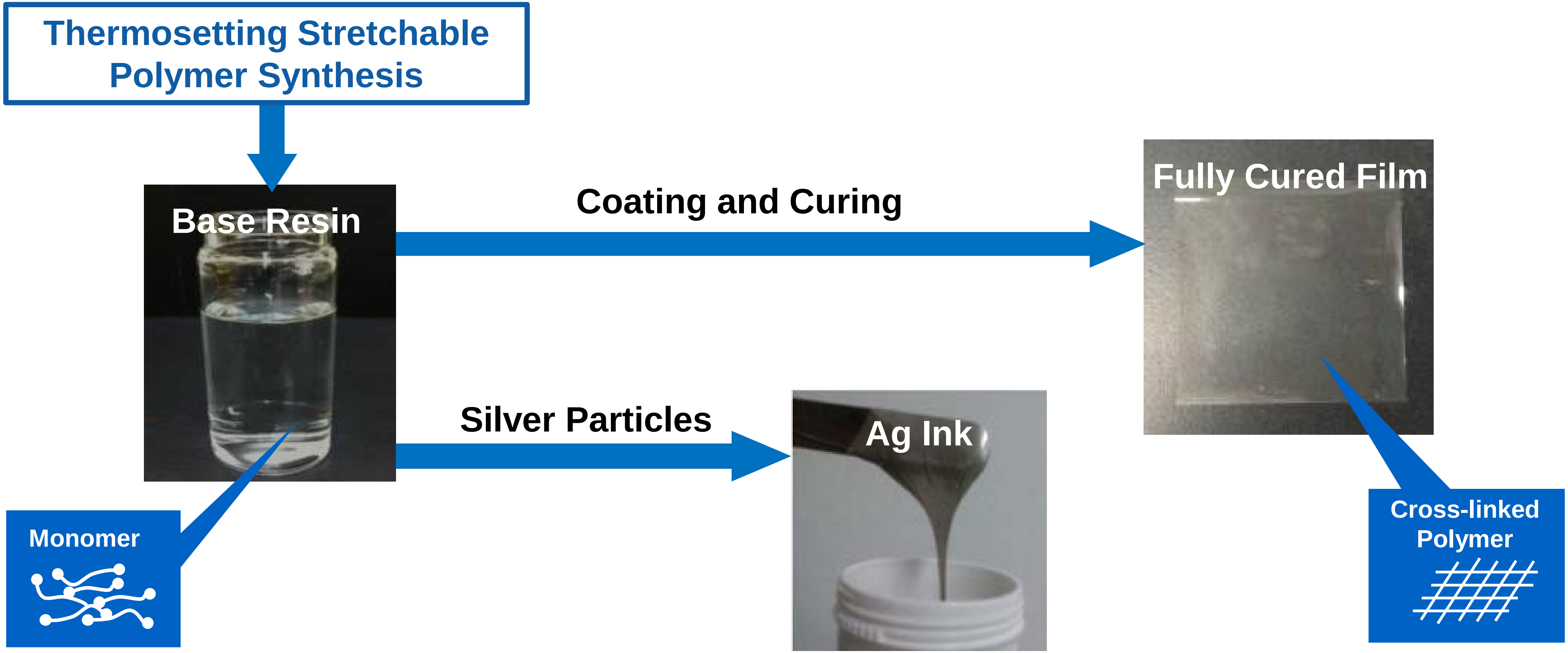
- Evaluate the room temperature stretchability of thermoset silver ink printed on thermoset film substrate *without* preconditioning
- Evaluate the room temperature stretchability of thermoset silver ink printed on thermoset film substrate *with* preconditioning (high T/high humidity 96 hours) before stretching
- Evaluate the high temperature stretchability(85°C) of thermoset silver ink printed on thermoset film substrate
- Compare the evaluation results with previous evaluations of thermoplastic materials
- Understand the fundamental issue with stretching, especially the microstructure change

Introduction – Thermoset and Thermoplastic Polymers

Qualities		Typical Thermoplastic	Typical Thermoset	Stretchable Thermoset
Physical Properties	Melting Point	Low	High	High
	Tensil Strength	Low	High	Medium
	Thermal Stability	Low (Reforms to solid when cooled)	(Decomposes at high temperatures	(Decomposes at high temperatures
	Stiffness	Low	High	Low
	Brittleness	Low	High	Low
	Rigidity	Low	High	Low
	Solubility	Soluble in organic solvents	Insoluble in organic solvents	Insoluble in organic solvents
	Durability	Low	High	Medium



Introduction – Film and Conductive Ink Manufacturing Process



Introduction – Film and Conductive Ink Properties

Thermoset Stretchable Film Properties

Substrate	Thermoset Film		TPU
Thickness [microns]	50	100	100
Elongation ratio at break [%]	201.7	212.6	>1,000
Elastic modulus [Mpa]	6.8	21.5	7.2
Stress relaxation value [%]	37.3	60.4	25.3
Hysteresis [%]	0.1	1.7	5.9

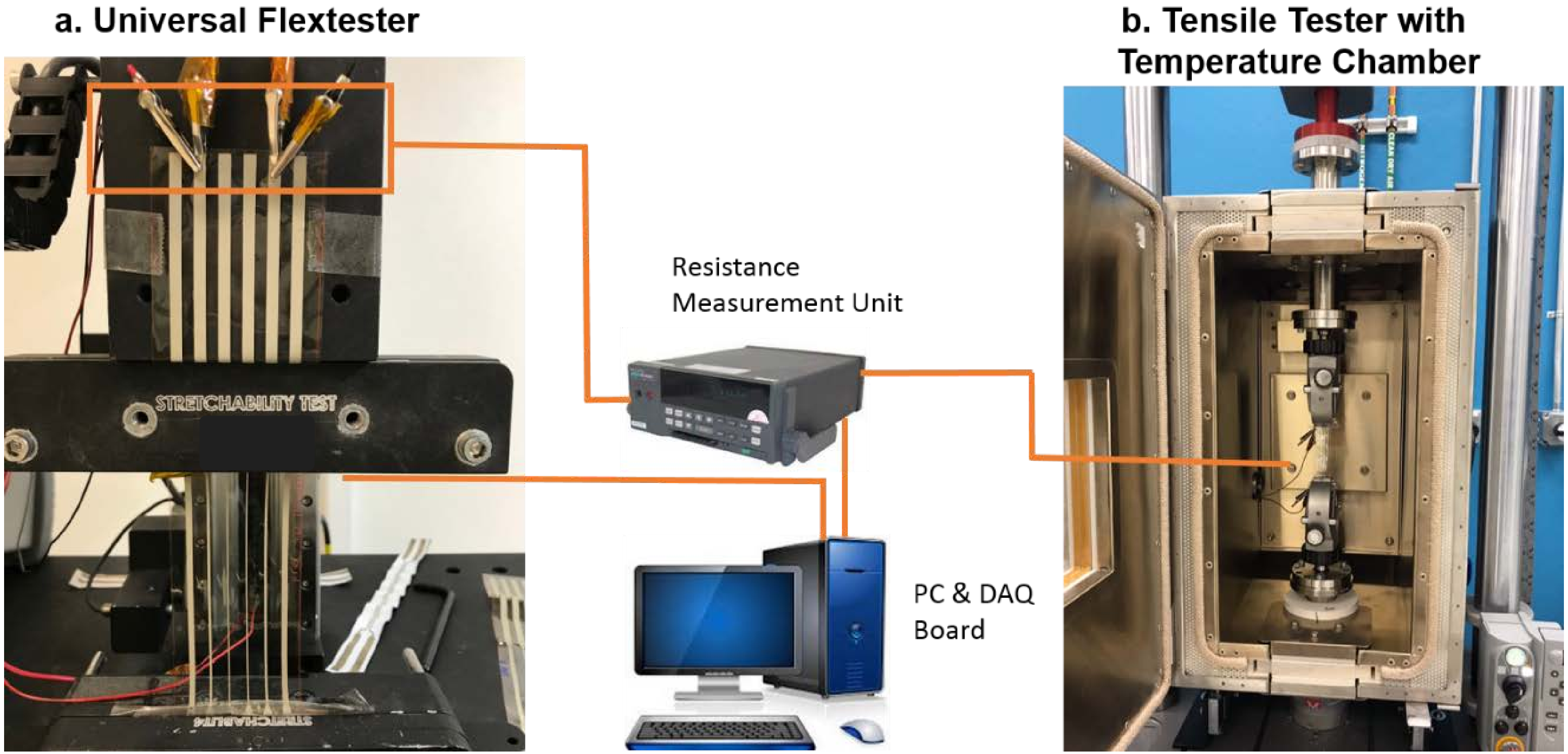
- Good Elongation
- Stress Relaxation
- Low Hysteresis
- High Temperature Resistance
- High Surface Energy

Thermoset Stretchable Conductive Ink Properties

Silver Ink		Condition
Viscosity [Pa·s]	20	Paste
Elongation ratio at break [%]	71.7	Cured
Elastic modulus [MPa]	20.1	Cured
Volume resistivity [Ω·m]	0.3×10^{-5}	Cured

- Good Elongation
- Low Resistance
- High Adhesion
- High Temperature Resistance

Experiment – Stretching Test Set Up



Constant speed of 1 mm/s, strain rate of 0.01 s^{-1}
20% elongation - up to 2,000 stretching cycles

Experiment – Stretchable Ink Printing Conditions

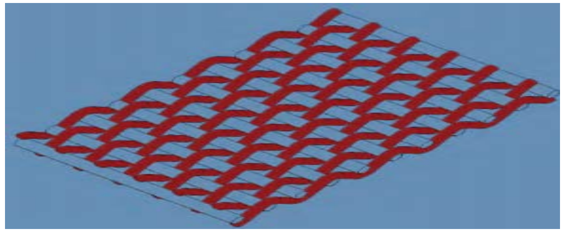
- Automated Screen Printer
- Printing parameters
  - Pressure 14-16 Kg
  - Speed: 35 mm/s
  - Gap: 4mm
  - Double printing
- Substrate
  - Stretchable Thermoset Film

Print 1

•Standard Polyester Mesh Screen
•305 Thread Count
•34 mm Thread dia.
•Angle: 45°
•10 mm Emulsion

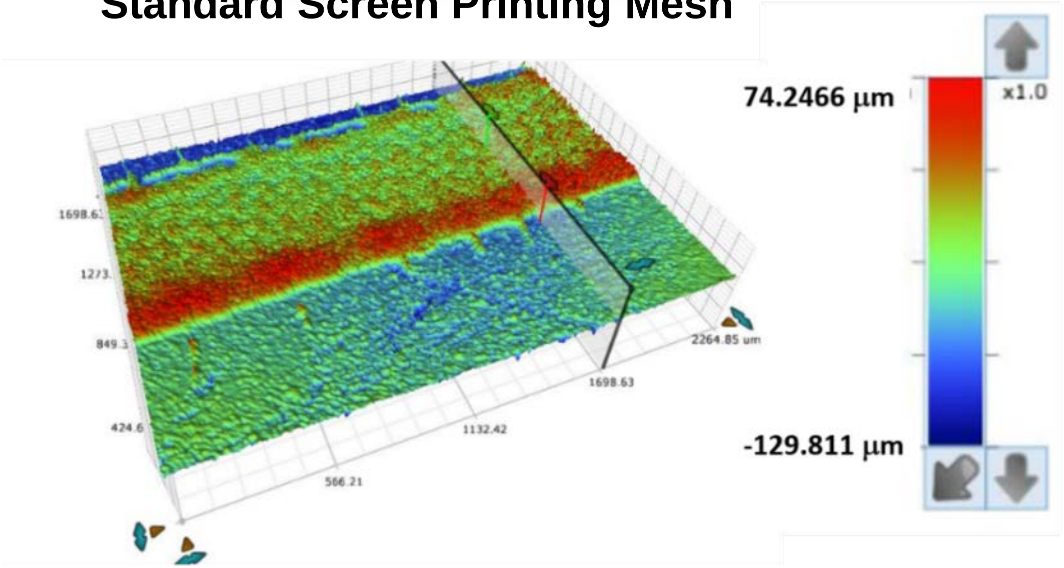
Print 2

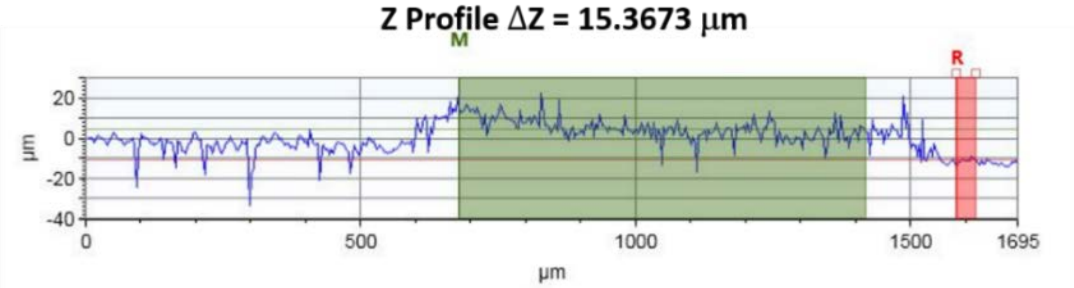
- 3D Stainless Steel Mesh Screen
- Special weave pattern results in a higher ink deposition volume



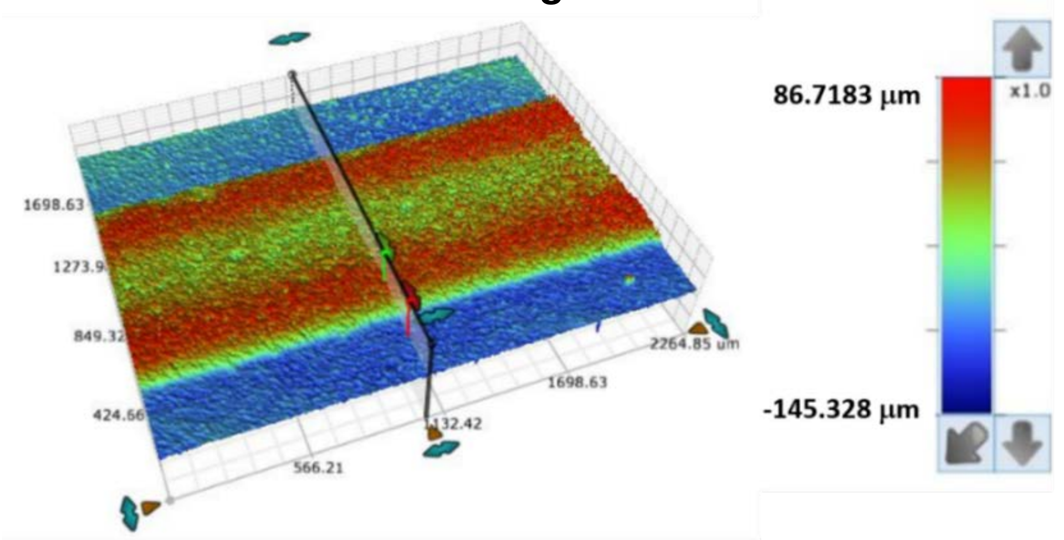
Results – Ink Thickness Measurement

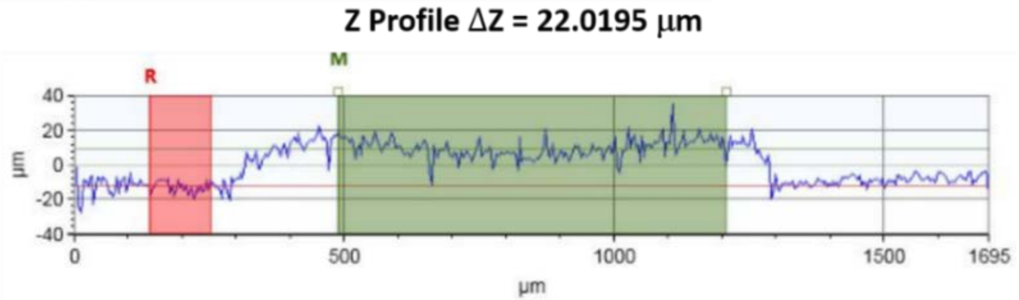
Standard Screen Printing Mesh





3D Screen Printing Mesh





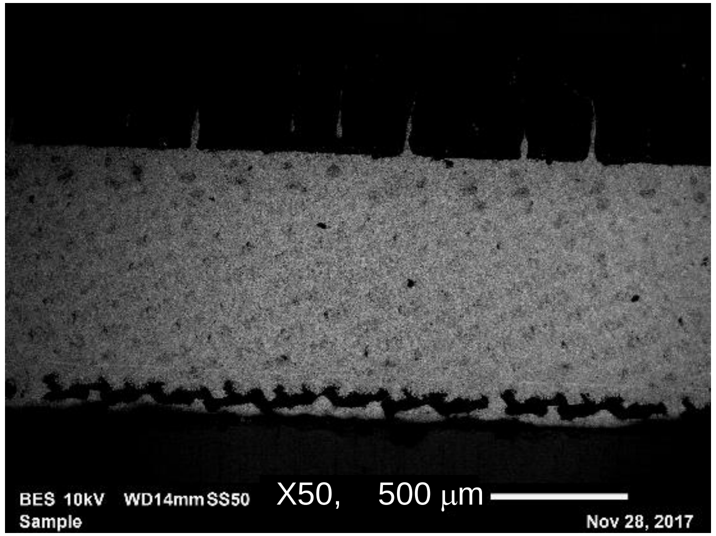
Normal Screen Mesh
Ink Deposition Thickness = 15.37 mm

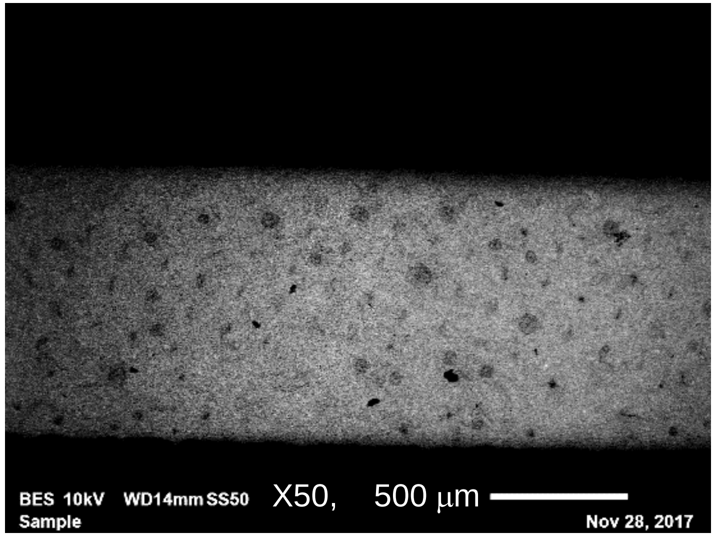
3D Screen Mesh
Ink Deposition Thickness = 22.02 mm

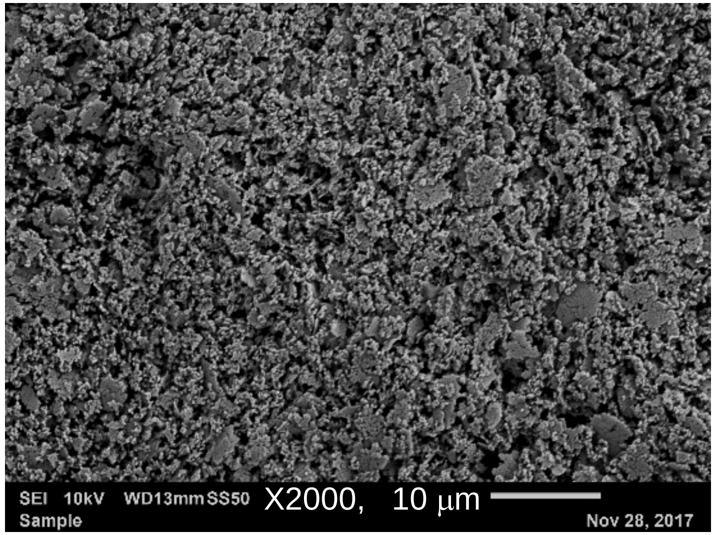
Delta = 43%

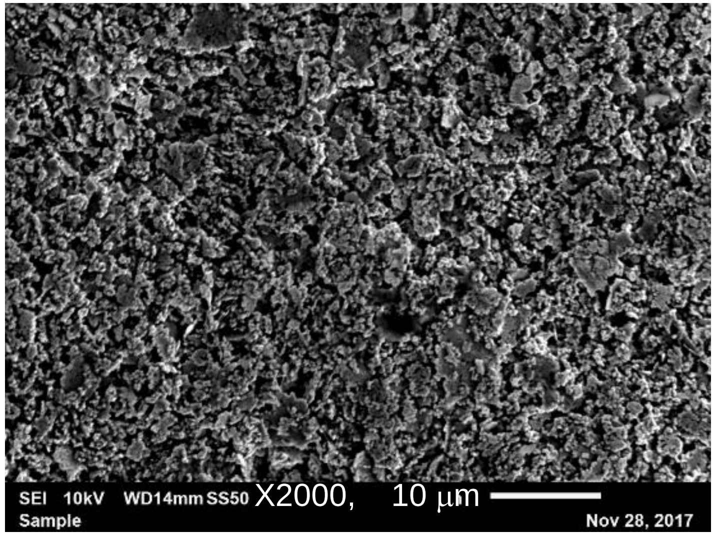
Results – Microstructure at 0% Stretch

Traditional Screen Printed Ink | **3D Screen Printed Ink**









<u>Observations</u>

- Similar surface microstructure in the inks with two printing methods
- Trace width 1mm
- Silver particle size ranged from 0.5-5 mm, homogenous

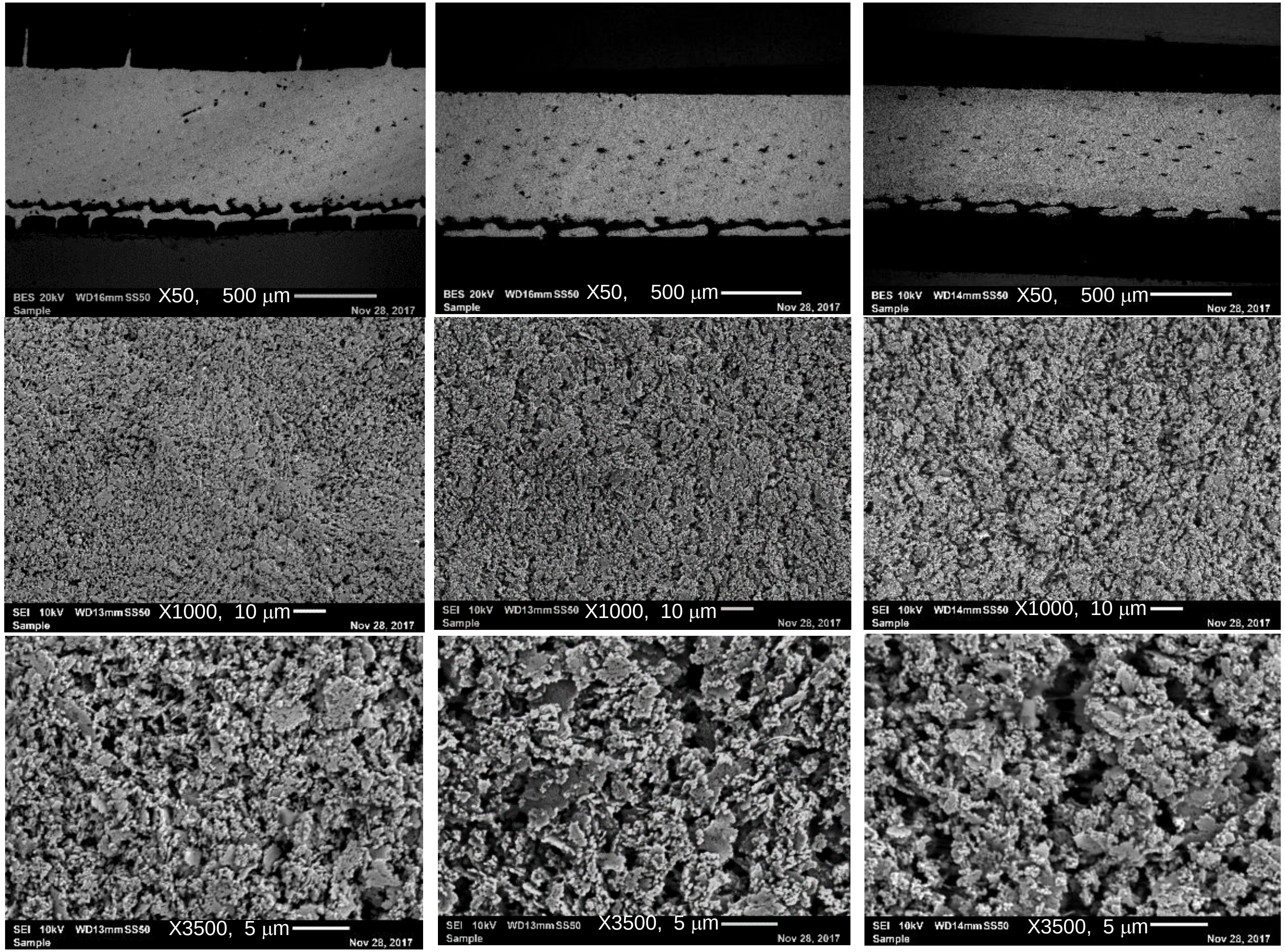
Experiment – Ink Printed with Standard Mesh Under Stretch

0% | 50% | 100%

X50

X1000

X3500



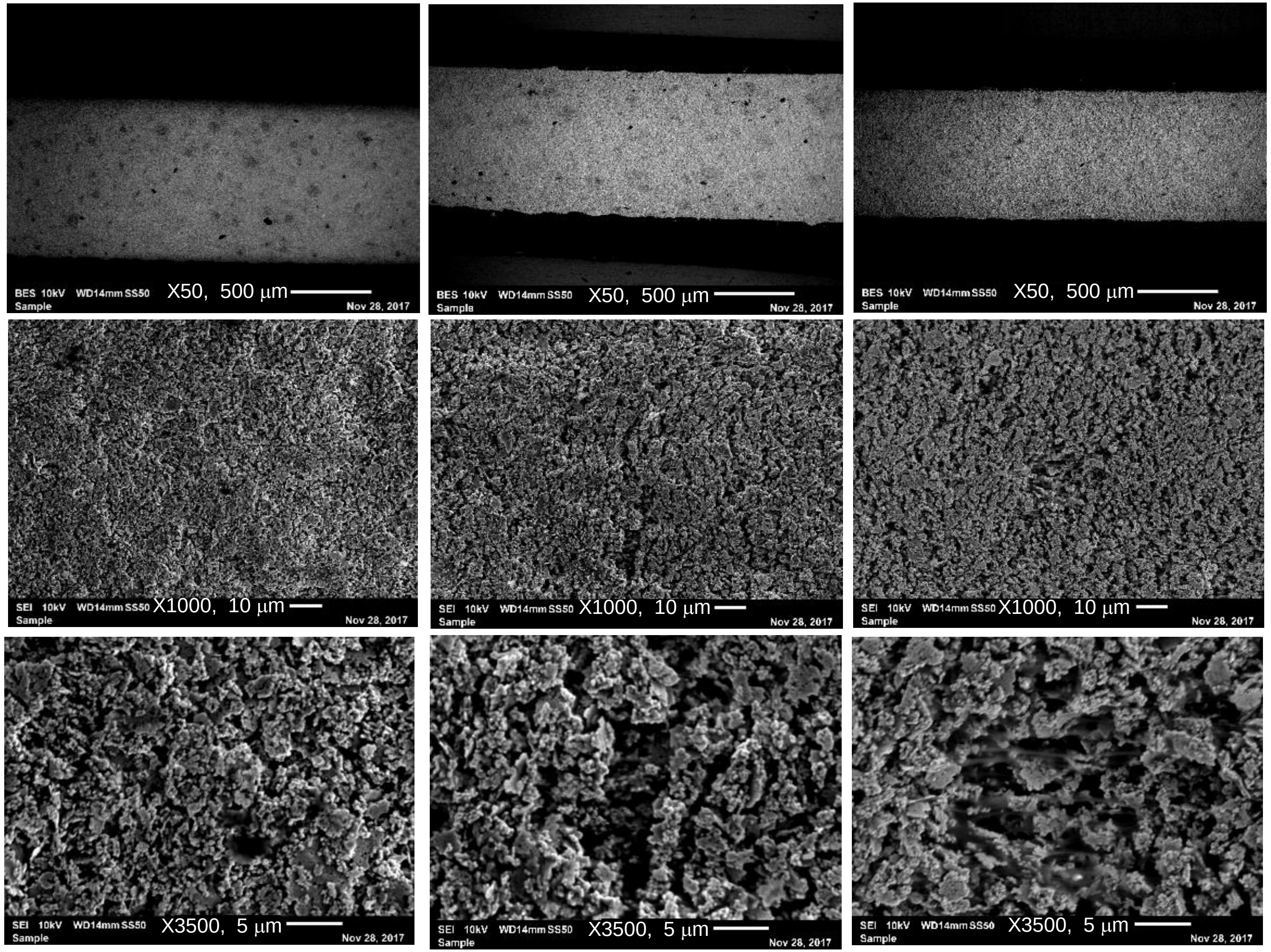
Observations

- Multiple vertically aligned cracks
- Higher elongation correlated with wider cracks

Results – Ink Printed with 3D Mesh Under Stretch

0% | 50% | 100%

X50, X1000, X3500



<u>Observations</u>

- Multiple vertically aligned cracks
- Higher elongations correlated with wider cracks

Surface morphology is similar in both print conditions

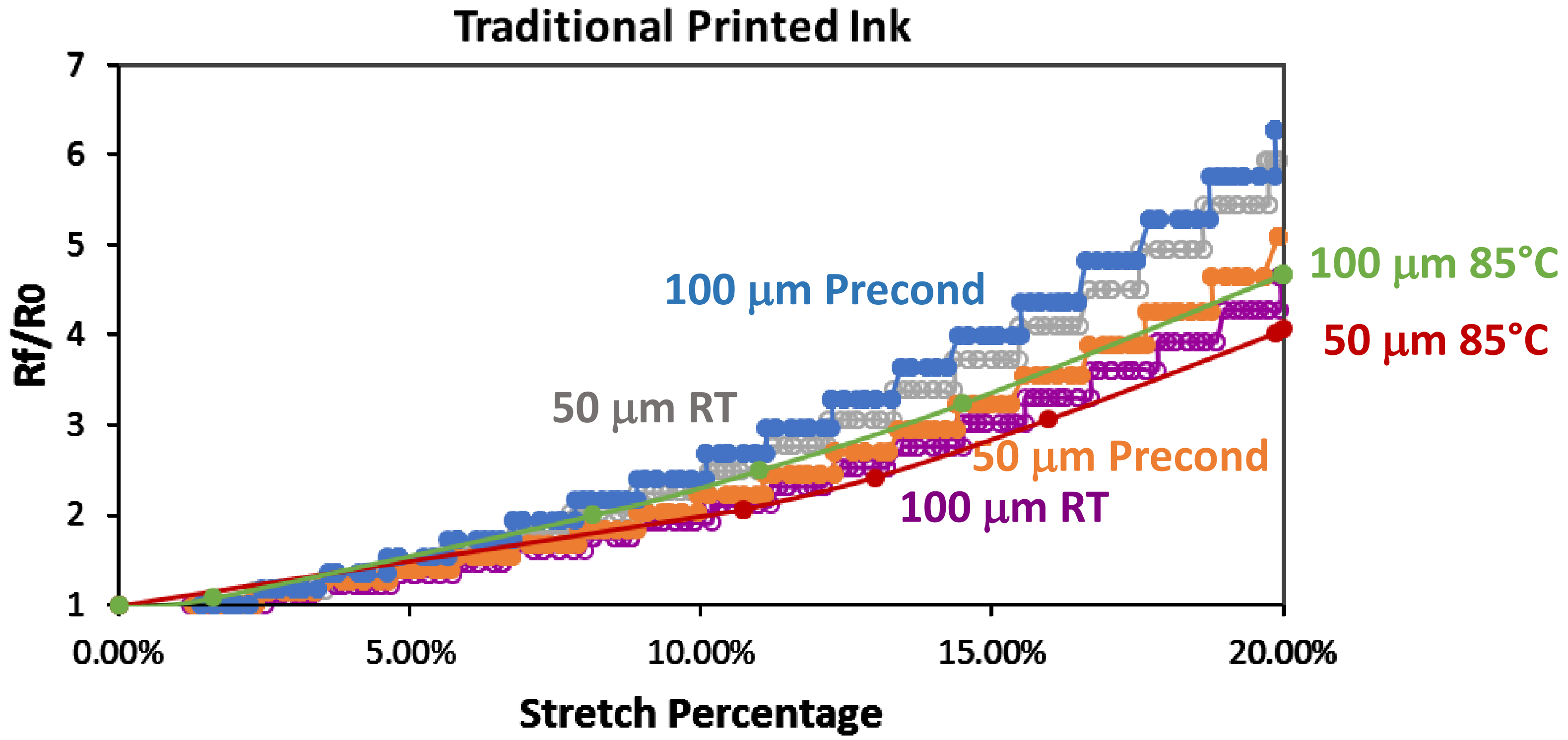
Results – Resistance Change During 1st Stretch Cycle: Standard Mesh

Three test conditions

- Room temperature - no sample preconditioning
- Room temperature - with sample preconditioning
- Elevated temperature - stretching in 85ºC temperature chamber

Two substrate film thicknesses

- 50 µm
- 100 µm



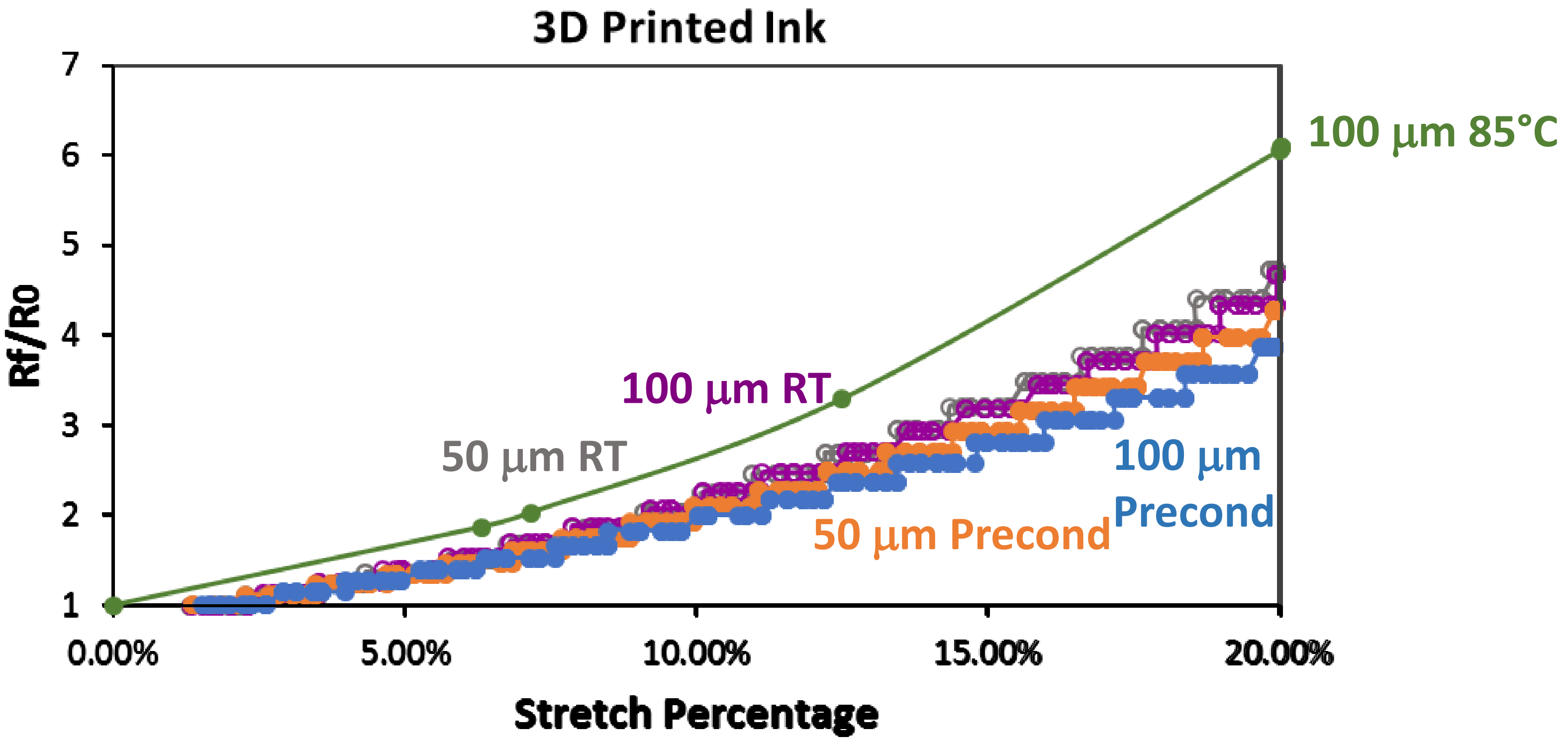
Observations

- Similar resistance curves for different test conditions and different substrate thickness
- Preconditioning and elevated operating temperatures didn't have a significant impact on the ink resistance

Results – Resistance Change During 1st Stretch Cycle: 3D Mesh

Three test conditions
•Room temperature - no sample preconditioning
•Room temperature - with sample preconditioning
•Elevated temperature - stretching in 85ºC temperature chamber

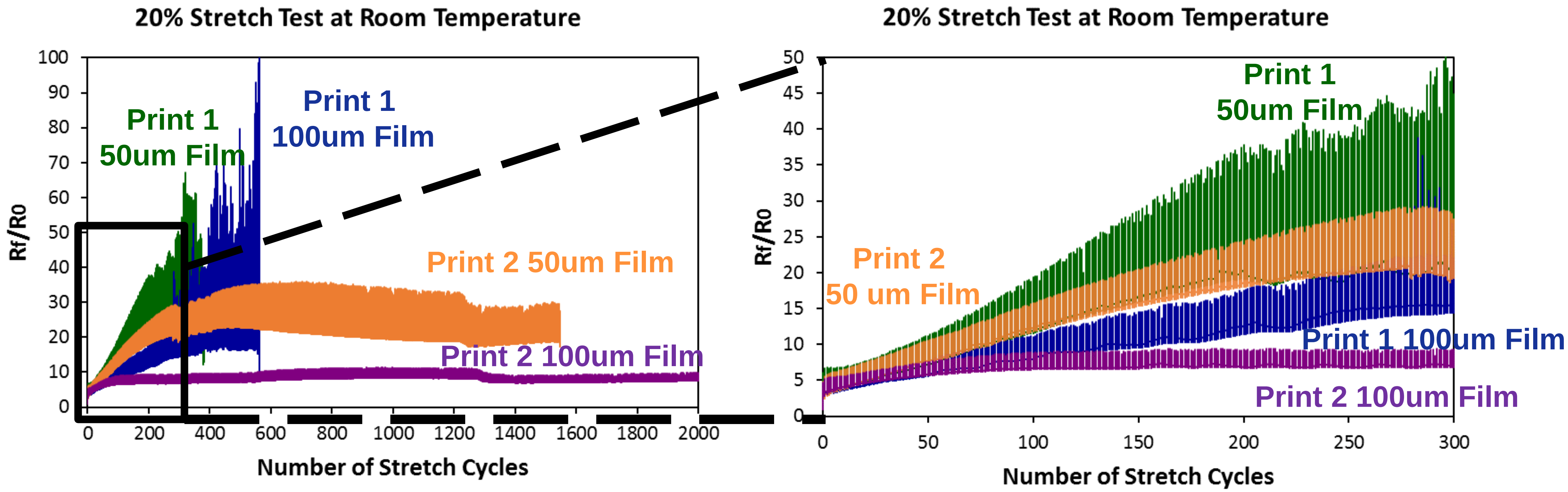
Two substrate film thicknesses
•50µm
•100µm



Observations
- Similar resistance curves for different test conditions and different substrate thickness
- Preconditioning and elevated operating temperatures didn't have a significant impact on the ink resistance

Results – Resistance Change at Room Temperature: No Preconditioning

Print 1 = Standard Screen Mesh
Print 2 = 3D Screen Mesh

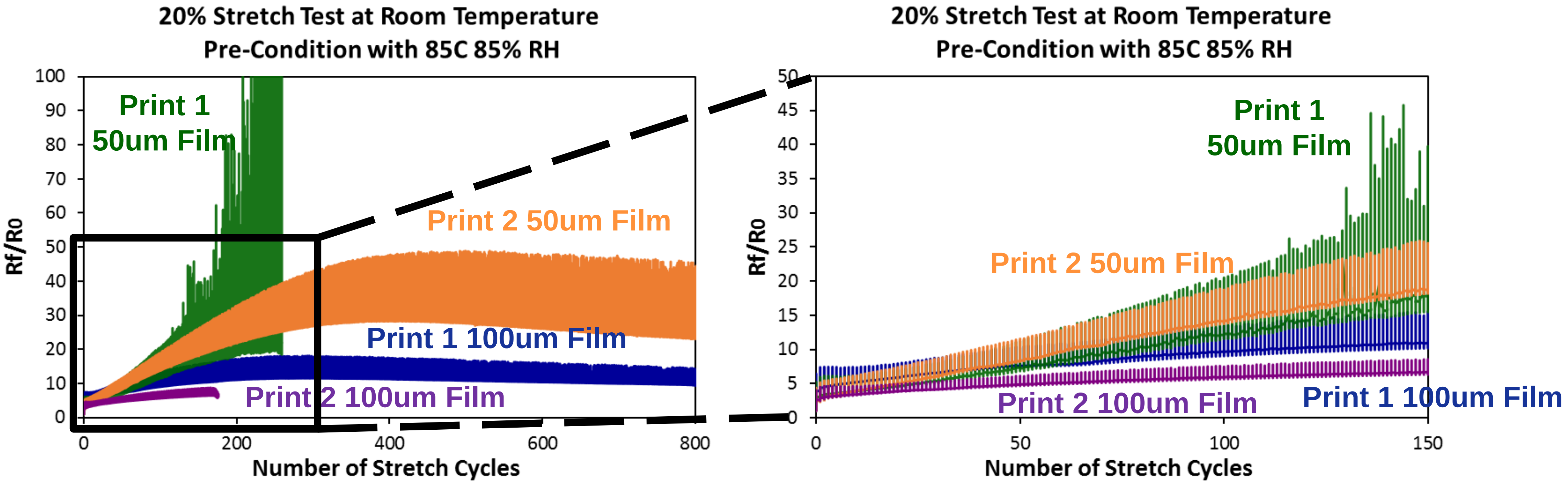


<u>Observations</u>

- Initial resistance is very similar for both ink thicknesses
- Early stage (<~75 cycles) of resistance are curves similar for all print and substrate thicknesses
- Thicker ink print (22 µm) and thicker substrate result in the lowest resistance curve and the lowest in-cycle resistance variation

Results – Resistance Change at Room Temperature: Pre-conditioned samples

Print 1 = Standard Screen Mesh
Print 2 = 3D Screen Mesh

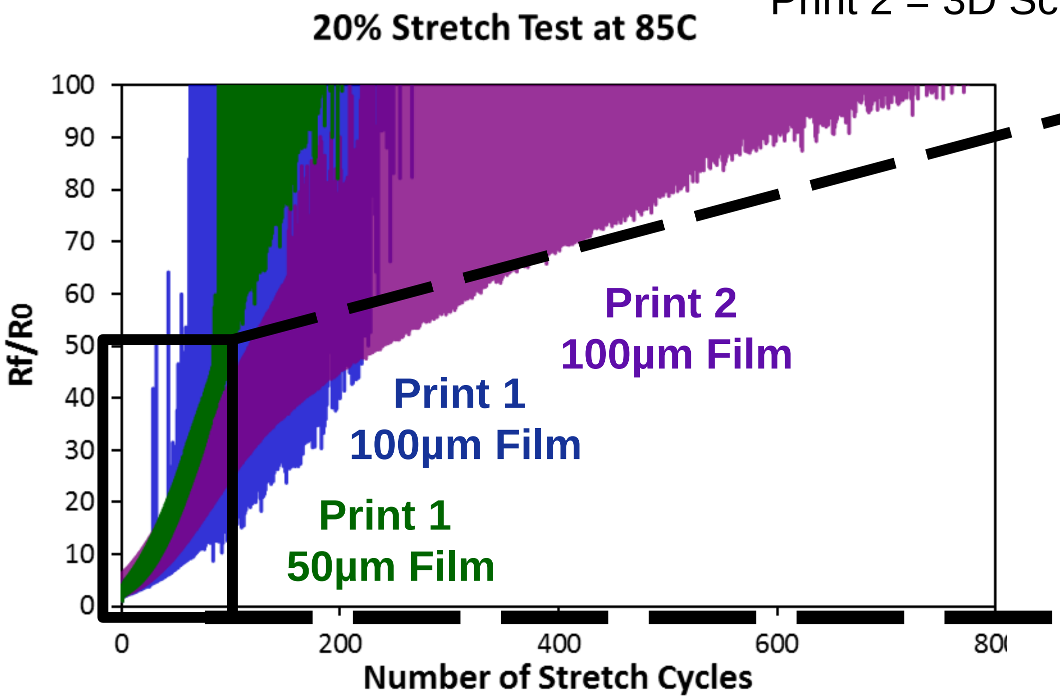


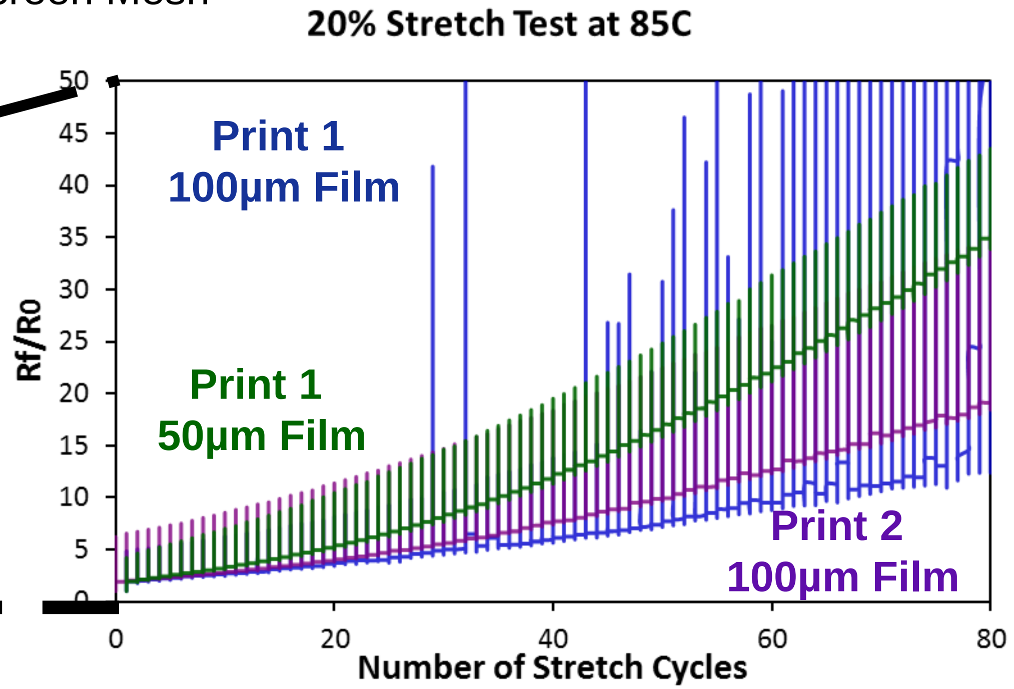
Observations

- Initial resistance is very similar for both ink thicknesses
- Early stage (<~30 cycles) of resistance are curves similar for all print and substrate thicknesses
- Thinner ink print (15µm) and thicker substrate result in the lowest resistance curve and the lowest in-cycle resistance variation out to 150 cycles

Results – Resistance Change at 85 °C

Print 1 = Standard Screen Mesh
Print 2 = 3D Screen Mesh



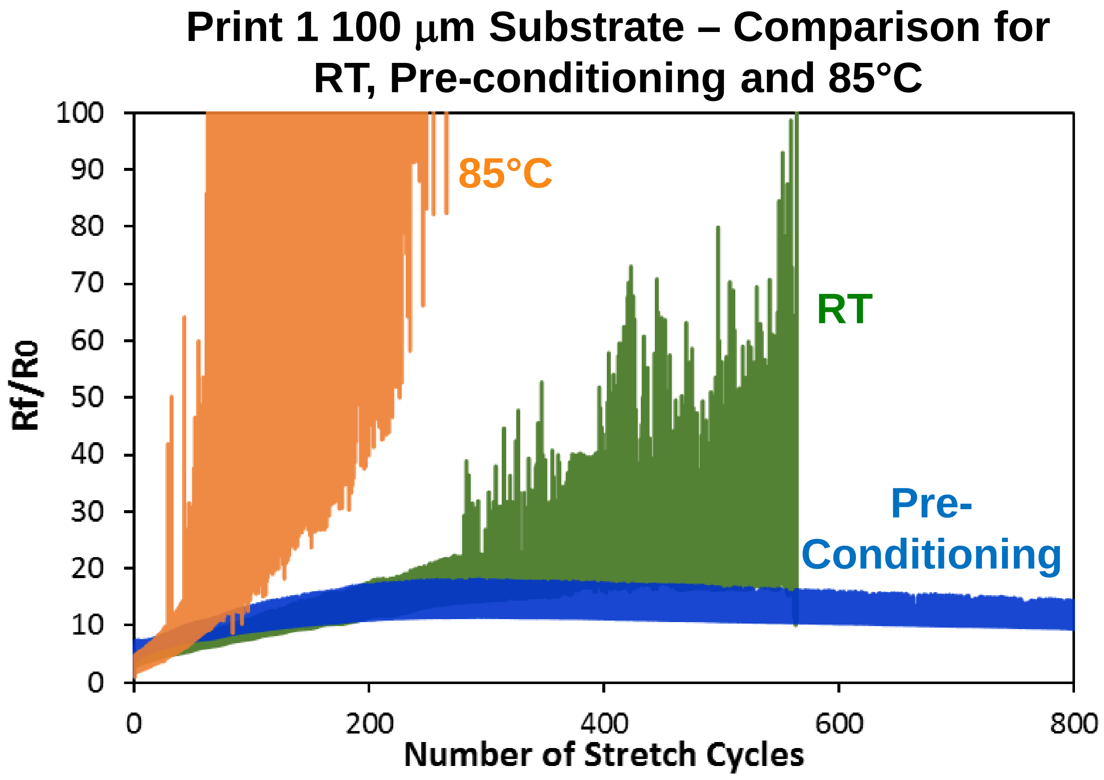


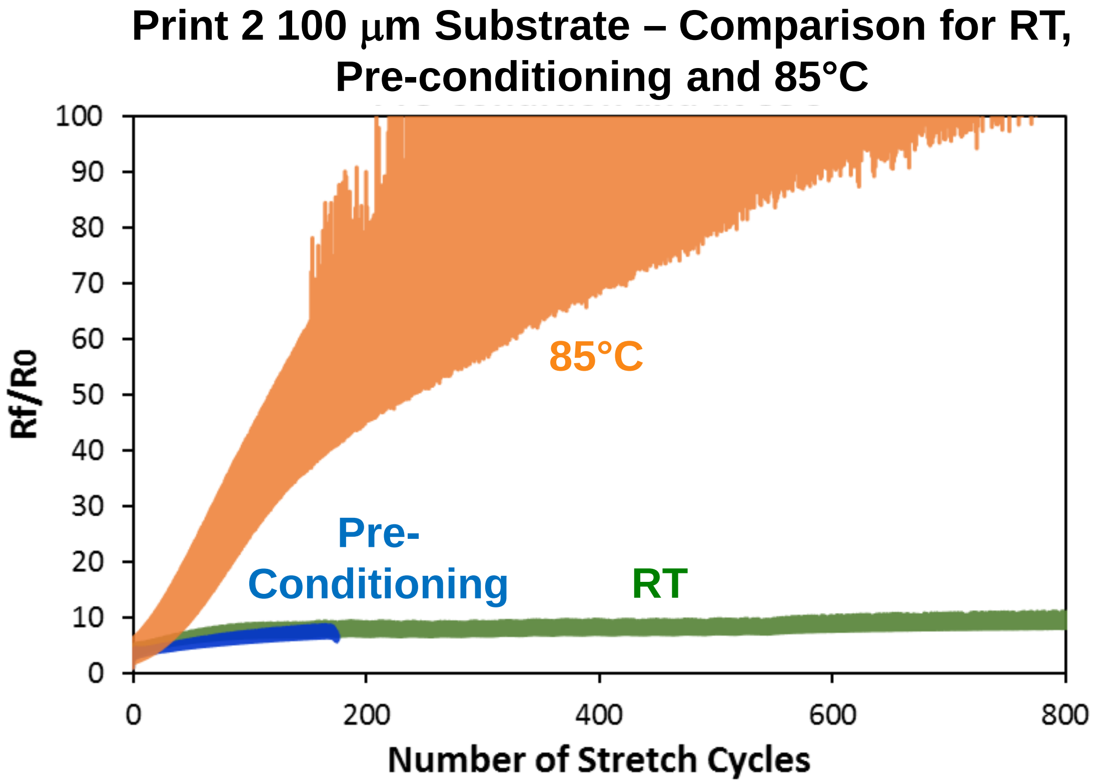
Observations

- Initial measured resistance is very similar for both ink thicknesses and both substrate thicknesses
- Early stage (<~30 cycles) resistance curves are similar for all conditions
- Difficult to determine/interpret extended cycle results
  - First time performing this test condition
  - There may be testing artifacts as a result of test probe contact consistency

Results – Ink thickness (on 100 µm film) versus test protocol

Print 1 = Standard Screen Mesh
Print 2 = 3D Screen Mesh





Observations

- Initial resistance and early cycling curves are similar for all conditions
- For standard print (Ink 1) the pre conditioned sample was more stable than RT sample (counter-intuitive)
- For 3D mesh print (Ink 2) RT sample resistance curve is very stable over 800 cycles
- Stretch cycling at elevated temperature (85°C) significantly accelerates the Rf/R_0 increase

Results – Comparison to Our Previous Studies with Thermoplastic Materials

Previous Studies

•Conductive ink printed on 100 µm thermoplastic polyurethane (TPU) substrates

•Rf/R0 > 200 after 500 cycles of 20% stretch

•More and larger cracks observed on surface morphology of conductive ink

Current Study

•The R_f/R_0 ratio for all samples were less than 100 after 500 cycles at 20% stretch

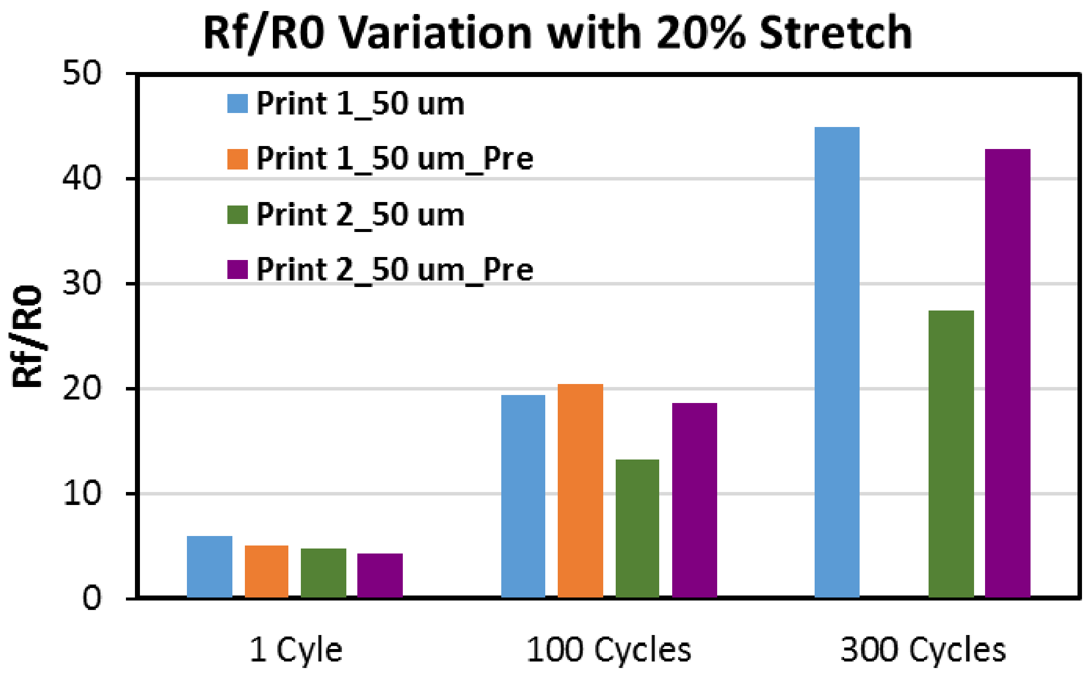
•For 22 µm (Print 2) with 100 µm substrate testing at room temperature, R_f/R_0 ratio is only 9.4 after 2000 cycles

Percolation Theory $\sigma = \sigma_{silver}(V_f - V_c)^S$

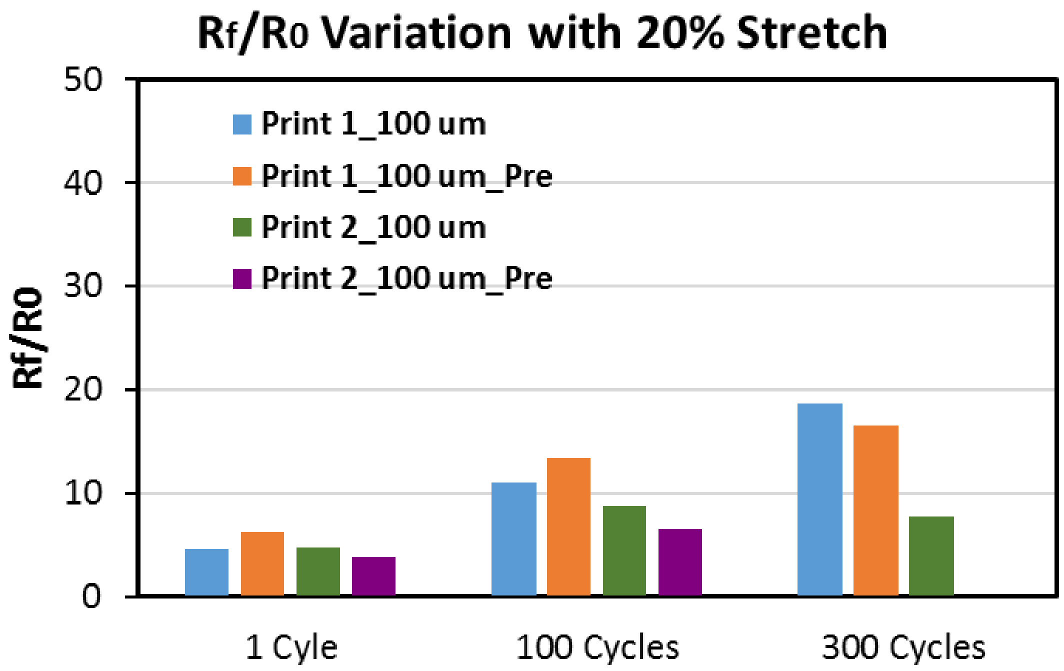
Summary

Print 1 = Standard Screen Mesh
Print 2 = 3D Screen Mesh

50 μm substrate



100 μm substrate



- Resistance changes during the first stretch cycle are quite similar in all conditions
- 3D mesh results in a thicker ink deposit and shows less resistance change over more stretch cycles
- In test conditions of RT and Pre-conditioning, the R_f/R_0 ratios were < 45 for samples with 50 μm substrate, and all < 20 for samples with 100 μm substrate

Conclusions and Discussion - 1/3

1. Test vehicles made with the developmental stretchable substrate film and conductive ink made with the thermosetting polymer resin demonstrated significantly better stretchability than thermoplastic materials studied in previous evaluations
   A. Rf/R0 ratio of 9.4 after 2000 cycles was achieved with 22 µm (Ink 2) with a 100 µm substrate tested at room temperature,
   B. For all constructions tested, the Rf/R0 ratio was less 100 for up to 500 cycles of stretch

Theory:

Cross-linked thermoset polymer is resistant to hysteresis in the ink form (maintaining conductive particle contact) and film form (relieving induced strain on the ink)

Conclusions and Discussion – 2/3

2. 85/85 Pre-conditioning does not significantly change the resistance performance during stretch testing

Theory:

Cross-linked thermoset polymer is resistant to heat and humidity

3. A thicker ink print on a thicker substrate provided better performance

Theory:

A thicker ink deposition is more mechanically resistant to cracking during stretch cycling

Theory:

A thicker substrate has greater residual strain and relieves stress on the conductive ink

Conclusions and Discussion – 3/3

4. For In-situ stretch testing at 85 C, the ink abrasion at the probe contact area and thermo-mechanical fatigue could degrade the stretchability testing
   - This test method may need more development

5. This was a preliminary study with one sample prepared for each test condition
   - Evaluations with larger sample sizes should be conducted in the future