Oil & Natural Gas Technology

Development of High Temperature Capacitor Technology and Manufacturing Capability

Final Technical Report

October 2006 - May 2011

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August 2011

DOE Award No.: DE-FC26-06NT42949

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Prepared for:

United States Department of Energy
National Energy Technology Laboratory

Office of Fossil Energy

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Abstract

The goal of the Development of High Temperature Capacitor Technology and Manufacturing Capability program was to mature a production-ready supply chain for reliable 250°C FPE (fluorinated polyester) film capacitors by 2011. These high-temperature film capacitors enable both the down hole drilling and aerospace industries by enabling a variety of benefits including:

- Deeper oil exploration in higher temperature and pressure environments
- Enabling power electronic and control equipment to operate in higher temperature environments
- Enabling reduced cooling requirements of electronics
- Increasing reliability and life of capacitors operating below rated temperature
- Enabling capacitors to handle higher electrical losses without overheating.

The key challenges to bringing the FPE film capacitors to market have been manufacturing challenges including:

- FPE Film is difficult to handle and wind, resulting in poor yields
- Voltage breakdown strength decreases when the film is wound into capacitors (~70% decrease)
- Encapsulation technologies must be improved to enable higher temperature operation
- Manufacturing and test cycle time is very long

As a direct result of this program most of the manufacturing challenges have been met. The FPE film production metalization and winding yield has increased to over 82% from 70%, and the voltage breakdown strength of the wound capacitors has increased 270% to 189 V/µm. The high temperature packaging concepts are showing significant progress including promising results for lead attachments and hermetic packages at 200°C and non-hermetic packages at 250°C. Manufacturing and test cycle time will decrease as the market for FPE capacitors develops.

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1 Executive Summary

The goal of the Development of High Temperature Capacitor Technology and Manufacturing Capability program was to mature a production-ready supply chain for reliable 250°C FPE (fluorinated polyester) film capacitors by 2011. These high-temperature film capacitors enable both the down hole drilling and aerospace industries by enabling a variety of benefits including:

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The key challenges to bringing the FPE film capacitors to market have been manufacturing challenges including:

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- Encapsulation technologies must be improved to enable higher temperature operation
- Manufacturing and test cycle time is very long

To solve the key manufacturing challenges for high temperature FPE film capacitors a premier team was assembled consisting of:

- Brady Corporation: The only company in the world with demonstrated capability of casting FPE polymer film in consistent thin (<5 μm) sheets.
- Steinerfilm: The premier company capable of metalization of thin films for electronics use.
- Dearborn Electronics: A premier producer of high quality, high temperature aerospace and military specification capacitor products.
- Hamilton Sundstrand: A premier provider of aerospace and industrial electronics components and systems.

This team conducted a series of experiments designed to optimize the production process, shown in Fig. 1-1, of high temperature FPE film capacitors. The experiments were divided into four production batches, where each batch focused on improving a different step of the production process with the end goal of a mature, production-ready supply chain for high temperature film capacitors.

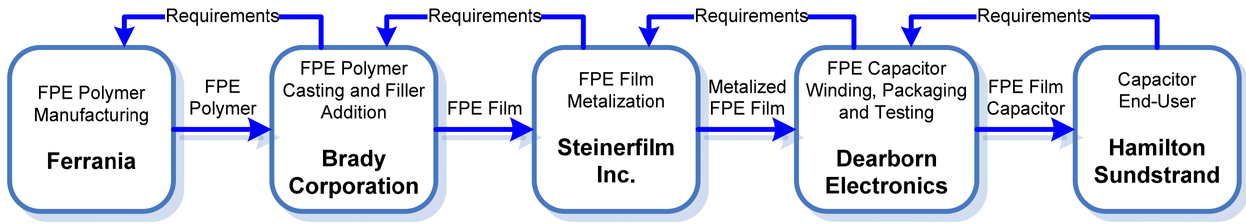


Fig. 1-1: FPE Film Capacitor Production Steps

As a direct result of this program most of the manufacturing challenges have been met. The FPE film production metalization and winding yield has increased to over 82% from 70%, and the voltage breakdown strength of the wound capacitors has increased 270% to 189 V/µm. The high temperature packaging concepts are showing significant progress including promising results for lead attachments and hermetic packages at 200°C and non-hermetic packages at 250°C. Manufacturing and test cycle time will decrease as the market for FPE capacitors develops.

Research continues even after this program to further optimize the final assembly steps of the capacitors. Meanwhile, Hamilton Sundstrand (HS) is actively transferring the FPE capacitor technology into power electronics systems. HS has tested FPE capacitors in an existing power electronics converter in a laboratory setting and is actively designing FPE capacitors into a ground-up design of a power electronics converter to demonstrate the technology alongside silicon carbide (SiC) switches and other high temperature electronics technologies. Simultaneously Dearborn Electronics is actively marketing FPE capacitors to its wide industrial, aerospace and petrochemical customers.

2 Introduction

2.1 Background

On April 5-6, 2005, DOD's Wright Patterson Air Force Research Laboratories (AFRL) and DOE's Sandia National Laboratories conducted a workshop to bring together two major industries with a common interest in high temperature power electronics. The workshop produced two major outcomes relative to high temperature capacitors:

> Reliable, consistently available, 250°C capacitors are needed in the 3-5 year time frame, and 300°C capacitors are needed in the 5-8 year time frame. Deeper down hole requirements as well as the More Electric Aircraft (MEA) require this fundamental technology.

To meet these requirements, Hamilton Sundstrand along with our partners Ferrania, Brady Corporation, Steinerfilm, and Dearborn Electronics embarked on this program to optimize the manufacturing process of an existing high temperature technology to meet the needs of the down hole and aerospace industries.

The aerospace industry has been using metalized film capacitors for power conditioning, filtering and energy storage applications for decades. Metalized film capacitors have the ability to "clear" small defects, have high reliabilities, have a long useful life, and fail in a controlled and manageable fashion at the end of life. Alternatively, multi-layer ceramic (MLC) capacitors have violent failure modes, and are highly susceptible to vibration and thermal cycling environments. Electrolytic capacitors have difficulties with "dry-out", a short life, and low reliabilities at elevated temperatures. Because of this, the aerospace industry focused on metalized film capacitor as the most viable solution.

Fluorine isophthalate terephalate (or Fluorine Polyester - FPE) was originally developed by 3M in the mid-1980s, but could not be manufactured in sufficiently thin gauges to make the compact energy dense capacitors required for aerospace applications. This changed in ~2000, when Brady Corporation began successfully producing thin gauges of the film (5 microns and below). Shortly thereafter, Hamilton Sundstrand, Dearborn Electronics, Steinerfilm, Brady Corporation, and Ferrania partnered with AFRL to produce the 250 V, 16 µF, 200°C capacitor shown in Fig. 2-1.

Fig. 2-1: 250V, 16µF, 200°C, Hermetic, Low Inductance Capacitor

In the past, various metalized film FPE capacitors were manufactured for Hamilton Sundstrand, NASA, AFRL, and the Army; however, these were "one of, special orders" and suffered from poor manufacturing yields at all stages of the manufacturing process.

This high temperature capacitor development program was built on the strong partnerships already in place among Ferrania (FPE resin manufacturing), Brady Corporation (film casting), Steinerfilm (film metalization), Dearborn Electronics (capacitor manufacturing), and Hamilton Sundstrand (applications and systems expert). High temperature FPE film capacitors manufacturing went through four stages of production optimization including film composition, metalization and capacitor assembly to arrive at a refined process for producing 250°C FPE film capacitors.

2.1.1 The Need for High Temperature Capacitors

High temperature capacitors are a fundamental building block required for high temperature power electronics. Both the down hole drilling industry and the aerospace industry are relying on the development of high temperature power electronics to develop the next generation products.

For the down hole drilling industry, deeper oil exploration forces higher temperature and pressure requirements to be met by the drills. In this case, high temperature capacitors are a direct enabler of deeper exploration.

For the aerospace industry, high temperature capacitors (and high temperature power electronics) bring about several major advantages:

– Similar to the down hole drilling industry, the availability of high temperature power electronics are an enabler for the aerospace industry. For example: integrate motor drives with motors to eliminate large and heavy EMI filters, place power electronics in close proximity to turbine engines to achieve greater controllability, and design power electronics for high Mach air vehicles (which have high temperatures due to the frictional heating that occurs at higher speeds).

– With high temperature capacitors, the cooling requirements internal to each Line Replaceable Unit (LRU) are greatly reduced by enabling a higher temperature gradient between heatsink and capacitors. Heatsink weight and volume reductions are an obvious advantage; however, reductions in bus bar cooling and magnetics cooling also bring about weight and volume savings.

– A major advantage that high temperature capacitors will bring the aerospace industry is an increased capacitor reliability when operated at below rated temperature (~130°C). Today, it is typical to operate a 125°C capacitor at 110°C. Utilizing a 250°C capacitor in this same 110°C application will result in a dramatic increase in reliability and life (~10x improvement is predicted utilizing the appropriate Eyring, Arrhenius and Coffin-Mason models for humidity, elevated temperature and temperature cycling respectively.)

– Finally, a high temperature capacitor can handle higher electrical losses without overheating.

It is also worthy to note that the government and industry are investing Millions of dollars each year on the development of Silicon Carbide (SiC) power electronic switching device research. It is true that SiC will enable high temperature motor drives and power supplies; however, this investment cannot be taken advantage of without high temperature capacitors. The "dc-link" capacitor is a fundamental building block of a voltage source inverter (VSI). Similarly, AC capacitors are required to be within close proximity of the switching devices in a Matrix converter or current source inverter (CSI).

2.1.2 High Temperature Capacitor Film Materials (125°C to 300°C)

2.1.2.1 Polycarbonate

Since Bayer's announcement to cease production of PC film, Electronic Concepts now claims the ability to manufacture PC film from General Electric (GE) resin. In addition to standard 125°C PC, GE also claims the ability to manufacture a higher temperature PC (150°C). To date, these films have not been compared with existing capacitors made from Bayer's PC film. Small changes in the resin manufacturing process, the solvents used to cast the film, or the resin filtering used in the casting process can change the final PC capacitor's integrity and electrical properties. In particular, a lower molecular weight will produce drastically lower DWV capabilities. Also, the requirements of the HTHP Drilling Program (> 400°F or 204°C) eliminate this film from consideration for the downhole industry.

2.1.2.2 Polyetherimide

(PEI) was investigated by Electronic Concepts (Capfilm, Inc) in 1991. A preliminary evaluation of the film shows 180°C capability and favorable electrical characteristics. The known disadvantage to PEI is its high moisture absorption rate. This forces PEI capacitor packaging to be fully hermetic. Again, the HTHP Drilling Program requirements (> 400°F or 204°C) eliminate this as a successful candidate.

2.1.2.3 Kapton or Teflon Paper - Foil Capacitors

Capacitors available for downhole drilling applications are being manufactured from Kapton or Teflon (Du Pont Company's trademark names for polyimide – PI and polytetrafluoroethylene – PTFE respectively). Capacitors wound with these dielectrics are most often standard paper-foil construction because the materials are only available in thick films with large thickness variations for any one casting. Although this technology is stable and readily available, it produces a large and expensive capacitor. A non-hermetic 200°C, 400V, 30µF capacitor would be approximately 3 inches in diameter and 4.4 inches long. The material and manufacturing costs of this capacitor are estimated at >$700 (cost estimated in 2003).

2.1.2.4 DLC & PCD

Although DLC (Diamond Like Carbon) & PCD (Polycrystalline Diamond) Capacitors have the potential of achieving size and weight reductions compared to present PC capacitors (assuming very thin supporting base metals), the required lengths of film are presently not available for large value (30 – 45µF), 400V capacitors. Additionally, since DLC and PCD are deposited dielectric material, on a supporting base metal, the size, weight, and resulting performance of the capacitor is dependent upon the base metal selected.

2.1.2.5 FPE

FPE is the most promising high temperature capacitor film and has born positive results in this program.

In the mid 1980's, the Air Force recognized the tremendous need they would have for high temperature capacitors. In the early 1990's, capacitor failures grounded aircraft during Desert Storm. Those failures, caused by overheating, dramatically underscored another need for higher temperature capacitors in current aircraft systems. AFRL/PRPE funded a program called the High Temperature Dielectric Program to develop a high temperature capacitor film for an operating temperature of >500K (>227°C). Under the program, plain and metalized FPE films were produced and wound into capacitors. These capacitors were tested to be thermally stable at greater than 225°C with DC and AC voltages applied (and the sheet samples of FPE were tested to 300°C). The total cost for the development of the FPE film approached $1M; with the program starting in 1988 and concluding in 1994. Unfortunately, due to web-handling limitations, FPE film thinner than ~10μm was not possible.

3M's formula for FPE (Fluorene Poly Ester or Poly 9,9-bis-(4-hydroxphenyl)-fluorine iso/terephthalate) was spun off into a company named Imation, which later became Ferrania, which now holds the FPE license, and decided it was an attractive market to enter based on the potential financial reward. As a side note, the first 3M patent on FPE (#4,967,306) ended in 2007 and the second patent (#5,115,372) ended in 2009.

It is important to note that the original work with 3M was interesting, although not useful to Hamilton Sundstrand aerospace (or downhole drilling) products since they could not produce films thinner than ~10μm (theoretically, only 2.35μm film is required for 400Vdc capacitors). In ~2000, Brady Corporation was integrated into the team, and they quickly produced 5, 3.5 and even 2.35μm film (and demonstrated voltage breakdown strengths of > 500 V/μm at temperatures to 250°C). Brady Corporation brought a tremendous amount of polymer experience and expertise to the table, along with their own internal funds, which greatly minimized DOD dollar expenditures. Without a doubt, Brady was key to the recent success of the FPE material given the total Air Force, Army and NASA expenditure of less than $800K.

FPE has a higher dielectric constant than PC (3.4 instead of 3); which translates to a 13% increase in capacitance (given the same film thickness). In addition, since FPE has a breakdown voltage of 12kV/mil (as opposed to PC's 8kV/mil) an even greater increase in capacitance (or decrease in size) can be achieved by going to a thinner gauge of film.

2.2 Scope

This project is specifically targeted to solve the key production challenges inherent with FPE film to allow for higher yield production with reliable capacitor performance.

2.2.1 Capacitor Production Process

A flowchart for the high temperature FPE capacitor production program is shown in Fig. 2-2. The process begins with Ferrania producing the FPE polymer and shipping it to Brady Corporation. Brady Corporation then adds fillers to the film to improve film handling and casts the polymer into a thin film ≤ 5µm in thickness. The film is then passed on to Steinerfilm who slits the film into the appropriate widths for capacitor manufacture and metalizes the film with a thin layer of aluminum. The metalized film is then passed to Dearborn Electronics where it is rolled into capacitors, packaged, and tested.

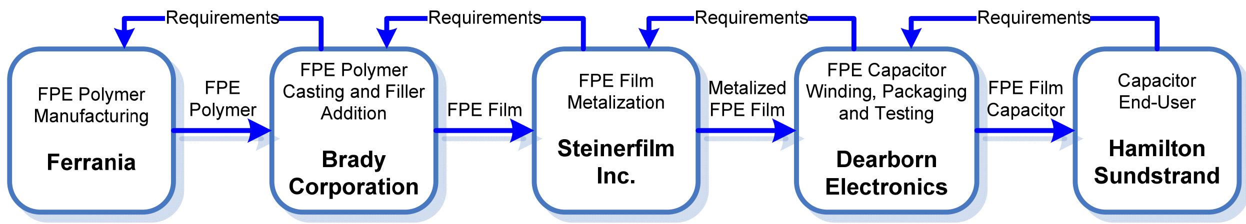


Fig. 2-2: FPE Film Capacitor Production Steps

2.2.2 FPE Production Key Challenge

Metalization of capacitor film typically occurs on a clean, free standing film (PP, PPS, MPF, PEN, …). Directly after the metalization layer is applied to film (typically only 500 Angstroms of metal is required), voltage is applied to the film to "clear" any defects from the bare, single layer of film. Clearing the film at this stage of the process (as opposed to clearing the film when it is wrapped in a capacitor) allows any large defects to fully clear, and debris is free to escape into the atmosphere or even onto the mfg floor.

Previously, pure FPE was being solvent cast onto a 2 mil polyester liner (also referred to as a backer, or a carrier), metalized, slit, and then removed from the liner. Because the FPE was on the liner during the metalization process, clearing of the bare film could not occur. This means the first time the FPE metalized film was energized with voltage was when it was in the capacitor, which obviously was not as beneficial as the process described in the previous paragraph. This was being done because pure FPE, not on a liner, is very "smooth" (it has a low coefficient of friction – COF and very low Atomic Force Microscopy - AFM … see Table 2-1) which makes it difficult to handle. Because pure FPE is so smooth, it tends to "stick" to itself in the form of wrinkles, and pure FPE also tends to "stick" to the rollers of the metalizer, and capacitor manufacturing processes. These characteristics are described generically as poor "web-handling" properties, meaning the FPE web is difficult to handle as it is passed through a sequence of rollers where tension, speed, and position must be accurately controlled. These poor web-handling properties often lead to film stretching or other harmful effects during the manufacturing processes.

Table 2-1: AFM Measurements Comparing PPS and FPE Capacitor Films

AFM Surface Roughness Results of Dearborn Materials			
	R_{rms}	R_{ave}	R_{max}
Metalized PPS 2 µm x 21 mm	53.2 nm	35.0 nm	472 nm
Metalized PPS 2.5 µm x 25.4 mm	74.1 nm	35.7 nm	998 nm
Metalized PPS 3.5 µm x 25.4 mm	74.0 nm	61.2 nm	600 nm
Metalized PPS 6 µm x 31.7 mm	66.1 nm	49.2 nm	556 nm
Metalized PPS 9 µm x 25.4 mm	37.8 nm	25.5 nm	481 nm
Metalized PPS 12 µm x 25.4 mm	48.5 nm	37.8 nm	415 nm
Metalized PPS 12 µm x 25.4 mm	51.1 nm	39.4 nm	429 nm
FPE	9.76 nm	5.65 nm	225 nm

Several options exist to improve the web-handling properties, and these options have been utilized, proven, and optimized over time with the standard low temperature capacitor films of PP, PPS, and PEN. The most promising option involves adding a "filler" to the FPE during the solvent casting process. Fillers are used in other films for exactly this reason, and adding the correct filler was shown to solve the web-handling issues previously experienced with FPE.

Typical fillers tended to increase the dielectric constant of the resulting film construction, but they also tended to decrease the dielectric withstand Voltage (DWV) capability by up to 10%. This small decrease in voltage breakdown is insignificant considering sheet samples of FPE were showing >500 V/µm; whereas the final capacitor was only handling approximately 75 V/µm. The large decrease was attributed to the lack of clearing of the bare film, as well as other detrimental effects caused by the poor web-handling properties of pure FPE. Obviously, if the 500 V/µm sheet sample comes down to ~450 V/µm, but the bare film clearing is possible and web-handling is greatly improved (greatly improving process yields), final capacitors were able to handle >150 V/µm.

2.3 Application

NETL and the downhole industry need a dependable source of high temperature capacitors to meet the requirements outlined in Table 2-2. This enables the industry to design reliable, repeatable, power electronics for high temperature drilling and aerospace applications. Additionally, the commercialization of high temperature FPE capacitors is directly applicable to the business plans and strategic planning of Ferrania, Brady Corporation, Steinerfilm and Dearborn Electronics.

Ferrania, Brady Corporation, Steinerfilm and Dearborn Electronics invested some private IR&D funds to advance FPE technology. These funds, coupled with small amounts of government funding from AFRL, NASA, and the Army have brought FPE technology to where it is today. Prior to this program, more IR&D funding was applied to the technology. Small web-handling experiments are occurring between Brady Corporation (utilizing a small, non production pilot coater) and Steinerfilm. Additionally, Dearborn Electronics is experimenting with high temperature connections using Teflon as a base material (in preparation for the upcoming FPE production runs). These efforts, as well as, their commitments to "cost share" with Hamilton Sundstrand in the HTHP Drilling Program, demonstrate the commitment, support and commercialization potential of FPE high temperature capacitors.

Table 2-2: Minimum Capacitor Technology Requirements for Unified Technology Maturation

Capability Requirements	Downhole	Aerospace
250°C operation	~300 to 500 hrs	>2000 hrs
-55°C operation	N/A	Yes
-55 to 250°C thermal cycling	N/A	~500 cycles
Low Dissipation Factor	Yes	Yes
High Energy Density	Yes	Yes
Manageable Failure Modes	N/A	Yes
Operation at increased Pressure	Packaging Dependent	N/A
Operation at reduced Pressure	N/A	Packaging Dependent
Long Life	~300 to 500 hrs	~20,000 hrs
High Reliability (MTBF)	Desired	> 1e6 hrs
Withstand High Vibration	Yes (Packaging Dependent)	Yes (Packaging Dependent)
Thermal Shock	N/A	Yes

3 Experimental Methods

To optimize the capacitor production process, this program was divided into four Batches, each was designed to optimize a specific step of the process shown in Fig. 3-1. In the first batch, initial web handling experiments were performed. The goal of the web handling experiments was to create a film that could be effectively handled by the film processing equipment without significant sticking, stretching, wrinkling or breaking and with little to no degradation of capacitor electrical and thermal performance. In the second batch, web handling was further optimized based on feedback from the first batch. In the third batch, metallization was optimized. Finally, in the fourth batch capacitor manufacturing and packaging was optimized. Due to programmatic challenges and the opportunity to leverage additional FPE capacitor development funding by AFRL to further advance batch 2, Batches 3 & 4 were combined into a single run with twice the volume.

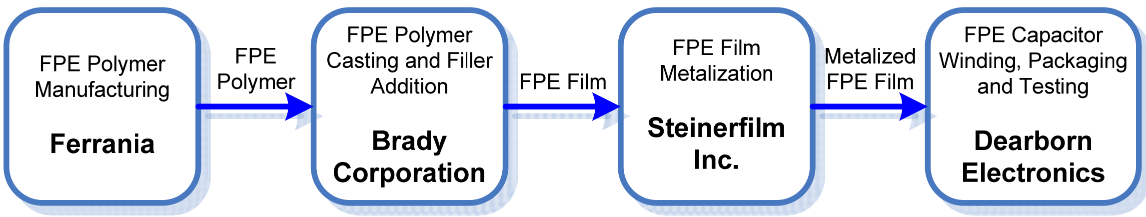


Fig. 3-1: FPE Film Capacitor Production Steps

3.1 Batch 1: Web Handling Experiments

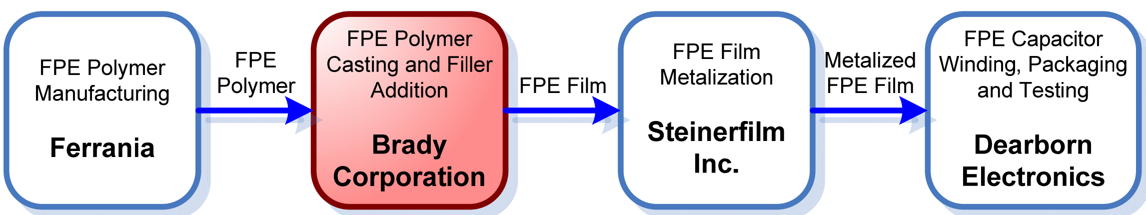


Fig. 3-2: Batch 1 Web Handling Experiments with Focus on Filler Addition

The objective of Batch 1 was focused on improving the polymer casting and filler portion of the process performed by Brady Corporation and highlighted in Fig. 3-2.

3.1.1 Batch 1: Film Casting (Brady Corporation)

Brady Corporation produced six different formulations of FPE film with varied amounts and types of fillers. Each of the six formulations were produced in a version with a carrier backing, designed to improve material handling for the metalization process, and a version without the backing.

3.1.2 Batch 1: Metalization (Steinerfilm)

The films from Brady Corporation were passed to Steinerfilm where they were slit to the appropriate widths and lengths and metalized using an aluminum metalization process. Steinerfilm then provided web handling feedback to Brady.

3.1.3 Batch 1: Capacitor Manufacture and Testing (Dearborn Electronics)

The metalized films were then shipped to Dearborn Electronics for film property and capacitor testing. At Dearborn, a sequence of tests was performed as shown in Fig. 3-3.

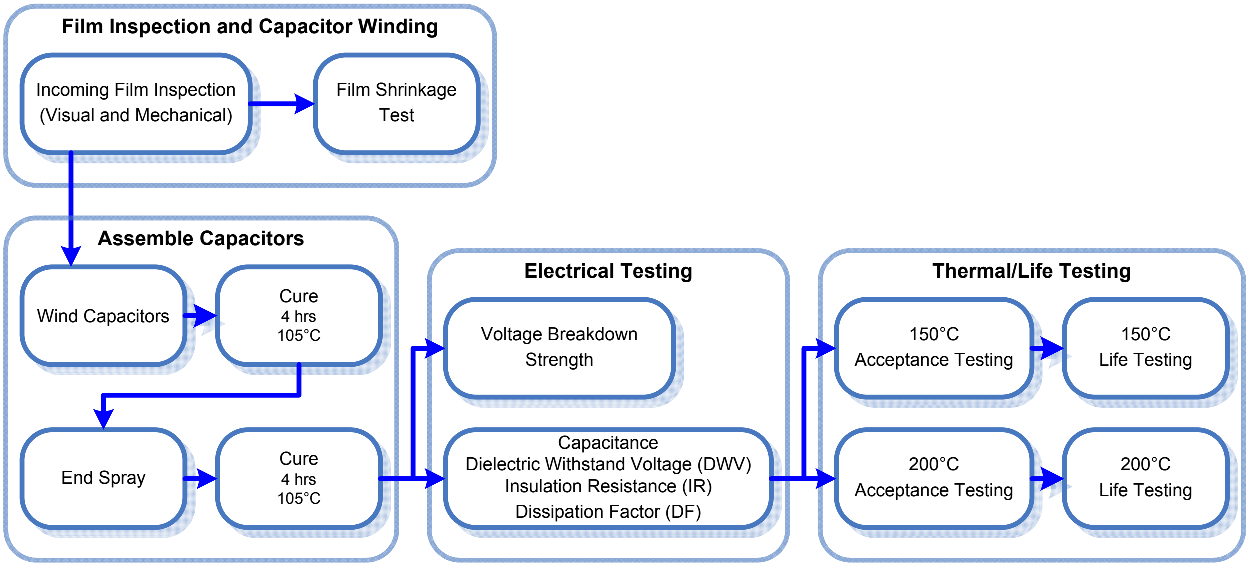


Fig. 3-3: Batch 1 Dearborn Electronics Testing

3.1.3.1 Film Inspection and Capacitor Winding

The first test was an incoming material inspection where they were examined visually and mechanically in the same manner as standard dielectrics. Next film shrinkage testing was performed on a sample of the film. Then, capacitors of approximately 7 µF were wound from the films. The wound Film was then cured at 105°C, end sprayed and cured again at 105°C.

3.1.3.2 Electrical Testing

Wound capacitor electrical testing started with determination of the voltage breakdown strength. This is a test to failure (short circuit) to establish voltage ratings for subsequent testing of Dielectric Withstand Voltage (DWV) and Insulation Resistance (IR). Capacitance readings were taken and recorded at a frequency of 1 kHz, and Dissipation Factor (DF) readings were measured and recorded at frequencies of 100 Hz, 1 kHz and 10 kHz.

3.1.3.3 Thermal/Life Testing

After the initial Electrical testing, acceptance and life testing was performed. The acceptance test was a 48 hour test during which some capacitors were subjected to 150°C temperatures and others to 200°C temperatures with 250 VDC applied. The life test is the same but the duration is increased to 250 hours.

3.2 Batch 2: Web Handling Optimization

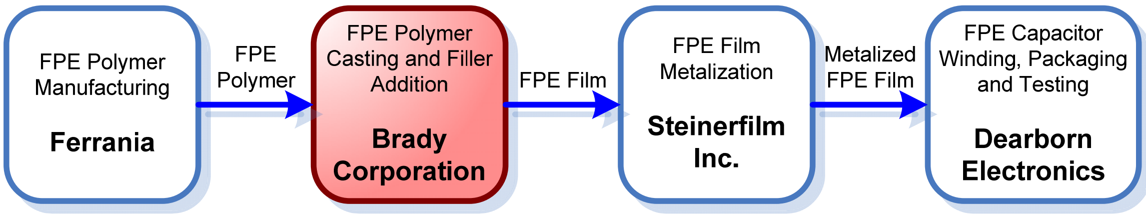


Fig. 3-4: Batch 2 Web Handling Experiments with Focus on Filler Optimization

The objective of Batch 2 was to further optimize the casting process of FPE film on the Brady's production casting equipment, highlighted in Fig. 3-4. As with Batch 1, six different FPE constructions were examined; however, with the knowledge gained from Batch 1, the variations between constructions was not as great. Again, web-handling properties and the high temperature capacitor properties (in particular, DWV and elevated temperature characteristics of the final capacitor assembly) were the desired optimization metrics.

3.2.1 Batch 2: Film Casting (Brady Corporation)

Additional funding was available from the Air Force Research Labs (AFRL) for FPE capacitor development and the team took advantage of this funding to improve the film casting process. After the AFRL film optimization study, Brady cast the DOE NETL Batch 2 FPE film with six different formulations.

3.2.2 Batch 2: Metalization (Steinerfilm)

For Batch 2 production, Steinerfilm moved the metalization process to production metalization lines from the demo lines. Otherwise the process was similar to that described in 4.1.2.

3.2.3 Batch 2: Capacitor Manufacture and Testing (Dearborn Electronics)

Batch 2 testing at Dearborn Electronics was the same as Batch 1 testing described in 3.1.3 except that the first curing was performed at 200°C and the second curing was eliminated as shown in Fig. 3-5 based on lessons learned from the AFRL FPE development program.

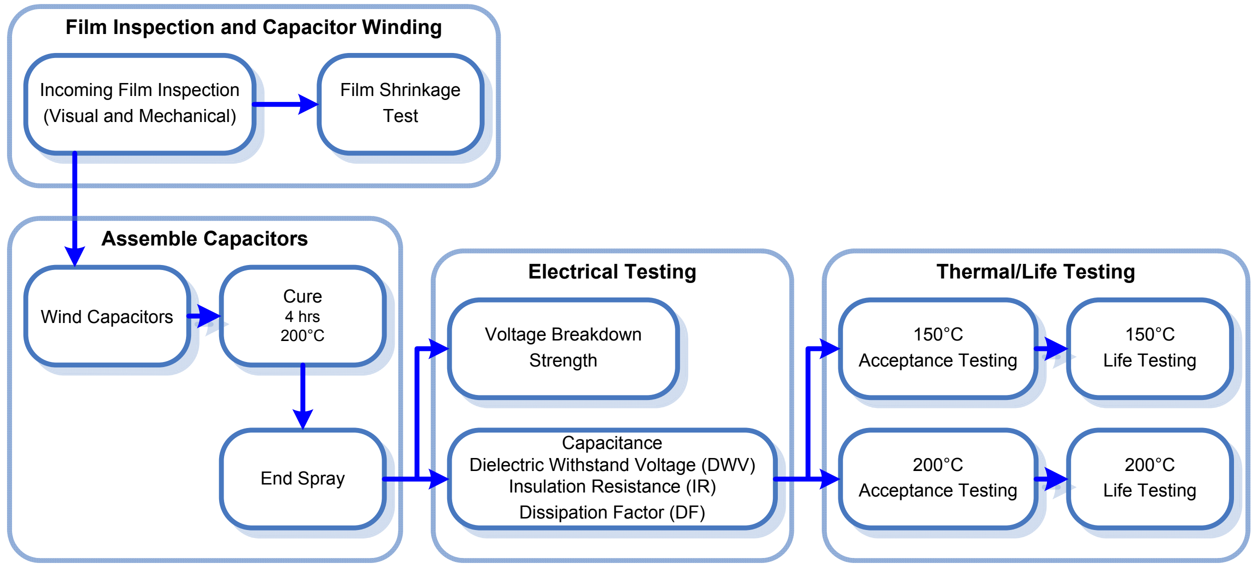


Fig. 3-5: Batch 2 Dearborn Electronics Testing

3.3 Batches 3 & 4: Metalization and Capacitor Manufacturing Optimization

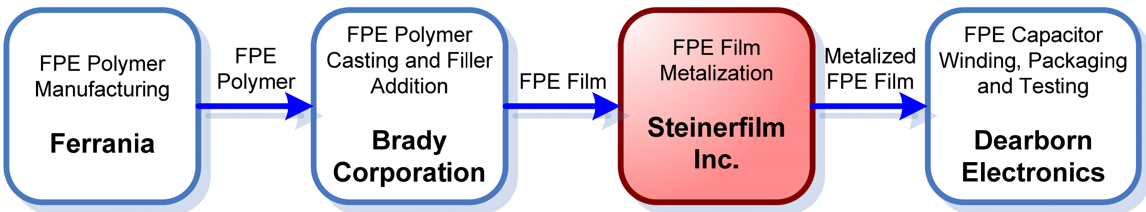


Fig. 3-6: Batch 3 Metalization Optimization

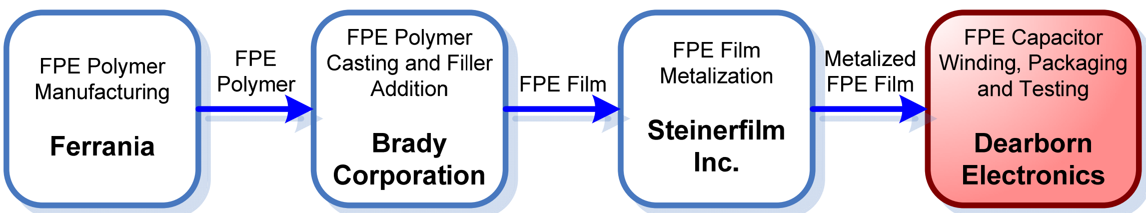


Fig. 3-7: Batch 4 Capacitor Manufacturing Optimization

The objective of Batch 3 was to optimize the metalization process performed by Steinerfilm, highlighted in Fig. 3-6, and the objective of Batch 4 was to optimize the capacitor manufacturing process at Dearborn Electronics, highlighted in Fig. 3-7. Due to schedule constraints a plan was developed to combine Batches 3 and 4 into a single production run optimizing both metalization and capacitor manufacturing as well as test four new film material variations. A total of 12 samples were created and were to be processed as shown in Table 3-1.

In Batches 1 and 2, Steinerfilm was held to a fixed amount of acrylate coating, a fixed metalization thickness, a fixed clearing voltage, as well as fixed tensions and other proprietary process variables. The fixed acrylate coating and metalization thickness are denoted as STD in

Table 3-1. This was done to avoid influencing the experiments being performed by Brady Corporation in Batches 1 and 2. In Batch 3, Steinerfilm performed the experiments necessary to optimize the metalization process. Dearborn Electronics manufactured capacitors from this FPE film and provided Hamilton Sundstrand the results of the experiments which are documented herein.

During Batches 1 and 2, Dearborn Electronics maintained a constant and pre-defined capacitor manufacturing process. This was done to avoid influencing the experiments being performed by Brady Corporation and Steinerfilm. In Batches 3 & 4, Dearborn Electronics manufactured capacitors from FPE film with varying manufacturing process experiments to optimize the Dearborn process.

Table 3-1: Batch 3 and 4 Material Plan

Brady Batch #	Sample #	Run #	NETL #	FPE Variation	Acrylate Coating				Al Metal		
					LT	MED	HVY	Strip B4 Met.	LT	MED	HVY
201568-A	33	33	3	Std	X				X		
201569-A	34	34	3	Std	X						X
201570-A	35	35	3	Std		X				X	
201571-A	36	36	3	Std			X		X		
201574-A	37	37	3	Std			X				X
201576-A	38	38	3	Std		X		X		X	
201578-A	39	39	4	Std		X				X	
201584-A	40	40	4	Var. 1		X				X	
201586-A	41	41	4	Var. 2		X				X	
201581-A	42	42	4	Std		X				X	
201605-A	43	43	4	Var. 3		X				X	
201621-A	44	44	4	Var. 4		X				X	
201786-A	Setup Materials for Steiner										

3.3.1 Batches 3 & 4: Film Casting (Brady Corporation)

Brady cast the DOE NETL Batch 3 and 4 FPE film with twelve rolls of five different formulations using the production processes matured in Batches 1 and 2.

3.3.2 Batches 3 & 4: Metalization (Steinerfilm)

For Batch 3 & 4 production, Steinerfilm used production metalization equipment to metalize the FPE films with variations as shown in Table 3-1.

3.3.3 Batches 3 & 4: Capacitor Manufacture and Testing (Dearborn)

The Batch 3 &4 capacitor manufacturing and testing for Dearborn was split into three parts:

Part 1 - Electrical characterization of wound, cured, and end sprayed FPE sections

Part 2 - Lead attachment and housing possibilities for FPE capacitor assemblies

Part 3 - Manufacturing of the final product and electrical evaluation

The first round of testing focused on the electrical and thermal performance of the capacitor windings similar to the previous batches and followed the process shown in Fig. 3-8. Thirty sections, where a section is a capacitor prior to packaging, were wound from each material type. Conditioning, curing, end spray and electrical testing were performed after completion of winding.

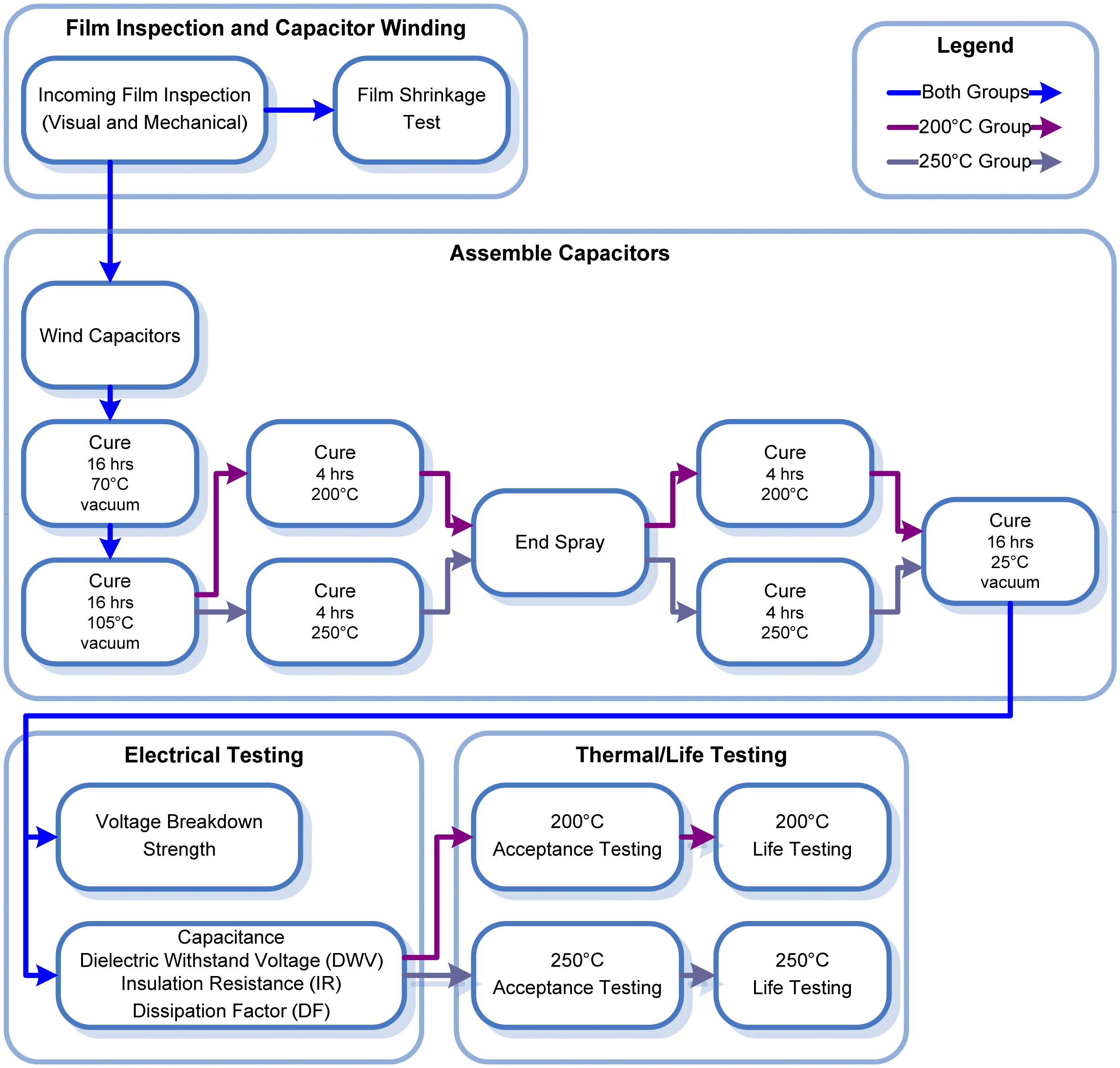


Fig. 3-8: Batch 3 & 4 Part 1 Dearborn Electronics Testing

Next, in Part 2, the investigation focused on the connection and assembly possibilities. For this investigation new FPE sections were wound following the winding instructions established during the winding of the sections prepared for Part 1. Several choices of leads / connectors, together with a several choices for the final housings were selected for this examination. There were a total of eight experiments performed, the procedures for which are shown in Fig. 3-9 to Fig. 3-16.

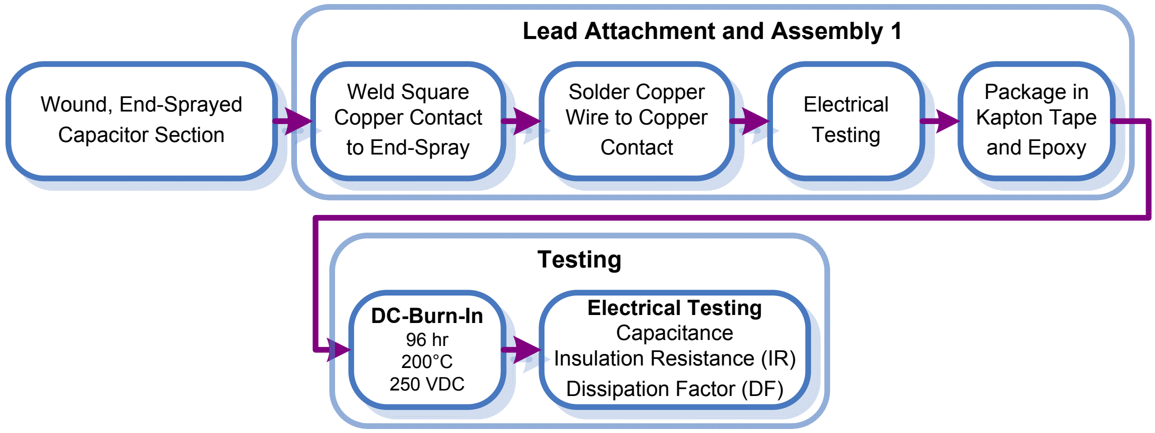


Fig. 3-9: Batch 3 & 4 Lead Attach and Assembly Experiment #1

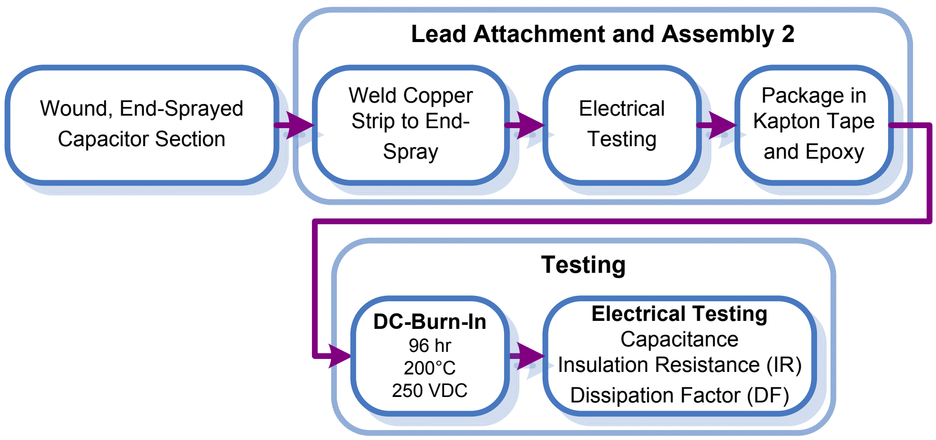


Fig. 3-10: Batch 3 & 4 Lead Attach and Assembly Experiment #2

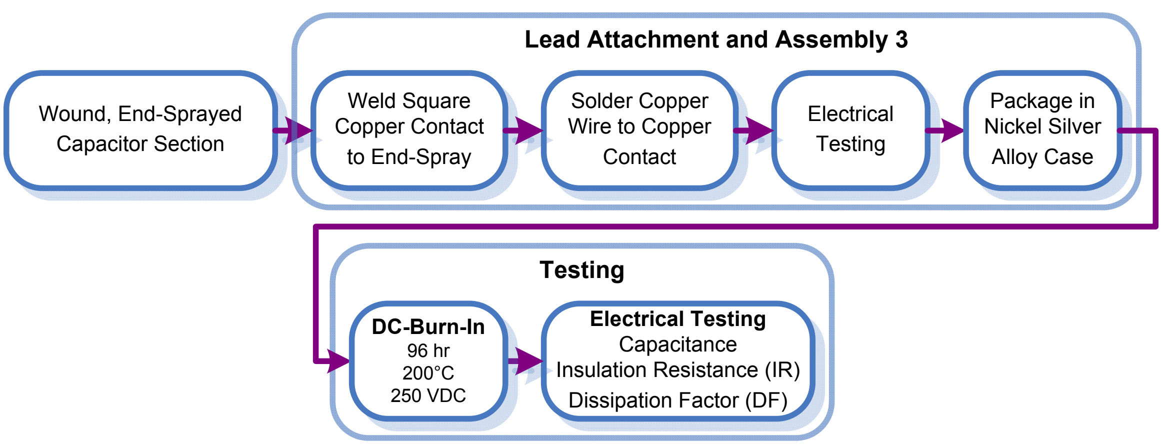


Fig. 3-11: Batch 3 & 4 Lead Attach and Assembly Experiment #3

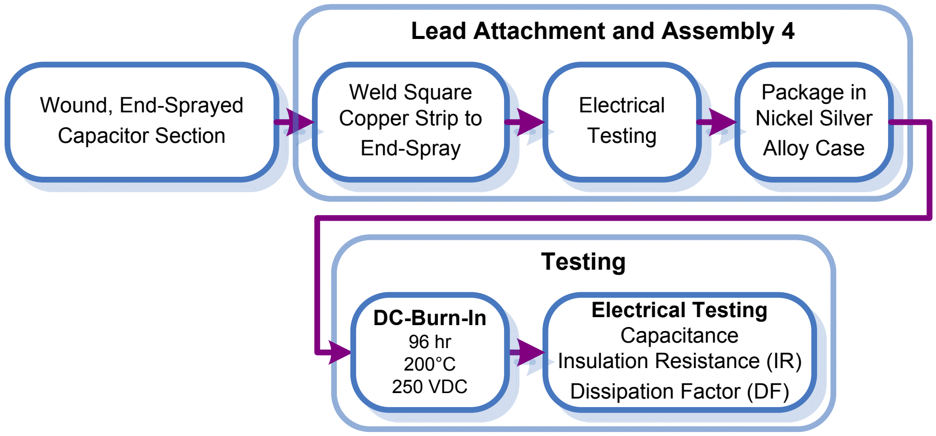


Fig. 3-12: Batch 3 & 4 Lead Attach and Assembly Experiment #4

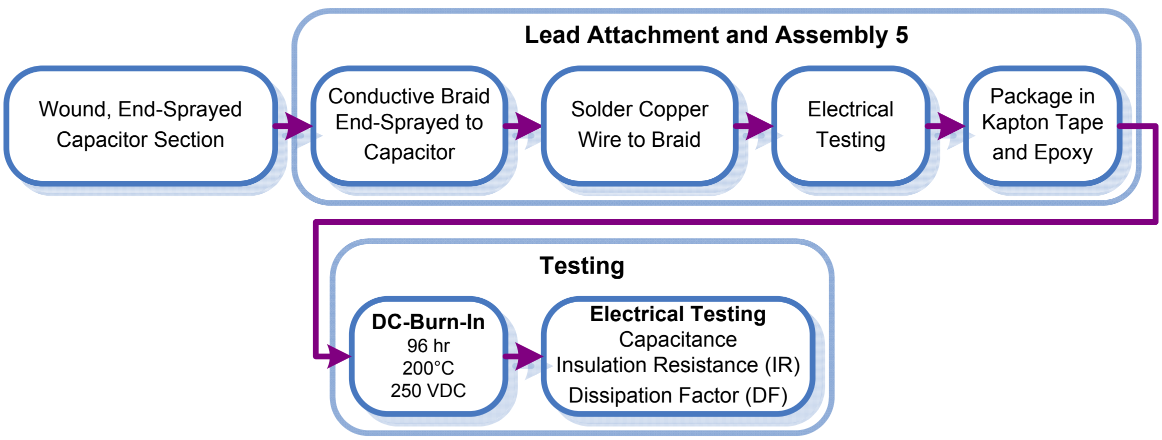


Fig. 3-13: Batch 3 & 4 Lead Attach and Assembly Experiment #5

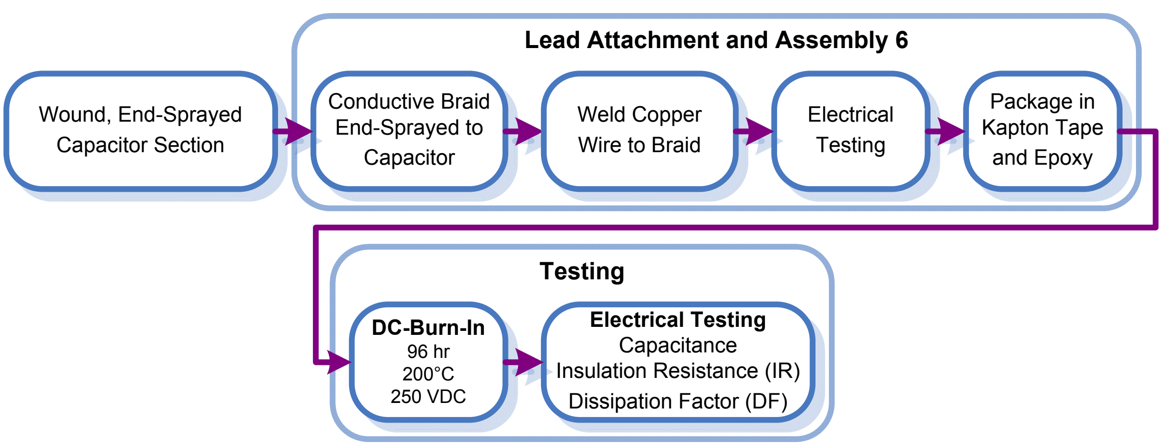


Fig. 3-14: Batch 3 & 4 Lead Attach and Assembly Experiment #6

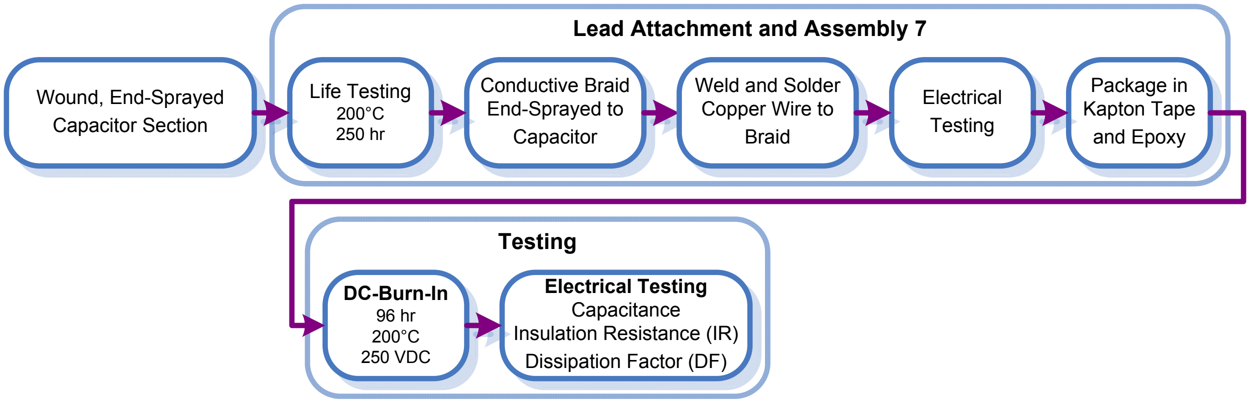


Fig. 3-15: Batch 3 & 4 Lead Attach and Assembly Experiment #7

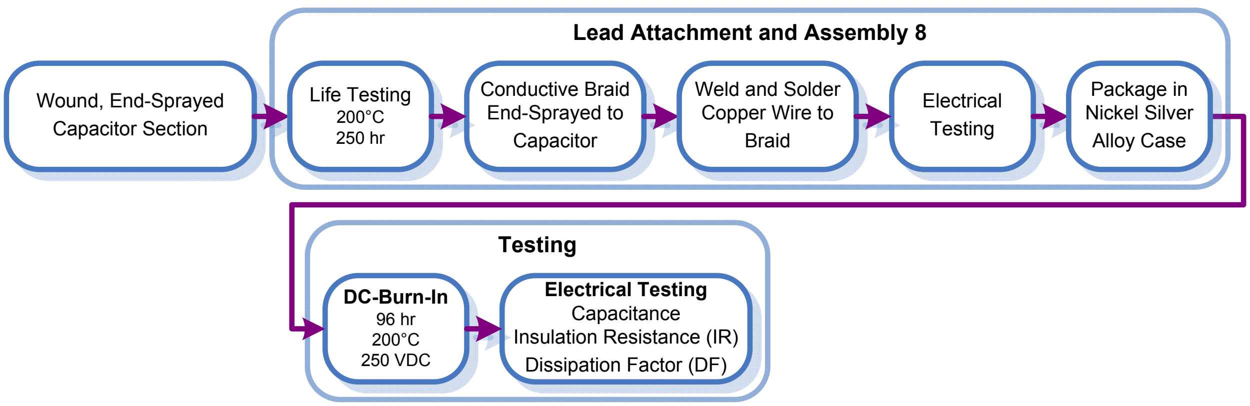


Fig. 3-16: Batch 3 & 4 Lead Attach and Assembly Experiment #8

Part 3 involved the manufacture of the FPE capacitors using the most efficient terminal attachment and packaging using the best selected housing. Electrical evaluation of the packaged FPE capacitors was included in this part.

4 Results and Discussion

In this section the results of the testing are presented. In each main section of the report the results of each batch are summarized, then additional details provided in the sub-sections.

4.1 Batch 1: Web Handling Experiments

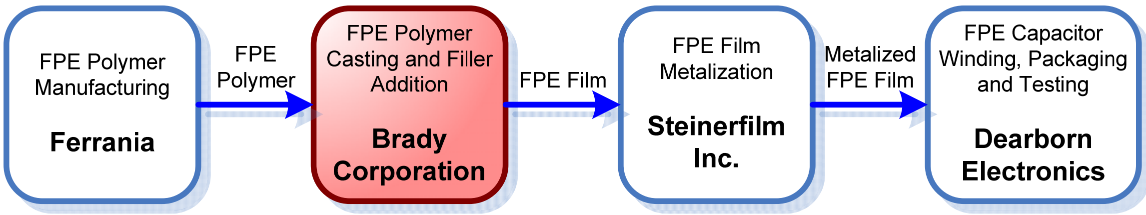


Fig. 4-1: Batch 1 Web Handling Experiments with Focus on Filler Addition

In Batch 1, six different FPE constructions were examined for web-handling and final capacitor assembly properties with focus on the polymer casting and filler formulation, highlighted in Fig. 4-1.

For Batch 1, Brady cast 30 lbs of FPE resin into twelve 2.5 lb. rolls, 5 µm thick with different fillers. Subsequently Steinerfilm metalized and evaluated the different FPE constructions. Finally, Dearborn Electronics manufactured 7µF capacitors from the FPE film. The capacitors with the highest Insulation Resistance (IR) and DWV were tested at 25°C, 200°C, and 250°C for electrical properties.

The film production process resulted in 6 of 12 rolls producing usable capacitors. Several rolls had film imperfections including extreme weave, wrinkles, scratches, and core extension beyond the edge. The winding yield, or number of testable capacitors after film winding as a percentage of all wound capacitors, was ~70% (185/268).

Of the capacitor film rolls, 2 of 12 batches had breakdown voltage of > 500 VDC. The electrical testing results from these two batches are shown in Table 4-1. All of the capacitor sections failed the acceptance and life tests due to separation of the end spray surfaces from the electrodes or extremely high DF, an example of which is shown in Fig. 4-2.

Table 4-1: Batch 1 Electrical Testing Results of Two Best Candidates

NETL/Batch 1 material	DWV @ 500 VDC	IR @ 250 VDC MΩ	Cap @ 1 kHz µF	DF @ 1 kHz %	Yield %
Y554259A	Pass	32,000	6.9	0.8	70
Y554261A	Pass	22,000	7.0	0.3	50

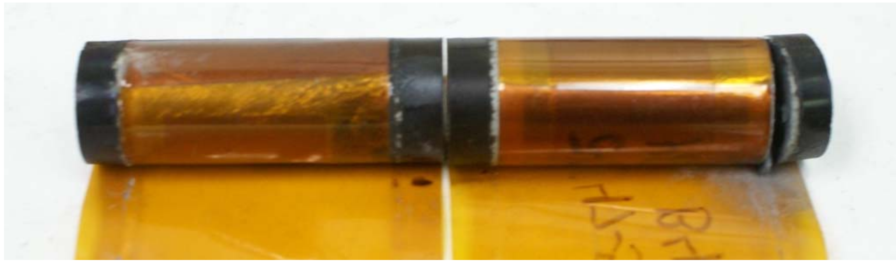


Fig. 4-2: Capacitors with Separated End Spray

It could be concluded that Batch 1 materials still possess poor qualities for manufacturability and reliability.

4.1.1 Batch 1: Film Casting (Brady Corporation)

Brady Corporation produced a total of twelve rolls of six different FPE formulations. A short summary of the FPE rolls is shown in Table 4-2. The “A” samples included a carrier backing in an attempt to make metalization easier where the “B” samples did not include the carrier for metalization. A 1 mil carrier backing was also tested, but failed during production at Brady.

Table 4-2: Batch 1 Brady Feedback

Batch Number	Slit Length (m)	Thickness (μm)	Breakdown Strength (V)
Y554257A	360	5.1	1627
Y554257B	360	5.1	1627
Y554258A	695	5.4	2051
Y554258B	695	5.4	2051
Y554259A	600	5.6	1911
Y554259B	600	5.6	1911
Y554260A	760	4.6	1221
Y554260B	760	4.6	1221
Y554261A	860	4.8	1326
Y554261B	860	4.8	1326
1 mil carrier failed at Brady			
1 mil carrier failed at Brady			

4.1.2 Batch 1: Metalization (Steinerfilm)

At Steinerfilm, the ten surviving material rolls were metalized. The materials were difficult to work with exhibiting many of the problems observed with FPE film prior to this program. Two batches Y554258B and Y554261B did not survive the metalization process.

4.1.3 Batch 1: Capacitor Manufacture and Testing (Dearborn Electronics)

The metalized FPE film was then sent to Dearborn Electronics for capacitor construction and testing. The film was found to have several imperfections including: extreme weave, wrinkles, scratches in the film, and core extension beyond the roll edge. The material did have correctly located blended heavy edges (H.E.) with clean, sharply defined margins. The poor condition of some material rolls limited Dearborn’s capability of winding required number of sections.

A total of 268 sections of Batch 1 were assembled as described in section 3.1.3. Of those, 83 were rejected during the winding process, and 185 were used for the testing (winding yield: 70%).

A material shrinkage test was performed. It was found that approximately 3% shrinkage occurred in both directions (MD- Machine Direction (down the length) and TD- Transverse Direction (across the film)). The shrinkage test consisted of measuring the length and width of samples from materials representative of both batches. The samples were measured in Machine Direction (MD) and Transverse Direction (TD) before and after heating. The temperatures used for the shrinkage tests were: 125°C, 150°C, 200°C, and 250°C. In Fig. 4-3 the test results for Batch 1 A group are shown. All tested FPE material experienced significant shrinkage in both directions, MD and TD.

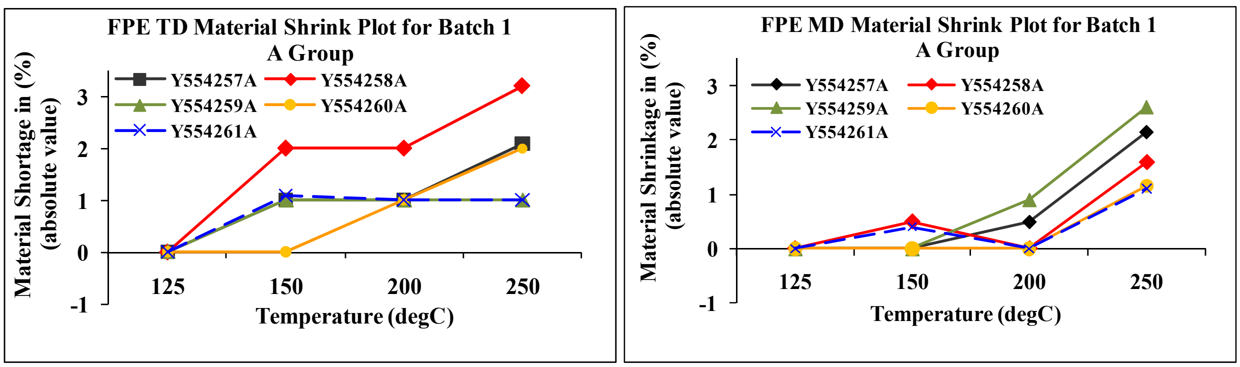


Fig. 4-3: FPE material shrinkage (TD-Transverse Direction & MD-Mechanical Direction) vs. temperature (Batch 1, A group)

Next, electrical testing was performed. Batch 1 had two FPE material candidates that passed the Dielectric Withstand Voltage (DWV) test at 500 VDC and the Insulation Resistance (IR) test at 250 VDC. These tests were performed at room temperature and the electrical data of the best two FPE materials from Batch 1 is shown in Table 4-3. In Table 4-3 the yield percentage denotes the number of capacitors that passed the DWV and IR tests relative to the total number tested.

Table 4-3: Batch 1 Best FPE Material Candidate Electrical Properties

Batch Number	DWV @ 500 VDC	IR @ 250 VDC (MΩ)	Cap @ 1 kHz (µF)	DF @ 1 kHz (%)	Yield (%)
Y554259A	Pass	32,000	6.9	0.8	70
Y554261A	Pass	22,000	7.0	0.3	50

The rest of material from Batch 1 resulted either in breakdown voltages in the range of 100-200 VDC or the breakdown voltage was not determined due to failure of the tested sections at very low voltage (≤100 VDC).

After the initial DWV and IR testing, acceptance and life testing was performed. The acceptance test was a 48 hour test during which some capacitors were subjected to 150°C temperatures and others to 200°C temperatures with 250 VDC applied. The life test is the same but the duration is increased to 250 hours. During these tests Batch 1 parts built from two the best material representatives (Y554259A and Y554261A) failed during the acceptance test mostly either due to separation between the end spray surfaces from the electrodes, shown in Fig. 4-2, or extremely high DF.

4.1.4 Batch 1: Summary

In summary, Batch 1 FPE capacitors did not perform as expected either in production, where stretching, tearing and wrinkling were problematic, or in the electrical and thermal characteristics where the electrically sound capacitors failed thermally.

4.2 Batch 2: Web Handling Optimization

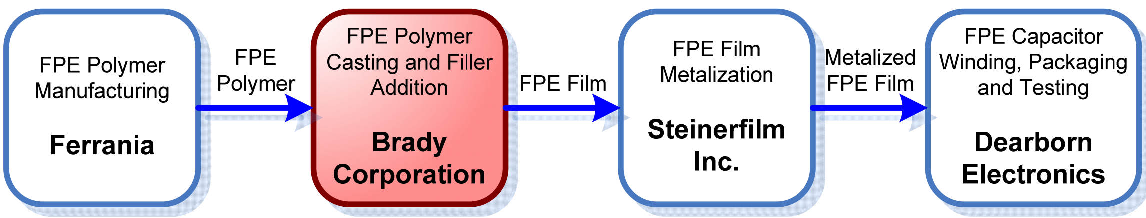


Fig. 4-4: Batch 2 Web Handling Experiments with Focus on Filler Optimization

In Batch 2, the casting process of FPE film on Brady Corporation's production casting equipment was optimized and the web-handling properties and the high temperature capacitor properties were examined.

For Batch 2, Brady cast 30 lbs of FPE resin into 6 5 lb. rolls, 5 µm thick with different fillers. Subsequently Steinerfilm metalized and evaluated the different FPE constructions. Finally, Dearborn Electronics manufactured 7µF capacitors from the FPE film. The capacitors with the highest Insulation Resistance (IR) and DWV were tested at 25°C, 200°C, and 250°C for electrical properties.

The film production process resulted in all 6 rolls producing usable capacitors. Film imperfections were minimized in this batch. The winding yield was ~80%. During burn-in of the capacitors a strong odor was present, but after further examination, the out-gassing components were within OSHA and EPA limits and more aggressive ventilation of the ovens allowed testing to continue.

All of the capacitor film rolls had breakdown voltage of >500 VDC and 5 batches had >1000 VDC breakdown. The electrical testing results from the best two batches are shown in Table 4-4. Results from the life and acceptance tests are shown in Table 4-5 and Table 4-6 respectively.

Table 4-4: Batch 2 Electrical Testing Results of Two Best Candidates

NETL/Batch 2 material	DWV @ 500 VDC	IR @ 250 VDC (MΩ)	Cap @ 1 kHz (µF)	DF @ 1 kHz (%)	Yield (%)
163457-A	Pass	18,000	7.5	1.3	70
171142	Pass	2,800	7.6	1.0	80

Table 4-5: Batch 2 Acceptance Test (48 hrs @ 200°C) Results of Two Best Candidates

NETL/Batch 2 material	IR @ 250 VDC (MΩ)	Cap @ 1 kHz (µF)	DF @ 1 kHz (%)	Yield (%)
163457-A	47,000	7.7	1.0	60
171142	26,000	7.7	1.0	80

Table 4-6: Batch 2 Life Test (250 hrs @ 200°C) Results of Two Best Candidates

NETL/Batch 2 material	IR @ 250 VDC (MΩ)	Cap @ 1 kHz (µF)	DF @ 1 kHz (%)	Yield (%)
163457-A	20,000	7.9	2.6	100
171142	10,000	8.4	1.1	70

Batch 2 materials are substantially better than batch 1 materials with regard to manufacturability, reliability and electrical performance though they still had high failure rate at 250°C. A 200°C limit for this material is more practical.

4.2.1 Batch 2: Film Casting (Brady Corporation)

Additional funding was available from the Air Force Research Labs (AFRL) for FPE capacitor development and the team took advantage of this funding to improve the film casting process. After the AFRL film optimization study, Brady cast the DOE NETL Batch 2 FPE film with six different formulations summarized in Table 4-7.

Table 4-7: Batch 2 Brady Feedback

Batch Number	Slit Length (m)	Thickness (µm)	Breakdown Strength (V)
163231A	800	4.6	1252
163457A	500	4.7	1217
163470A	500	4.5	1304
171142	666	4.1	
163480A	500	3.1	1201
171143	1025	4.1	

4.2.2 Batch 2: Metalization (Steinerfilm)

For Batch 2 production, Steinerfilm was able to move the metalization process to production metalization lines from the demo lines. The improved web handling characteristics of the Batch 2 material made it much easier to work with.

4.2.3 Batch 2: Capacitor Manufacture and Testing (Dearborn Electronics)

4.2.3.1 Capacitor Winding and Shrinkage

Batch 2 resulted in better winding yield, ~80%, where out of 238 total wound capacitors, 42 were rejected for poor winding, and the 196 good windings were used for further testing.

The material shrinkage test was repeated for Batch 2 with similar results as those from Batch 1. It was found that approximately 3% shrinkage occurred in both directions (MD- Machine Direction (down the length) and TD- Transverse Direction (across the film)).

4.2.3.2 Capacitor Electrical Testing

Electrically Batch 2 performed better than Batch 1. The Dielectric Withstand Voltage (DWV) test at 500 VDC and the Insulation Resistance (IR) test at 250 VDC were performed on four out of six tested FPE materials from Batch 2. The other two Batch 2 materials were tested for DWV at 250 VDC and IR at 125 VDC. The electrical data of the best four FPE materials from Batch 2 is shown in Table 4-8.

Table 4-8: Batch 1 Best FPE Material Candidate Electrical Properties

Batch Number	DWV @ 500 VDC	IR @ 250 VDC (MΩ)	Capacitance @ 1 kHz (µF)	DF @ 1 kHz (%)	Yield (%)
163231A	Pass	34,000	7.6	1.0	60
163457A	Pass	18,000	7.5	1.3	70
163470A	Pass	21,000	7.4	1.1	60
171142	Pass	2,800	7.6	1.0	80

4.2.3.3 Capacitor Thermal Testing

After the initial DWV and IR testing, acceptance and life testing was performed. The acceptance test was a 48 hour test during which some capacitors were subjected to 150°C temperatures and others to 200°C temperatures with 250 VDC applied. The life test is the same but the duration is increased to 250 hours. The Batch 2 capacitors performed significantly better than those from Batch 1. Out of six FPE materials tested for 48hr, all passed through the temperature point of 150°C, but three failed the test at 200°C temperature because of very low IR. Two material representatives from Batch 2 passed through the 250hr Life Test at 150°C and the 250hr Life Test at 200°C. All data related to these two materials 48hr and 250hr Life Tests are shown in Table 4-9, Table 4-10, Table 4-11, and Table 4-12.

Table 4-9: Batch 2 Acceptance Test Results (48 hours @ 150°C)

Batch Number	IR @ 250 VDC (MΩ)	Capacitance @ 1 kHz (µF)	DF @ 1 kHz (%)	Yield (%)
163457A	47,000	7.7	1.0	60
171142	26,000	7.7	1.0	80

Table 4-10: Batch 2 Life Test Results (250 hours @ 150°C)

Batch Number	IR @ 250 VDC (MΩ)	Capacitance @ 1 kHz (µF)	DF @ 1 kHz (%)	Yield (%)
163457A	13,000	7.9	1.0	70
171142	16,000	7.6	1.0	100

Table 4-11: Batch 2 Acceptance Test Results (48 hours @ 200°C)

Batch Number	IR @ 250 VDC (MΩ)	Capacitance @ 1 kHz (µF)	DF @ 1 kHz (%)	Yield (%)
163457A	17,000	7.8	1.6	40
171142	38,000	8.0	1.0	70

Table 4-12: Batch 2 Life Test Results (250 hours @ 200°C)

Batch Number	IR @ 250 VDC (MΩ)	Capacitance @ 1 kHz (µF)	DF @ 1 kHz (%)	Yield (%)
163457A	20,000	7.9	2.6	100
171142	10,000	8.4	1.1	70

Two candidates from Batch 2 demonstrated the most promising improvement in the FPE dielectric. Performance at elevated temperature was stable and consistent for all tested parts from the 163457A and 171142 material groups.

4.2.4 Batch 2: Summary

In general, there was a tremendous improvement in the breakdown strength of the FPE film in Batch 2 compared to Batch 1. Based on this data it can be concluded that the FPE capacitors can reach a maximum voltage of 1000 VDC with a Dielectric Withstanding Voltage of 500 VDC.

These two material lots from Batch 2 met the goal of capacitor manufacturability, consistent performance after an extended period of temperature under load, and yields of greater than fifty percent.

Table 4-13: Batch 2 Yields Results

Winding Yield	80%

Best Batches	163457A	1711142
DWV and IR Testing Yield	70%	80%
150°C Acceptance Test Yield	60%	80%
150°C Life Test Yield	70%	100%
200°C Acceptance Test Yield	40%	70%
200°C Life Test Yield	100%	70%

4.3 Batch 3 & 4: Metalization and Capacitor Manufacturing Optimization

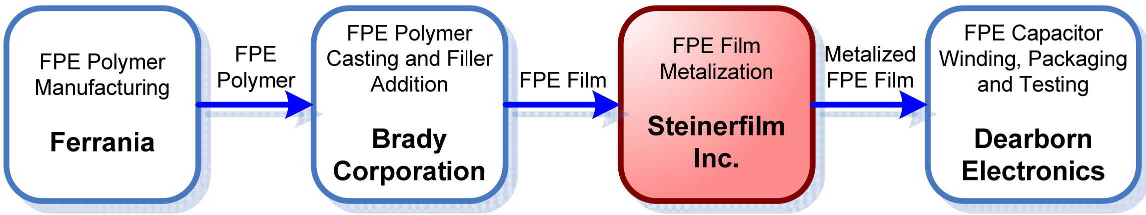


Fig. 4-5: Batch 3 Metalization Optimization

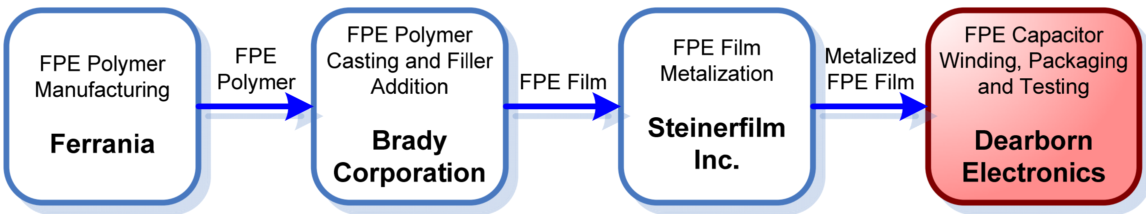


Fig. 4-6: Batch 4 Capacitor Manufacturing Optimization

Due to delays with Batches 1 & 2, which enabled additional learning during those batches, Batches 3 & 4 were combined in such a way that enabled both metalization and capacitor manufacturing optimization, highlighted in Fig. 4-5 and Fig. 4-6. The experiments were designed as shown in Table 3-1.

For Batch 3 & 4, Brady cast 60 lbs of FPE resin into 12 5 lb. rolls, 5 µm thick with 5 variations of fillers. Subsequently, Steinerfilm metalized and evaluated the different FPE constructions varying the acrylate and metalization thicknesses. Finally, Dearborn Electronics manufactured 7 µF capacitors from the FPE film. The capacitors with the highest Insulation Resistance (IR) and DWV were tested at 25°C, 200°C, and 250°C for electrical properties.

The film production process resulted in all 12 rolls producing usable capacitors. Film imperfections were minimized in this batch.

All of the capacitor film rolls had breakdown voltage of >500 VDC. The electrical testing results from the best two batches are shown in Table 4-14. Results from the life and acceptance tests are shown in Table 4-15 and Table 4-16 respectively.

Table 4-14: Batch 3 & 4 Electrical Testing Results of Two Best Candidates

NETL/Batch 2 material	DWV @ 500 VDC	IR @ 250 VDC (MΩ)	Cap @ 1 kHz (µF)	DF @ 1 kHz (%)	Yield (%)
37	pass	3,600	7.6	0.8	85
43	pass	370	7.4	0.7	80

Table 4-15: Batch 3 & 4 Acceptance Test (48 hrs @ 200°C) Results of Two Best Candidates

NETL/Batch 2 material	IR @ 250 VDC (MΩ)	Cap @ 1 kHz (µF)	DF @ 1 kHz (%)	Yield (%)
37	11,000	7.6	1.0	100
43	11,000	7.6	0.8	100

Table 4-16: Batch 3 & 4 Life Test (250 hrs @ 200°C) Results of Two Best Candidates

NETL/Batch 2 material	IR @ 250 VDC (MΩ)	Cap @ 1 kHz (µF)	DF @ 1 kHz (%)	Yield (%)
37	19,200	7.8	1.2	100
43	18,800	8.0	1.0	100

Batch 3 & 4 materials have best manufacturability characteristics of all batches produced. The Batch 3 & 4 materials had good electrical performance at 200°C, but a high failure rate at 250°C electrical testing. A 200°C limit for this material is more practical for this FPE film formulation.

Batch 3 & 4 development also focused on high temperature capacitor packaging. Several experiments were performed to test end connection and housing options. The best packaging technique to date was determined to include a zinc end-spray on the capacitor film. The film is then heated for 250 hours at 200°C before attaching a solder braid using the end-spray process. Finally a lead wire or copper strip is attached to this braid and the capacitor is packaged in either a kapton or Nickel alloy casing and filled with high temperature epoxy. This technique still has a relatively high failure rate (40% after initial 250 hr. curing), and other techniques are still under investigation as a follow-on efforts.

4.3.1 Batch 3 & 4: Film Casting (Brady Corporation)

Brady cast the DOE NETL Batch 3 and 4 FPE film with twelve rolls of five different formulations. A short summary of the FPE rolls is shown in Table 4-17.

Table 4-17: Batch 3 & 4 Brady Feedback

NETL Sample	Batch Number	Slit Length (m)	Thickness (µm)	Breakdown Strength (V)	Coefficient of Friction (COF)	Tensile Strength (psi)	Elongation (%)
33	201568-A	500	4.5	1616	0.545	9367	6
34	201569-A	400	4.6	1361	0.549	5631	2
35	201570-A	500	4.4	1332	0.497	12922	5
36	201571-A	502	4.3	1280	0.532	10068	3
37	201574-A	515	4.4	1358	0.522	10738	3
38	201576-A	500	4.6	1515	0.538	8510	3
39	201578-A	505	4.2	1677	0.557	7805	3
40	201584-A	500	4.5	1327	0.678	21176	5
41	201586-A	500	4.4	1594	0.642	12114	4
42	201581-A	514	4.7	1401	0.474	11543	4
43	201605-A	1515	4.8	1175	0.560	8066	4
44	201621-A	809	4.0	1282	0.678	17983	4

4.3.2 Batch 3 & 4: Metalization (Steinerfilm)

For Batch 3 & 4 production, Steinerfilm used production metalization equipment to metalize the FPE films as described in Table 3-1. The film was much easier to work with when compared to previous FPE constructions, especially FPE films without fillers.

4.3.3 Batch 3 & 4: Capacitor Manufacture and Testing (Dearborn)

After metalization the twelve FPE material samples were investigated by Dearborn Electronics. The testing consisted of three parts:

Part 1 - Electrical characterization of wound, cured, and end sprayed FPE sections

Part 2 - Lead attachment and housing possibilities for FPE capacitor assemblies

Part 3 - Manufacturing of the final product and electrical evaluation

Part 1 focused on the electrical and thermal performance of the capacitor windings. Half of the windings were cured at +200°C and the other half at +250°C. Thirty sections, where a section is a capacitor prior to packaging, were wound from each material type. Conditioning, curing, end spray and electrical testing were performed after completion of winding.

4.3.3.1 Part 1 - Electrical characterization of wound, cured, and end sprayed FPE sections

4.3.3.1.1 Voltage Breakdown Strength in Section Form

Three units from each FPE material group and each temperature group were used in voltage breakdown testing. This test was performed on a Dearborn proprietary voltage bridge. Voltage was increased in 10 VDC increments until a catastrophic failure occurred. A dead short that occurs during the test is considered to be a catastrophic failure. Not all FPE materials responded the same way to this test. The summary of voltage breakdown test for all tested FPE materials from Batch 3 is shown in Fig. 4-7 and Batch 4 in Fig. 4-8.

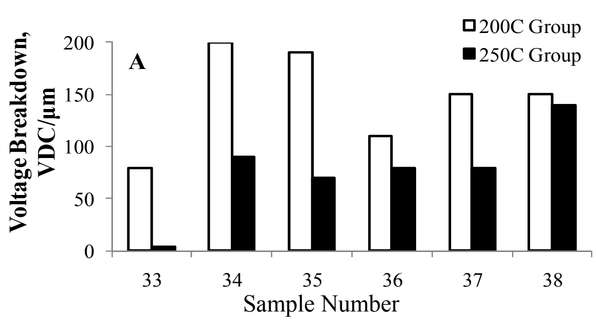


Fig. 4-7: Batch 3 FPE Film Voltage Breakdown in VDC/µm

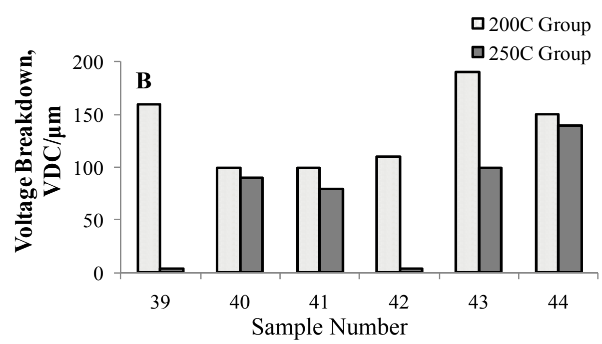


Fig. 4-8: Batch 4 FPE Film Voltage Breakdown in VDC/µm

Most of the FPE materials cured at +200°C and +250°C reached a maximum voltage of 500 VDC total and 400 VDC respectively, which corresponds to voltage breakdown strengths of 100 V/µm and 80 V/µm.

Some batches had voltage breakdown strength of 1000 VDC, which confirmed success in FPE material development. For instance, NETL sample #34 had a voltage breakdown of 870 VDC or 174 V/µm when tested in sheet level. The sections wound from the same material, baked for four hours in air circulating oven at +200°C and +250°C, had voltage breakdown of 1000 VDC or 200 V/µm and 450 VDC or 90 V/µm, respectively.

In the case of the NETL sample #43, sheet level voltage breakdown reached was 963 VDC or 190 V/µm. The sections wound from the same material, baked for four hours in air circulating oven at +200°C and +250°C, had voltage breakdown of 950 VDC or 190 V/µm and 500 VDC or 100 V/µm, respectively.

From the examples presented, high temp (250°C) degrades the FPE capacitor electrical performance. The majority of the FPE materials, cured at this temperature, did not perform well resulting in very low voltage breakdown, and the sections cured at this temperature could not be electrical characterized.

4.3.3.1.2 Volt Testing, Insulation Resistance, Capacitance, Dissipation Factor

After voltage breakdown testing to failure, the data was used to establish voltages for two tests, DWV and IR. Capacitors are tested for hi-pot or Dielectric Withstand Voltage (DWV) and Insulation Resistance (IR) at rated voltage. In general, the DWV voltage is much less, up to 50% lower, than the breakdown voltage and the DWV much higher, usually 2x, than the rated or Insulation Resistance test voltage. In addition, FPE batches showed very good results for DWV, IR, Cap, and DF.

These calculated voltage values were used for all further electrical testing of each batch. Table 4-18 and Table 4-19 show initial test readings for each FPE material and the cure temperature for each.

Table 4-18: Batch 3 & 4 Initial Electrical Ratings of Each FPE Material cured at +200°C

	NETL Sample Number	DWV @ 400 VDC	IR @ 250 VDC (MΩ)	Cap at 1 kHz (µF)	DF at 1 kHz
Batch 3	33	short	N/A	N/A	N/A
	34	pass	3,890	7.5	0.6
	35	pass	3,530	7.5	0.9
	36	short	N/A	N/A	N/A
	37	pass	3,600	7.6	0.8
	38	pass	1,500	7.3	0.5
Batch 4	39	pass	1,400	7.5	0.6
	40	pass	35	7.5	0.4
	41	pass	25	7.4	0.4
	42	pass	25	7.3	0.3
	43	pass	370	7.4	0.7
	44	pass	135	7.4	0.9

Table 4-19: Batch 3 & 4 Initial Electrical Ratings of Each FPE Material cured at +250°C

	NETL Sample Number	DWV @ 400 VDC	IR @ 250 VDC (MΩ)	Cap at 1 kHz (μF)	DF at 1 kHz
Batch 3	33	short	N/A	N/A	N/A
	34	short	N/A	N/A	N/A
	35	short	N/A	N/A	N/A
	36	short	N/A	N/A	N/A
	37	pass	3,600	7.6	0.8
	38	pass	55	7.3	0.5
Batch 4	39	short	N/A	N/A	N/A
	40	short	N/A	N/A	N/A
	41	short	N/A	N/A	N/A
	42	short	N/A	N/A	N/A
	43	pass	215	7.4	0.7
	44	pass	85	7.4	0.9

Using the number of the capacitors tested and the number of capacitors that passed the initial testing the yield of each FPE material was determined; see Table 4-20 and Fig. 4-9. Capacitors were defined as "passed" if they withstood the DWV voltage, IR voltage, and had Cap, and DF values within 10% of the typical values.

The best initialized FPE materials cured at +200°C, were Sample #34 which had a DWV of 400 VDC, typical Insulation Resistance value of 3,890 MΩ when tested at 250 VDC, typical Cap value of 7.5 μF at 1 kHz, typical DF value of 0.6% at 1 kHz, and a yield of 50% and Sample # 37 that had a typical Insulation Resistance value of 3,600 MΩ at 250 VDC, typical Cap value of 7.6 μF at 1 kHz, typical DF value of 0.8% at 1 kHz, and a yield of 85%.

Whereas the best initialized FPE material cured at +250°C, was Sample #37 that had a DWV of 400 VDC, a typical Insulation Resistance value of 3,600 MΩ when tested at 250 VDC, typical Cap value of 7.6 μF at 1 kHz, typical DF value of 0.8% at 1 kHz, but it resulted in very poor yield of 7%.

Capacitors were defined as "passed" if they withstood the DWV voltage, IR voltage, and had Cap, and DF values within 10% of the typical values.

Table 4-20: Batch 3 & 4 FPE Film Yield

		+200°C Group		+250°C Group	
	NETL Sample Number	# Units Passed / # Units Tested	Yield (%)	# Units Passed / # Units Tested	Yield (%)
Batch 3	33	0 / 14	0	0 / 15	0
	34	7 / 14	50	0 / 13	0
	35	9 / 9	100	0 / 15	0
	36	0 / 23	0	0 / 22	0
	37	22 / 25	85	2 / 27	7
	38	5 / 21	20	7 / 19	35
Batch 4	39	6 / 17	35	0 / 13	0
	40	4 / 11	36	0 / 12	0
	41	7 / 16	45	0 / 16	0
	42	5 / 16	30	0 / 14	0
	43	17 / 21	80	17 / 20	85
	44	11 / 14	70	9 / 14	60

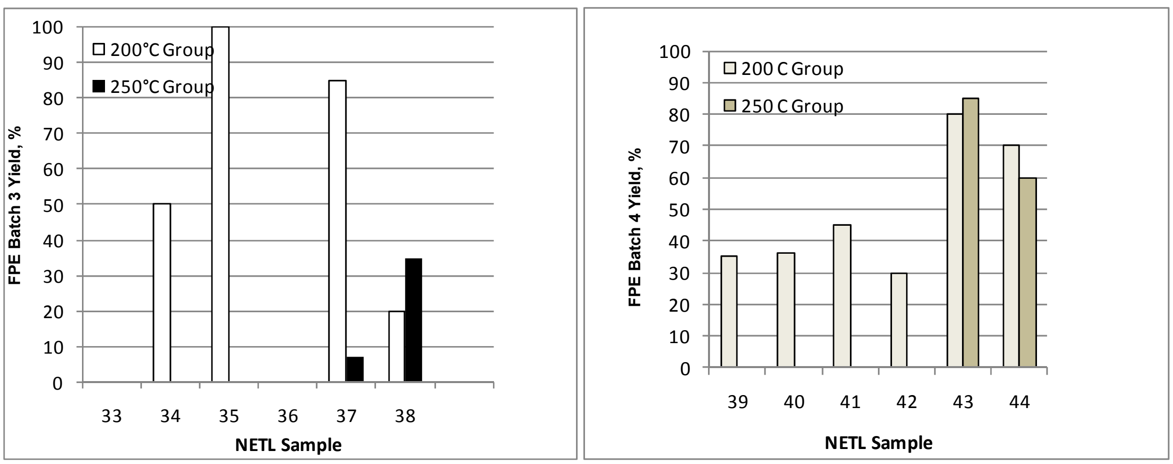


Fig. 4-9: Batch 3 & 4 FPE Film Yield

As it can be seen from Table 3 and Fig 2, FPE / NETL sample #33, #34 #36, #38,# 39, #40, and #42 resulted in very low yields, due to the poor electrical performance during initializing of the samples cured at +200°C. In addition, FPE / NETL sample #33 through #37 from Batch 3 and #39 through #42 from Batch 4 cured at +250°C resulted in low or “zero” yield.

4.3.3.1.3 High Temperature Characterization

High temperature characterization of FPE capacitors followed initialization of the units. The capacitors that remained good electrically after initial testing were placed in a temperature chamber matching their curing temperature. After reaching the test temperature of +200°C for one group and+250°C for other group, the FPE capacitors were maintained at that temperature for a period of 60 minutes to allow temperature stabilization of the test chamber and the capacitors before the test voltage of 250 VDC was applied.

Sections were fully charged for 3 minutes and IR at elevated temperature was measured and recorded. With completion of the IR testing, the FPE capacitors were allowed to cool to room temperature. The 25°C IR was measured and recorded followed by Cap and DF test at frequency of 1 kHz. All collected data was compared to the initial readings and is presented in Fig. 4-10 below.

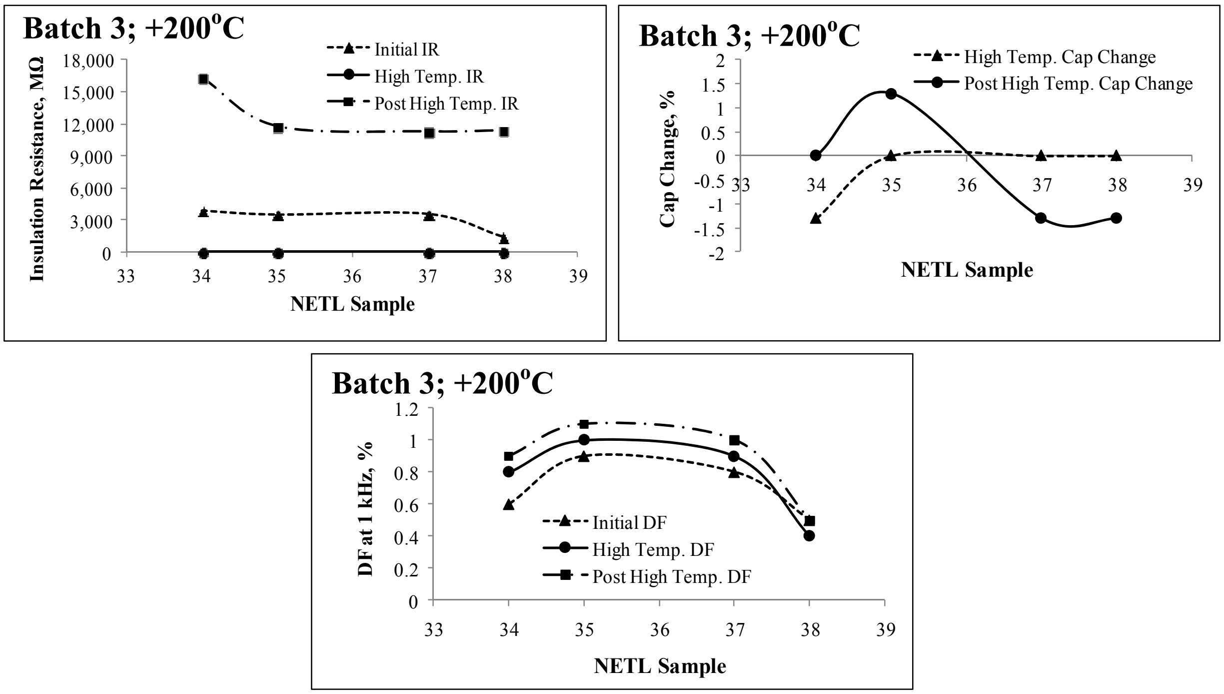


Fig. 4-10: Batch 3 FPE Film IR (top left), Cap change (top right), and DF (bottom) Measurements of FPE Batch 3 Materials cured at +200°C Taken at Elevated Temperature and at Room Temperature After High Temperature Readings, and Compared to the Initial values

As it can be seen from the graphs, there was a significant improvement in electrical performance of the Batch 3 FPE materials cured at +200°C relative to the initial performance

Insulation Resistance data shows very low values as the FPE sections were tested at the elevated temperature, but as the capacitors were brought back to room temperature and tested, the IR values increased by tens of thousands of Mega Ohms.

In addition, capacitance changed very little as a result of the test cycle, and the DF measured was close to the initial values.

From the data collected and presented here it may be concluded that the temperature of +200°C allowed the FPE capacitor to self heal to the extent that the electrical characteristics improved.

However, there were no survivors from the Batch 3 FPE capacitor group cured at +250°C, and no data was collected from those units.

Similar behavior occurred with Batch 4 FPE sections exposed to the same testing. The data collected is presented in Fig. 4-11.

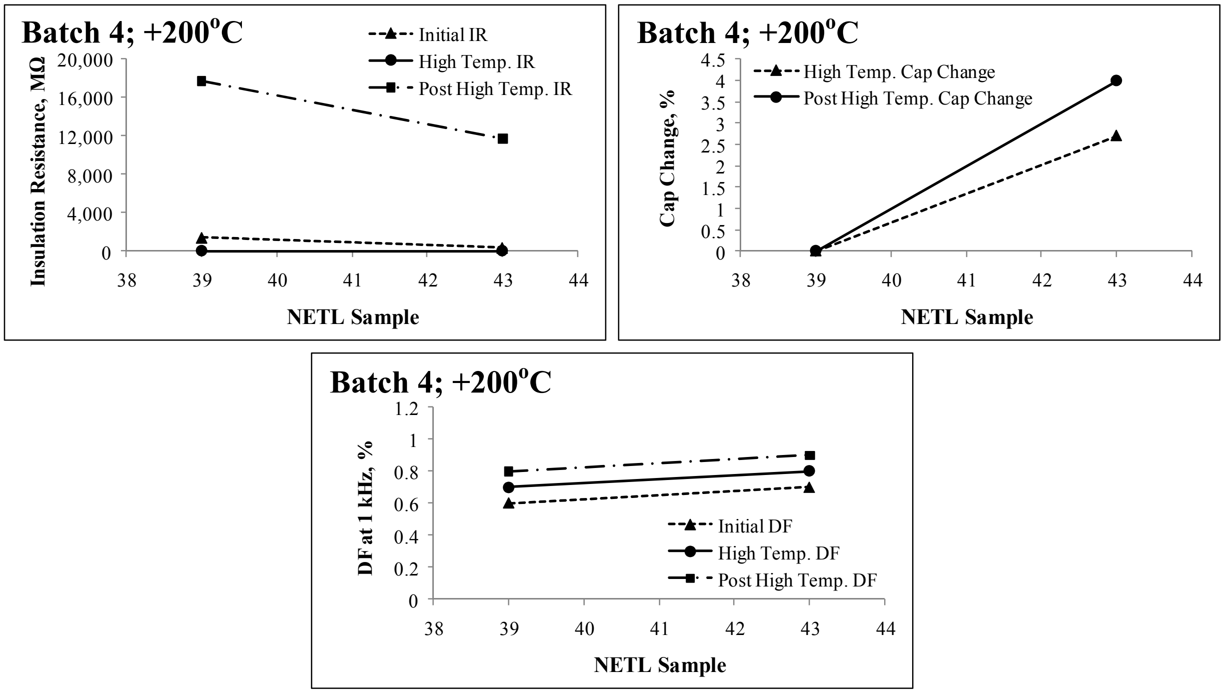


Fig. 4-11: Batch 4 FPE Film IR (top left), Cap change (top right), and DF (bottom) Measurements of FPE Batch 4 Materials cured at +200°C Taken at Elevated Temperature and at Room Temperature After High Temperature Readings, and Compared to the Initial values

4.3.3.1.4 Acceptance Test & Life Test

The 48 hours Acceptance Testing was performed on the FPE sections to cull out early failures prior to the 250 hours Life Test. All FPE unpackaged sections that survived the high temperature test were assigned to these two tests.

The 48 hours Acceptance Test was repeated at the elevated temperature and the voltage was maintained on the capacitors, at the elevated test temperature, for a period of 48 hours.

After completion of 48 hours at +200°C and 250 VDC the capacitors were cooled down to room temperature for room temperature readings. The capacitors were tested for IR, Cap, and DF and the data was recorded.

As the next and final step the FPE sections were returned to the elevated test temperature (+200°C) and the test voltage (250 VDC) for the completion of the 250 hours Life Test. Once again, the sections were brought back to room temperature for measurement. The data was compared to the initial readings for final characterizing of the FPE unpackaged capacitors.

Using the number of the capacitors tested and the number of capacitors that passed acceptance and life testing, the yield of each FPE material was determined. The following tables show the yield results.

Table 4-21: Batch 3 & 4 Acceptance Test (48 hr) Yield

		+200°C Group		+250°C Group	
	NETL Sample Number	# Units Passed / # Units Tested	Yield (%)	# Units Passed / # Units Tested	Yield (%)
Batch 3	33	n/a	n/a	n/a	n/a
	34	7 / 7	100	n/a	n/a
	35	4 / 5	80	n/a	n/a
	36	n/a	n/a	n/a	n/a
	37	17 / 17	100	n/a	n/a
	38	4 / 4	100	n/a	n/a
Batch 4	39	4 / 6	67	n/a	n/a
	40	n/a	n/a	n/a	n/a
	41	n/a	n/a	n/a	n/a
	42	n/a	n/a	n/a	n/a
	43	11 / 11	100	n/a	n/a
	44	n/a	n/a	n/a	n/a

Table 4-22: Batch 3 & 4 Life Test (250 hr) Yield

		+200°C Group		+250°C Group	
	NETL Sample Number	# Units Passed / #Units Tested	Yield (%)	# Units Passed / # Units Tested	Yield (%)
Batch 3	33	n/a	n/a	n/a	n/a
	34	5 / 7	71	n/a	n/a
	35	3 / 4	75	n/a	n/a
	36	n/a	n/a	n/a	n/a
	37	17 / 17	100	n/a	n/a
	38	4 / 4	100	n/a	n/a
Batch 4	39	4 / 4	100	n/a	n/a
	40	n/a	n/a	n/a	n/a
	41	n/a	n/a	n/a	n/a
	42	n/a	n/a	n/a	n/a
	43	11 / 11	100	n/a	n/a
	44	n/a	n/a	n/a	n/a

The following figures illustrate the Acceptance Test and Life Test data for FPE materials from Batch 3 & 4 cured at +200°C. No data was collected from the sections cured at +250°C due to poor performance of the FPE materials at that temperature.

Fig. 4-12 and Fig. 4-13 show the data collected after 48 hours Acceptance Test for Batch 3 & 4 and Fig. 4-14 and Fig. 4-15 show the data collected after 250 hours Life Test, respectfully.

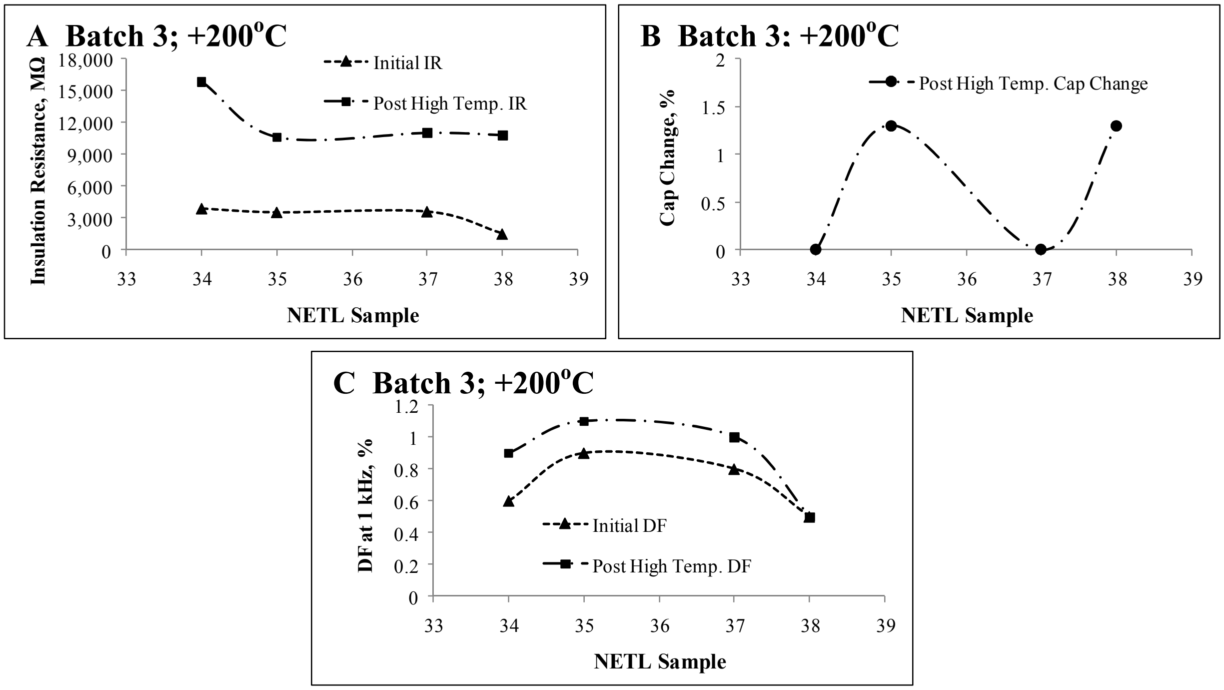


Fig. 4-12: Batch 3 IR (A), Cap change (B), and DF (C) Measurements Taken at Room Temperature After +200°C Curing for 48 hr (Acceptance Test), and Compared to the Initial Values.

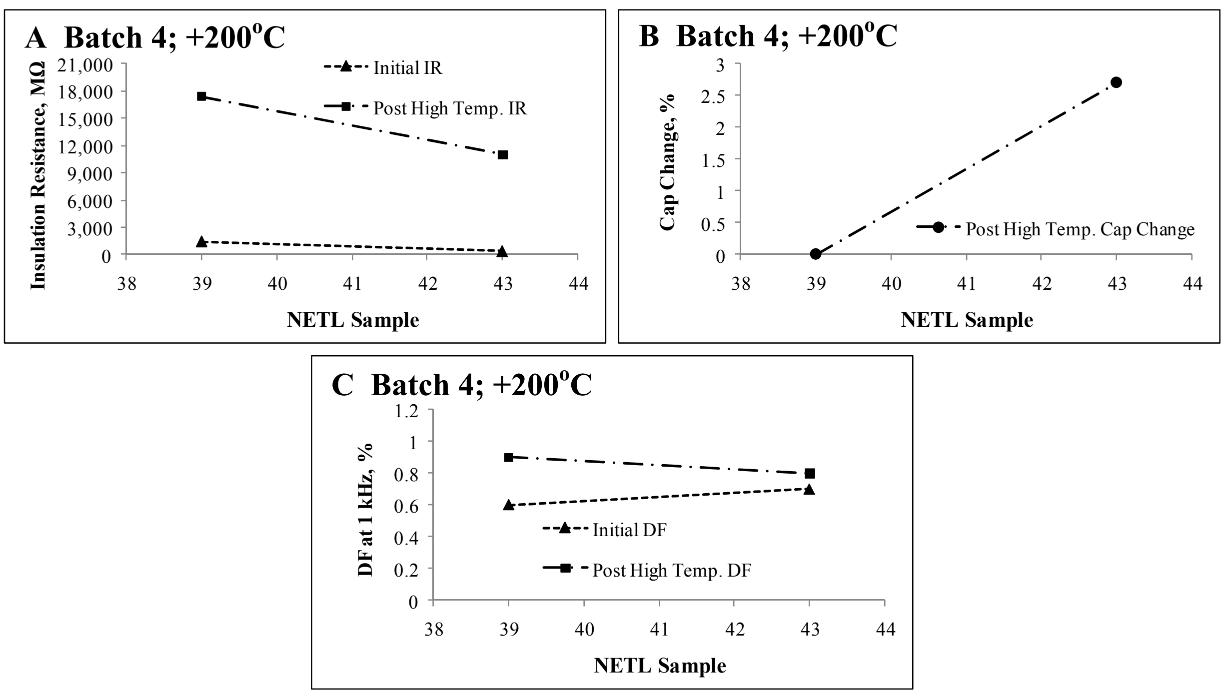


Fig. 4-13: Batch 4 IR (A), Cap change (B), and DF (C) Measurements Taken at Room Temperature After +200°C Curing for 48 hr (Acceptance Test), and Compared to the Initial Values.

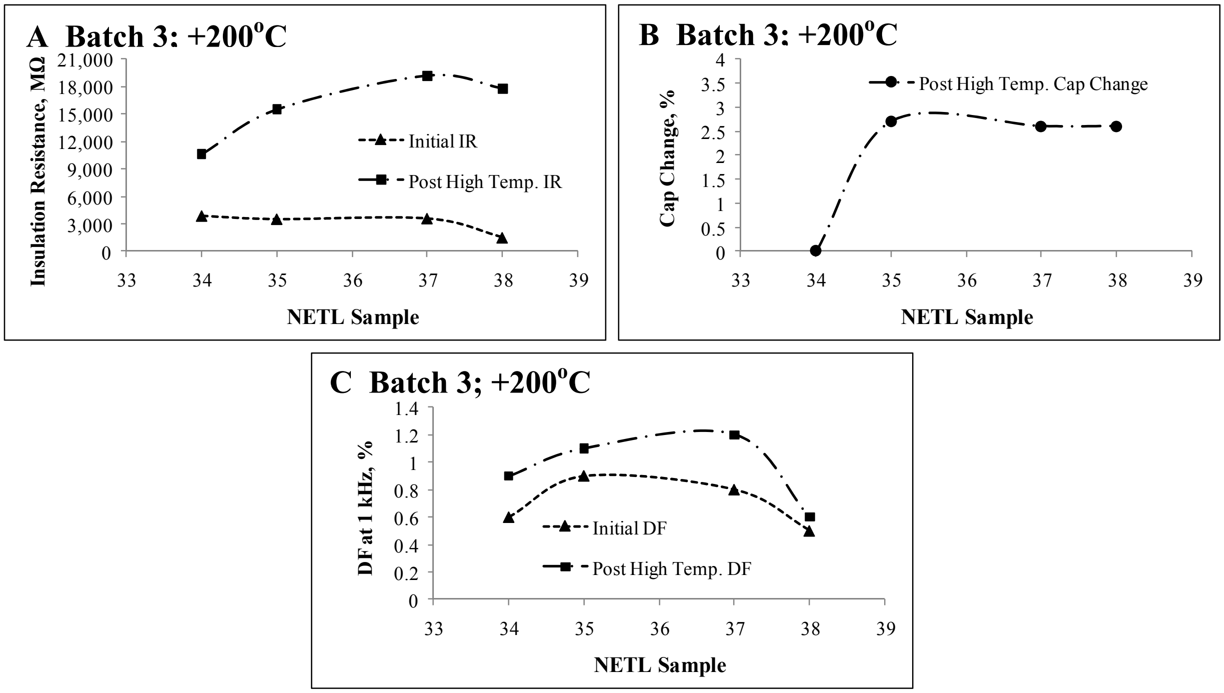


Fig. 4-14: Batch 3 IR (A), Cap change (B), and DF (C) Measurements Taken at Room Temperature After +200°C Curing for 250 hr (Life Test), and Compared to the Initial Values.

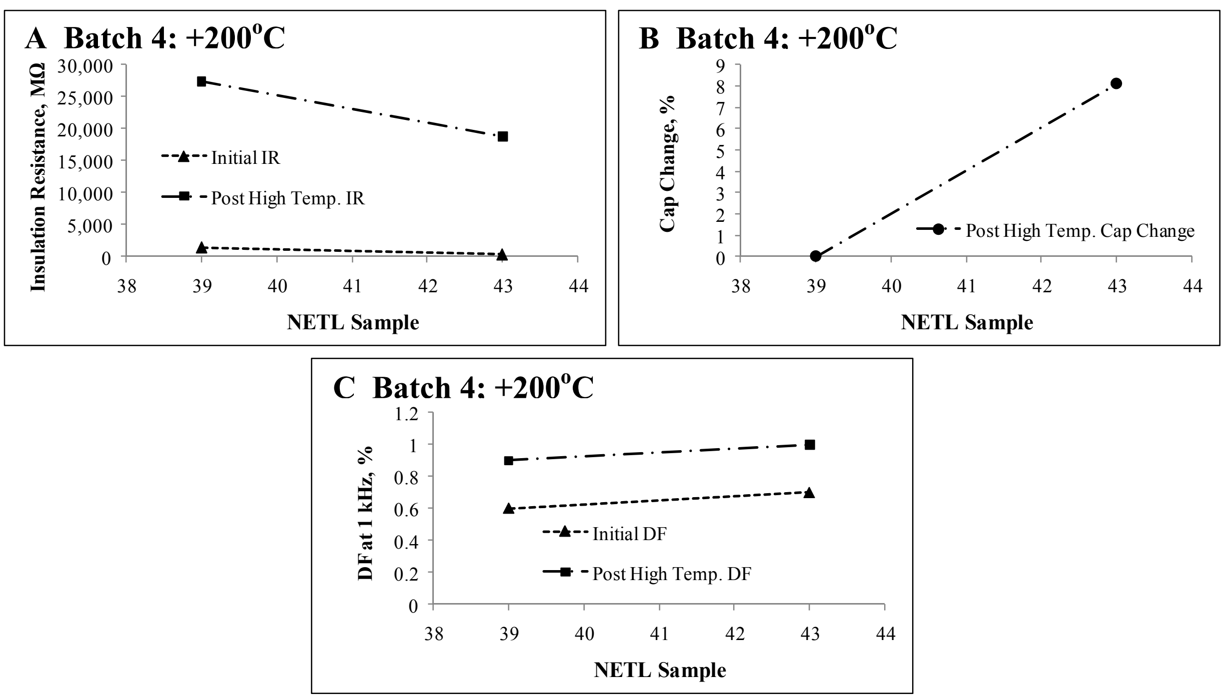


Fig. 4-15: Batch 4 IR (A), Cap change (B), and DF (C) Measurements Taken at Room Temperature After +200°C Curing for 250 hr (Life Test), and Compared to the Initial Values.

4.3.3.2 Part 2 - Lead attachment and housing possibilities for FPE capacitor assemblies

Part 2 of the packaging study involved examination of lead attachment and housing possibilities for FPE capacitor assembly by Dearborn Electronics. To perform the FPE packaging study new capacitor sections were wound and the winding; conditioning; curing; and end spray processes established during Part 1.

Each FPE material type from Batch 3 & 4 were converted into approximately sixty wound capacitor sections with the exception of #44 from Batch 4. This material was not wound due to lack of high temperature insert cores and this report does not contain any data from this FPE group. The total number of FPE wound sections used in Part 1 and Part 2 study is shown in Table 4-23.

Table 4-23: Batch 3 & 4 Number of Sections Wound and Used in FPE Investigation; Part 1 and Part 2

		Part 1		Part 2		
	NETL Sample Number	Number of Sections Wound for 200°C Group	Number of Sections Wound for 250°C Group	Number of Sections Wound for 200°C Group	Number of Sections Wound for 250°C Group	Total Number of Sections Wound for Part 1&2 per NETL Sample
Batch 3	33	16	16	33	30	95
	34	13	13	28	28	82
	35	16	16	32	32	96
	36	17	16	28	28	89
	37	21	20	35	33	109
	38	20	21	27	27	95
Batch 4	39	16	16	26	25	83
	40	13	13	32	32	90
	41	16	16	21	21	74
	42	16	15	26	26	83
	43	20	21	28	29	98
	44	21	20	*	*	41
Total Number of Sections Wound for Part 1&2						**1035**

4.3.3.2.1 Electrical Evaluation of FPE Sections Prior to Packaging Study

Three randomly selected sections from each FPE material sample and each temperature group were tested to voltage breakdown. The +250°C group was closely observed through this step since performance of this group in Part 1 was very poor. This test, as described in 4.3.3.1.1 was performed on a Dearborn proprietary voltage bridge. Voltage was increased in 10 VDC increments until failure. Recorded results confirm similar behavior of each FPE material, as observed in Part 1.

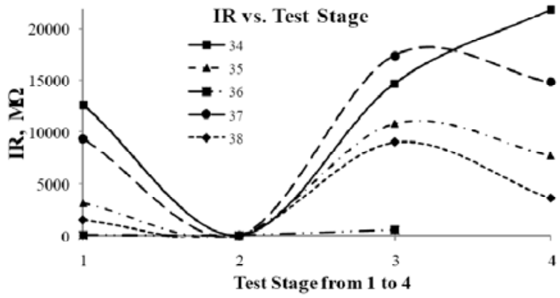
In the +250°C group, voltage breakdown was decreased to half of the preliminary voltage rating established in Part 1. The concept behind this was to prepare some representatives from this group that would be able to perform at a lower voltage but at a higher temperature. As a reminder, it was reported in 4.3.3.1 that +200°C and +250°C groups had voltage breakdown of 1000 VDC or 200 V/µm and 450 VDC or 90 V/µm, respectively.

The +200°C group testing closely matched the results from previous testing. However, the +250°C group performed poorly even when tested at half of the value determined in Part 1. Therefore, an even greater voltage reduction for the +250°C group was tried, but poor performance of the tested sections still resulted. In conclusion, the FPE materials from Batch 3 & 4 are not yet ready to handle +250°C temperatures. There will be no data collected from capacitor sections prepared for this temperature group.

Following the test procedures developed in Part 1, and initial measurements of all surviving FPE sections prepared for Part 2, the +200°C and +250°C groups were submitted to high temperature characterization. Only capacitors that remained good for electrical conditions were placed on to this test. The test conditions and settings are detailed in 4.3.3.1.3. Insulation Resistance (IR) was measured and recorded at 250 VDC for the +200°C group, and at 100 VDC for the +250°C group. Then, FPE capacitors were brought back to room temperature and IR measurements were recorded followed by Capacitance (Cap) and Dissipation Factor (DF) at 1 kHz. DC Burn-In test for 96 hours followed as the next step in preparation of FPE sections for the lead attachment and packaging investigation. During this test, +200°C group capacitors were tested at 250 VDC and +200°C for 96 hours, and +250°C group capacitors were tested at 100 VDC and +250°C for 96 hours. After completion of burn-in all tested FPE sections were cooled down to room temperature for post DC burn-in evaluation. Again, IR measurements were recorded followed by Capacitance (Cap) and Dissipation Factor (DF) at 1 kHz.

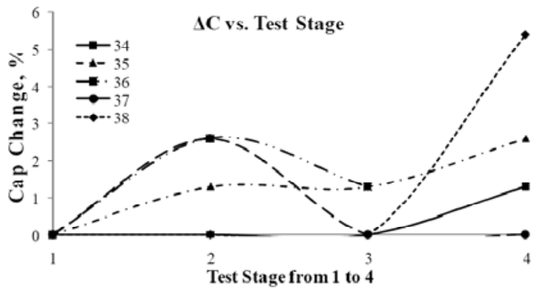
From the collected data, four FPE materials (NETL samples 34, 35, 37 and 38) were selected as Batch 3 representatives for further testing. These same NETL material samples performed well after investigation of Part 1.

Three FPE materials from Batch 4 (NETL samples 39, 41, and 43) behaved as expected during the testing of Part 2 based on their performance and data collected in Part 1. However, none of the FPE materials from the +250°C group survived high temperature tests. Therefore, no data was collected from capacitor sections prepared for this temperature group. The results collected from the +200°C group of Batch 3 & 4 are presented in Fig. 4-16 and Fig. 4-17, respectively.



Test Stage - Batch 3:

1 - Initial readings
2 - Electrical test readings at 200°C
3 - Electrical test readings at 25°C post high temperature testing
4 - Electrical test readings at 25°C post 96 hr DC-Burn-In at 200°C & 250 VDC



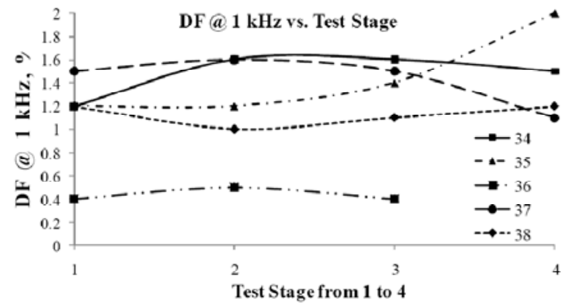
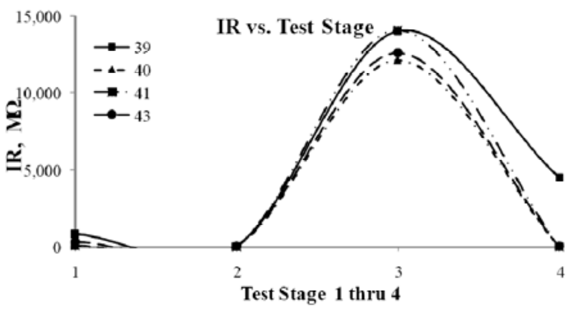
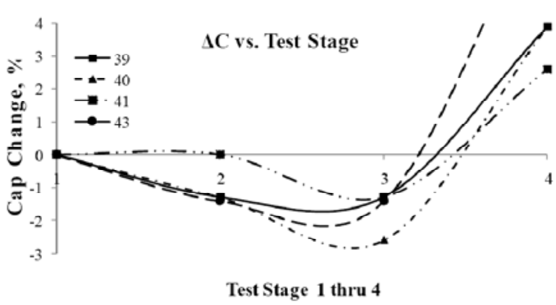


Fig. 4-16: Batch 3 Test Evaluation Through Four Steps of the Testing



Test Stage - Batch 4:

1 - Initial readings
2 - Electrical test readings at 200°C
3 - Electrical test readings at 25°C post high temperature testing
4 - Electrical test readings at 25°C post 96 hr DC-Burn-In at 200°C & 250 VDC



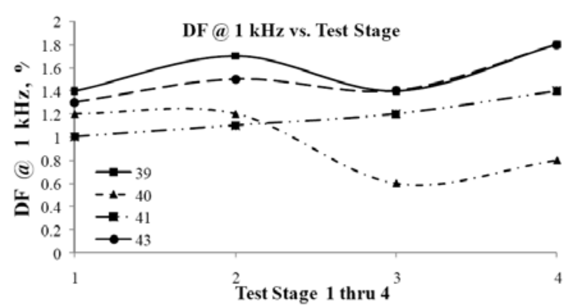


Fig. 4-17: Batch 4 Test Evaluation Through Four Steps of the Testing

4.3.3.2.2 Lead Attachment Investigation and Final Assembly Evaluation

This portion of the Part 2 testing provides information about interface connections ("end spray to terminal") for FPE capacitor assembly. The purpose of this investigation was to select the most suitable way of terminal connection and final packaging for the FPE capacitors.

Lead Attachment and Final Assembly 1

Lead Attachment and Final Assembly 1 consisted of a tinned copper contact square strip 0.002 in. thick, 18 AWG oxygen free, high conductivity copper wire, Kapton tape, and high temperature (200°C) epoxy resin system. Six FPE capacitor sections were used in this experiment, three from batch 3 and three from Batch 4.

In this configuration a 0.002 in. thick square copper contact strip was welded to each section's end surface using an ultrasonic resistance welder. Next, the 18 AWG copper wires were soldered to the squares with 5%Sn 95%Pb high temperature solder. After lead attachment all units were electrically tested on the leads, to evaluate the welding and soldering process. Then, sections were taped with Kapton tape and sealed with epoxy. Upon completion of the epoxy curing process, the first FPE assembled capacitors were manufactured. Drawings of the assembly are presented in Fig. 4-18.

Fig. 4-18: Batch 3 & 4 Lead Attachment and Final Assembly 1 – Capacitor Assembly

Completed Final Assembly 1 capacitors were electrically verified for IR, Cap, and DF at room temperature, and then placed on DC Burn-In test for 96 hours at 250 VDC and +200°C. This high temperature test was selected to evaluate connection of all interfaces included in this assembly; end spray to leads and end spray to epoxy. Previous studies of Batch 2 documented that separation between the end spray surface and capacitor electrode occurred when enclosed in epoxy and heated to high temperature. This separation between the two interfaces led directly to capacitor failure.

During the burn-in all six FPE assemblies experienced separation induced failures. In Fig. 4-19 an example of this malfunction is shown.

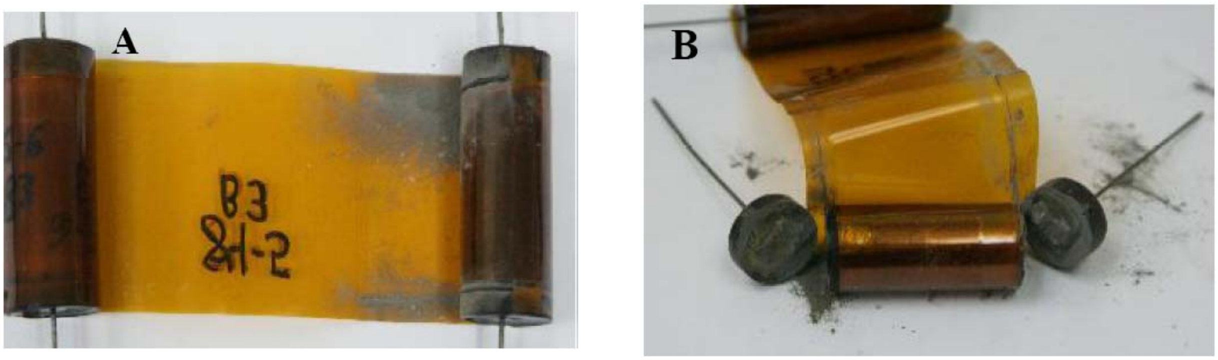


Fig. 4-19: Batch 3 & 4 FPE Capacitor Failure: A) End Spray Turned Into Powder; B) Complete Separation of the Epoxy From the Capacitor Section; End Spray Interface Completely Converted Into Powder.

Lead Attachment and Final Assembly 2

Lead Attachment and Final Assembly 2 was performed in the same manner as the Lead Attachment and Final Assembly 1 with attachment of a 2 in. long copper strip rather than the 18 AWG copper wire. The strip was welded to each section's end spray, wrapped with Kapton tape, and enclosed with high temperature epoxy resin. Electrical tests were repeated for Final Assembly 2 capacitors. Eight FPE sections from Batch 3 & 4 were selected for this trial, and all resulted in failure either during or after the 96 hours burn-in test. In Fig. 4-20 a sample failure is shown.

Fig. 4-20: Batch 3 & 4 Lead Attachment and Final Assembly 2 Failure Example: Epoxy Separated from the Section on one Side; Other Side Remains Connected

Lead Attachment and Final Assembly 3

Final assembly 3 consisted of packing FPE sections in a metal housing after applying all connection types from Final Assembly 1. A nickel silver alloy case was selected for the housing. Electrical tests were repeated for Final Assembly 3. Six FPE sections from Batch 3 & 4 were selected for this trial. All assemblies failed after 96 hours burn-in test when tested at room temperature.

In this experiment epoxy was pushed out of the housing. After cutting the case open, the cracking between epoxy and end spray was seen again. In Fig. 4-21 a sample of Lead Attachment and Final Assembly 3 is shown.

Fig. 4-21: Batch 3 & 4 Lead Attachment and Final Assembly 3; FPE Capacitor with Lead Wires Assembled into a Metal Case

Lead Attachment and Final Assembly 4

Similar to Final Assembly 3, FPE capacitor sections with 2” long copper contact strip welded to the end spray surface were packaged into Ni-Ag alloy cases and enclosed with epoxy. Again, after DC burn-in for 96 hours at 250 VDC and +200°C all samples from Batch 3 & 4 failed electrical evaluation when tested for IR, Cap, and DF at room temperature.

Lead Attachment and Final Assembly 5 & 6

From the four assembly configurations presented herein none were suitable as a packaging option for the manufacture of the FPE capacitors. Regardless of the choice of housing, FPE capacitors failed during or after 96 hour burn-in test. Separation and disconnection between capacitor section and the end spray layer always occurred during capacitors exposure to +200°C for extended periods.

To establish failure mechanism and eliminate the test voltage of 250 VDC as the cause of failure, two FPE capacitors from each of four assembly configurations were placed at room temperature DC Burn-In for 96 hours. During and after the burn-in time no physical damage or disconnections among the interfaces of each assembly was noticed. It might be concluded that the only cause for poor performance and the “capacitor section – epoxy” separations presented above was the result of high temperature.

To investigate this problem further, another experiment was set up. In this case FPE sections had a braid attached to the capacitor by the end spray process. The concept behind this was to eliminate welding or soldering processes to the end spray surface and to reduce additional stress to the capacitors. A copper wire was then either welded or soldered to a silver coated nickel braid, and the four capacitors with leads, in section form, were placed under DC Burn-In test for 96 hours at 250 VDC and +200°C. After completion of the burn-in, no disconnection of the unsealed windings was observed. Next, two capacitor sections with leads were assembled with Kapton tape and two capacitor sections with leads were assembled in the metal case, both sets enclosed with high temperature epoxy, cured, and put on an additional 96 hour DC Burn-In test under the same test voltage of 250 VDC and temperature of +200°C. Three out of four tested FPE capacitors failed DC Burn-In due to poor electrical performance and one FPE capacitor survived the test with no signs of losing connection. In Fig. 4-22 the samples are shown as they were prepared for this experimental step.

Fig. 4-22: Batch 3 & 4 FPE Capacitors with Braids Assembled Through the End Spray Process and the Copper Wires Attached to the Braids Either by Welding or Soldering.

In Fig. 4-23 is shown one of three FPE capacitors that failed DC Burn-In test. As it can be seen, the end spray surface and epoxy are in very good condition. No separation or cracks were noticed. Poor electrical performance of this capacitor came from its initial readings. The purpose of this experiment was to estimate high temperature effect on the final FPE assembly when no stress was applied directly to the end spray surface, and the sections selected did have higher DC leakage than desired.

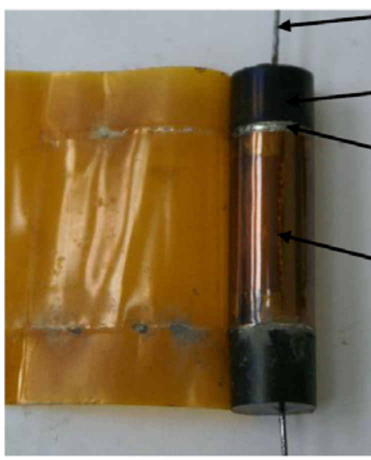


Fig. 4-23: Batch 3 & 4 Lead Attachment and Final Assembly 5: FPE Capacitor That Failed 96 Hours DC Burn-In Test at 250 VDC and +200°C; No Signs of Cracks or Disconnection Among Capacitor's Elements

However, it might be concluded that the stress applied to the end spray surface through the welding process has a negative effect on the final output of the packaged FPE capacitors when exposed to the temperature of +200°C for time of 96 hours. As a reminder, the tested FPE capacitors were kept at high temperature (+200°C) for 96 hours, and at this point it is hard to say how they would have performed during the Life Test when the capacitors would be exposed to +200°C at a voltage of 250 VDC for 250 hours.

Lead Attachment and Final Assembly 7 & 8

Due to the nature of the failures and the FPE manufacturing process, it could be assumed that the out-gassing of the FPE capacitor sections has an important role in the development of a successful packaging system. All FPE sections used in the lead attachment and final assembly trials have been exposed to the temperature of interest for a period of 96 hours. The assumption was that the FPE capacitor sections needed longer time at the temperature to allow release of the gasses, which build up at rated temperatures. The thought process was if the impurities present in the FPE film were out gassed for a longer time at temperature, the greater the possibility for success of the FPE packaged capacitor.

To investigate this theory, another experiment was performed. Five FPE units from Batch 3 & 4 which were used for Part 1 of this report were selected for the examination. All five FPE capacitors survived 250 hours Life Test in the section form at +200°C and 250 VDC. Leads were attached through the process of welding and soldering. An improvement of the leads being attached to a braid was demonstrated in the previous experiment and to explore the extension of time at the high temperature the lead attachment through welding was selected.

Some of the FPE Life Test sections were housed in Kapton tape and some in Ni-Ag alloy cases; all were enclosed with the high temperature epoxy resin, and cured. Assembled capacitors were then placed to 250 hours Life Test at 250 VDC and +200°C. During this test capacitors were closely watched on a daily basis by controlling the power supply and test settings in order to catch a failure. Two out of five FPE capacitors failed Life Test in the first 96 hours. The other three successfully completed the 250 hours Life Test.

After physical examination of the surviving samples no separation or major cracks between the end spray layer and the epoxy layer was noted. All connections were in place and remained in good condition.

From this test it was concluded that when FPE sections are exposed to high temperature for an extended period of time (250 hours) it allows them to out-gas to the point when they might be packaged and survive in the final form.

From all the results collected it is recommended the 250 hours Life Test be performed at the temperature and rated voltage when the FPE capacitors are in the section form prior to their final assembly. In addition, the lead attachment should be soldering a copper wire or a copper connect strip to a solder braid installed on the FPE section through the end spray process. It is strongly recommended these two steps be applied in the assembling process of the FPE capacitors in order to establish a successful manufacturing mechanism.

Continued Lead Attachment and Final Assembly Experiments

Lead Attachment and Final Assembly experiments are ongoing even after the conclusion of this program and are presently focused on different non-metallic housing materials.

4.3.3.2.3 Conclusion

Many experiments were closely observed in order to develop a successful packaging system for the FPE capacitors. Dearborn's investigation pointed out a few things which need to be carefully executed in order to collect desirable data and to develop a successful device capable of operation at +200°C and 250 VDC when fully assembled.

First, a braid needs to be attached to the FPE section through the end spray process and a lead wire or copper connection strip should be soldered to the braid. This lead attachment technique reduces stress to the end spray surface which would otherwise occur during the welding process.

In addition to this, the FPE sections should be subjected to a 250 hours Life Test at 250 VDC and +200°C to eliminate potential failures and to allow the FPE sections to out-gas prior to encapsulation.

4.3.3.3 Part 3 - Manufacturing of the final product and electrical evaluation

In Part 3, further research was performed to find an optimum assembly technique for FPE capacitors. In Part 2 several housing possibilities were tested, however, end connection deterioration was still encountered. Dusting/end disconnection was occurring every time the capacitor sections were exposed to heat and voltage following end spray and epoxy. Several steps were taken to try to understand the mechanism of the end disconnection. A new design was established to avoid the dusting, and lead attach and electrical measurement studies continued. In this new phase of testing, Babbitt, a tin, antimony, copper alloy, was selected for the end spray rather than the zinc used in Part 2. The dusting or end disconnection was not experienced after changing the end spray to Babbitt only.

Since all FPE capacitor inventory was sprayed with zinc, new sections were wound from the following NETL samples and Batch #s: NETL 37, Batch 3 and NETL 43, Batch 4.

4.3.3.3.1 Dusting/end Disconnection

Although elemental analysis results of the dusting samples were not received, it is known that the dusting was only visible when end spraying with zinc. When units were epoxied with no end spray, there was no dusting. Sixteen units were wound from the three different batches, eight with no end spray, and eight with zinc on one end and Babbitt on the other. The dusting only occurred on the zinc ends. The Babbitt/FPE connection always remains intact, no matter what time/temperature/voltage the parts were exposed to.

4.3.3.3.2 Chosen Batches and New Windings

Upon review of the Part 1 and Part 2 data, it is clear that the best yields from 200°C Initial, Acceptance and Life Tests were achieved with NETL # 37 - Batch 3 and NETL #43 - Batch 4. Therefore, several capacitors were wound from each of those batches to continue the study. There were no winding issues with any of the batches. The film winding yield was 100% and winding conditions were clearly established.

There were a few differences between the batches including the resistivity (ohms/sq) and FPE film variation. See Table 4-24 below for differences between batches.

Table 4-24: Batch 3 & 4 NETL Batch Properties for Part 3 Films

NETL #	Batch #	FPE Variation	Acrylate Coating	Al Metal
37	3	STD	HVY	HVY (25 ohm/sq)
43	4	Var. 3	STD	STD (15 ohms/sq)

4.3.3.3.3 Electrical Evaluation of Babbitt-only End Spray vs. Zinc

Babbitt end spray does sacrifice DF performance compared to zinc. In a study with a non-FPE (PPS) dielectric of a standard Dearborn part, the DF for Babbitt was found to be 30% higher than that of zinc. The electrical conductivity of zinc and Babbitt are 1.67E+07 S/m and 1.17E+07 S/m, respectively, or a difference of 29%. See Fig. 4-24 for a comparison of end spray electrical testing over a 96-hr DC Burn-In test.

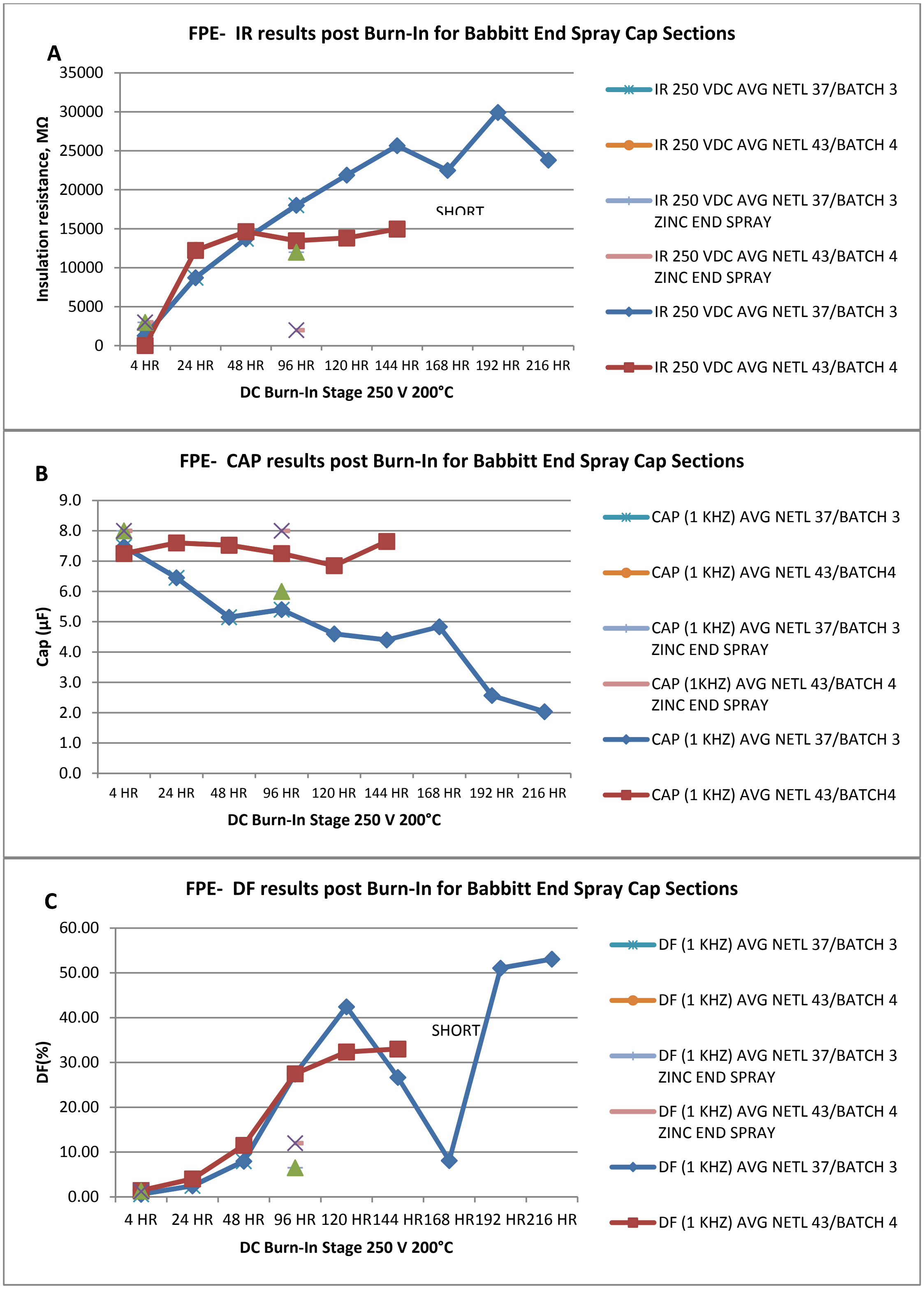


Fig. 4-24: Batch 3 & 4 IR (A), Cap (B), and DF (C) Measurements of Materials End Sprayed with Babbitt

It should be noted that IR increased over burn-in time, especially for Batch 3 with Babbitt end spray. DF percent increased dramatically as the burn-in time increased. The rise was relatively independent of Batch #, however significantly higher for the Babbitt end spray. Additionally, capacitance decreased substantially over burn-in time. Beyond the scope of this program, life testing continued to observe how DF changes with increased burn-in time.

4.3.3.3.4 Lead Attach to Babbitt End Spray

Leads were attached with three different methods to the Babbitt. Welding, soldering or attachment in the end spray was achieved without sacrificing electrical properties. The conclusion was made that using silver plated wire (rated at a temperature of 200°C with silver-plated conductors or to 260°C with nickel-plated conductors) was preferable due to its ease of welding, soldering and attachment in the end spray versus the nickel plated wire. Therefore, this wire will be chosen for any future designs.

4.3.3.3.5 Increase of DF with Burn-in testing

Based on the dramatic increase in DF values with increasing time at temperature, it was believed that the capacitor end connection at the film/electrode to Babbitt interface was being lost. However, mechanically the Babbitt adhesion to the capacitor ends is very strong. Additionally, no metalized electrode loss was observed on the film surface. When unwinding several sections to investigate the increase in DF, some apparent defects were seen. Defects similar to the left-most image in Fig. 4-25 appear randomly in the film with approximately six for every twelve inches of film. The center and right-most images in Fig. 4-25 are a repeating defect for several feet of film and then disappear. The defects are visible without a microscope and appear as an uneven surface of the film or coating, possibly static in the base film which caused an uneven metallization (a defect called "hoof" in polyester film manufacturing).

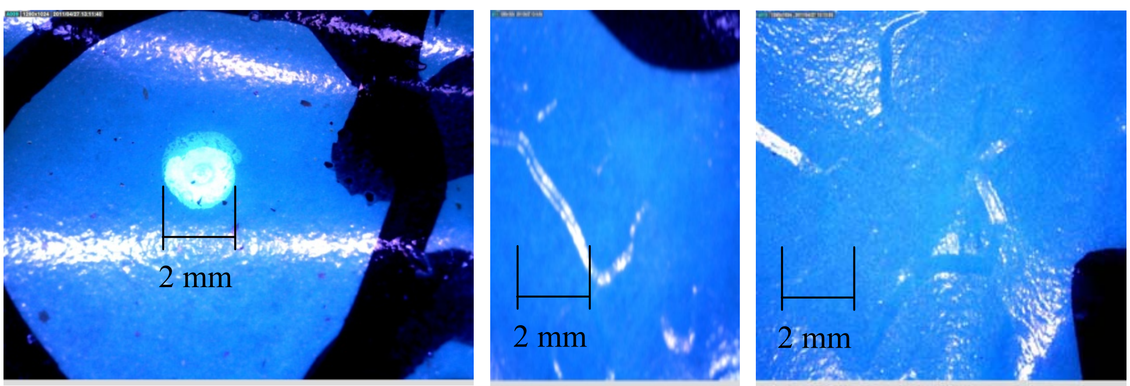


Fig. 4-25: Batch 3 & 4 Film Defects with Babbitt End-Spray

4.3.3.3.6 Packaging Status

Materials are available in-house at Dearborn Electronics to continue the packaging study including non-metallic tubes and boxes, nickel plated terminals, and high temperature wire. High temperature tests confirm that these materials will remain stable at 200°C. Measurements taken before and after the 200°C cure confirm that no significant change in dimension will take place. Additionally, FPE inside a hermetically sealed metal case housing was heated for several days at 200°C with no compromise to the seal. There are several potential housing options available.

There are 50 lbs of Batch 2, 8.6 lbs of Batch 3 and 20.5 lbs of Batch 4 FPE material remaining. However, packaging is placed on hold until the DF issue is investigated further.

4.3.3.3.7 Continuation of Study at 250°C

Parts end sprayed with Babbitt were placed in a 250°C oven under voltage of 250 VDC. Within a few hours, the ends of the parts were visibly losing end spray with large sections of end spray depositing at the bottom of the rack. One additional part was put in the oven under heat only with the same result. The Babbitt is experiencing a phase change in the 250°C range.

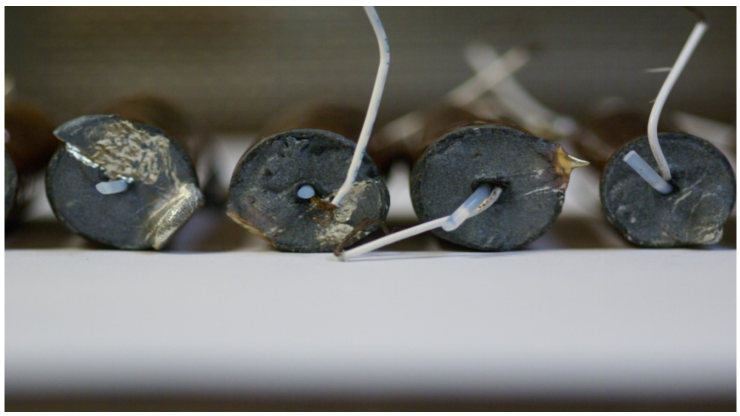


Fig. 4-26: Batch 3 & 4 Capacitors with Babbitt End-Spray at 250°C

4.3.3.3.8 Conclusion

In this last time period of testing, some positive results with regards to solving the dusting/end disconnection issue have been obtained, as well as finding a high temperature wire which works well with a new end spray material. However, the DF results are not acceptable and more investigation is required. Also, in the pursuit of a capacitor that will survive 250°C, Babbitt end spray is not the right choice.

Research is underway for a new end spray material with adequate conductivity, which will withstand high temperatures and allow good connectivity to the chosen lead wire material.

Future activity will include additional testing of zinc failures, testing of different end spray materials, testing of parts at 250°C, and finally housing of the capacitor sections once we solidify the resulting material. The material for future testing will be Batch 3, NETL #33-39, as this is the standard FPE variation and there is more available for metallization.

4.3.4 Batch 3 & 4: Summary

In general, there was a tremendous improvement in the consistency of the FPE film production and metalization between Batch 2 and Batch 3 & 4. The electrical performance of the films was consistent with high yields for most of the formulations at 200°C.

Packaging of the final capacitors was investigated with a preferred end connection and packaging scheme developed as follows:

- Capacitor winding built and tested as shown in Fig. 3-8 for 200°C parts.
- Windings successfully passing the 250 hour life test are to be packaged using the procedure shown in Fig. 3-15 or Fig. 3-16.

The key outstanding issue with the capacitor is a deficiency in the end-spray material. The zinc end-spray had significant disconnection issues, and Babbitt had significant degradation of DF and IR when held at 200°C for extended time periods. The best solution tested today requires at least 250 hours of life test at 200°C prior to packaging. Future research will focus on new end-spray materials that may be compatible with the FPE material.

5 Conclusions

Through DOE/NETL Development of High Temperature Capacitor Technology and Manufacturing Capability program, the manufacturing processes of high temperature FPE film capacitors have been drastically improved.

The key challenges to bringing the FPE film capacitors to market have been manufacturing challenges, specifically:

- FPE Film is difficult to handle and wind, resulting in poor yields
- Voltage breakdown strength decreases when the film is wound into capacitors (~70% decrease)
- Encapsulation technologies must be improved to enable higher temperature operation
- Manufacturing and test cycle time is very long

As a direct result of this program most of the manufacturing challenges have been met. The FPE film production metalization and winding yield has increased to over 82% from 70%, and the voltage breakdown strength of the wound capacitors has increased 270% to 189 V/µm. The high temperature packaging concepts are showing significant progress including promising results for lead attachments and hermetic packages at 200°C and non-hermetic packages at 250°C. Manufacturing and test cycle time will decrease as the market for FPE capacitors develops. Dearborn Electronics is continuing the optimization of the FPE capacitor packaging to further increase yields.

Hamilton Sundstrand is actively transferring the FPE capacitor technology into power electronics systems. HS has tested FPE capacitors in an existing power electronics converter in a laboratory setting and is actively designing FPE capacitors into a ground-up design of a power electronics converter to demonstrate the technology alongside SiC and other high temperature electronics technologies. Simultaneously Dearborn Electronics is actively marketing FPE capacitors to its wide industrial, aerospace and petrochemical customers, see APPENDIX A.

List of Figures

List of Tables

List of Acronyms and Abbreviations

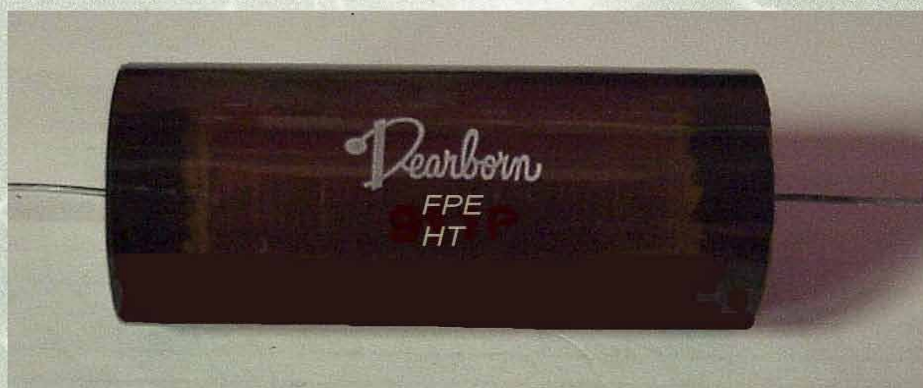
AFM	Atomic Force Microscopy
AFRL	Air Force Research Laboratories
Cap	Capacitance
COF	Coefficient of Friction
CSI	Current Source Inverter
DF	Dissipation Factor
DLC	Diamond Like Carbon
DOD	Department of Defense
DOE	Department of Energy
DWV	Dielectric Withstand Voltage
EMI	Electromagnetic Interference
EPA	Environmental Protection Agency
FPE	Fluorine Polyester
GE	General Electric
H.E.	Heavy Edge
HS	Hamilton Sundstrand
HTHP	High Temperature High Pressure
HVY	Heavy
IR	Insulation Resistance
IR&D	Internal Research & Development
LRU	Line Replaceable Unit
LT	Light
MD	Machine Direction
MEA	More Electric Aircraft
MLC	Multi-Layer Ceramic
MPF	Monolithic Polymer Film
NASA	National Aeronautics and Space Administration
NETL	National Energy Technology Laboratory
OSHA	Occupational Safety and Health Administration
PC	Polycarbonate
PCD	Polycrystalline Diamond
PEI	Polyetherimide
PEN	Polyethylene Naphthalate
PI	Polyimide
PP	Polypropylene
PPS	Polyphenylene Sulfide
PRPE	Electrical Technology & Plasma Physics Branch (AFRL)
PTFE	Polytetrafluoroethylene
SiC	Silicon Carbide
STD	Standard
TD	Transverse Direction
VSI	Voltage Source Inverter

APPENDIX A Dearborn FPE Capacitor Bulletin

Dearborn electronics, *inc.*

ISO9001 Dearborn electronics, inc. CERTIFIED

FPE…200°C Operation Without Derating



DEARBORN'S FLUORENE POLYESTER FILM (FPE) capacitors provide the electronic designer with a new choice for applications with operating conditions of 200°C.

High Stability | **Capacitance up to 33µF**

Ratings up to 200VDC | **-65°C to +200°C Operation**

Dearborn Electronics, Inc. – 1221 N. Highway 17/92 – Longwood, Florida 32750

Tel: (407) 695-6562 Fax: (407) 695-6889 E-Mail: dearborn92@att.net Web site: www.dearbornelectronics.com

Dearborn electronics, inc.

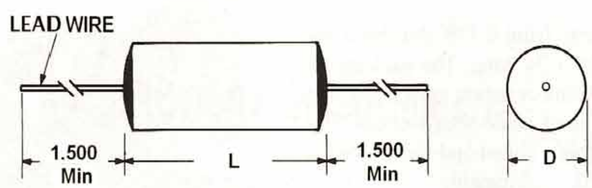
METALIZED FLUORENE POLYESTER FILM CAPACITORS

TYPE FPEP

Wrap-and-Fill High Temperature Metalized Fluorene Polyester Capacitors

Features—
- **High Stability**
- **High Insulation Resistance**
- **Low Series Resistance**

DIMENSIONS IN INCHES



Major Applications:
Polycarbonate replacement for timing and integrating circuitry, high frequency coupling, and other applications where severe environments require high operating temperature range without voltage derating.

PHYSICAL CHARACTERISTICS —

Construction:
Axial wrap-and-fill non-inductive wound metalized fluorene polyester; flame retardant tape wrap and epoxy end fill.

Lead material:
Solder coated lead wire

Lead Pull:
5lbs. (2.3Kg) for one minute; No physical damage

Lead Bend:
After three complete consecutive bends. No damage

Marking:
Dearborn trademark, Type or catalog number, capacitance, tolerance and voltage

ELECTRICAL SPECIFICATIONS —

Operating Temperature:
-65°C to +200°C

Capacitance Range:
.010 µf to 33 µf

Capacitance Tolerance:
±20%, ±10%, ±5%

Dissipation Factor:
1.0% maximum @ 1KHz

Voltage Rating:
63 VDC to 200 VDC

DC Voltage Test:
150% of rated voltage for 2 minutes

Insulation Resistance:
Measure at rated VDC following a 2 minute charge
At +25°C, 50,000 Megohm-Microfarads, need not Exceed 100,000 Megohms
At +200°C, 200 Megohm-Microfarads typical, need not Exceed 400 Megohms

Dearborn electronics, inc.

METALIZED FLUORENE POLYESTER FILM CAPACITORS

TYPE FPEP

CATALOG NUMBERING SYSTEM

EXAMPLE:

FPEP 683 X9 140

- 140 — **DC VOLTAGE RATING:** EXPRESSED IN VOLTS. SEE STANDARD RATINGS CHARTS FOR VOLTAGE CODE.
- X9 — **CAPACITANCE TOLERANCE:** X0 = ± 20%; X9 = ±10%; X5 = ± 5%;
- 683 — **CAPACITANCE:** EXPRESSED IN PICOFARADS. THE FIRST TWO DIGITS ARE SIGNIFICANT FIGURES; THE THIRD IS THE NUMBER OF ZEROS FOLLOWING. SEE STANDARD RATINGS TABLES FOR CAPACITANCE CODE
- FPEP — **DEARBORN TYPE NUMBER:** IDENTIFIES THE BASIC CAPACITOR.

STANDARD RATINGS

Capacitance µF	Capacitance Code	Voltage Code 063 63VDC		Voltage Code 140 140VDC		Voltage Code 200 200VDC	
		D	L	D	L	D	L
0.010	103	.170	.562	.170	.562	.170	.562
0.022	223	.170	.562	.170	.562	.235	.687
0.047	473	.170	.562	.235	.687	.235	.687
0.056	563	.170	.562	.235	.687	.235	.687
0.068	683	.170	.562	.235	.687	.312	.813
0.120	124	.312	.813	.312	.813	.312	.813
0.150	154	.235	.687	.312	.813	.312	.813
0.180	184	.235	.687	.312	.813	.400	1.063
0.220	224	.235	.687	.312	.813	.400	1.063
0.270	274	.235	.687	.312	.813	.400	1.063
0.330	334	.312	.813	.312	.813	.400	1.063
0.470	474	.400	1.063	.400	1.063	.400	1.063
0.560	564	.400	1.063	400	1.063	.400	1.063
0.680	684	.312	.813	.500	1.125	.500	1.125
0.820	824	.312	.813	.500	1.125-	.500	1.125
1.000	105	.312	.813	.500	1.125	.500	1.125
1.500	155	.500	1.125	.500	1.125	.670	1.375
2.000	205	.400	1.063	.670	1.375	.670	1.375
3.000	305	.400	1.063	.670	1.375	.670	1.375
4.000	405	.500	1.125	.670	1.375	.670	1.375
5.000	505	.500	1.125	.670	1.375	.750	1.875
6.000	65	.750	1.875	.750	1.875	.750	1.875
12.000	126	.670	1.375	1.00	2.375	1.00	2.375
15.000	156	.670	1.375	1.00	2.375	1.00	2.375
16.000	166	.670	1.375	1.00	2.375	-	-
25.000	256	.750	1.875	1.00	2.375	-	-
30.000	306	.750	1.875	1.00	2.375	-	-
33.000	336	.750	1.875	-	-	-	-

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