EVALUATION OF POLYETHYLENE SUBSURFACE DRIP IRRIGATION DRIPLINES AFTER 26 YEARS OF SERVICE IN A CORN FIELD IN NORTHWESTERN KANSAS

Adriana Velasquez, Mark Jablonka, Freddie Lamm, Eddy Garcia-Meitin
The Dow Chemical Company / KSU Northwest Research Extension Center

Subsurface drip irrigation (SDI) delivers increased crop yields, improves utilization of farmland, reduces fertilizer run off and more efficiently uses water. One concern commodity growers have when investing in an SDI system is the longevity of the dripline, requiring a long life to be economically practical. The use of high quality raw materials is a key factor in maximizing the value of an investment in SDI.

This paper contains an actual example of how the use of high quality materials enabled driplines to last 26 years in service. A new dripline was installed in Kansas as part of an SDI system in 1989. The system operated with the same dripline until 2015, when it began to fail. The overall dripline held up well and remained ductile over its 26 year life, demonstrating longevity when quality raw materials are used.

Through this real life example, the key inputs to maximizing the longevity of driplines are identified and outcomes of the aging process described.

INTRODUCTION

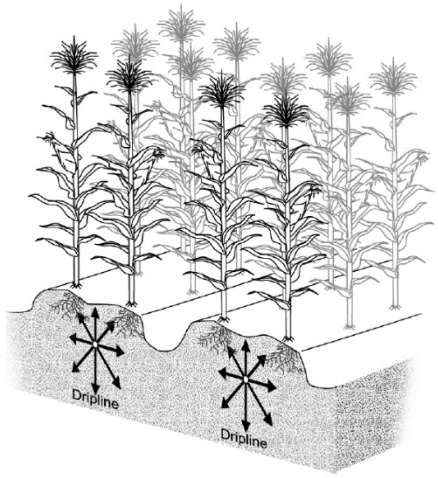
A polyethylene Subsurface Drip Irrigation (SDI) system that was installed in a corn field in 1989 in the Northwest region of Kansas was taken out of service due to leaks in 2015. Portions of the dripline were analyzed for characterization. The purpose of this work is to highlight the importance and advantages in the use of high quality materials to manufacture the polyethylene driplines for such application. This report documents the characterization of this dripline and the failure analysis.

BACKGROUND

Dow was the first company to develop polyethylene grades specifically for the microirrigation segment in the 1970's. The products Dow developed greatly extended the longevity of tubing at the time. As the segment continued to develop, so did Dow's product offering. FINGERPRINT™ are Dow's resins for microirrigation that have been used to improve the way watering of crops is done, which has contibuted to increasing the utilization of farmland and also has help growers save in the usage of fertilizers.

One of the key application areas for growth within the microirrigation dripline segment is referred to as Subsurface Drip Irrigation (SDI). In this application, driplines are buried up to 24 inches below the surface of the soil, water is pumped through the dripline and delivered to the roots of the crops via emitters built into the driplines. Figure 1 illustrates where the dripline is placed relative to the crop. The dripline is indicated by the circle below the surface with arrows indicating soil water redistribution radiating outward from it.

Figure 1. Image of Subsurface Drip Irrigation system placement for growing corn.[i]



Research in the area of subsurface drip irrigation has been ongoing for over 30 years. Kansas State University began studying and developing subsurface drip irrigation (SDI) techniques for growing commodity crops in 1989.[ii] The driver for this technology is to make more efficient use of water, water conservation is one of the most effective ways to positively impact the environment these days.

For over 25 years the Northwest Research-Extension Center in Colby, Kansas has been focused on subsurface drip irrigation (SDI). The dripline evaluated in this study was installed in a corn field in Colby in 1989. Dripline longevity is a key concern for growers investing in an SDI system, especially when commodity prices are low, which makes the fact that this dripline lasted 26 years very compelling.

EXPERIMENTAL PROCEDURES

Materials

The dripline materials are listed in Table 1. Sample 24-1 is a dripline manufactured in the 1989-1990 time frame but was never put in service, sample 24-2, is the in service dripline that failed, had been buried underground during its lifetime. It was used to carry water to the roots of corn plants. Sodium hypochlorite (NaOCl) was used in the dripline as a disinfectant periodically to remove algae/growth. In addition, urea-ammonium nitrate fertilizer was applied through the dripline. Water, sodium hypochlorite and urea-ammonium nitrate are the only chemicals used in the dripline during its lifetime. Sample 24-3 is a standard polyethylene resin produced and sold by Dow into drip irrigation systems today and was used in this study as control.

Table 1. Summary of materials analyzed.

Databook No.	Sample Name	Sample Description
201500084-24-1	Control dripline (unused)	15mil thick Dripline[iii] purchased in 1990 and stored inside a building at Kansas State University's Northwest Research-Extension Center in Colby, Kansas.
201500084-24-2	Failed dripline	15mil thick Dripline installed in 1989 as part of Kansas State University's Subsurface Drip Irrigation (SDI) system in their corn fields at their Northwest Research-Extension Center in Colby, Kansas.
201500084-24-3	FINGERPRINT™ Resin	Dow's resin used to fabricate driplines.

Test Methods

Differential Scanning Calorimetry (DSC)

Since the samples were in the form of dripline, the specimens were punched out and pressed into a pan. TA instrument DSC Q 2000 series was used. The first step is an equilibration step to remove thermal history, then the sample is cooled at 10°C/min to -90°C, isothermal time is 5 minutes, the sample is then heated at 10°C/min to 290°C, isothermal time of 5 minutes. This was done under a nitrogen atmosphere (nitrogen at 50mL/min).

Differential Scanning Calorimetry (DSC) – OIT

Using a DSC instrument, specimens were heated to 200^{o}C in a 100% nitrogen environment, then the nitrogen was replaced by oxygen and the time to full oxidation recorded.

Dynamic Mechanical Spectroscopy (DMS)

Frequency Sweep, viscoelastic properties are measured under controlled strain at 190C, with varying frequencies from 0.1 to 100 rad/s with 10% strain.

Thermogravimetric Analysis (TGA)

A TGA based method was used to quantify carbon black and inorganic residue levels in the dripline. In this method, material from the dripline is heated in an inert environment until it reaches 520^{o}C, at which time the environment is changed to air, the carbon black oxidizes and the weight loss is determined. Residue represents the material left at 800^{o}C.

X-Ray Fluorescence (XRF)

This test was used for determining catalyst residues and relevant additives in all polyolefin samples. The following elements can be requested as an individual test: Al, Ba, Ca, Cl, Mg, Mo, Na, P, S, Si, Ti, and Zn. The XRF is calibrated with polymer standards. Based upon method development data, the error in the accuracy is typically less than +/-10%, but is dependent upon the concentration. The precision (%RSD) of XRF analysis is usually better than +/-5%, but is also dependent upon concentration. The precision and accuracy are also dependent upon sample homogeneity.

Antioxidant (AO)

Resin was extracted using TDM. Extract was analyzed by LC with UV/Vis detector to identify active Antioxidants and oxidized antioxidants as well as their concentration level. Concentration is reported as parts-per-million (ppm). Analysis follows DOWM 102408-I10B.

Tensile

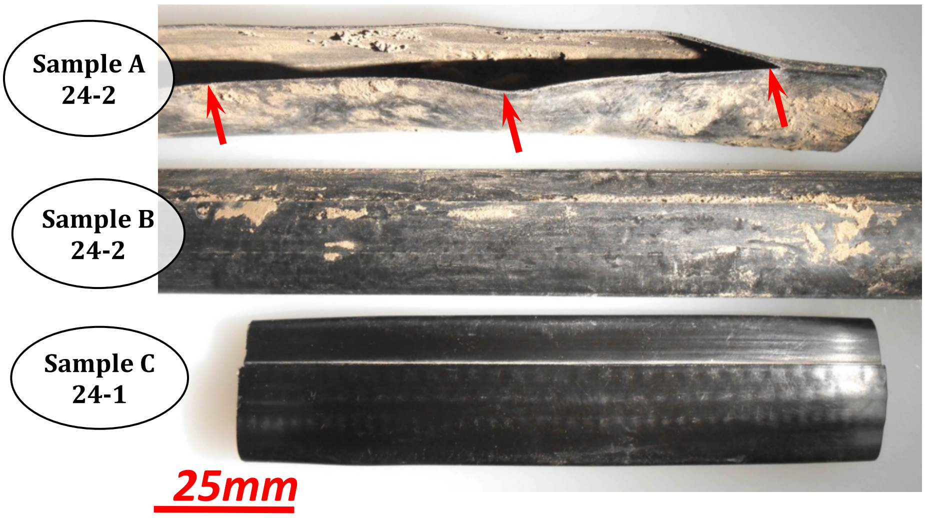
Tensile tests were performed on electromechanical tensile tester. Load cell was 50 lb (~220N). The test was carried out on the full dripline samples with a 2” gauge length using line grips and at a speed of 20”/min.

RESULTS AND DISCUSSION

Characterization

The failed dripline was coated with dirt on both interior and exterior surfaces as received. The presence of dirt on the inside of the dripline was likely caused by either the dripline failing or being cut and removed from the field. Figure 2 is a photo of pieces of the materials received. Sample A is a portion of the failed dripline that was split. A sharp linear failure occurred along one of the two edge creases indicated by the red arrows in the photo. This failure is a machine direction split in the dripline. Sample B is a portion of the failed dripline that did not contain a failure and sample C is the control dripline which was manufactured back in 1989 but was never put in service, instead it was stored for 26 years.

Figure 2. As received dripline samples.



The driplines were confirmed to have been made with 100% Dow resins by identification of the tracer which is added to Dow’s microirrigation FINGERPRINT™ products as well as pressure pipe products, in order to positively identify the material as being from Dow in the case of a failure.

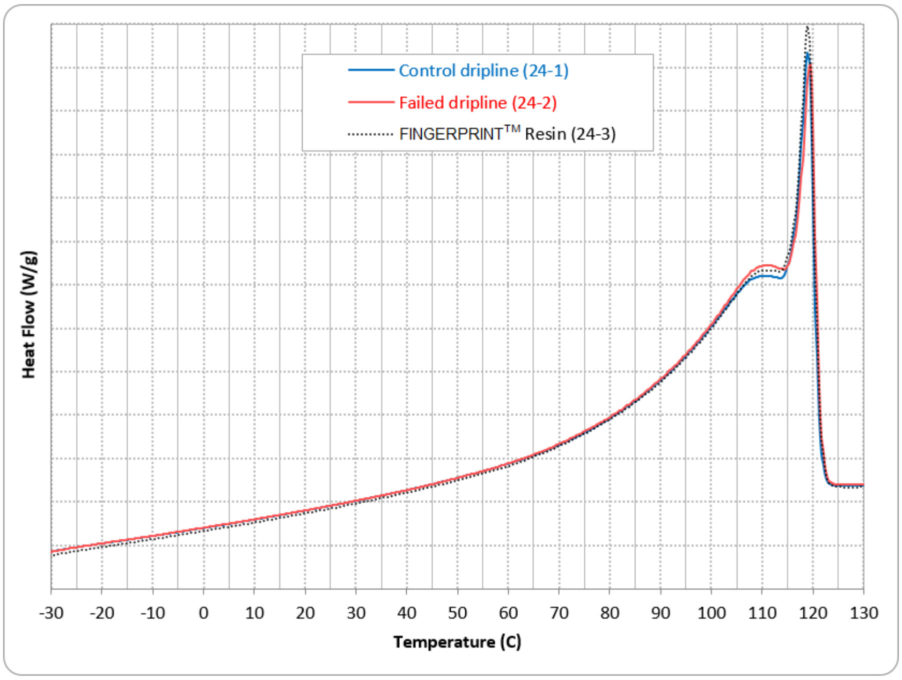
Today FINGERPRINT™ resins continue to be the leading resins used in the manufacturing of microirrigation driplines. DSC was used to characterize thermal properties of the driplines and results are summarized in Table 2. The failed and control driplines both had similar melting characteristics which were comparable to FINGERPRINT™ providing evidence that FINGERPRINT™ was used to make this dripline. Usually as PE ages its density slightly increases, but the DSC measurements of the failed and control driplines relative to the FINGERPRINT™ control do not indicate this, suggesting this material aged very well, perhaps due to the quality of the resin, it's AO package and the protection from UV light afforded it by being buried in the soil.

Table 2. Summary of DSC melting and crystallization temperature measurements.

	Tm (°C)	Tc (°C)
Control dripline (24-1)	119.0	108.3
Failed dripline (24-2)	119.4	108.8
FINGERPRINT™ Control (24-3)	119.0	107.7

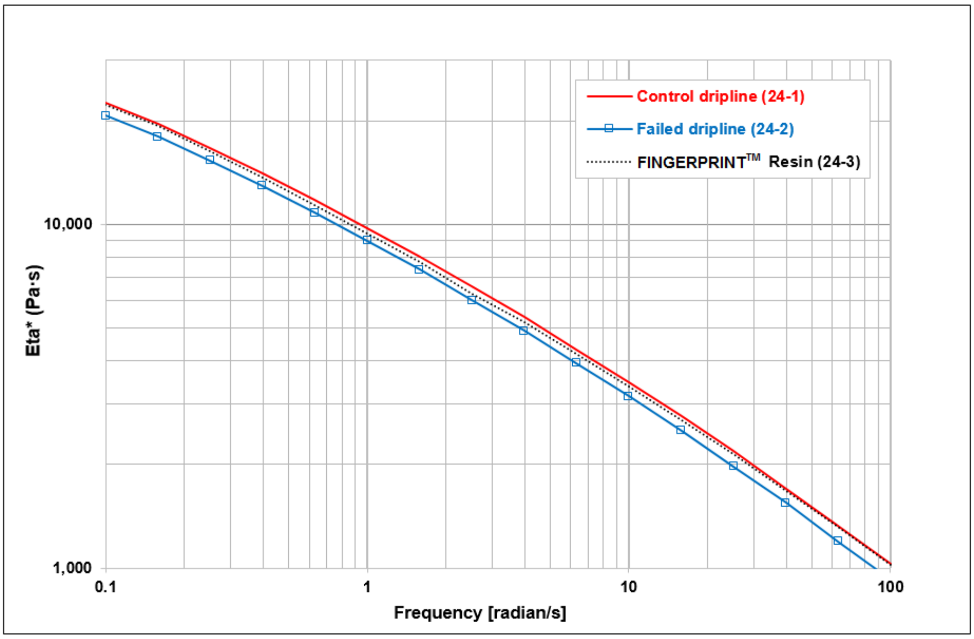
Figure 3 contains a plot of the DSC second heat melting curve comparing the control dripline, failed dripline and the FINGERPRINT™ resin. This plot illustrates the equivalent thermal characteristics of all three materials.

Figure 3. DSC Second Heat Melting Curve Comparison.



DMS rheology was performed on the materials and the viscosity curves are summarized in Figure 4. The overall viscosity of the material in the failed dripline (24-2) is about 7% lower than FINGERPRINT™ in the low shear region and about 10% lower in the high shear region. Typically the viscosity of carbon black containing polyethylene is greater than polyethylene absent of carbon black. The control dripline (24-1) which was never put in service has a viscosity profile very close to that of FINGERPRINT™. This difference is likely due to normal variation during the process of extruding the dripline, and doesn't necessarily indicate that there is degradation by chain scission.

Figure 4. DMS Viscosity Overlay at 190°C.



Standard resin characterization tests were performed on the control and failed driplines along with FINGERPRINT™ resin and results are summarized in Table 3. The failed dripline measured density is greater than the Fingeprint™ resin because it contains 2.3% carbon black. The measured melt index of the failed dripline is greater than the FINGERPRINT™, which suggests it has an overall lower molecular weight since typically the addition of carbon black would yield a lower melt index, more viscous, product. Carbon black levels in both the failed dripline and control dripline were within the expected range of 2.0-3.0 wt% and residue levels were extremely low at 0.05 wt%, indicating the carbon black used was very clean and no fillers were used in the manufacturing of the dripline.

Table 3. Density, Flow Rate and Carbon Black Measurements on the dripline samples.

	Density (g/cm^3)	Melt Index (dg/min)	Flow Index at 190°C, 21.6kg (dg/min)	MFR (I21/I2)	Carbon Black (wt%)	Inorganic Residue (wt%)
Failed dripline (24-2)	0.934	0.68	49.3	72.5	2.33	0.05
FINGERPRINT™ resin (24-3)	0.922	0.53	45.8	86.4	0	0
Control dripline (24-1)	Not Measured	Not Measured	Not Measured	Not Measured	2.37	0.05

Additive type and levels were determined in the failed dripline indicating that there were still 46 ppm of active secondary antioxidant in the dripline, However all of the primary AO had been oxidized.

Environmental stress crack resistance (ESCR) properties were measured on compression molded specimens created with material from the 26 year old dripline. The method used is described in ASTM D-1693 and specified in ASAE S553.[iv] The ASAE S553 standard requires the material used in the dripline to have an ESCR greater than 1,000 hours using condition A (regardless of the density of the material) at 50°C. In this case, the ESCR of the 26 year old material was measured to be greater than 1,000 hours. Test specimens were taken off test at 1,000 hours and not allowed to go to failure. This result provides evidence that the bulk material in the dripline continued to be of very high quality, even after being in service for 26 years.

Tensile properties of the walls of the dripline were characterized and results are summarized in Table 4. Both the failed dripline and control dripline remained strong even after 26 years. Both driplines exhibited equivalent strain behavior. The control dripline exhibited about 400 psi greater yield stress and 300 psi greater break stress than the failed dripline. In general, the failed dripline exhibited strain at break in the 260 to 350% range. Two of the control driplines exhibited results in the 325% range, but three of them were quite a bit less in the 140 to 200% range. Overall, the failed dripline exhibited relatively consistent tensile performance, suggesting it remained strong even after 26 years of service.

Table 4. Micro tensile result summary of Control (24-1) and Failed (24-2) driplines.

	Failed dripline (24-2)	Control dripline (24-1)
Stress at Yield (psi)	2,030	2,598
Strain at Yield (%)	13	13
Stress at Break (psi)	3,185	3,520
Strain at Break (%)	280	223
Peak Load (lbf)	8.5	9.6

The characterization performed confirms that the failed dripline was fabricated with a Dow FINGERPRINT™ resin. The dripline contained the tracer used in FINGERPRINT™ products at a level consistent with what had been using since the introduction of the products in the market. Thermal characteristics of the dripline are comparable to FINGERPRINT™, with an equivalent melting shoulder at around 110°C and peak at around 119°C. Rheological characterization indicates the material used in the dripline has a lower overall viscosity than FINGERPRINT™. This may be due to the fact that the comparison was made between an extruded dripline and natural resin. Residue levels were less than 0.1 wt%, suggesting the carbon black used was very clean and no foreign material such as recycle was used in the fabrication of the dripline. Additive characterization indicates the failed dripline contained 46ppm of active antioxidant, suggesting the antioxidant package in the dripline had held up extremely well over the years.

Failure Analysis

Although dripline failures occurred along a crease the overall dripline remained pliable and visual evidence of degradation did not exist[12]. Figure 5 includes a cross section of the failed dripline along with a cross section of the control dripline used, with the red arrow indicating where the dripline failed. This area is where the dripline is creased as it is rolled onto the spool after being fabricated. It remains on the spool in a collapsed state until it is installed in the field.

Figure 5. Cross sections of failed and control dripline samples.

Failed dripline Cross Section (24-2) | Control dripline Cross Section (24-1)

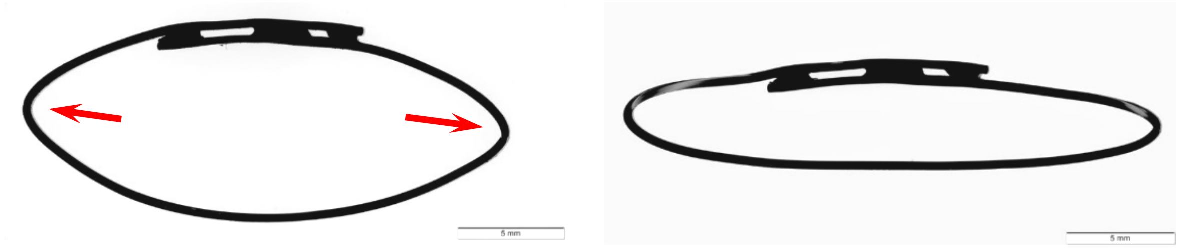


Figure 6 contains dripline cross sectional optical microscopy images taken at the failure region of the dripline. Surface striations were present along the inner surface of a control dripline which followed edge creases (blue arrow). These inner surface striations acted as crack initiation sites for the failures as shown by the internal cracks (red arrows) in the failed in-service dripline.

Figure 6. Cross sectional optical microscopy images of failure region in both Failed (24-2) and Control (24-1) dripline Specimens.

Failed dripline Cross Section (24-2) | Control dripline Cross Section (24-1)

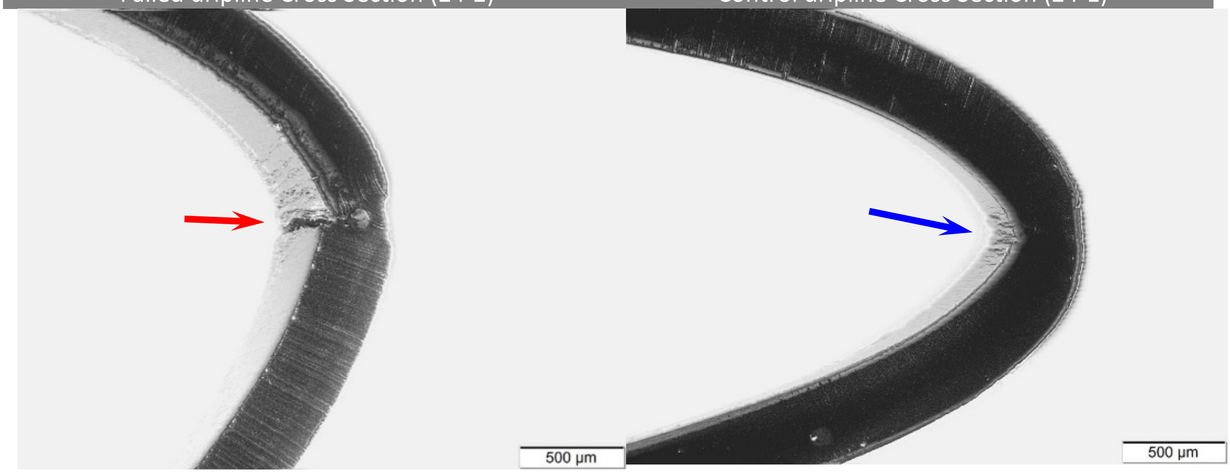


Figure 7 contains higher magnification views of the cross section of the failure region of both the failed and control driplines. Surface striations were present along the inner surface of a control dripline which followed the edge crease (blue arrows). Outer surface deformations were also observed in the control dripline. Sharp internal cracks (red arrows) were present on the inside surface of the failed dripline in the crease region, but not on the outside surface of the dripline. Crack penetration was approximately 40um along the inner dripline surface.

Figure 7. Cross sectional optical microscopy images of failure region in both failed (24-2) and control (24-1) dripline specimens

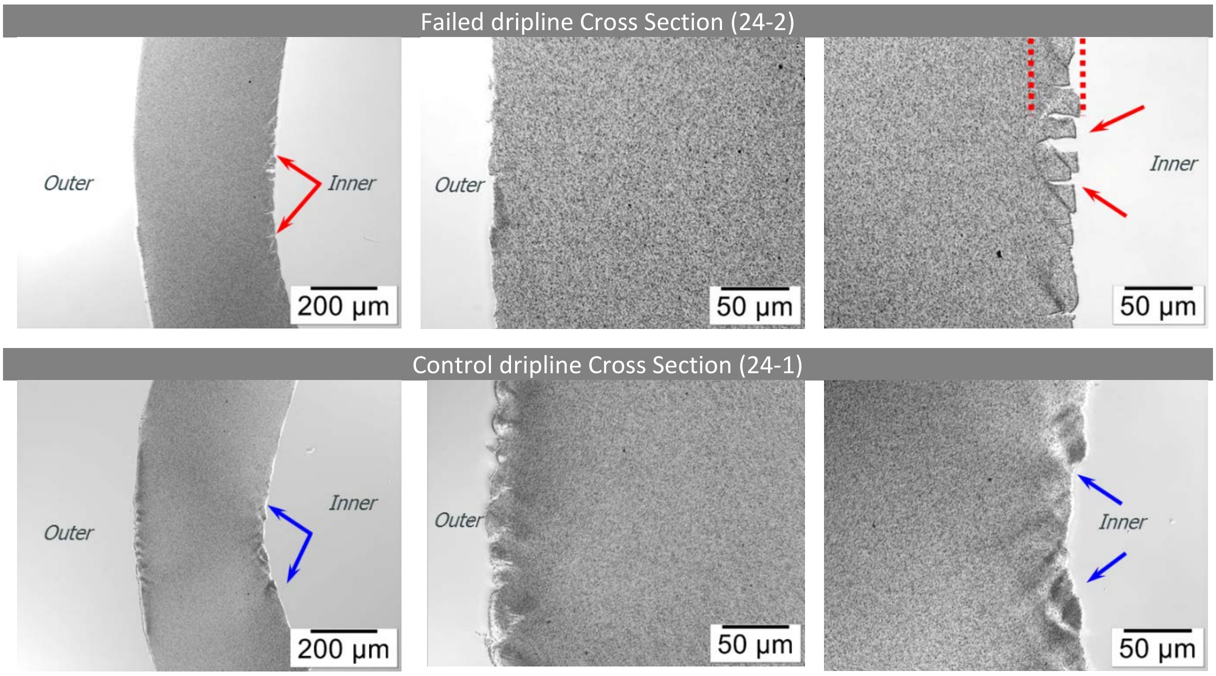
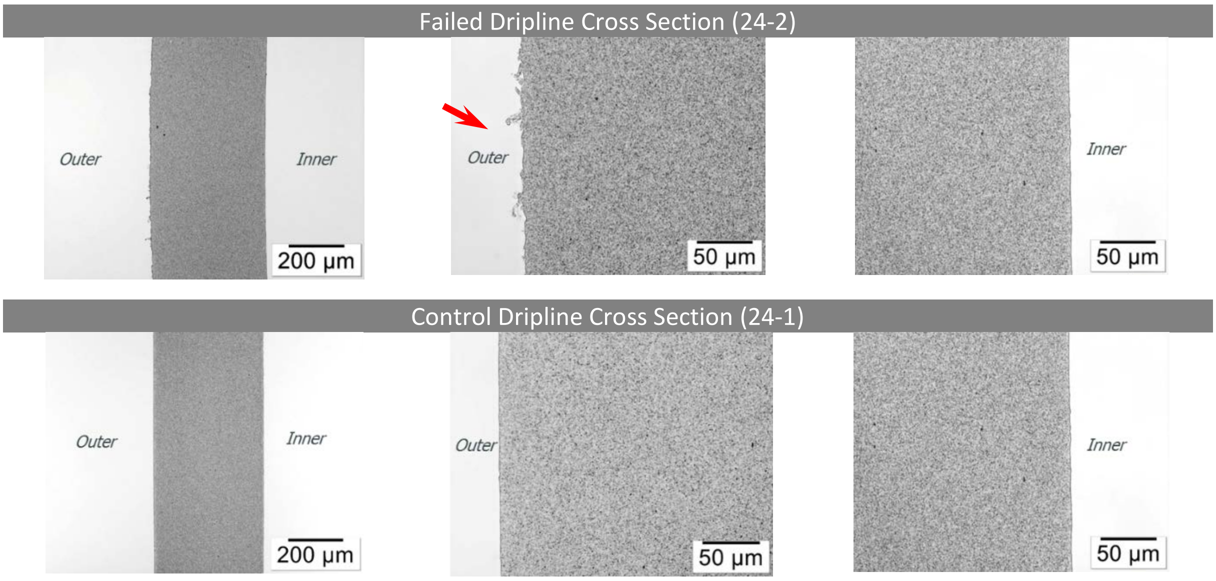


Figure 8 contains cross sectional images of the flat region of both failed and control driplines. The internal surface of both the failed and control driplines in areas away from the crease were absent of any striations, cracks or defects, suggesting the strain in the crease area coupled with exposure to the liquid medium pumped through the dripline over the years led to the failure of the dripline in the failed dripline sample which is expected. Evidence of dripline degradation or cracking was not observed in areas away from the crease. Prior studies have shown an increase in the rate of degradation of polyethylene when a constant strain has been applied.[v] Slight abrasion was observed (indicated by red arrow in Figure 8) along the outer surface of the failed dripline, but is not present on the inner surface of the failed dripline. This abrasion likely occurred during the installation of the dripline because it is not present in the control dripline. There was no evidence of failures initiating from the slightly abraded surface of the failed dripline. This abrasion must have occurred during installation or normal use over time.

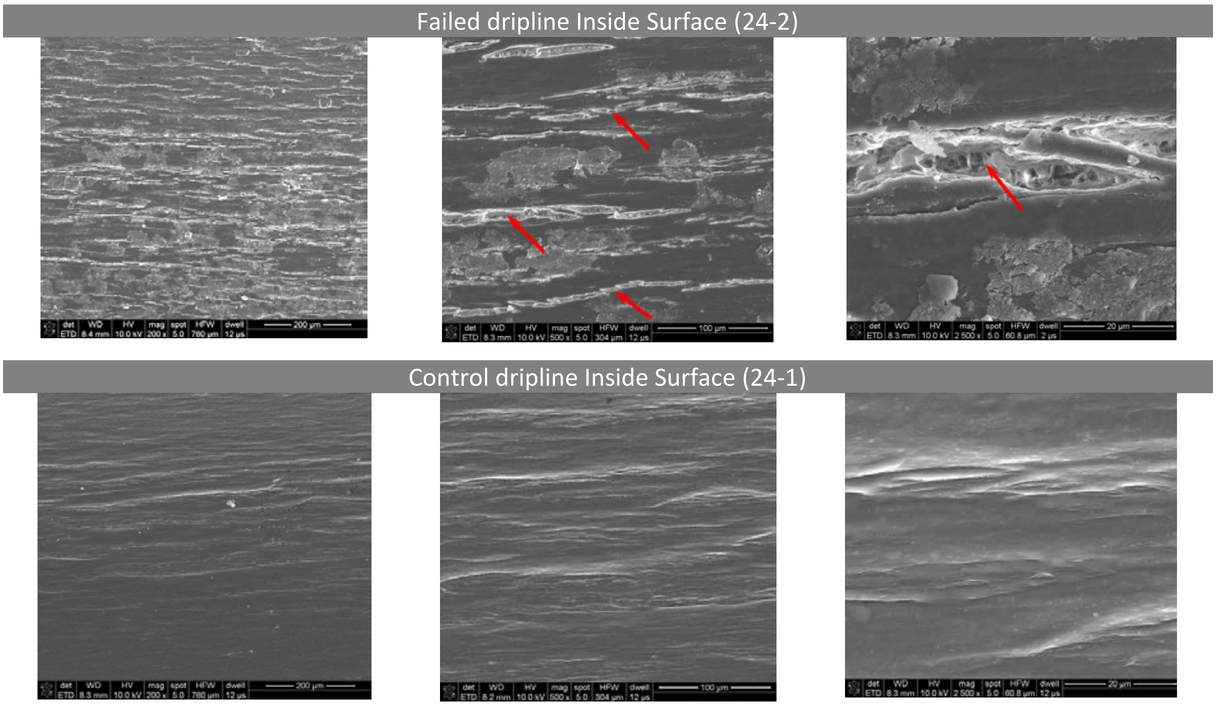
Figure 8. Cross sectional optical microscopy images of flat (non-failure) region in both failed (24-2) and control (24-1) dripline specimens



A review of cross section images shown in Figure 7 and Figure 8 indicate remarkable level of carbon black dispersion and homogeneity by today's standards in dripline material used.

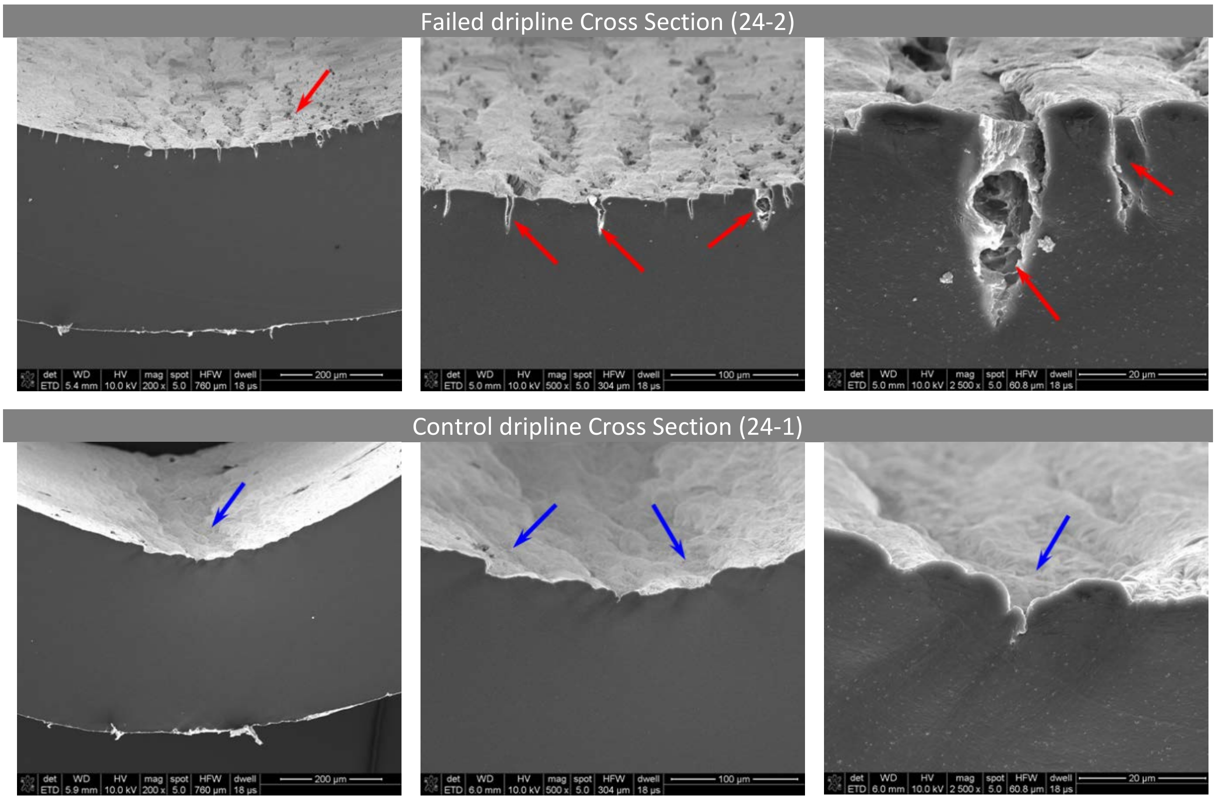
An evaluation of the inner dripline surfaces showed parallel striations were present along edge creases, as shown in optical cross sections. Figure 9 contains scanning electron microscope (SEM) images of the inner surface of the crease region in the failed and control dripline specimens. Surface cracks were not observed along creases in the control dripline. Sharp parallel cracks (red arrows) were present along the creases of the failed dripline.

Figure 9. SEM Images of Inner Surface of Crease Region in both Failed and Control Dripline Specimens.



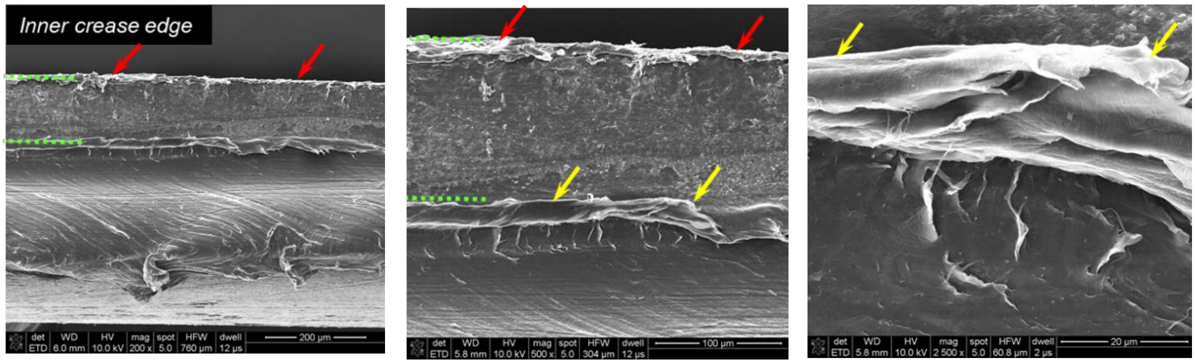
Comparison of the inner surface of the crease region of the dripline using SEM shows the surface striations present in the control driplines, shown in Figure 10 and highlighted with blue arrows. The cracks on the inner surface crease region are also shown in the failed dripline (red arrows), with cracks penetrating approximately 40um into the wall of the dripline.

Figure 10. SEM cross section images of crease region.



Examination of a fracture surface of the failed dripline showed that failure had initiated at the inner surface of the crease, inside the dripline, and propagated outward. The red arrows in Figure 11 indicate where the fracture initiated. A fracture surface associated with brittle failure was observed to a depth of approximately 125um or 5 mils (indicated by the area between the green dotted lines). This region was associated with parallel striations which formed sharp linear surface cracks along the dripline crease. Beyond the initial brittle fracture, a ductile shear lip was present (indicated by the yellow arrows in Figure 11) which extended to the outer dripline surface. Evidence of ductile tearing was observed beyond the initial brittle fracture zone.

Figure 11. SEM Images of fracture surface along crease of failed dripline (24-2)



Oxidation induction time (OIT) was measured at the fracture and flat locations of the failed dripline as well as at the flat region of the control dripline. Specimens were carefully cut using scissors. Due to the relatively low thickness of the dripline (15mils) and amount needed for the OIT measurement, the specimen taken at the fracture was a cross sectional specimen representing both the inside, middle and outer areas in the fracture region. A linear direct relationship exists between phenolic concentration and OIT, so this technique can be used to assess the relative amount of active phenolic antioxidant present.[vi] The results are shown in Table 5. A greater concentration of phenolics were present away from the fracture versus at the fracture, suggesting the antioxidants were either consumed or extracted from the dripline wall at a greater rate in the crease region.

Table 5. Oxygen Induction Time of failed (24-2) and control (24-1) dripline specimens.

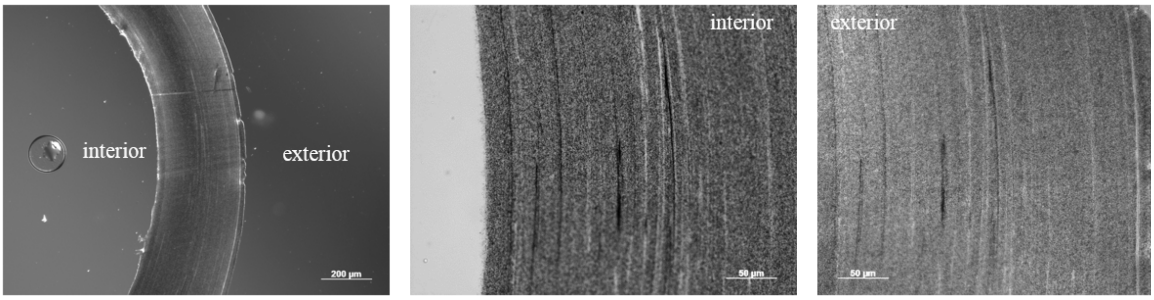
Description	OIT at 200°C (min)
Specimen Near Fracture of Failed Dripline (24-2)	1.0
Specimen on flat region of failed dripline (24-2)	1.3
Specimen on flat region of control dripline (24-1)	3.3

It is noted that the fracture surface was found to be extremely brittle, as shown in Figure 11, and it is therefore unlikely any antioxidants were present in this brittle area. The material in the crease region had reached stage 3 of the classical chemical degradation model for polyethylene, the stage at which all antioxidants are depleted, chemical degradation of the polymer is occurring, ultimately leading to an engineering failure.[vii]

It is concluded that the dripline likely failed due to the strain in the crease area coupled with exposure to the liquid medium pumped through the dripline during its lifetime. Evidence of dripline degradation or cracking was not observed in areas away from the crease, nor was any evidence of degradation found on the outside of the dripline. The overall dripline held up extremely well over its 26 year life, suggesting if the dripline had not contained a crease it likely would still be in service today.

Analysis of a dripline manufactured in 2017 with Dow's FINGERPRINT™ resin was done. The results are shown in Figure 12 below. The pictures show that the dripline does not have striations on the crease, there is no evidence of manufacturing deficiencies. Therefore the striations observed on the 26 year old dripline which initiated the failure likely appeared over time due to the depletion of the AO and localized compressive stress in the fold area during its lifetime. That is why the control tape also presented the striations, but didn't fail due to the fact that was not in use. This is the same issue any collapsible/folded tape would have.

Figure 12. Cross sectional optical microscopy images of Dripline made in 2017 with 100% Dow FINGERPRINT™ Resin



CONCLUSIONS

The failed dripline was fabricated with Dow FINGERPRINT™ resin. Thermal characteristics of the dripline are comparable to Dow's FINGERPRINT™ resin, with an equivalent melting shoulder at around 110^{o}C and peak at around 119^{o}C. Rheological characterization indicates the material used in the dripline has a lower overall viscosity than Dow's FINGERPRINT™ resin. This may be due to the fact that the comparison was made between an extruded dripline and natural resin. Residue levels were less than 0.1 wt%, suggesting the carbon black used was very clean and no foreign material such as recycle was used in the fabrication of the dripline. Additive characterization indicates the failed dripline still contained some 46 ppm of active antioxidant, suggesting the antioxidant package in the dripline had held up well over the years.

ESCR was measured to be greater than 1,000 hours on the failed dripline. Even after 26 years of service the material used to fabricate the dripline still met ASAE S553 dripline standard.

It is concluded that the dripline likely failed due to the strain in the crease region coupled with exposure to the liquid medium pumped through the dripline during its lifetime. Evidence of dripline degradation or cracking was not observed in areas away from the crease, nor was any evidence of degradation found on the outside of the dripline. The overall dripline held up well over its 26 year life, suggesting if the dripline had not contained a crease it would likely still be in service today.

There is no evidence of manufacturing deficiencies that could have been the cause of the failure of the 26 year old dripline. The striations that initiated the failure likely appeared over time due to the localized compressive stress in the fold area during its lifetime. This is the same issue any collapsible/folded tape would have.

RECOMMENDATIONS/FUTURE WORK

As next generation products are developed to enable thinner wall dripline for SDI applications, understanding the impact of strain on antioxidant depletion and the degradation of polyethylene in a water environment could be useful. Few studies have been reported and published literature regarding this topic. The importance of using a clean resin and masterbatch is valuable and the advantages include higher performance dripline that will last far longer.

ACKNOWLEDGMENTS

Thanks to Babli Kapur, Rajen Patel, Cosme Llop, David Gillespie, Teresa Karjala and Kalyan Sehanobish for reviewing this case and sharing insight into the cause of this failure.

REFERENCES

---

[i] Lamm, Freddie R., Danny H. Rogers, "*SDI, The Basics of Successful Systems*", Proceedings of the 24th Annual Central Plains Irrigation Conference; Colby, Kansas; February 21-22, 2012.

[ii] Kansas State University Research and Extension Website: http://www.k-state.edu/sdi/

[iii] United States Patent 4,572,756 titled Drip Irrigation System Employing Adjacently Arranged Flow-Restricting Passages; Richard D. Chapin; January 16, 1985.

[iv] ASAE S553 Standard "Collapsible Emitting Hose (Dripline) – Specifications and Performance Testing" December 2000 (R2012).

[v] Llop, Cosme, Antonio Manrique, "*Cast Silage Wrap Film UV Evaluation*" LAIT-4032; October 22, 1990; CRI 907151.

[vi] Iten, Cicile, Jaime Setien, Carlos Alonso, Bernard Fehr, Cosme Llop, *"Oxygen Induction Time (OIT): What it really measures on Dow's polyethylene?"* LACAT 2006-001; June 22, 2006, CRI 2006004035.

[vii] Ewais, A.M.R., R. Kerry Rowe, "Effect of again on the stress crack resistance of an HDPE geomembrane" Polymer Degradation and Stability 109 (2014) 194-208.

[12] Garcia-Meitin, Eddy, "Failure Analysis of 26 Year Old Drip Tape Made from Union Carbide Resin at Chapin (Jain)", May 13, 2016; Analytical Report AL-2016-001783.