University of Victoria
Engineering & Computer Science Co-op
Work Term Report
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SMEFA LEAKY ANALYSIS

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6th August 2021

In partial fulfillment of the academic requirements of this co-op term

Supervisor's Approval: To be completed by Co-op Employer

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Signature:____________________ Position: Manufacturing Engineering Date: 6th August2020

Name (print): Dr. Taryn Biggs E-Mail: tarynbiggs@cellcentric.net

For (Company Name): Cellcentric

Letter of Transmittal

Cellcentric Canada
4343 North Fraser Way
Burnaby, Canada, V5J 5K7

August 6th 2021

Dear Dr. Taryn,

Please accept the attached report entitled “SMEFA LEAKY ANALYSIS”.

The following report is the result of work at Cellcentric Canada during my second co-op term of my engineering studies at the University of Victoria. Fuel Cell stack development and production is one of the DTFCC’s programs for the next generation of emission free Trucks. Alongside with the development of prototype fuel cell stacks with a focus on reduced costs and increased performance.

This report discusses SMEFA leaky issues and its analysis. Root cause analysis has been presented along with its detailed analysis using Keyence and SEM. Process of Bubble jig has been also introduced which was used to observe leak location in SMEFA.

Please note this report contains confidential information of Cellcentric Canada. Therefore, creating copies of the report, including electronic copies is prohibited and any exceptions must be approved by Cellcentric Canada.

I would like to thank you for your help and patience during my work term, as well as your knowledge and advice on the manufacturing engineering.

Sincerely,

____

Sanjeev Kumar

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Abstract

Fuel cell electric vehicles (FCEVs) are powered by hydrogen and offer a promising approach to zero-emission driving, especially for heavy-duty commercial vehicles. FCEVs are more efficient than conventional internal combustion engine vehicles and produce no tailpipe emissions; they only emit water vapor and warm air. Besides the lack of hydrogen infrastructures around the cities, the cost of high-volume **production** and durability are the major challenges to fuel cell commercialization. The gas diffusion layer (GDL) is an important component to the proton exchange membrane fuel cell (PEMFC). It has to direct the flow of electrons to an external circuit as well as distribute hydrogen and oxygen gas evenly across the electrode surfaces. The current GDL supplier of the MBFC UC360 stacks is Toray. This report gives overview and analysis of leaky SMEFA issues, which is very critical for better performance of fuel cell. Bubble jig procedure and SEM methodologies were employed in order to study analyze leaky MEFA issue.

Glossary

BEV – Battery electric vehicle

DTFCC – Daimler Truck Fuel Cell Canada

EV – Electric vehicle

FCEV – Fuel cell electric vehicle

GHG – Greenhouse gas

MEA – Membrane electrode assembly

MEFA – Membrane electrode frame assembly

PEMFC – Proton exchange membrane fuel cell

SMEFA – Sealed membrane electrode assembly

CCM – Catalyst Coated Membrane

CFP – Carbon Fiber Paper

CFCP – Common Fuel Cell Program

CMD – Cross Machine Direction

GDL – Gas Diffusion Layer

MBFC – Mercedes-Benz Fuel Cell

MD – Machine Direction

MEA – Membrane Electrode Assembly

MPL – Micro Porous Layer

PEM – Proton Exchange Membrane

UC360 – Unit Cell 360

1 Introduction

1.1 Membrane electrode assembly

In proton exchange membrane fuel cells (PEMFC), the electrochemical reactions that generate electrical power occur in the membrane electrode assembly (MEA). The standard MEA construction, shown in Figure 1 , contains a proton exchange membrane (PEM) in between two catalyst layers all in between two gas diffusion layers (GDL). These components facilitate the reaction between hydrogen and oxygen gas as well as directing the flow of electrons to external circuit. The GDL has to be made of a material that not only evenly distributes the reactive gases across the electrode surfaces but also direct the flow of electrons to the external circuit [2]. The best material that fits these requirements is carbon fiber so the GDL is primarily made of carbon fiber paper (CFP). Carbon fiber has the specific porous, hydrophobic and conductive properties that the GDL needs. GDL also provides the support structure for the MEA; it provides it rigidity and stability alongside the PEM. However, the GDL itself is composed of two layers; the aforementioned CFP and the microporous layer (MPL). Although GDL is composed almost entirely of CFP, the MPL is a thin layer that makes contact with the catalyst-coated membrane (CCM). The main purpose of the MPL is to protect the PEM from protruding carbon fibers.

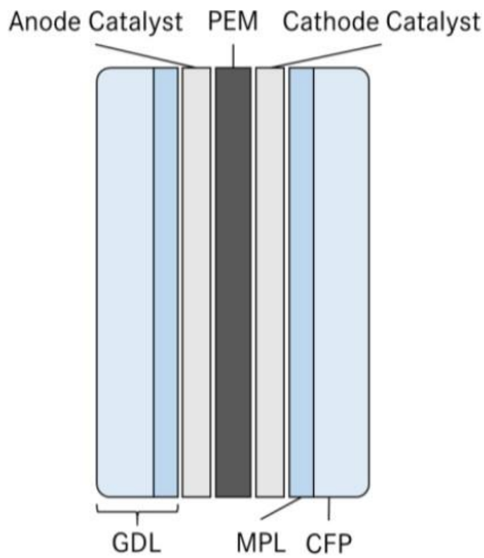


Figure 1 MEA , labeled with the different parts**.**

The PEM is situated between two catalyst layers and GDL. The GDL itself is composed of two layers, the MPL and CFP[4].

Hydrogen is supplied from the anode side of the cell, and later it is catalytically split into protons and electrons according to the following reaction:

$$\boldsymbol{H_2 \rightarrow 2H^+ + 2e^-}$$

Atmospheric oxygen is supplied from the air into the cathode side. Afterwards, due to the potential difference between anode and cathode sides, electrons flow as an electrical current to load the required power for automotive applications (cell stacking increases the

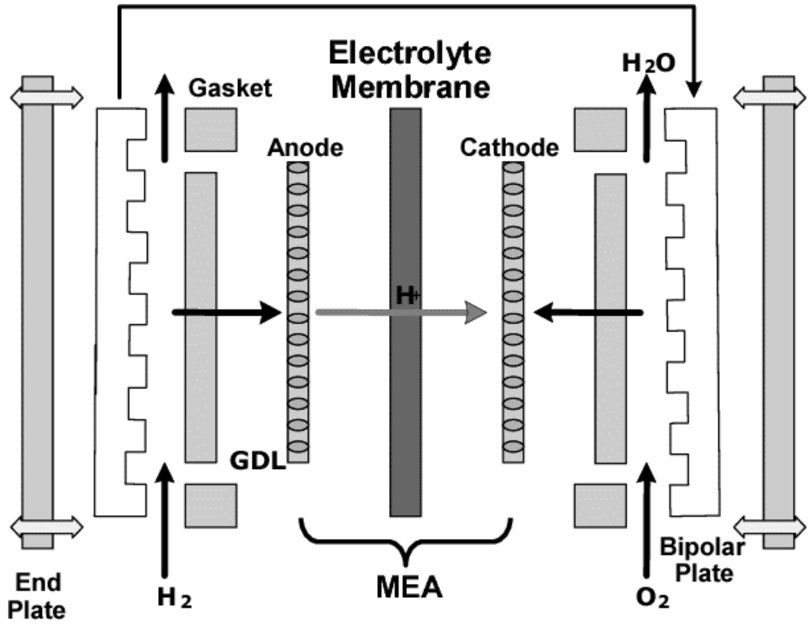


Figure 2 Overview of PEMFC Structure.

provided voltage, can be seen in Figure 2). At the same time the protons diffuse through the membrane. The oxygen molecule is catalytically split at the cathode side into the oxygen ions so that they can connect with two protons to form a water molecule based on the following reaction:

$$\mathbf{1/2\ \boldsymbol{O_2} + 2\boldsymbol{e^-} + 2\boldsymbol{H^+} \rightarrow\ \boldsymbol{H_2O}}$$

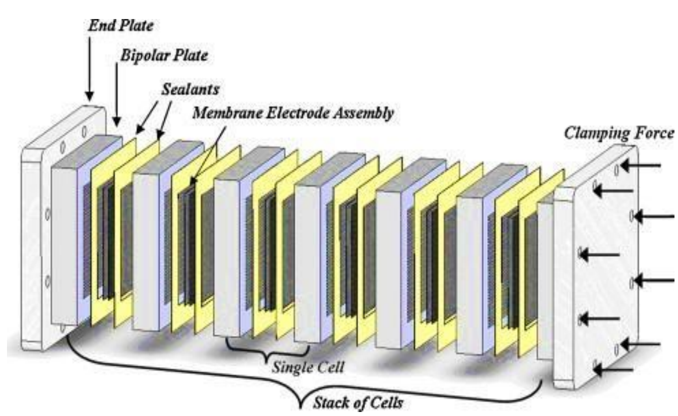


Figure 3 PEMFC Stack design

The water is carried out of the fuel cell by the hot airflow, which leads to exhaust water vapor at the end.

1.2 Working

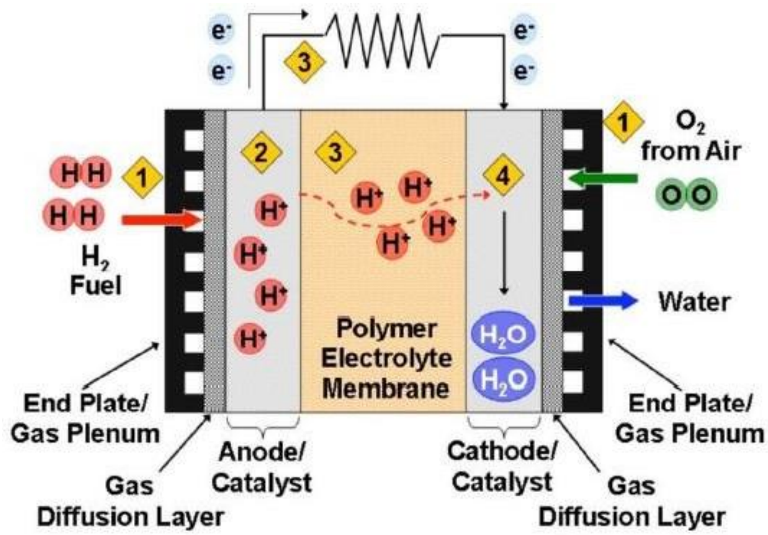


Figure 4 Schematic diagram of PEMFC

The functional principle of a fuel cell is shown schematically in Figure 4. The incoming hydrogen is supplied from the anode side and oxygen from the atmosphere from the cathode side (1). Hydrogen molecules (H2) are split by a platinum catalyst into two protons (2) diffusing through the membrane, while the electrons flow to the load (3). Electrical voltage is generated due to the potential difference between anode and cathode. The voltage of several cells can be combined by stacking them up. At the cathode side, the oxygen molecules (O2) are split up into two ions because of the acceptance of four electrons. Every oxygen ion connects with two protons forming a water molecule (4).

1.3 Production Environment at MBFC

The current production line for the common fuel cell program (CFCP) is located in an ISO 8 classified cleanroom. Maximum allowable particles per cubic meter and air changes per hour are determined by the classification. The cleanroom layout has a size of approximately 1,000 m^2 (16m x 63m), shown in Figure 5.

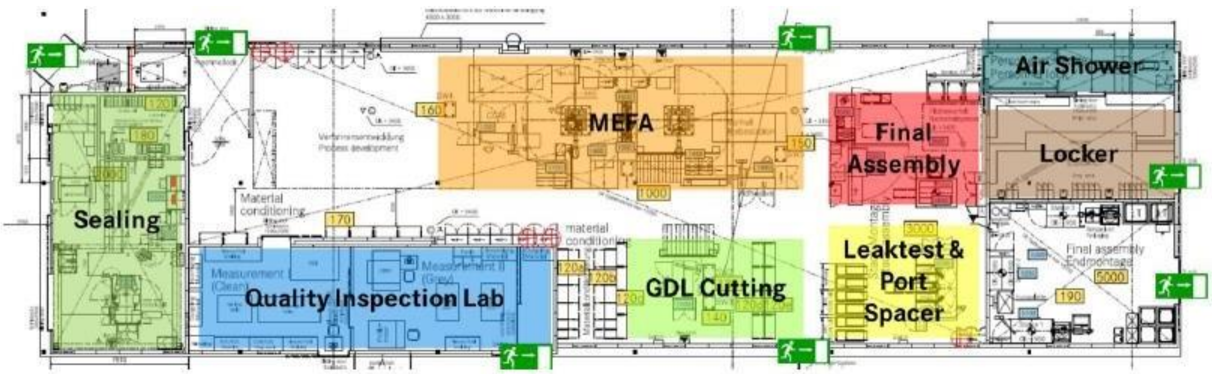


Figure 5 Cleanroom Layout MBFC

2 Leak in SMEFA

2.1 AIM

The leak in SMEFA cell causes unbalanced flow of oxygen and hydrogen while operation that results in poor performance of cell in stack assembly. Thus, it is extremely important to minimize such leaks in both inlet and outlet side of SMEFA. The leak/hole in the active region is major concern as it affects the properties and performance of fuel cell during operation. Before stacking 15 cell leak testing is done in order to identify the leak in SMEFA if it exits on inlet or outlet side.

2.2 Testing Method

As the leak/hole is very small in magnitude and it is very difficult to observe and locate it normally. In order to overcome this challenge Bubble jig experiment (explained in subsequent sections) is performed and which gives the idea about location of hole in SMEFA.

Once hole/leak location has been located in SMEFA, further analysis about its characteristic of hole can be found and analyzed using Keyence microscope and SEM (Electron microscope). Based on the data available and various possibilities IS/IS NOT tabulation was done along with fish bone diagram analysis.

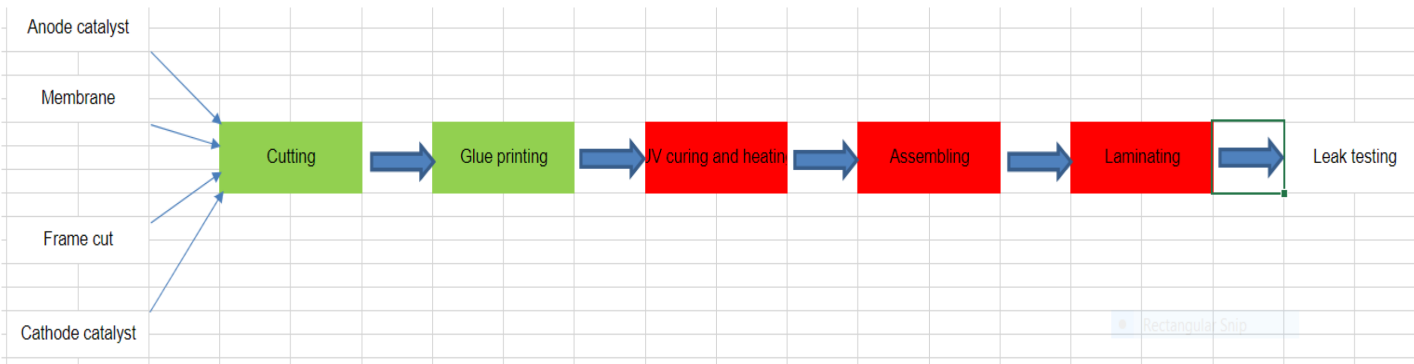


Figure 6 Process overview

2.3 BUBBLE JIG TEST PROCEDURE

Working procedure:

- Safety equipment - safety glasses and steel toe shoes required. Ensure the area is clear of hazards and items that may be damaged by water. Ensure absorbent cloths are nearby to clean up water.
- Inspect equipment before use and note any signs of wear, especially on the tubing or valves.
- Install the tubing mainfolds in the push-to-connect fittings on the top plate. Ensure datum pins and seals are properly installed. Check that the "spacers" on the fasteners are installed.
- Place lower polycarbonate plate in the water
- Lower UC360 plate onto the polycarbonate plate in the water. Take care not to trap any bubbles under the plate (lower it into the water "at a slant"). Align with the datum pins.
- Connect the tubing to the pressure system, checking carefully for any other lines in the system that may be holding pressure, and that the valves are set correctly (Close all valves to ensure water does not escape from the circuit)
- Install the upper polycarbonate plate in the water, in the same slanted method to avoid trapping air bubbles. Take care that seals don't become dislodged in the process, and that the UC360 plate remains corner-crowded. Align to the datum pins.
- Install and tighten the knobs to secure the polycarbonate plates together.
- With inlet valves shut, increase the distribution pressure to 0.1bar (1.5psi)
- Ensuring that the pressure is set to 1.5psi, slowly open the inlet valves and outlet valves one at a time. This will purge the Cx circuit of water. This will purge the Cx circuit of water.

  (a) Close the Cx Out valve, pressurizing the Cx circuit. This will test for external leaks.

  (b) Open the Fx & Ox out valves. This will allow for air to leak from the Cx circuit if there are any cracks.

  (c) Increase pressure slowly to 10psi (0.7bar) constantly checking for leaks.

  (d) When a leak is observed note down pressure & location (square on grid, Fx or Ox side, or external leak).

- When observations are complete, shut the airflow and ensure that the outlet valve has been properly opened all the way to prevent trapped air.
- Unscrew the knobs to begin disassembly process. Dry all pieces before storing to prevent rust.

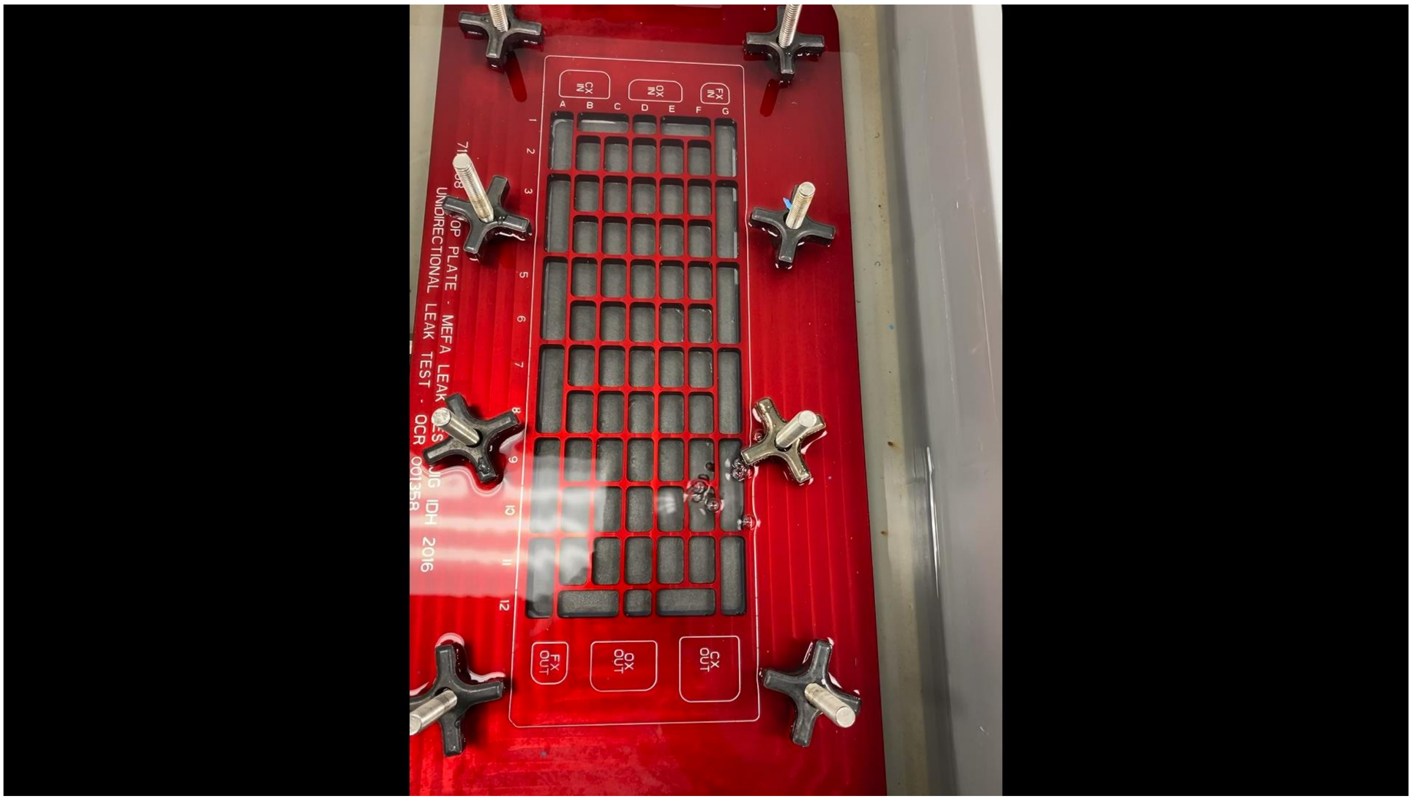


Figure 7 Bubble jig set up

3 ANALYSIS

3.1 Approach and overview

Different samples, which failed 15-cell leak testing after the production process were used for analysis. The details of samples are listed in the Table 1. Characteristics of properties and location of hole have been studied for different lots(for CCM) and its variation over the sample ids. After bubble jig , samples were analyzed under Keyence microscope and SEM. Short DMC is method of referring a date which isbeing in Julian calendar format and last three numbers denoting the sample Number.

MEFA produced	MEFA produced	Number of leaky MEFAS	Sequence	CCM lot	Found at Stacking
6 May 2021 (Thu)	21126	12	42-54	21-034-01	6 June (TBC)
14 May 2021 (Fri)	21134	26	71-98	21-040-01	16-17 June
17 May 2021 (Mon)	21137	46	43-88	21-040-01	17 June

Table 1 Samples for analysis

Table 2 shows the variation how samples are distributed for MEFA denotations and CCM lots. Different test, which are being performed, are shown for different samples. Bubble jig was first stage of testing for finding the location of hole in MEFA.

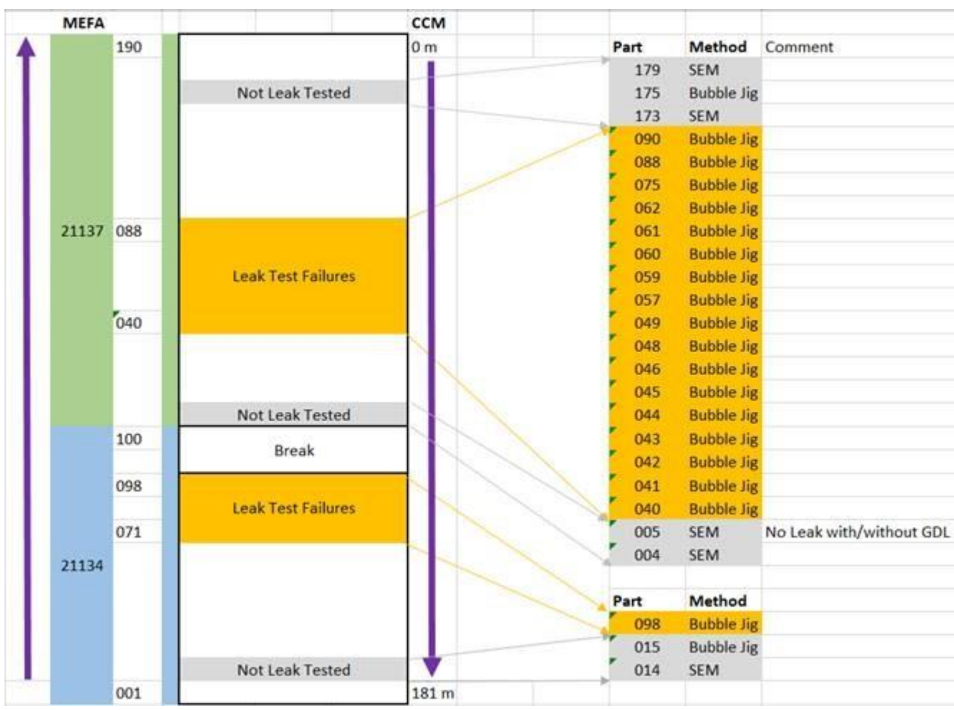
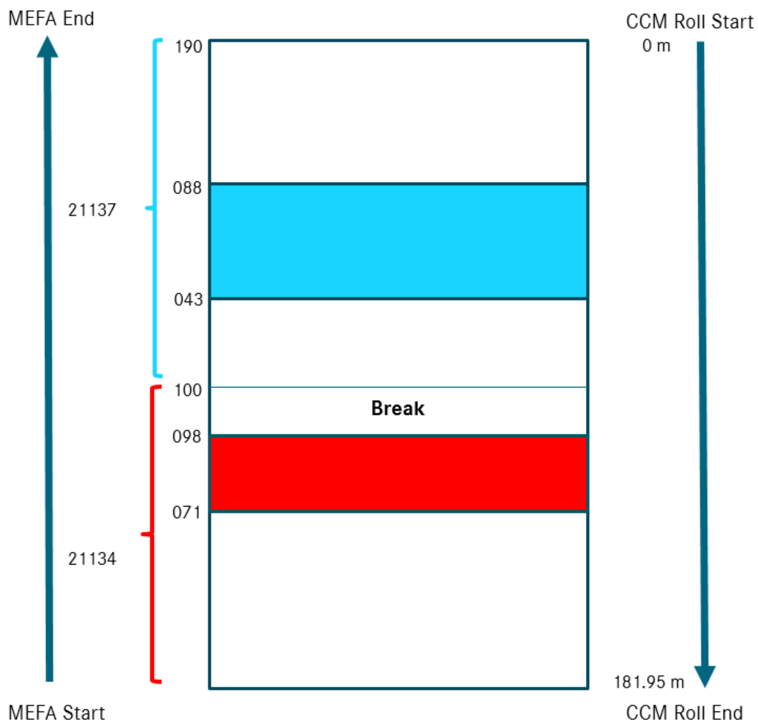


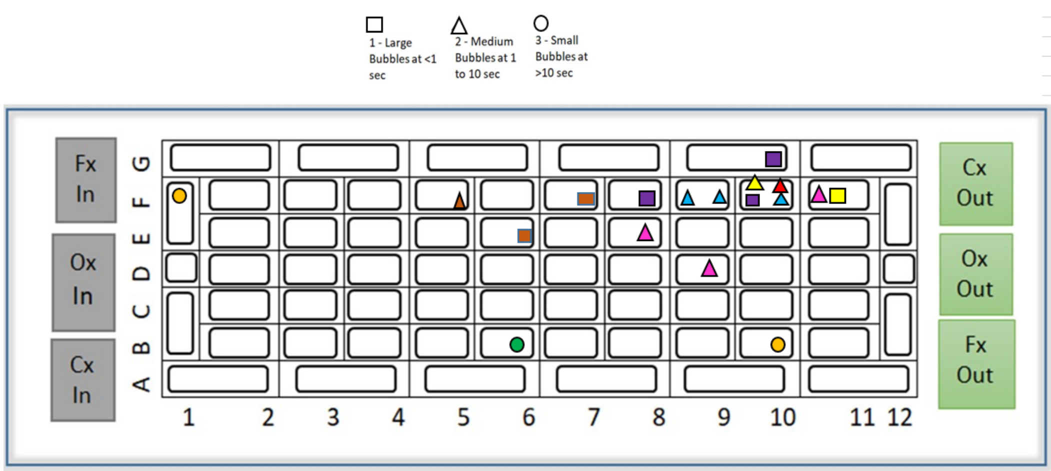
Table 2 Testing overview



- The map shows what sections of the CCM has leaks
- CCM roll is cut and used in reverse order
  - So the end of the CCM roll is used in earlier MEFA's
- The colored blocks on the map represent where leaks were found

Table 3 Map for CCM Rolls

BUBBLE JIG TESTING RESULTS



Samples	Color	Sample Number
SAMPLE 1		21126 042
SAMPLE 2		21126 047
SAMPLE 3		21126 051
SAMPLE 4		21126 050
SAMPLE 5		21126 045
SAMPLE 6		21126 054
SAMPLE 7		21126 049
SAMPLE 8		21126 046

Figure 8 Results of Bubble jig for different samples

From the results of bubble jig, it is clear that most of the leak locations were in the active region, which is evident from figure 7 showing the intensity of leak at different locations for different samples.

MEA information		
MEA	Leak Type	Leak Rate (ccm)
2113400072	Fuel-Air	0.5
2113400076	Fuel-Air	0.6
2113400080	Fuel-Air	0.5
2113400081	Fuel-Air	0.5
2113700066	N/A	N/A
2113700072	N/A	N/A
2113700073	N/A	N/A
2113700075	N/A	N/A
2113400077	Fuel-Air	0.75
2113700088	Fuel-Air	0.70

Figure 9 Leak rates for sample

3.2 Keyence microscope

Introduction

The VK-X100K/X200K Series uses a two-way light source comprised of a laser light source and a white light source.The two light sources together provide the colors, laser intensity and height information necessary to create deep field color images, laser intensity images, or height images (shown in figure 9).

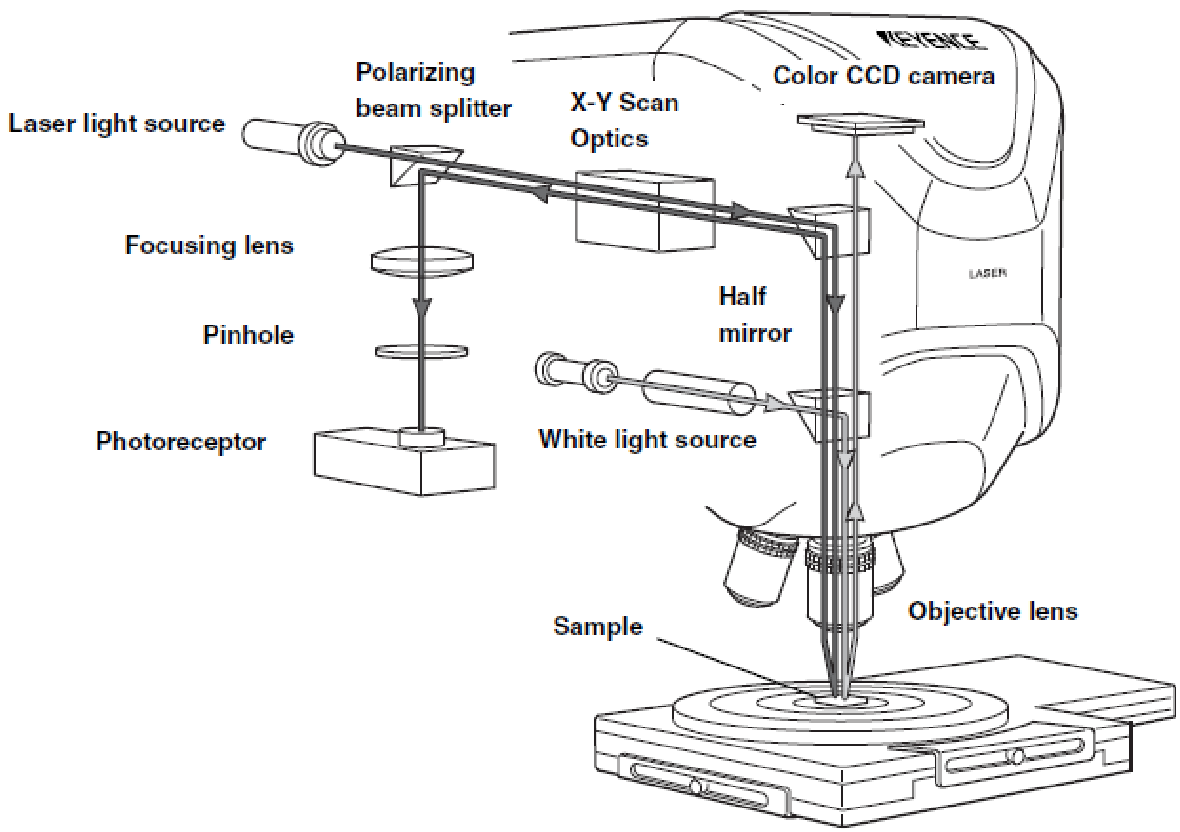


Figure 10 Keyence microscope

Sample 21137 043 : E10 (Location) large , Leak rate 0.8 with hole oriented in cross machine direction.

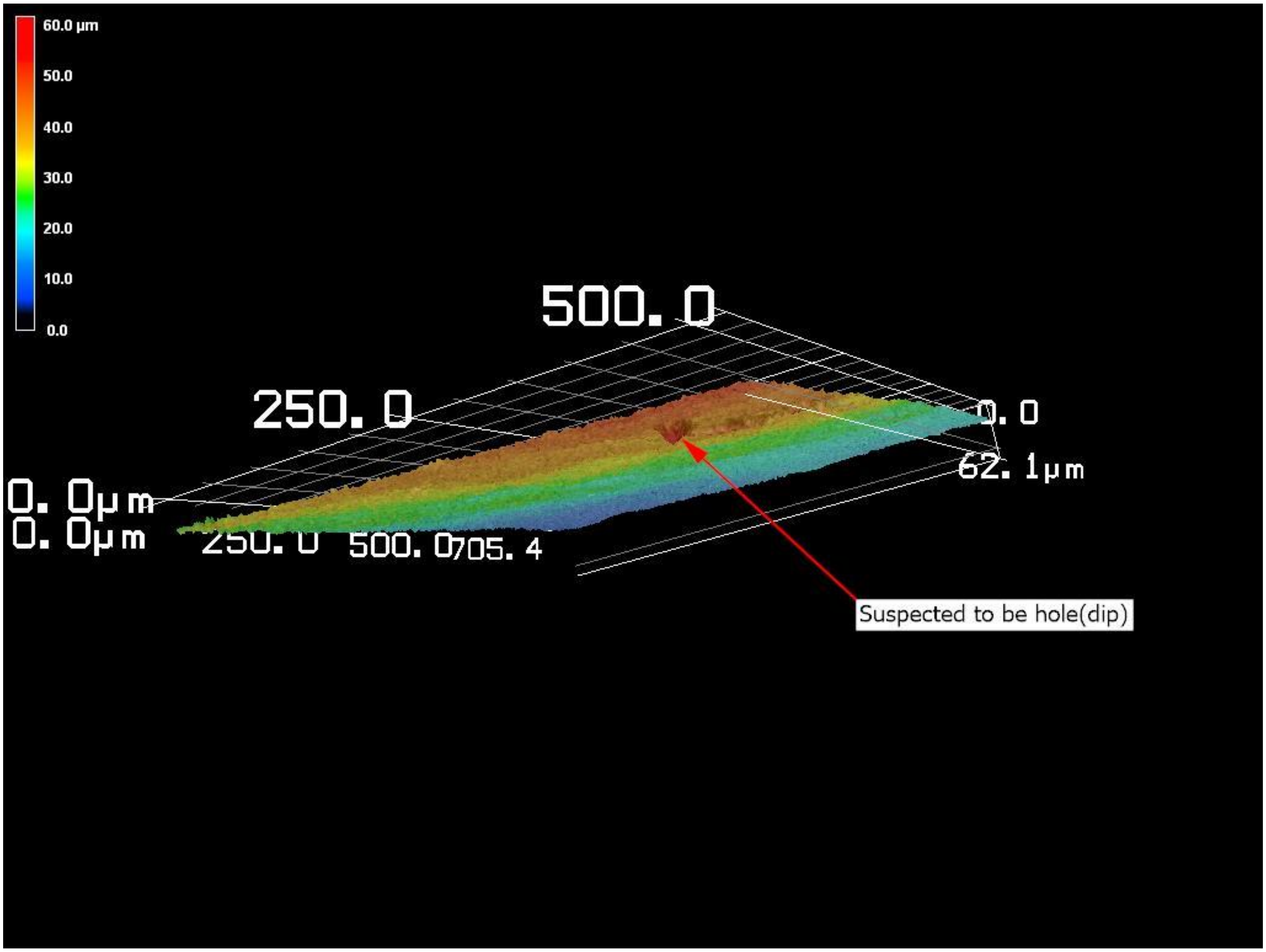


Figure 11 21137 sample 3D VIEW of location

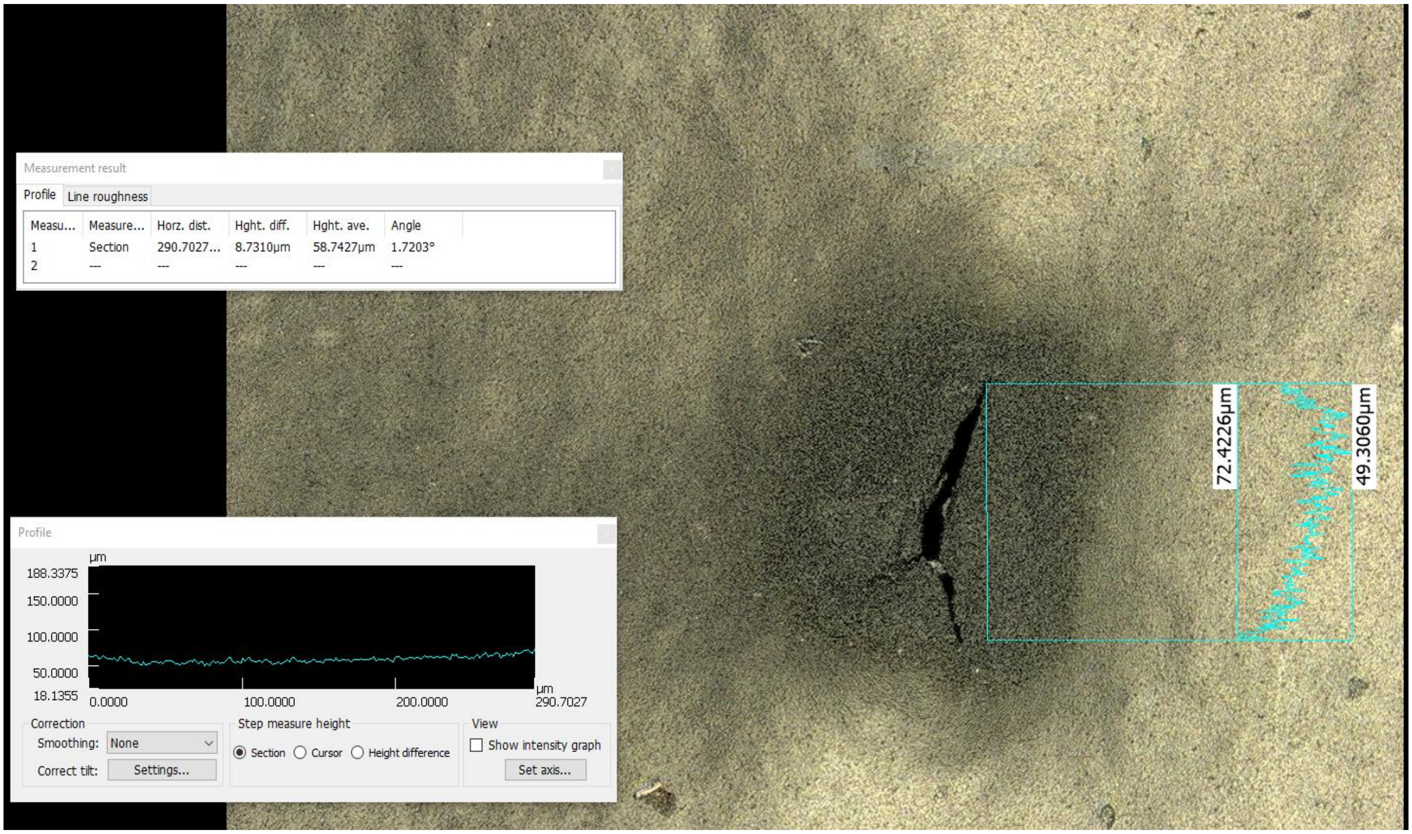


Figure 12 hole profile

Sample 21137 057 : F9(location) medium ,Cathode Side,hole orientation in Cross machine direction

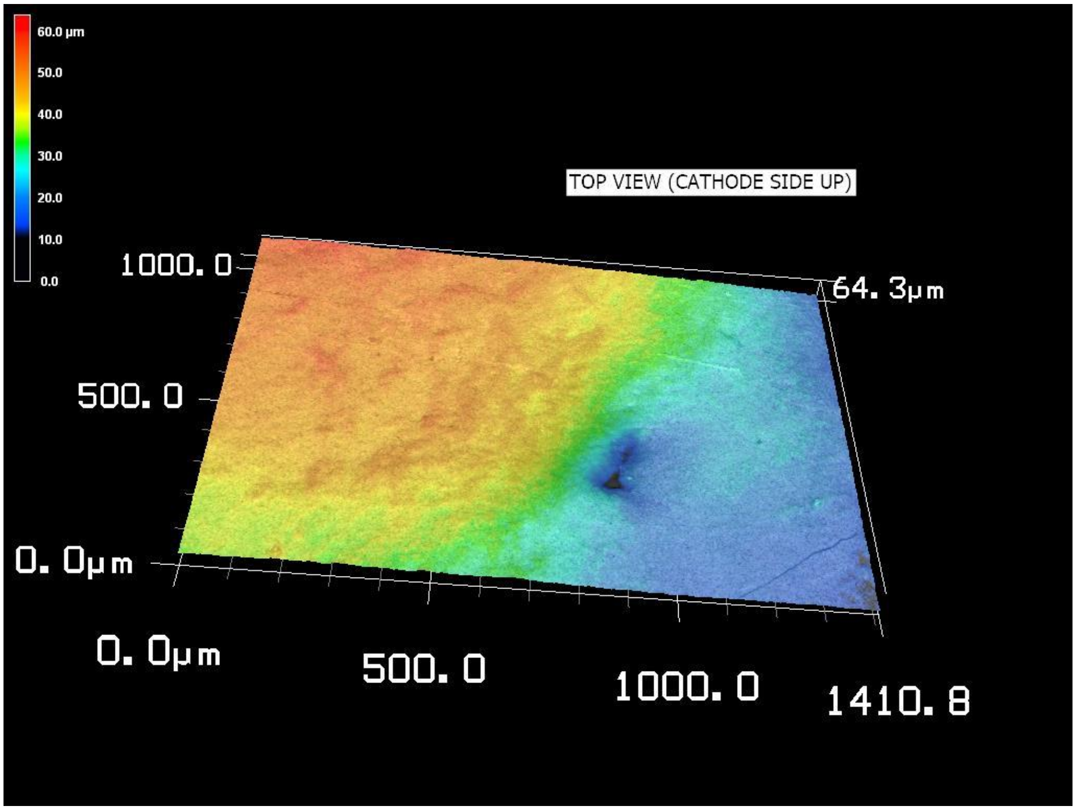


Figure 13 21137 057 cathode side top view

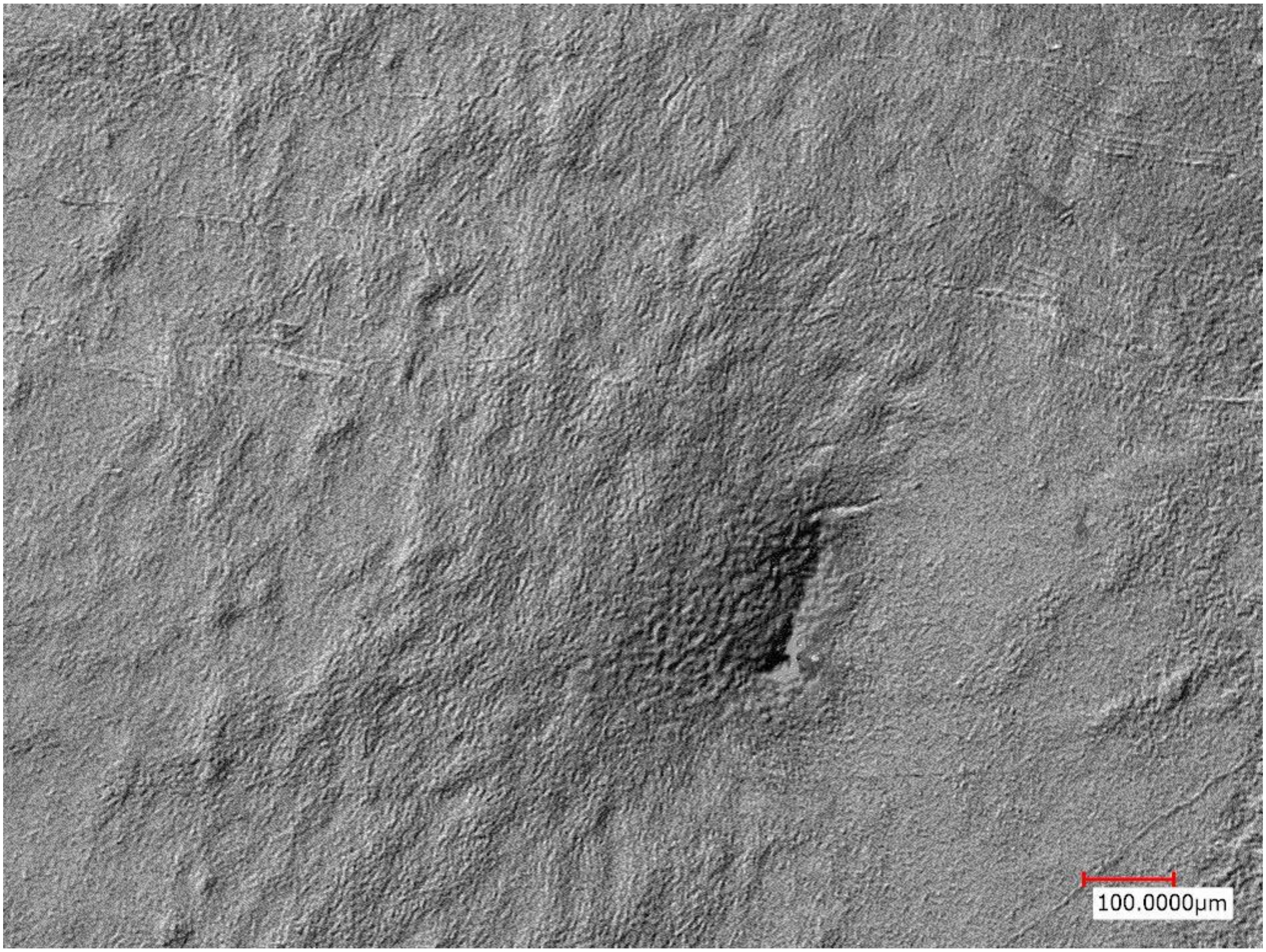


Figure 14 laser view of sample showing hole

Sample 21137 057 : F9(location) medium ,Anode Side View hole orientation cross machine direction

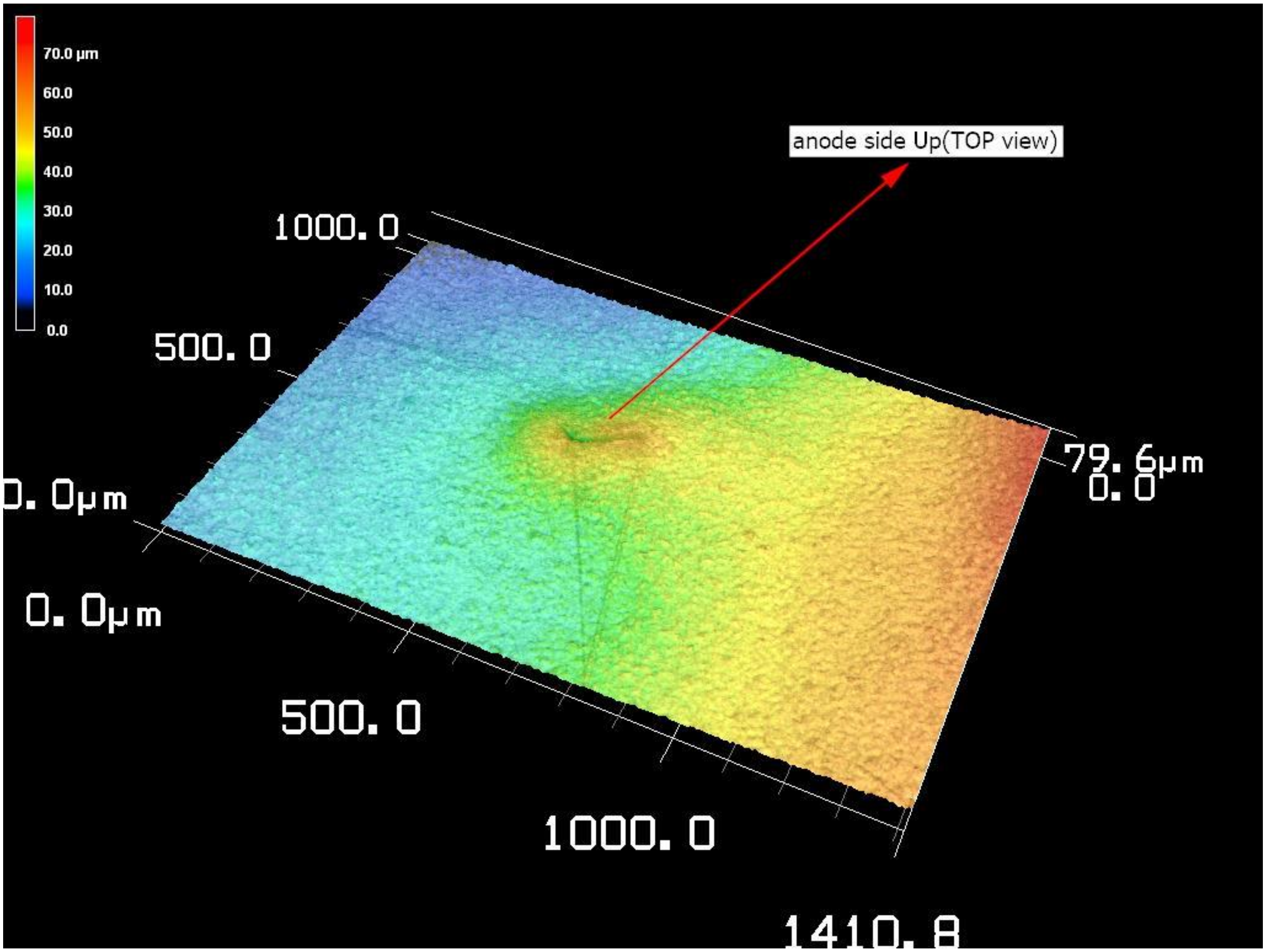


Figure 15 anode side view of sample

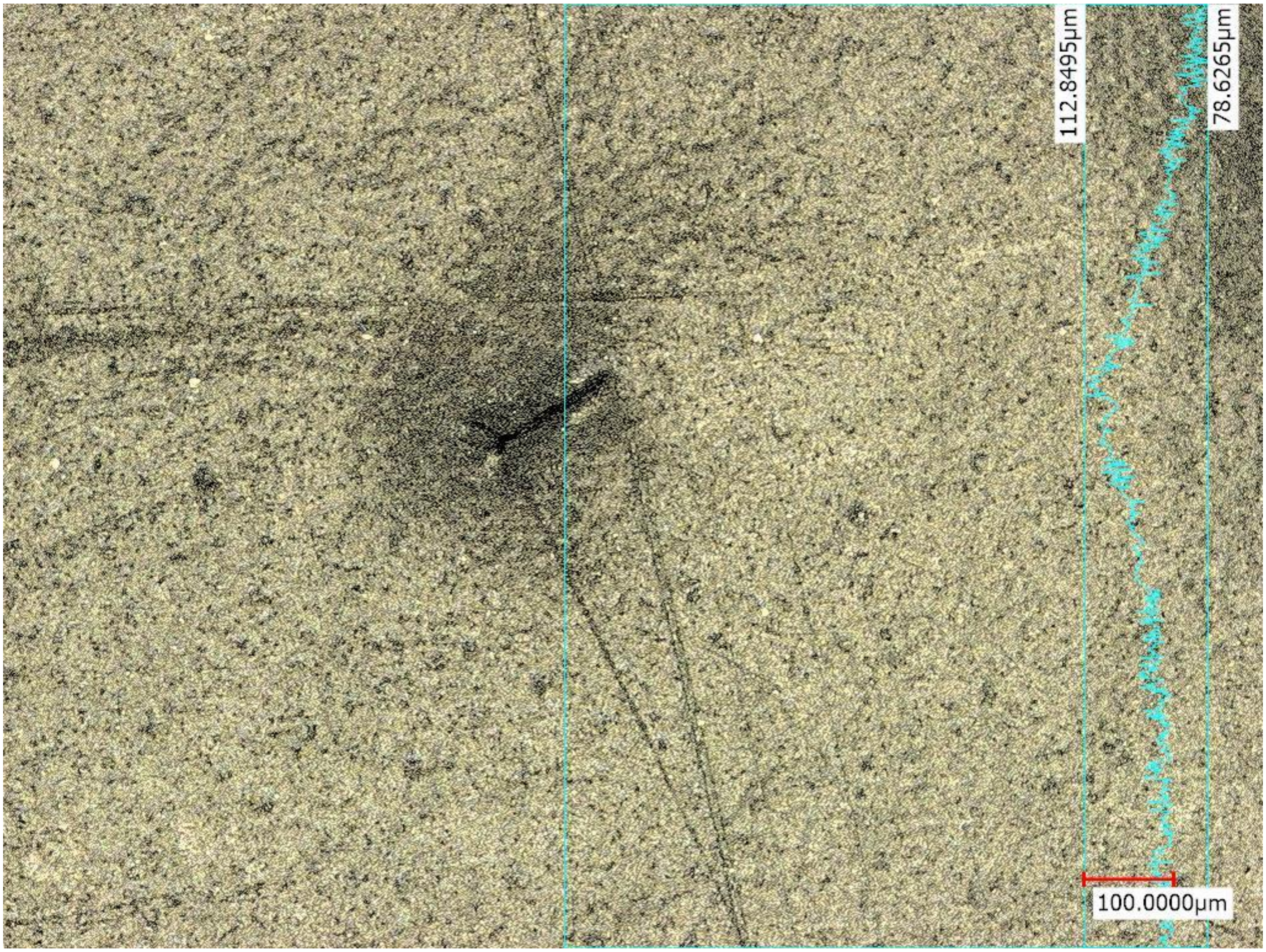


Figure 16 hole profile

Sample 21137 061 : F11(location) medium ,Cathode Side hole orientation cross machine direction.

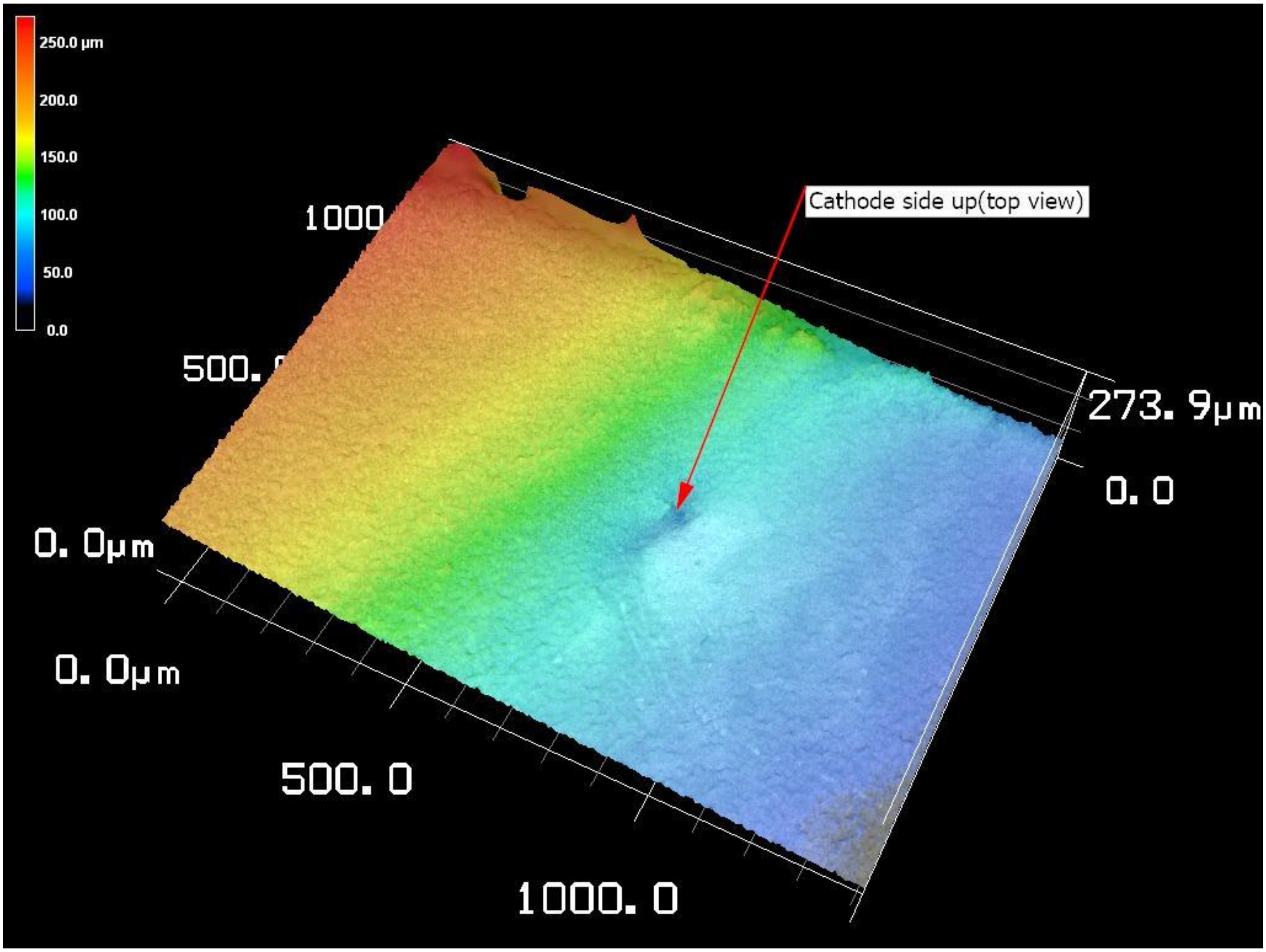


Figure 17 cathode side top view of sample

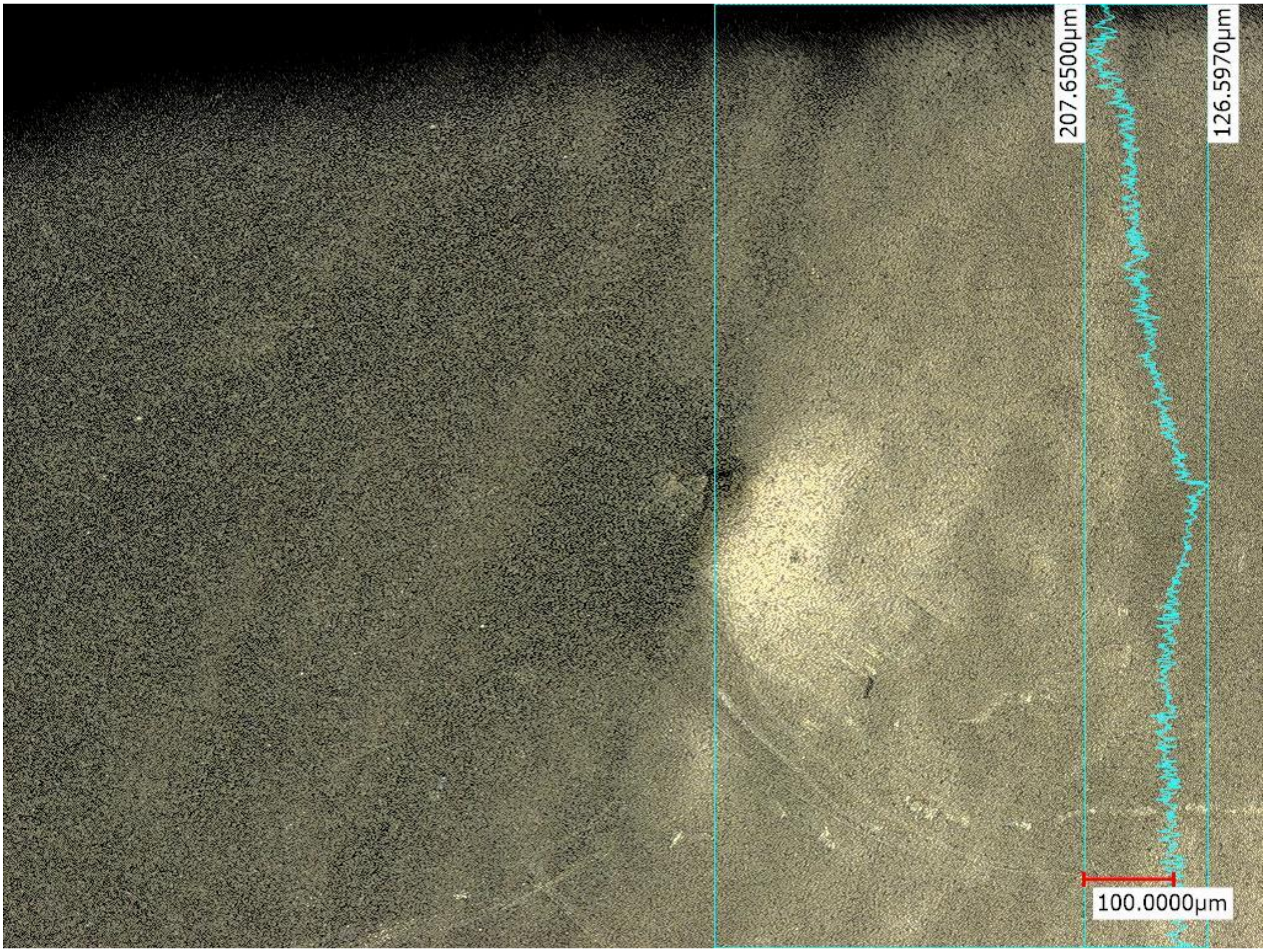


figure 18 hole profile of sample

ANODE SIDE VIEW OF SAMPLE 21137 **061:**

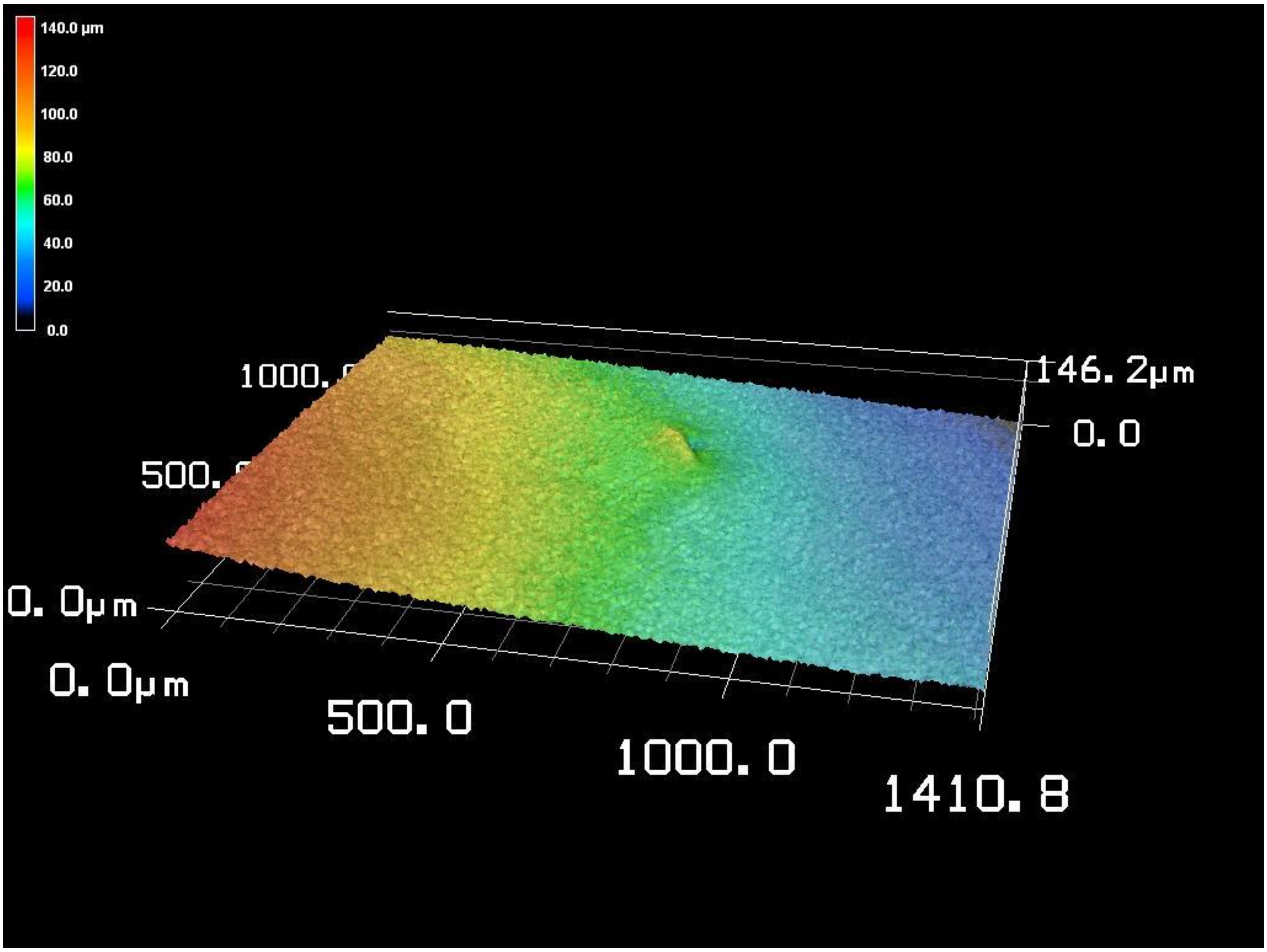


Figure 19 anode top view 3D

Figure 20 hole profile

KEYANCE PERFORMANCE FOR DIFFERENT SAMPLES

			ORIENTATION				Scratches				Surrounding Region				
Sample	Location	Size	Cathode side		Anode side		Cathode Side		Anode side		Cathode Side		Anode side		Observation/comments
			CMD	MD	CMD	MD	Yes	No	Yes	No	Normal	Scratches	Normal	Scratches	
21137 043	E10	large	✓	×	×	×	×	✓	×	✓	✓	×	✓	×	Hole in CMD Direction
21137 057	F9	medium	✓	×	✓	×	✓	×	✓	×	×	✓	×	✓	Protusions and **cross machine direction scratches** around hole observed.
21137 060	C7	medium	✓	×	×	×	✓	×	×	✓	✓	×	✓	×	-
21137 061	F11	medium	✓	×	✓	×			×	✓	✓	×	✓	×	-
21137 062	F9	small	✓	×	not done	not done	✓	×	not done	not done	×	✓	not done	not done	few scratches around hole observed around cathode

Figure 21 Keyence microscope performance tabulation

3.3 SEM

21134 Cell 72 SEM Surface Analysis

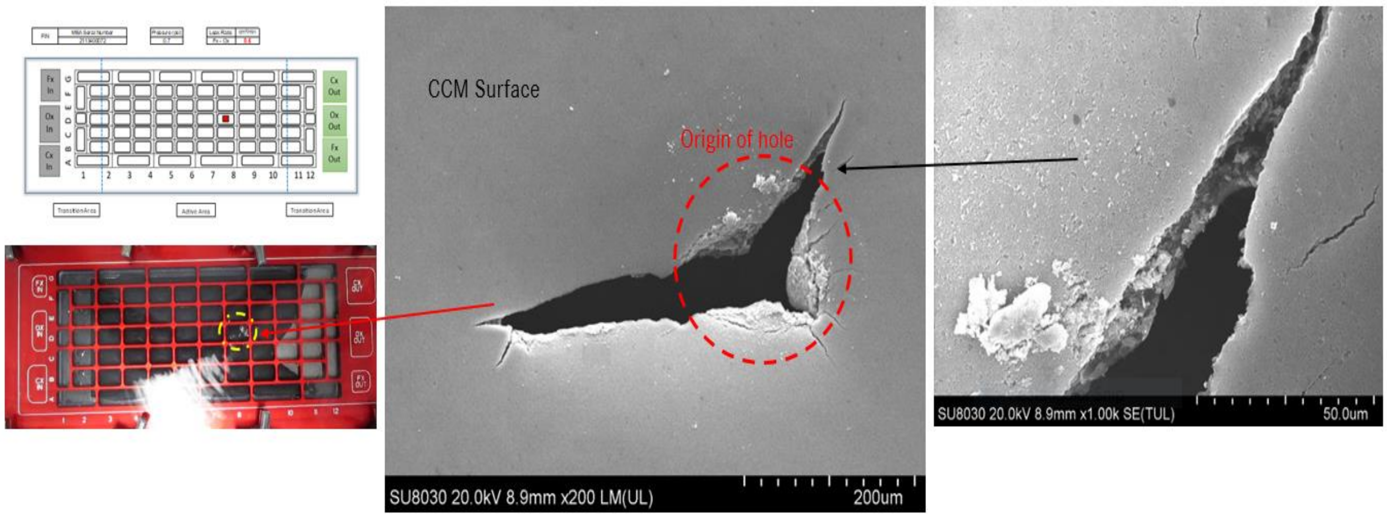


Figure 22 sample under microscope

Large hole with similar shape and dimensions to the hole observed in cell 21134 076, Similarly to cell 76, no big holes found in Anode GDL.

21134 Cell 76 SEM Surface Analysis

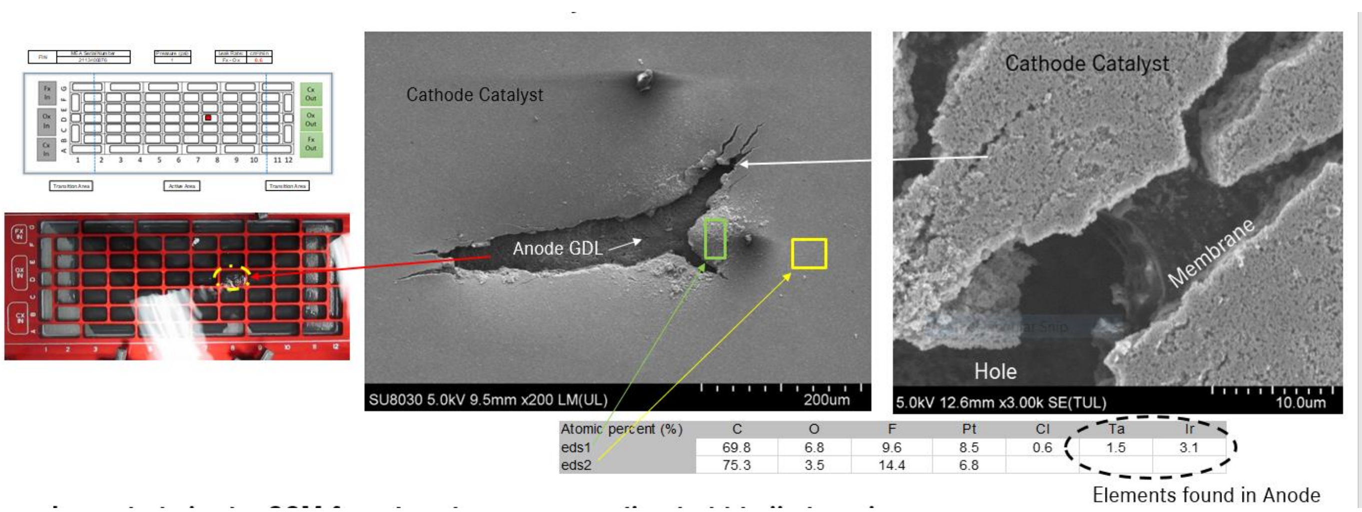


Figure 23 sample 21134 076

Observations:-

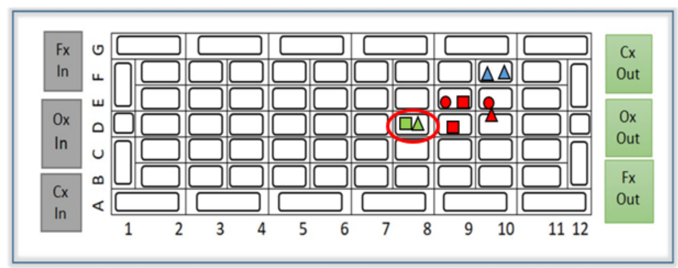
- Large hole in the CCM found at the corresponding bubble jig location
- Anode catalyst seen on cathode surface of CCM
- No big holes found in Anode GDL
- Some areas of missing MPL found in Anode GDL but not representative of the hole seen in the cathode catalyst

21134 - Ends

Leak observed 70-98

Samples 14, 15 no leaks
Sample 72, 76 leaks and hole on bubble jig D8
Sample 98 leak and small leak bubble jig D8

Next sample 77 failed leak test, NOT bubble jigged



21134 Cell 77 SEM Surface Analysis

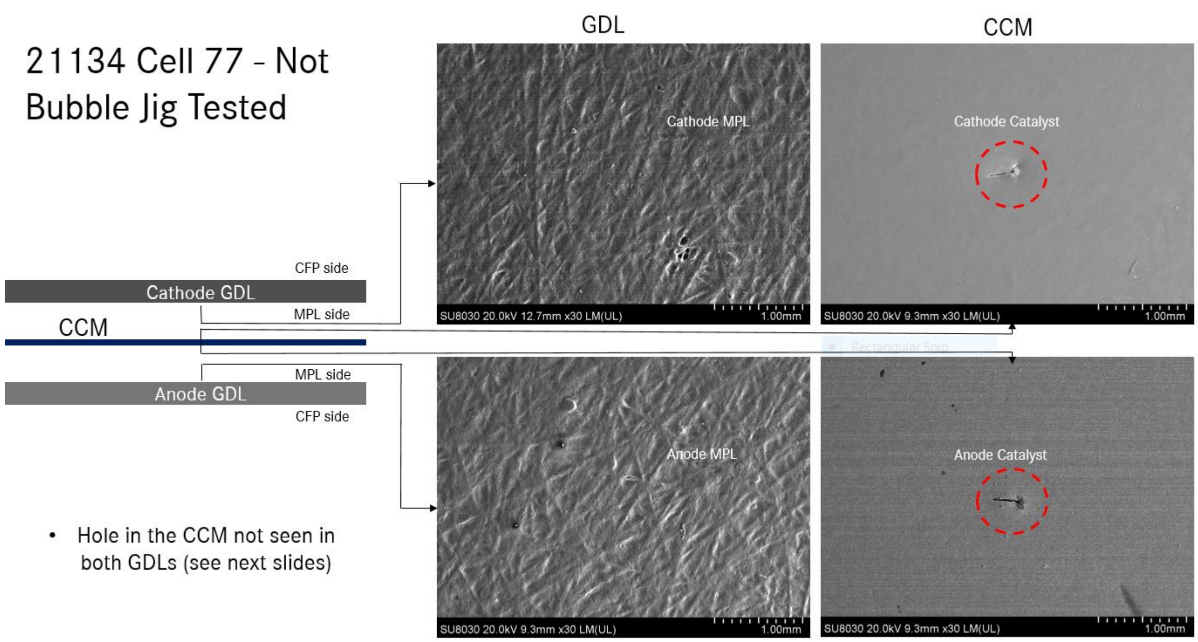
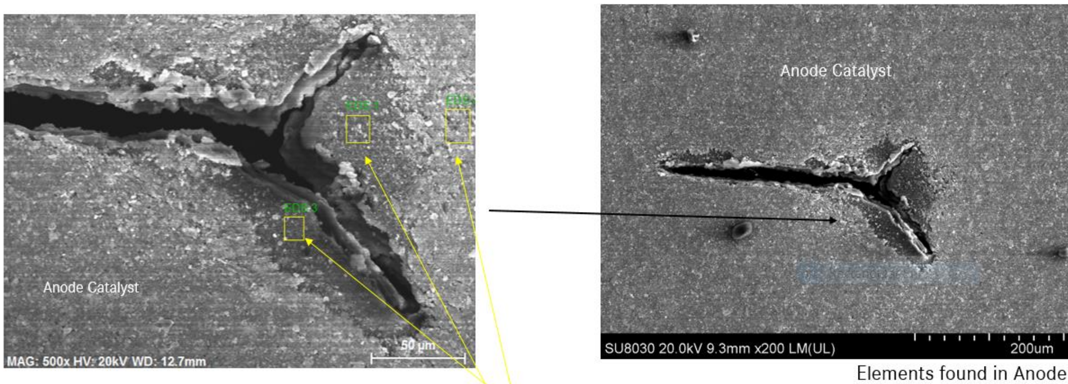


Figure 24 GDL & CCM Side

21134 Cell 77 SEM Surface Analysis – CCM Anode side



Elements found in Anode Catalyst

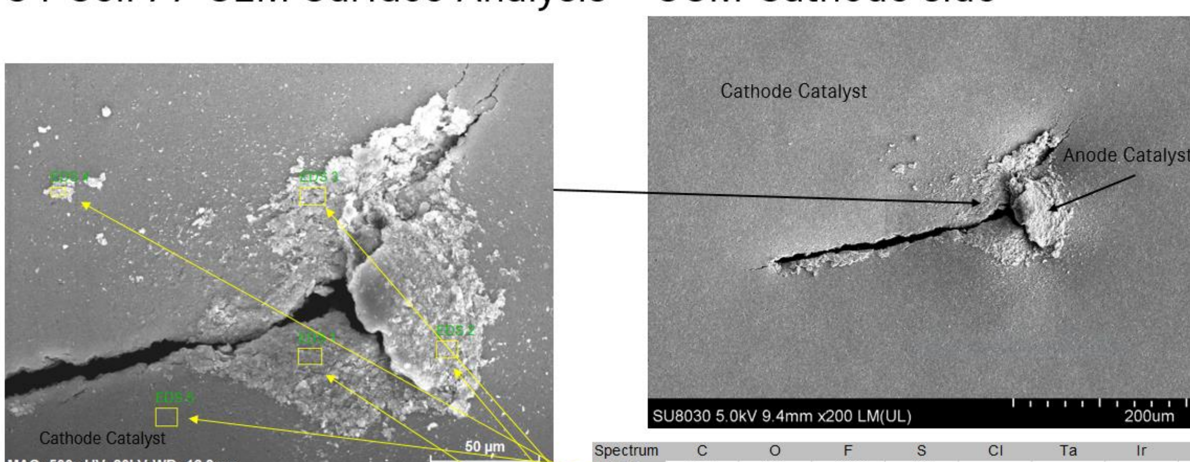
Spectrum	C	O	F	S	Cl	Ce	Ta	Ir	Pt
EDS 3	44.9	2.4	48.2	2.5	0.1	0.2	0.2	0.5	1.0
EDS 2	60.3	5.6	27.2	0.9	0.4	0.1	0.9	1.8	2.8
EDS 1	45.7	5.1	29.1	7.1	0.4	1.2	1.7	4.7	5.0

Figure 25 sample 21134 077(Anode)

Observations:

- Looking from the anode side, missing anode residue at the same spots where anode residue is found when looking from the cathode side
- No cathode catalyst transferred to the surface of the anode catalyst
- The configuration of the hole and the catalyst transfer from one side to another suggests that the hole is coming from the anode side

21134 Cell 77 SEM Surface Analysis – CCM Cathode side



Spectrum	C	O	F	S	Cl	Ta	Ir	Pt
EDS 5	76.8	2.8	11.9	1.0				7.5
EDS 4	75.4	5.2	10.5	0.9	0.2	0.6	1.0	6.3
EDS 3	72.7	6.5	9.6	0.8	0.4	0.9	1.7	7.4
EDS 2	73.8	6.1	8.1	0.8	0.4	1.2	2.3	7.2
EDS 1	73.8	5.3	9.2	1.0	0.3	0.7	1.2	8.5

Figure 26 sample 21034 77 (cathode)

Observations:

- **Large hole in the CCM found similar to previous cells.**
- **Anode Catalyst transfer through the hole to the surface of the cathode catalyst surface.**
- Hole not stretched as much as the other examples due to not being tested in the bubble jig and thus avoiding the exposure to water.

3.3 ISHIKAWA DIAGRAM:

The Ishikawa diagram was developed by Kaoru Ishikawa during the 1960s as a way of measuring quality control processes in the shipbuilding industry. An Ishikawa diagram is a diagram that shows the causes of an event and is often used in manufacturing and product development to outline the different steps in a process, demonstrate where quality control issues might arise and determine which resources are required at specific times.

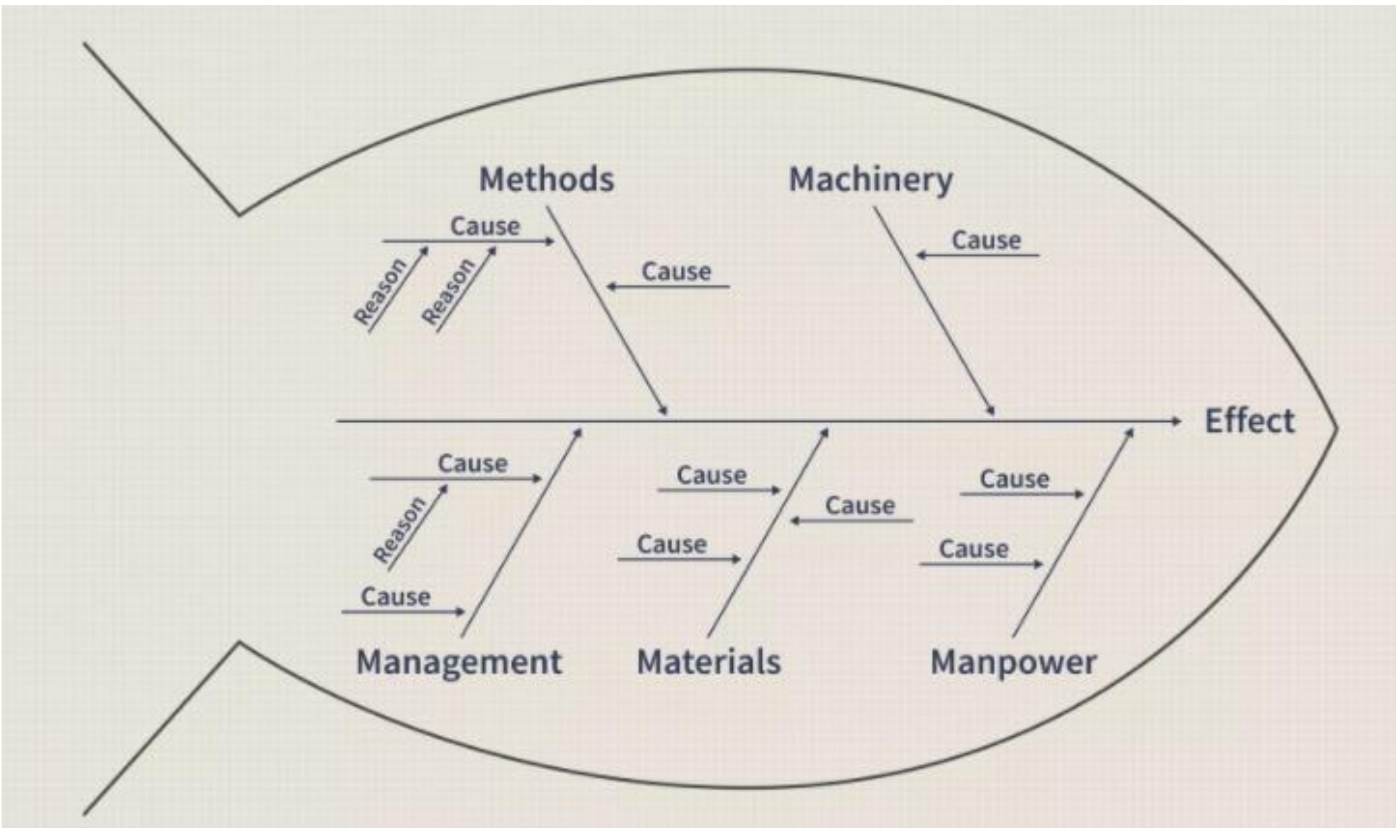


Figure 27 Generalized Ishikawa diagram for analysis.

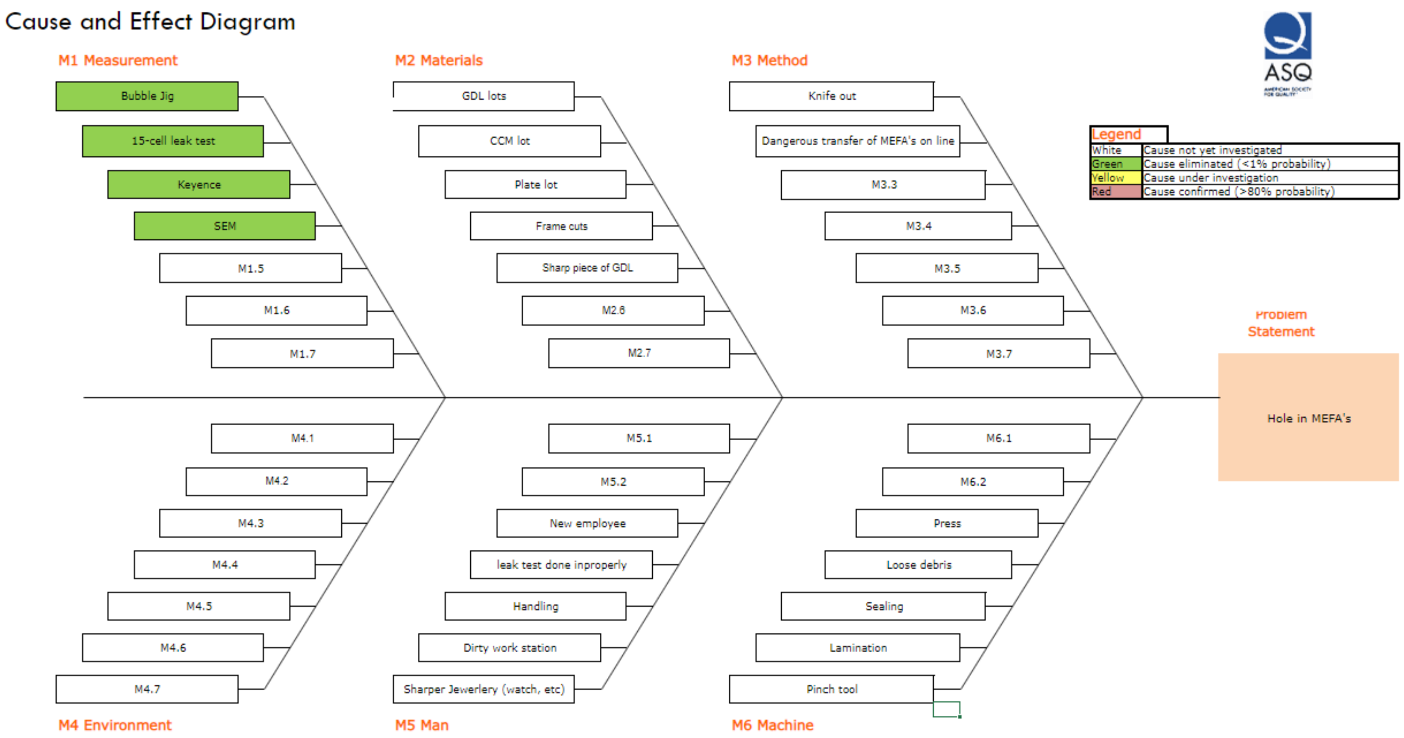


Figure 28 Ishikawa diagram for SMEFA leaky issue

Conclusion

- Originates on anode side, pushes anode catalyst through to cathode side
- Changes size as analysis proceeded (e.g 300 to 100 microns)
- Although clustered seems to be moving along
- No damage on GDLs. Fe, Cr on one GDL observed
- CCM only
- **Problem gone for now**
- **15 cell appears to be catching it**
- **Monitor for now but no further escalation or allocation of resources needed.**
- **Eliminated all MEFA and all sub supplier CCM**
- Down to near net and CCM cut & alignment at Burnaby
- Down to defect marking & packaging at PT.

References

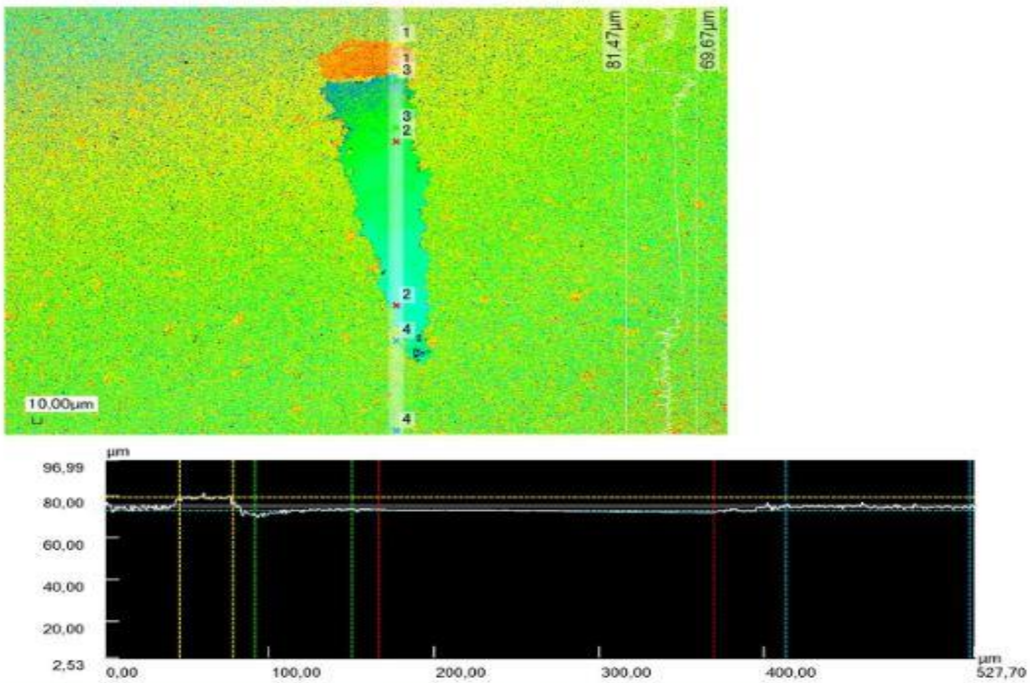
[1] Burnaby, BC – Mercedes-Benz Fuel Cell (A division of Mercedes-Benz Canada), "Locations," *Daimler*. [Online]. Available: https://www.daimler.com/career/about-us/locations/location-detail-page-104512.html

[2] B. Abderezzak, "Introduction to Hydrogen Technology," *Introduction to Transfer Phenomena in PEM Fuel Cell*, 1st ed. Khemis Miliana, Algeria: ISTE Press, 2018, ch. 1, pp. 1-51. [Online]. Available: https://www.sciencedirect.com/topics/engineering/gas-diffusion-layer. Accessed: June 30, 2020/

[3] MRS_GDL_41177_2021-03-31.doc ,SAP-PLM-Document number 003/41177/000/00. MRS(Material Requirement Specification) report for Cellcentric.

Appendix

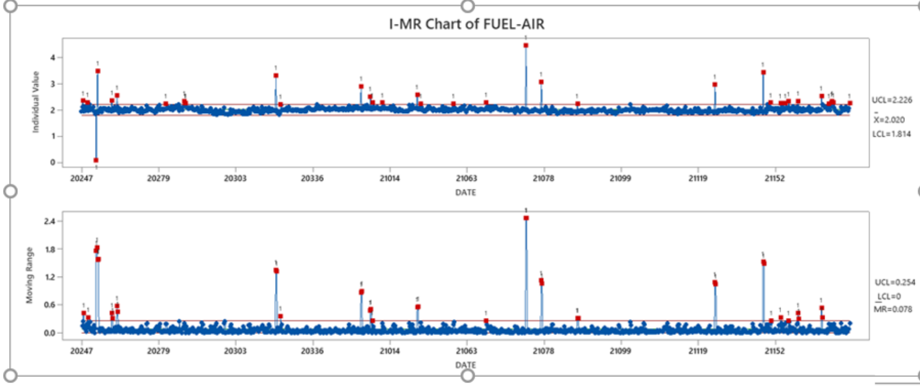
Appendix 1



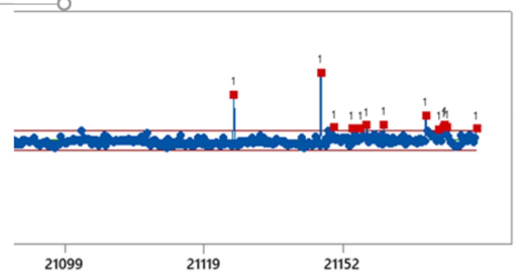
Profil1	Horiz.Abst.	Durchs.-H
Seg.1	32,13µm	78,87µm
Seg.2	202,80µm	72,75µm
Seg.3	58,55µm	72,58µm
Seg.4	111,39µm	74,67µm

Profil1
Linientyp: Vert.
Durchschn.: Bereich 10 Linien, Filter: 0 Linien überspringen
Korrektur : Intensität glätten Keine, DCL/BCL Keine, Höhenglättung Keine, Neigungskorrektur Keine
Bezugswert1 : 78,87µm
Bezugswert2 : 72,58µm
Höhe : 6,29µm

IM-R chart



- 21152 corresponds to when leaks in MEFA's were observed
- The plot shows that after 21153, the density of points over the indicator line is larger
- This is more visible in the cropped plot below



Variation of leak location in active region for different samples.