Identification of Service-Oriented Degradations using Non-Destructive Testing Methods on Passive Direct Methanol Fuel Cell Components

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Abstract

Direct Methanol Fuel Cells are considered a notably high promising one amongst energy sources in the renewable energy sector. Its ion or proton-conducting electrolyte is based exclusively on polymer electrolyte membrane fuel cell technology. Fuel cell components and their durability are affected by methanol solution, its concentration, evaporative conditions of water, carbon dioxide evaluation, heat generation, and its sealing components. Non-Destructive Testing is performed on Passive Direct Methanol Fuel Cell components to evaluate their performance and to ascertain their reliability, serviceability, durability, expected life &healthiness. Non-Destructive Testing such as Visual Testing, Liquid Penetrant Testing, Ultrasonic Thickness measurement, hardness measurement, and metallographic examination is used to identify direct or indirect means to find the size and to locate surface and subsurface discontinuities in the materials and components. The materials and components have been examined using Non-Destructive Testing, evaluated, and interpreted for acceptance/rejection or repair and to assure components' safety and reliability.

Keywords
Passive Direct Methanol Fuel Cell, Membrane Electrode Assembly, Current Collector, Non-Destructive Testing, Reliability

1. Introduction
A fuel cell converts the chemical energy of reactants into electrical energy (electricity) besides other reaction products (Ryan et al. 2016). It is one of the electrical power sources similar to a conversion device like a battery (Bincy et al. 2021). It differs from a battery in that it produces electricity continuously as long as the fuel and oxidant are supplied (Raghavaiah et al. 2022). It has advantages like clean by-products, extremely no/low emission of oxides of nitrogen and sulfur (Beatriz et al. 2020), operates quietly, does not have any moving parts, extra fuel processing to meet demand requirements, and high energy density (Raghavaiah et al. 2020). Methanol is a commonly used fuel for Passive Direct Methanol Fuel Cell (PDMFC). Methanol is relatively inexpensive and easily available as a fuel, has a high specific energy density, quick refueling, and good transport and storage facility (Boni et al. 2020). The cell process operates at ambient temperature and atmospheric pressure without adding additional liquid electrolyte requirements (Braz et al. 2020). Compacted fuel cell design allows it exclusively for porTable applications and easy handling (Junoh, 2020).

1.1 Objectives
Non-Destructive Testing (NDT) is used to identify direct and/or indirect means to find the size and locate surface and subsurface discontinuities in the materials and components of Passive direct methanol fuel cells. The cell with labeled components is shown in figure 1. The materials and components examined using NDT are interpreted for acceptance/rejection or repair and assure the safety and reliability of components (Raghavaiah, 2019). Various Non-Destructive Testing such as Visual Testing (VT), Liquid Penetrant Testing (PT), Ultrasonic Testing (UT) for Thickness measurement, hardness measurement, metallographic examination, etc., on Passive Direct Methanol Fuel Cell components are chosen based on the mode of failures and are considered in this analysis to ascertain their serviceability, durability, expected life, and healthiness.

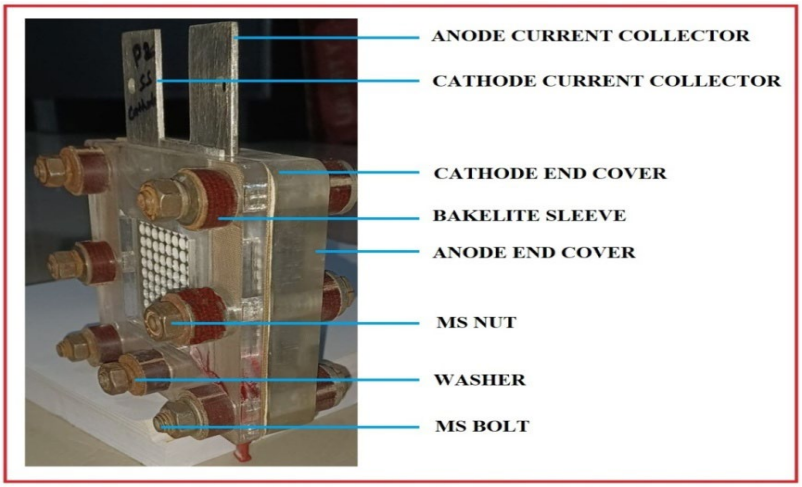

Figure 1. Direct Methanol Fuel Cell.

2. Literature Review

The following literature study is made on various topics covering different types of NDT on fuel cells.

J. R. Claycomb et al (2003) reported on electric and magnetic Non-Destructive Testing of Proton Exchange Membrane (PEM) fuel cells to locate flaws in the PEM from measured magnetic field maps. They explored several NDT techniques employing highly sensitive HTS and LTS SQUID and fluxgate magnetometers. The Magnetic fields produced by currents in the cell are investigated in spatial, frequency, and time domains under several operational conditions. Frequency domain magnetic and electric signals are related to extreme operating conditions including membrane adversity. This study aims at the membrane point of view, but not the total components of the cell.

Frikkie C. De Beer (2018) has presented optimization of Hydrogen Fuel Cells through NDT using Neutron Radiography. This presentation demonstrates efficient water management for optimizing fuel cells for energy production using radiation as the probe. Axenics (2019) indicated that Non-Destructive examination is an important part of the component production process. *Axenics* has evaluated the performance of the components of the three-pass heat exchanger of hydrogen fuel cells including, material corrosion, welding fissures, banding distortion, surface flaws, etc. Vacuum and Helium leak testing processes are used to ensure the components are free from defects. However, from the literature, it is clear that no extensive investigation has been performed on passive direct methanol fuel cells for the assessment of components except membrane electrode assembly. Hence it is necessary to know and identify in-Service originated degradations using Non-Destructive Testing Methods on Passive Direct Methanol Fuel Cell Components for ascertaining the integrity of cell components.

3. PDMFC components and probable degradations

Cell Component-wise envisaged degradation mechanisms, various non-destruction examination methods, and a plan of record for evaluation are tabulated in Table 1.

Table 1. NDE and Examination Methods for PDMFC Components and Methodology for Selection Criterion of Examination Method

Name of the component	Degradation Mechanism	Examination Methods	Record for evaluation
Anode End Cover (Acrylic)	A) Brittle Cracks under aging and bolt loading	a) Visual Examination of component b) Surface Examination using Penetrant Testing	i. VT-Image ii. PT-Image

Gasket between anode end cover & anode Current Collector (Viton)	A) Compression Set B) Lack of softness	a) Visual Examination b) Thickness Testing c) Hardness Testing	i. VT-Image (new and used gaskets) ii. Thickness- measurement for compression set iii. Hardness-ShoreA value
Anode Current Collector (SS, Nickel, Brass)	A) Uniform Corrosion B) Corrosion Erosion in openings C) Surface cracks	a) Visual Examination b) Thickness Testing c) Loss of weight in a given time.	i. VT-Image for SS, Ni, Brass ii. PT-Image iii. Corrosion-Images &Weight loss calculations
Gasket between anode Current Collector and MEA (Teflon coated woven cloth)	A) Compression Set B) Lack of softness	a) Thickness Testing b) Hardness Testing	i. VT-Image (new and used gaskets) ii. Thickness- measurement for compression set iii. Hardness-ShoreA value
MEA (Nafion-117)	A) Reduction of Performance	a) Durability	SEM and TEM images
Gasket between MEA and cathode Current Collector (Teflon coated woven cloth)	A) Compression Set B) Lack of softness	a) Thickness Testing b) Hardness Testing	i. VT-Image, (new and used gaskets) ii. Thickness-Compression set iii. Hardness-ShoreA
Cathode Current Collector (SS, Nickel, Brass)	A) Uniform Corrosion	a) Visual Examination b) Surface Examination using Penetrant Testing c) Thickness Testing	i. VT-Image for SS, Ni, Brass ii. PT- Image iii. Corrosion- Images
Gasket between cathode end cover & cathode Current Collector (PTFE, Viton)	A) Compression Set B) Lack of softness	a) Thickness Testing b) Hardness Testing	i. VT-Image (new and used gaskets) ii. Thickness- measurement for compression set iii. Hardness-ShoreA value
Cathode end cover (Acrylic)	A) Brittle Cracks	a) Visual Examination of component b) Surface Examination using Penetrant Testing	i. VT-Image ii. PT-Image
Fasteners (bolts, nuts, washers) (MS, SS)	A) Uniform Corrosion	a) Visual Examination	i. VT-Image ii. corrosion-weight basis
Wrapping on fasteners (Bakelite)	A) Cracks under compression B) Methanol Tolerance C) Swelling	a) Visual Examination (For condition of the part, component, or surface)	i. VT-Image (new & used)

4. Experimental Investigations

For performing the experimental investigation on PDMFC components, shore A hardness gauge for soft materials, color contrast penetrant system for identifying surface discontinuities, ultrasonic thickness gauge for thickness measurements, and digital balance for weight measurements are used.

4.1 Investigation of Anode End Cover

Anode end acrylic cover is shown in figure 2(a). This cover is subjected to a Visual Examination to identify visible surface discontinuities on the component. However, during the evaluation of the component, no visible discontinuities are identified.
Surface Examination is performed using color contrast type Penetrant Testing by the solvent removable method. However, no reporTable discontinuities are identified during the evaluation of the component. The component after the application of the developer while in the evaluation process is shown in figure 2(b).

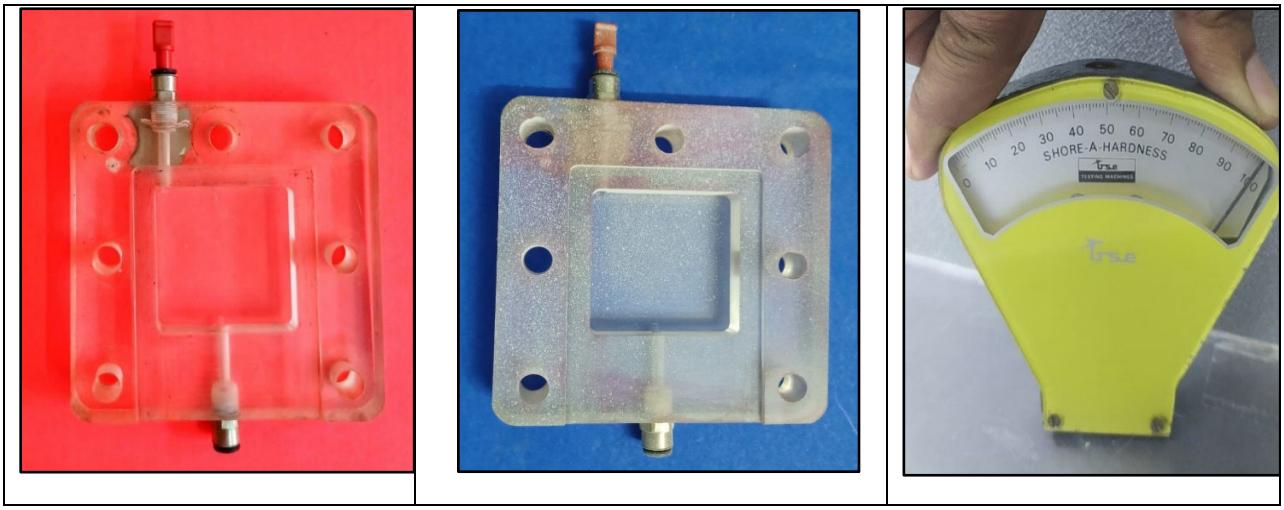

Figure 2. (a) VT-Image (left), (b) PT-Image (middle), (c) Hardness Testing (right)

Hardness testing is carried out on an anode end acrylic cover and shore A hardness is showing the maximum value of 100 shore A, indicating there is no change in the material in the methanol environment. The performed hardness testing is shown in figure 2(c).

Evaluation of test results: From the above interpretations, the anode end acrylic cover has no deterioration and is intact with the cell operating environment.

4.2 Gasket between anode end cover & anode current collector

The Viton gasket is used to arrest the leak between the anode end cover and the anode current collector. New and used Viton gaskets are subjected to visual Examination for identifying visible discontinuities if present on the component. During the evaluation of the component, no visible defects are identified. However, scratch marks and irregular surfaces are observed which are resulted from the rough surface of metallic current collectors. The failure of the serviced gasket is attributed to physical damage due to mishandling while removing from the cell.

Hardness testing is carried out on the new and serviced gaskets and hardness is showing the values of 74 shore A and 76 shore A respectively. These measurements are shown in figures 3(a) and 3(b). This variation in the hardness is very small, indicating no significant change in the Viton material in the methanol environment.
Thickness measurement is carried out on the new and serviced gasket. The new gasket is 2.00 mm, whereas the serviced one is 1.96 mm. Hence there is a compression of 2% (0.04 mm) due to bolting loads over two years of service.

Evaluation of test results: From the above interpretations, there is no deterioration of the Viton gasket, and is intact with the cell operating environment for a reported duration of two years.

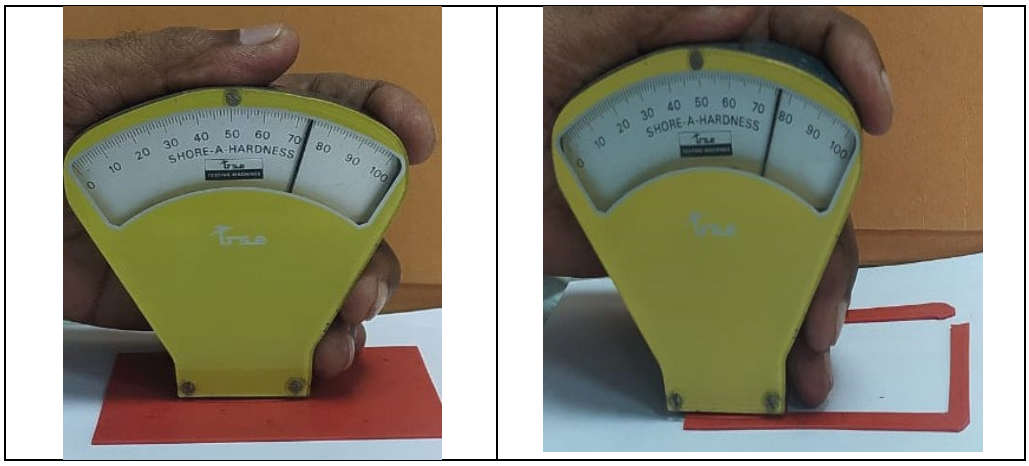

Figure 3. (a) Hardness testing on virgin Viton gasket (left), (b) Hardness testing on serviced Viton gasket (right)

4.3 Anode Current Collector

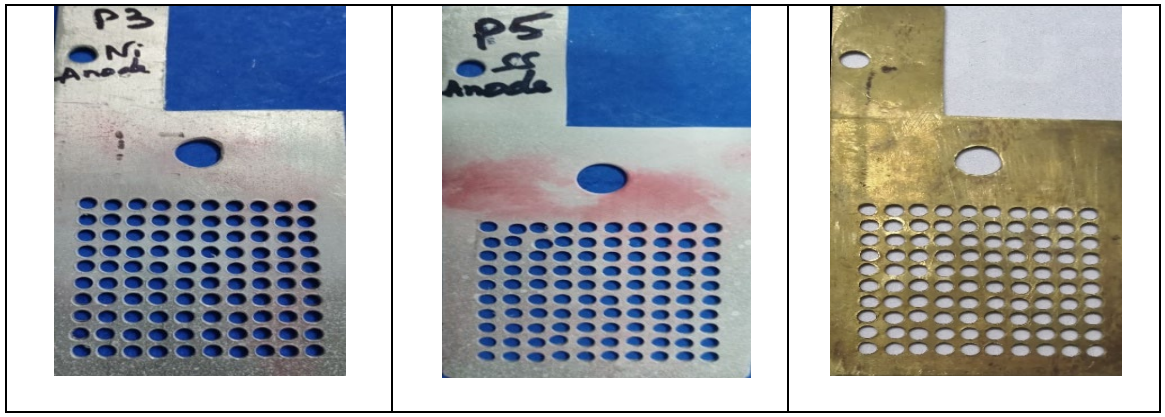

Figure 4. (a) Penetrant Test on SS current collector (Left), (b) Penetrant Test on Ni current collector (Middle), (c) Corrosion on Brass at anode side after exposure to 4 M methanol. (Right)

From the PDMFC, it is observed that the current collectors are getting corroded faster than that of the cathode side. Hence anodic current collectors are chosen for the study of corrosion and loss of material. The visual examination shows that the brass reacts with methanol quickly and forms surface oxides (Meenakshi 2021). So, the usage of brass materials specific to the anodic end may not be a good choice. An image of the brass CC after exposure to the methanol environment is shown in Figure 4(c).

Surface Examination is performed using Penetrant Testing by solvent removable method with color contrast type. However, no reporTable discontinuities are identified during the evaluation of the component. The component after the application of the developer during the evaluation process is shown in Figures 4(a) and 4(b).

The durability of Anode Current Collectors:
Short term experimental corrosion rate of the CC materials is calculated and tabulated by taking the weights before and after the experiment and the time of exposure to methanol solution during the test. A comparison of experimental corrosion rates concerning SS316L material is also calculated and provided in Table 2.

Calculation details:
Density of the material = γ g/cm^3
Thickness of CC = μ mm

Weight Before the experiment = θ1 g
Weight after the experiment = θ2 g
Weight Loss = (θ1- θ2) g
Duration of experiment (in hours) = Ƭ h
Constant, π =3.14
Effective surface area (Ă), mm^2 $\check{A} = 2\left(2500 - \frac{100\pi}{4}3.8^2\right) + 380\pi\mu$
Experimental Corrosion Rate (ξ), mm/year $\xi = \frac{(\theta 1-\theta 2)}{Ƭ}\ \frac{1}{1000\gamma\check{A}}$ x24x365

Table 2: Short-term corrosion measurement results

Description	SS-316L	Nickel-201	Brass (66-34)
Density of the material, γ g/cm^3	7.90	8.89	8.50
Thickness of CC, μ mm	2.01	1.98	1.99
Weight before the experiment, θ1 g	89.1572	97.8724	94.3231
Weight after the experiment, θ2 g	89.1546	97.8720	94.2901
Weight loss, (θ1- θ2) g	0.0026	0.0004	0.0330
Duration of exposure, Ƭ h	12	12	12
Effective Surface area of the current collector, Ămm^2	5131.25	5095.45	5107.39
Experimental Corrosion rate,ξ mm/year	0.047	0.006	0.555
Comparison of experimental corrosion rates w.r.t SS-316L material	100% (Assumed)	12.7% of SS316L corrosion rate	1180% of SS316L corrosion rate

Evaluation of test results: From the above interpretations, it is observed that there is a deterioration of anode current collectors. Out of the SS-316L, Ni-201, and brass current collectors, the corrosion rate on brass is 11.8 times higher than that of the SS-316L current collector, whereas Ni-201 has a corrosion rate of approximately 1/8 of that of SS-316L.

4.4 Gaskets between anode CC &MEA, between MEA & cathode CC, and between cathode CC & cathode end cover

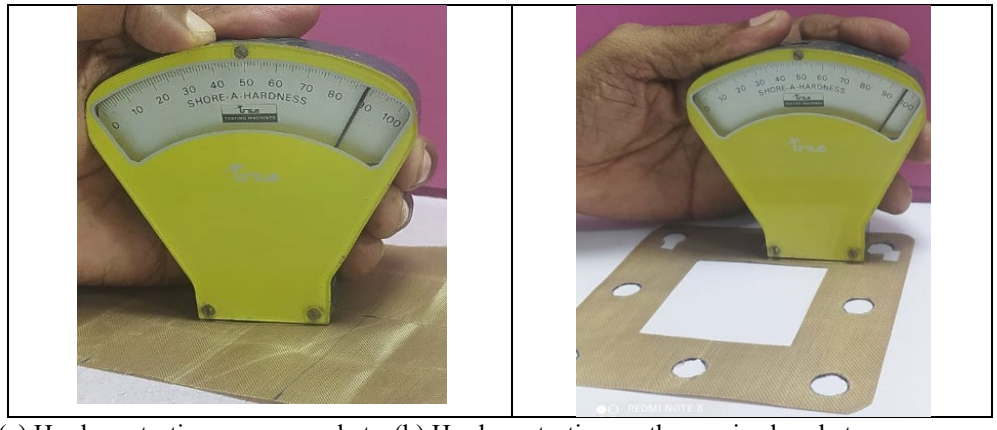

Figure 5. (a) Hardness testing on new gaskets, (b) Hardness testing on the serviced gasket

The Teflon-coated woven cloth gasket is used to arrest the leak between the anode current collector &MEA, MEA & cathode current collector, and cathode current collector & cathode end cover. The new gasket and the gasket which served for two years are subjected to this study. During the evaluation of the component, no visible defects are identified. However, pressing and scratch marks are observed which are resulted from the rough surface of metallic current collectors.

Hardness testing is carried out on the new and serviced gaskets and hardness is showing the value of 89 shore A and 96 shore A respectively. These measurements are shown in Figures 5(a) and 5(b) respectively. This variation in the hardness is small, indicating no significant change in the Teflon material in the methanol environment. Thickness measurement is carried out on the new and serviced gasket. The new gasket is 0.20 mm, whereas the serviced one is 0.18 mm. Hence there is a compression of 10% (0.02 mm) due to bolting loads over two years of service.
Evaluation of test results: From the above interpretations, there is no significant deterioration of the Teflon-coated woven cloth gasket, and is intact with the cell operating environment for a reported duration of two years.

4.5 Membrane Electrode Assembly

Membrane electrode assembly consists of an anode loaded with Platinum Ruthenium as a catalyst, a cathode loaded with Platinum as a catalyst, and a proton-conducting membrane in between the anode and cathode. MEA, facing the anode side is shown in figure 6(a). From the literature, (Liu et al. 2004) it is observed that the initial power density of PDMFC is lost about by 30% after a test period of 75 hours. This is due to an increase in resistance because of de-bonding of electrodes and MEA, and swelling of membrane and electrodes. SEM image of MEA having 75 hours of service is shown in figure 6(b). TEM image of electrocatalysts is shown in figure 6(c) of 75 hours of serviced MEA. Accumulation of electro-catalysts and metallic particles on the membrane indicates the aggressiveness of the methanol environment on the MEA and fuel cell internals.

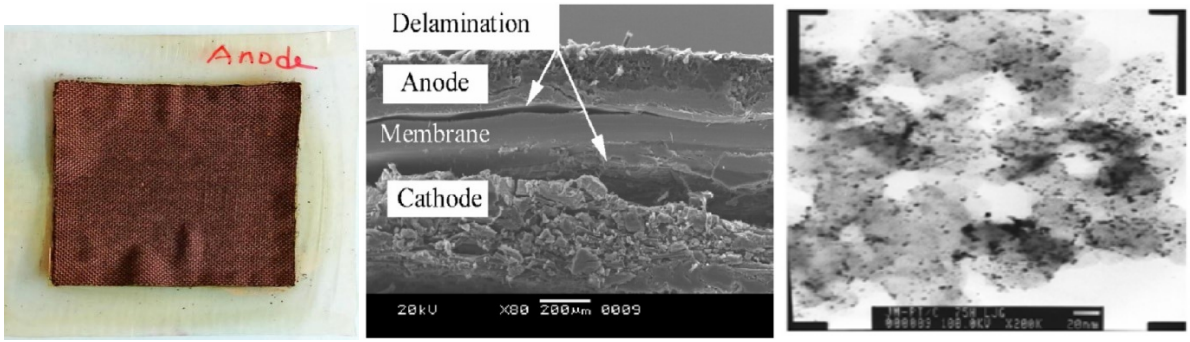

Figure 6. (a) MEA facing anode side (left), (b) After 75 hours of MEA in PDMFC, cross-sectional view under SEM (Middle) (Liu et al. 2004), (c) After 75 hours of MEA in the cell, electrocatalysts view under TEM (Right) (Liu et al. 2004)

4.6 Cathode Current Collector

From the PDMFC, it is observed that the cathode end current collectors are getting corroded slower than that of the anode side. The reason behind the cathode end CC corrosion is attributed to methanol cross-over and the formation of water at the cathode. The visual examination shows that the brass reacts with methanol and forms surface oxides (Nickel, 2021). However, Ni-201 and SS-316L are found free from visual imperfections. So, the usage of brass materials specific to the cathode end may be considered as long as the surface is free from methanol cross-over. But brass is not a good choice for higher methanol concentrations and the prolonged operation of cells due to methanol cross-over. Images of the Brass current collectors before the start of the experiment is shown in figure 7(d) and after exposure to the methanol environment is shown in figure 7(e).

Surface Examination is performed using Penetrant Testing by solvent removable method with color contrast type. However, no reporTable discontinuities are identified during the evaluation of the component. The SS, Nickel, and brass current collectors after the application of the developer during the evaluation process are shown in figures 7(a), 7(b), and 7(c) respectively.

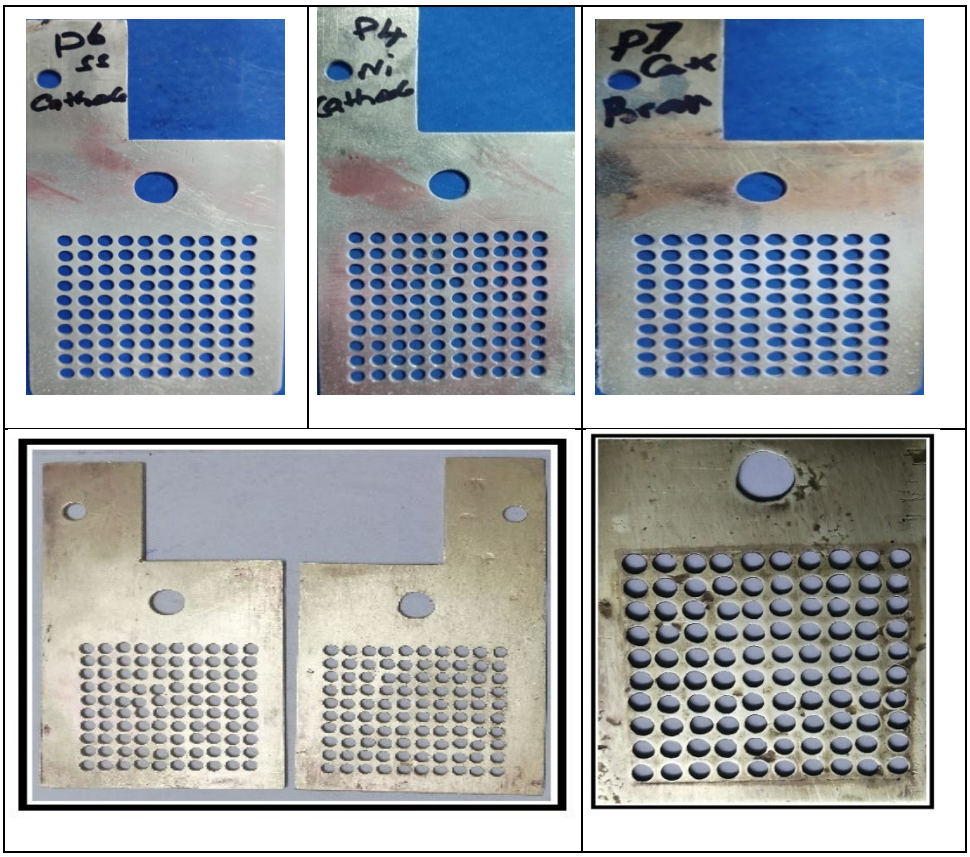

Figure 7. (a) Penetrant Test on SS current collector (Top, left), (b) Penetrant Test on Ni current collector (Top, middle), (c) Penetrant Test on Brass current collector (Top, right) (d) Brass CC before the experiment (Bottom, left) (e) Corrosion on Brass at cathode side after exposure to 4 M methanol. (Bottom, right)

Evaluation of test results: From the above interpretations, it is observed that there is no deterioration of cathode current collectors. Out of the SS-316L, Ni-201, and brass current collectors, the corrosion is seen on the brass current collector due to methanol crossover and further reaction of brass with methanol with the formation of surface oxides.

4.7 Cathode end cover

The cathode ends acrylic cover shown in figure 8(a) is subjected to visual examination to identify visible discontinuities on the component. However, during the evaluation of the component, a linear crack is visible as discontinuity at the top left corner of the drilled hole. Surface Examination is performed using Penetrant Testing by solvent removable method with color contrast type. However, two reporTable discontinuities are identified during the evaluation of the component. The component after the application of the developer during the evaluation process is shown in Figures 8(b) and 8(c). The evaluated crack length is 32 mm and one more crack above the hole is having a length of 8 mm.

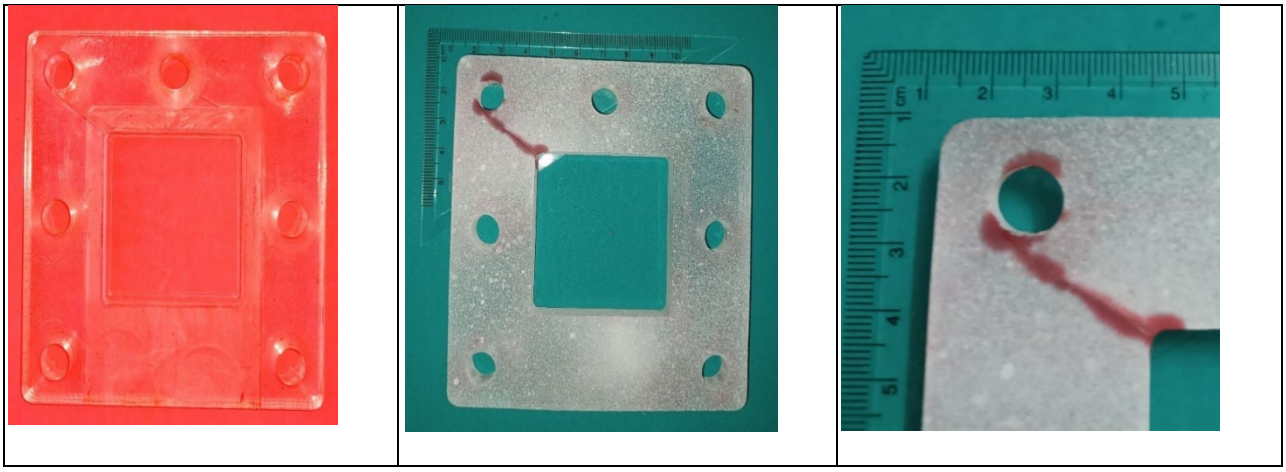

Figure 8. (a) VT cathode end acrylic cover, (b) Penetrant Test on cathode end acrylic cover, (c) Close shot of PT defect indication

Evaluation of test results: The cathode end acrylic cover has two linear defects from the above interpretations of penetrant testing. These defects may cause leakages. These defects originated due to excessive bolt loadings or uneven tightening of fasteners.

4.8 Fasteners (bolts, nuts, washers)

Figure 9. (a) As received MS Bolts with an insulating cover, (b) As received washers and nuts, (c) Corroded bolts after a service period of 2 years (d) Corroded nuts after a service period of 2 years, (e) Corroded spring washers after a service period of 2 years, (f) Corroded plain washers after a service period of 2 years

Within the PDMFC, it is observed that the fasteners such as bolts, washers, and nuts are getting corroded. The visual examination shows that the mild steel components react quickly and form surface oxides. Images of the bolts, nuts, and washers before the start of the experiment are shown in Figures 9(a) and 9(b), and after a service period of 2 years are shown in figures 9(c), 9(d), 9(e) and 9(f). The corrosion rate of each fastener is calculated by taking the weights before and after the experiment. An average of seven sets of fasteners are calculated and tabulated and provided in Table 3.

Calculation details:
Weight Before the experiment = $\theta 1$ g
Weight after the experiment = $\theta 2$ g
Weight Loss = $(\theta 1 - \theta 2)$ g
Duration of experiment = τ years
Experimental Corrosion Rate, ξ g/year: $\xi = \frac{(\theta 1 - \theta 2)}{\tau}$

Table 3: Short-Term Corrosion Measurement Data

Parameter / Component	MS Bolt	MS Nut	Spring washer	Plain Washer
Weight Before Experiment, g	16.736	4.391	1.125	1.080
Weight after two years, g	16.625	4.243	1.013	0.958
Weight loss, g	0.111	0.148	0.112	0.122
Duration of exposure, years	2	2	2	2
Established Corrosion rate, ξ g/year	0.0555	0.074	0.056	0.061

Evaluation of test results: From the above interpretations, it is observed that there is a deterioration of fasteners. The root cause of the deterioration in the corrosive environment over the cell.

4.9 Wrapping on fasteners

Figure 10. (a) Electrical insulating tube over MS Bolts after 2 years of service, (b) Bulged insulating sleeves after a service period of 2 years.

From the PDMFC, it is observed that the wrapped tubes and sleeves over bolts are getting bulged. From the visual examination, it is observed that the tubes are getting peeled off due to frequent assembling and dismantling. However, the sleeves are getting bulged under compressive loads of bolts and nuts. Images of the insulating tubes and sleeves after a service period of 2 years are shown in Figures 10(a) and 10(b).

Evaluation of test results: From the above interpretations, it is observed that there is damage to tubes and sleeves. The root cause of the deterioration is the compressive loading on sleeves and the rubbing of tubes during assembling and dismantling. Physically damaged insulating sleeves and tubes need to be replaced for equipment and personal protection.

5. Discussion

Non-Destructive Testing such as Visual Testing, Liquid Penetrant Testing, Ultrasonic Testing for Thickness measurement, hardness measurement, and metallographic examination is performed on Passive Direct Methanol Fuel Cell components to evaluate their performance and to ascertain their serviceability, durability, expected life & healthiness. Non-Destructive Testing is used to identify direct or indirect means to find the size and to locate surface and subsurface discontinuities. The materials and components have been examined using Non-Destructive Testing and interpreted for acceptance/rejection or repair and to assure components' safety and reliability.

6. Results and Conclusions

Passive Direct Methanol Fuel Cell is considered a notably high promising one amongst energy sources in the renewable energies sector. Its ion or proton-conducting electrolyte is based totally on polymer electrolyte membrane fuel cell technology. Fuel cell components and their durability are affected by methanol solution, its concentration, evaporative conditions of water, carbon dioxide evaluation, heat generation, and its sealing components. The concluded results of the identified Service-Oriented Degradations using Non-Destructive Testing Methods on Passive Direct Methanol Fuel Cell Components are tabulated in Table 4.

Table 4: Results of the identified Service-Oriented Degradations using Non-Destructive Testing Methods

Name of the component	Degradation Mechanism	Results
Anode End Cover	• Brittle Cracks under aging and bolt loading	The anode end acrylic cover has no deterioration and is intact with the cell operating environment
Gasket between anode end cover & anode	• Compression Set • Lack of softness	From the interpretations, there is no deterioration of the Viton gasket and is intact with the cell operating environment for a reported duration of two years.
Anode Current Collector	• Uniform Corrosion • Corrosion Erosion in openings • Surface cracks	From the investigations, it is observed that there is a deterioration of anode current collectors. Out of the SS-316L, Ni-201, and brass current collectors, the corrosion rate on brass is 11.8 times higher than SS-316L current collector, whereas Ni-201 has a corrosion rate of approximately 1/8 of that of SS-316L.
Gasket between anode & MEA, between MEA & cathode, and between cathode cover & cathode	• Compression Set • Lack of softness	From the interpretations, there is no significant deterioration of the Teflon -coated woven cloth gasket and is intact with the cell operating environment for a reported duration of two years.
MEA	• Reduction of Exchange Performance with time	From the interpretations, the performance degradation of MEA attributes to delamination of the MEA with electrodes and agglomeration of electro-catalyst and metals.
Cathode Current Collector	• Uniform Corrosion	From the interpretations, it is observed that there is no deterioration of cathode current collectors. Out of the SS-316L, Ni-201, and brass current collectors, the corrosion is seen on the brass current collector due to methanol crossover and further reaction of brass with methanol with the formation of surface oxides.
Cathode end cover	• Brittle Cracks	The cathode end acrylic cover has two linear defects from the above interpretations of penetrant testing. These defects may cause leakages. These defects originated due to excessive bolt loadings or uneven tightening of fasteners.
Fasteners (Bolts, nuts, washers)	• Uniform Corrosion	From the interpretations, it is observed that there is a deterioration of fasteners. The root cause of the deterioration in the corrosive environment over the cell.
Wrapping on fasteners	• Cracks under	From the interpretations, it is observed that there is

	compression • Methanol Tolerance • Swelling	damage to tubes and sleeves. The root cause of the deterioration is the compressive loading on sleeves and the rubbing of tubes during assembling and dismantling. Physically damaged insulating sleeves and tubes need to be replaced for equipment and personal protection.

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