Developing Failure Catalogues Using Signatures From Electrochemical Impedance Spectroscopy (EIS)

By Dr N. Patterson

To enable high-throughput manufacturing, detecting and sorting defective cells or stacks must be accelerated:
Stack qualification is a critical step to ensure performance and reliability, yet it remains a major constraint in scaling up manufacturing processes. On average, the time required to qualify the performance of a stack is **20 times longer** than the time it takes to assemble it!

To support the high-throughput manufacturing of reliable stacks, there is a need for accurate defect detection:
Current methods for performance testing, like electrical testing, lack the sensitivity required for assuring the performance of stacks confidently.

Electrochemical Impedance Spectroscopy (EIS) is a highly sensitive diagnostic technique capable of identifying even minor changes in cell and stack performance:
EIS is an extremely powerful diagnostic for detecting subtle performance changes, isolating for sources of performance loss and flagging early warning signs of failure. However, **traditional EIS systems are too slow and impractical for gigafactory environments**.

Pulsenics' EIS is engineered for industrial scale, decreasing the time it takes to qualify stacks from days to seconds:
Pulsenics has transformed EIS into a **rapid, plug-and-play, and industrially compatible** diagnostic solution. By using rapid pulses, Pulsenics' EIS reduces stack qualification time from hours to **seconds or minutes**, enabling manufacturers to accelerate production without compromising quality.

A case study: Using Pulsenics to detect and categorize manufacturing defects:
In this study, the Pulsenics EIS & Failure Cataloguer Platform was used to detect manufacturing and operational defects for a CO_2 electrolyzer.

Table 1 [1] showcases the failure modes characterized, with their key indicators. During this study, cell voltage data was not found to be an effective indicator of defects or failures. Not only did EIS present strong indicators to flag defects, but the different impedance parameters were also effective in identifying the type of failure exhibited!

Table 1. Failure modes and their key identifiers.									
Failure mode	Key identifiers (*: compared to baseline, ^{T}: with respect to time)								Detection time before visible in FE and voltage
	Electrochemical parameters				Voltage	FE			
	R0	R1	C_{eff}	W1	V	H_2	CO	C_2H_4	
Over-compressed	$\downarrow^{*}$	$\downarrow^{*}$	$\uparrow^{*}$			$\uparrow^{*}$	$\downarrow^{*}$		Immediate detection
Under-compressed	$\uparrow^{*}$	$\downarrow^{*}$	$\downarrow^{*}$			$\downarrow^{*}$	$\uparrow^{*}$		Immediate detection
Salt formation		$\downarrow^{T}$		$\uparrow^{T}$		$\uparrow^{T}$	$\downarrow^{T}$	$\uparrow^{T}$	~1 Hour
GDE flooding	$\uparrow^{T}$	$\uparrow^{T}$	$\downarrow^{T}$						Immediate detection
Cathode catalyst degradation	$\downarrow^{*}$		$\downarrow^{*}$ $\uparrow^{T}$			$\uparrow^{T}$	$\downarrow^{*T}$	$\uparrow^{*T}$	Immediate detection
Anode degradation		$\uparrow^{T}$	$\downarrow^{T}$		$\uparrow^{T}$	$\uparrow^{T}$			~11 Hours
Short circuit (prior)	$\uparrow^{T}$	$\uparrow^{T}$	$\uparrow^{T}$		$\downarrow^{T}$				~10 Hours
Short circuit (after)	$\downarrow^{T}$	$\downarrow^{T}$							

Developing the Failure Catalogue for Flagging & Categorizing Manufacturing Defects:
During cell assembly, cells need an optimal compression to prevent leaks but also not crush the cell components. This study compares signatures obtained from Pulsenics EIS compared to traditional performance testing methods, like cell voltage data, for under compressed and over compressed cells compared to a normally constructed cell.

Cell voltage measurements of different cells reveal negligible changes to their voltage. Fig. 1 gives the voltage measurements which show a less than 5% variation between the optimal and under compressed cell. Such a low margin would be difficult to confidently qualify cells off a production line.

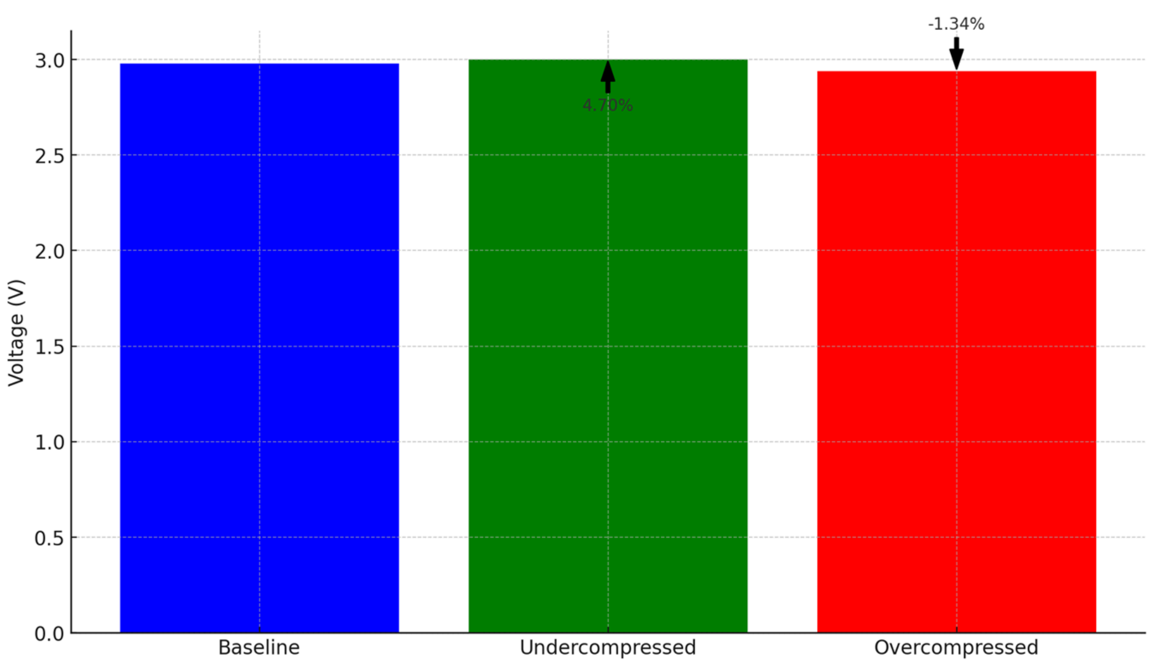

Fig 1: Voltage measurements of cells under three different compressions

With in-line EIS, non-optimal compression conditions showed a significant impact on cell impedance (Fig. 2). EIS separates not only impedance sources, but also their timescales leading to several distinct parameters that respond to cell compression.

Fig. 2 identifies two resistances from the impedance signature: the bulk resistance of the membrane and electrolyte, and the faradaic resistance of the redox reaction. Both see significant percentage changes compared to the baseline cell (>10%). Furthermore, bulk resistance has a unique response depending on under compression (53 % increase) vs over compression (13% decrease), which can be used to distinguish one error from the other.

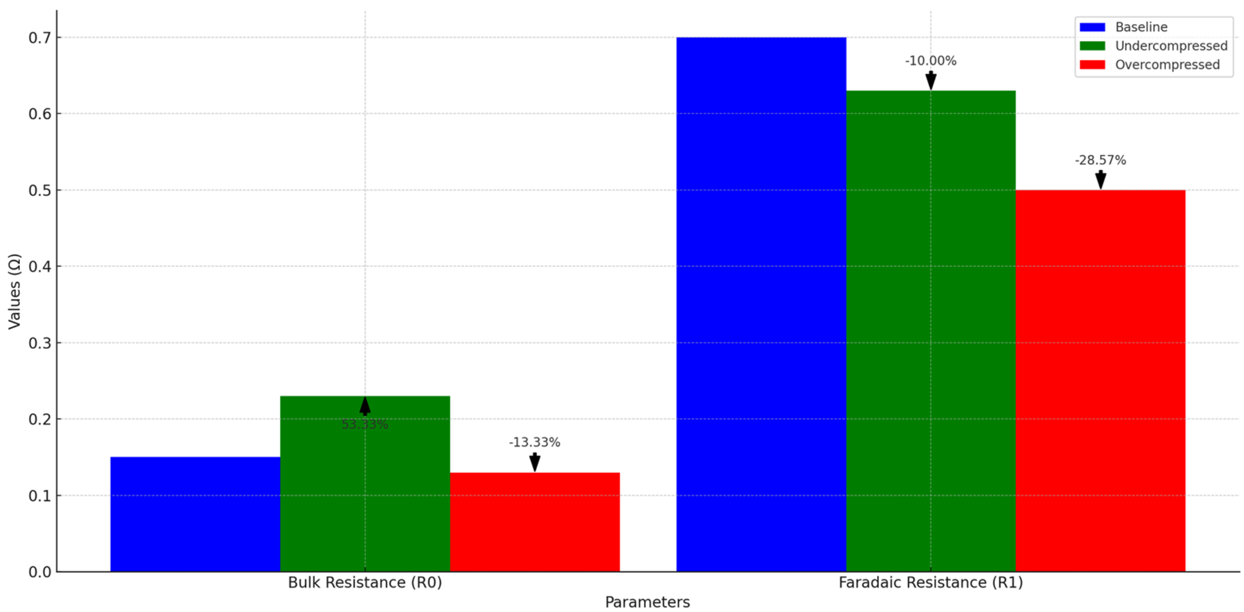

Fig 2: Bulk resistance and faradaic resistances derived from EIS under three different compressions

Impedance encompasses additional electrical behavior other than purely resistance. Capacitance, which is due to charge accumulation at the electrodes, is also captured in the impedance spectra and can serve as another failure signature. Fig. 3 shows the unique failure signatures of each compression. As with bulk resistance, under and over compression result in different capacitance signatures. This time, under compressed cells have reduced capacitance while over compressed cells have increased capacitance.

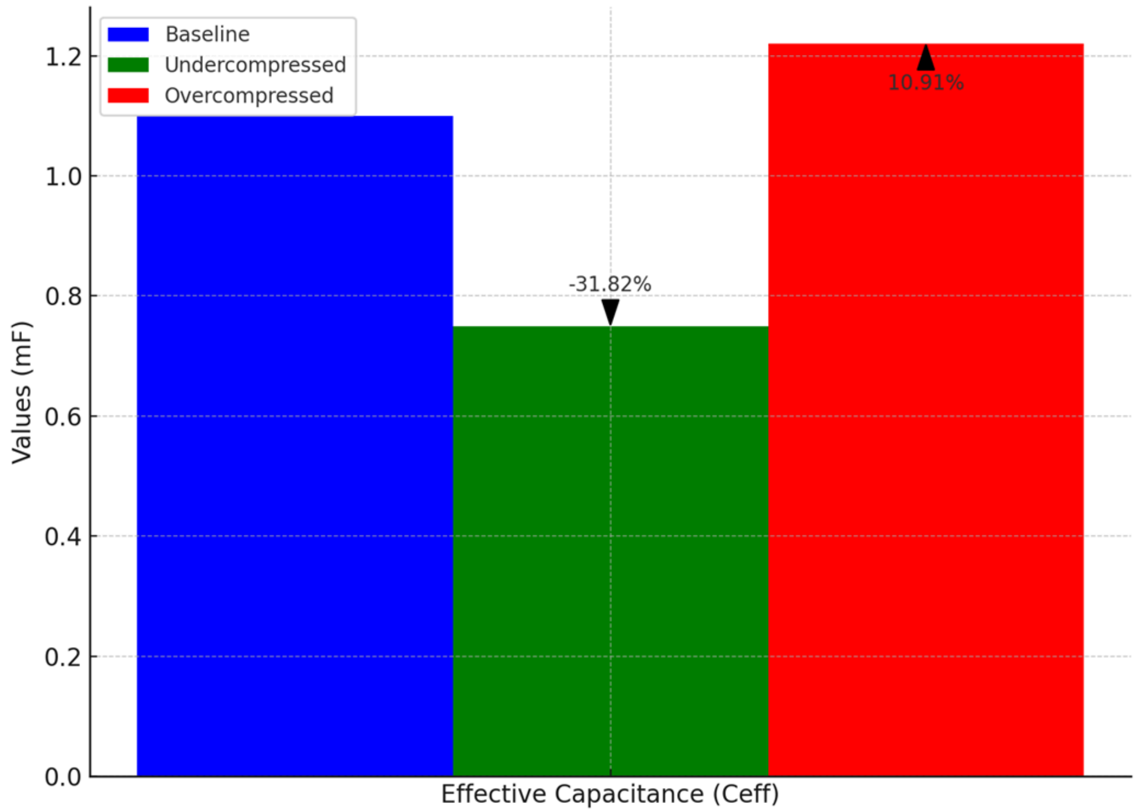

Fig 3: Capacitive impedance signatures for under and over compressed cells

The Pulsenics Failure Cataloguer takes data from Pulsenics' in-line EIS to automatically flag failures. Table 1 [1] highlights additional examples of failures detectable through EIS including flooding and catalyst degradation.

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Pulsenics' EIS is powerful for rapid and accurate determination of failures in QA/QC settings. The ability to detect subtle changes to cell electrochemistry, which are often hidden from other methods such as cell voltage, makes EIS a highly sensitive diagnostic tool. This is further enhanced through electrochemical parameter analysis, which isolates the impedance sources to specific components or mechanisms.

To learn more about identifying failures and building robust failure catalogues using EIS signatures, visit pulsenics.com or book a call with us.

[1] Warkentin et al., Early Warning for the Electrolyzer: Monitoring CO_2 Reduction via In-Line Electrochemical Impedance Spectroscopy, ChemSusChem, 2023, 16, e202300657