IEC TR 62282-7-3

Edition 1.0 2025-03

TECHNICAL REPORT

Fuel cell technologies –
Part 7-3: Test methods – Status of accelerated tests for fuel cell stacks and components and perspectives for standardization

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INTERNATIONAL
ELECTROTECHNICAL
COMMISSION

ICS 27.070

ISBN 978-2-8327-0277-2

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INTERNATIONAL ELECTROTECHNICAL COMMISSION

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FOREWORD

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IEC TR 62282-7-3 has been prepared by IEC technical committee 105: Fuel cell technologies. It is a Technical Report.

The text of this Technical Report is based on the following documents:

Draft	Report on voting
105/1091/DTR	105/1103/RVDTR

Full information on the voting for its approval can be found in the report on voting indicated in the above table.

The language used for the development of this Technical Report is English.

This document was drafted in accordance with ISO/IEC Directives, Part 2, and developed in accordance with ISO/IEC Directives, Part 1 and ISO/IEC Directives, IEC Supplement, available at www.iec.ch/members_experts/refdocs. The main document types developed by IEC are described in greater detail at www.iec.ch/publications.

A list of all parts in the IEC 62282 series, published under the general title *Fuel cell technologies*, can be found on the IEC website.

The committee has decided that the contents of this document will remain unchanged until the stability date indicated on the IEC website under webstore.iec.ch in the data related to the specific document. At this date, the document will be

- reconfirmed,
- withdrawn, or
- revised.

INTRODUCTION

The scope of accelerated testing is to reduce the time for qualification of the degradation or the long-term performance of a specific cell, cell component, stack, stack module or stack component compared to testing at nominal operating conditions. To generate an accelerated test, operating conditions stress a test item or one of its components, usually through the exaggeration of one of the testing parameters (the so-called stress factor). The results of this test are expected to provide a comparative assessment of the robustness (or degradation) of the test item and possibly – through an established transfer function – an estimation of the projected lifetime of the test item under nominal (non-stressed) conditions.

An accelerated test thus cannot be a self-standing experiment, since it cannot be applied universally to all cell technologies, architectures or material combinations. Degradation phenomena are different and occur heterogeneously for different fuel cell (stack) technologies and in different operating modes. Also, different fabrication process of cells and components can lead to different responses under stressed conditions (although the responses at nominal conditions could be similar). In any case, for each accelerated test, a benchmark test item (operated at nominal conditions and adequately characterized) is necessary in order to have a meaningful reference against which the accelerated test can provide the required understanding of long-term durability of that same item. Nevertheless, actual long term testing under normal operating conditions could be the only method to obtain an accurate degradation rate.

Tests on components (ex-situ tests) would only be relevant for comparison of such components, since the relevance for performance within a system can only be provided in the integrated assembly. For systems and end products, a generally applicable accelerated test for a given application (without the need for benchmarking) is certainly useful and is possible in the same way as there exist standardized drive cycles for vehicles/propulsion systems. However, it is still considered a major challenge to define an operating cycle that represents the actual application including events that contribute to degradation like start-stop cycles, air-air-starts, freeze starts, pressure and humidity cycles, temperature cycles etc. Nevertheless, standardized types of tests could be defined, generic for all types of fuel cells, with specific test conditions and cycles adapted to each application case and to each mission profile to be represented.

It is important to understand whether existing standards for testing performance of cells or stacks (e.g. IEC 62282-7-1 and IEC 62282-7-2, or IEC 62282-8-101 and IEC 62282-8-102) are sufficient for the definition of the testing approach, and only need a specific (quantified) definition of the required test parameters for them to be suitable for the accelerated estimation of durability or lifetime degradation. For example, an increase in operating temperature can be an accelerating test in solid oxide fuel cells (SOFCs). However, the measurements of electrochemical performance of cells/stacks at higher temperatures can be carried out according to the normal standard method (i.e. IEC 62282-7-2 or IEC 62282-8-101). The accelerated test is expected to enable an inference, from measurement of performance degradation at high temperature in this example, of the long-term durability of the tested cell/stack at nominal conditions (i.e. nominal temperature), and – crucially – in a shorter period of testing time than at nominal conditions. However, the increase of operation temperature (for example) can change not only cationic diffusion at the electrode/electrolyte interfaces but also the performance of electrodes/ionic diffusion. In this case, the one stress factor of increasing temperature can affect multiple performance degradation mechanisms of the SOFC, possibly with different time scales. Therefore, it will be difficult to univocally correlate an accelerating factor between degradation/lifetime in the test conditions and degradation/lifetime in nominal conditions. Defining a window of acceptance where the effects of one accelerating stress factor can be called representative of a single degradation mechanism can be a viable approach, even if other intrinsic mechanisms are affected to a lesser degree. To do this would likely require support from dedicated modelling activities, as well as by gathering the experience from manufacturers and comparing results with real long term durability tests.

In other cases, new, dedicated test procedures can be formulated to accelerate specific degradation mechanisms (e.g. controlled oxidation of solid oxide cell (SOC) electrodes to estimate redox stability).

In order to validate the reliability and representativeness of accelerated test procedures, post-test characterisation on samples having undergone tests are indispensable, also to compare the morphological/chemical state of test items after accelerated testing with samples from "real-time" ageing. Though this would fall outside the scope of TC 105, there is a description of such techniques and their applicability in Clause 4.

In this document, only intrinsic degradation mechanisms are considered, inherent to the operation of the cell/stack, and excluding degradation caused by externalities (impurities, shocks, etc.). Nevertheless, it is noted that an approach on contamination is still missing in the standards portfolio, both on fuel and air sides.

Finally, operating fuel cells in electrolysis mode will be considered, either for systems that can operate in reverse mode, or because it has been found that electrolysis mode can be a means to accelerate degradation mechanisms that occur in fuel cell mode.

This document has been compiled based on input from several National Committees and experts, convened in Ad Hoc Group 11 of IEC TC 105, and from two European projects funded by the Fuel Cells and Hydrogen Joint Undertaking (ID-FAST and AD ASTRA). The current Report represents a consensus on the status of accelerated tests for fuel cell stacks and components and outlines the perspectives for standardization of accelerated test procedures.

FUEL CELL TECHNOLOGIES –

Part 7-3: Test methods – Status of accelerated tests for fuel cell stacks and components and perspectives for standardization

1 Scope

The objective of this document is to answer several questions that have been circulating in the area of fuel cell development for many years: "What do we know about influencing fuel cell degradation, can we control it and accelerate it in a predictable way, is there a need for accelerated test procedures and can these be formulated adequately enough to be captured in international standards?"

This document is a generic assessment of the feasibility of standardizing accelerated test procedures (both proton exchange membrane (PEM) and oxide ion-conducting solid oxide cell (SOC) technologies) for fuel cell stacks that have been engineered for a specific system application. This document comprises a review of literature and projects, a discussion of the main physical phenomena of interest in accelerated testing campaigns (focusing on the cell and stack levels, not looking at the system as a black box), a compendium of measurement techniques that are applicable, and it suggests a macroscopic approach to the formulation of a representative accelerated testing campaign.

2 Normative references

There are no normative references in this document.

3 Terms, definitions, abbreviated terms and symbols

3.1 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

- IEC Electropedia: available at https://www.electropedia.org/
- ISO Online browsing platform: available at https://www.iso.org/obp

3.1.1
accelerated lifetime test
ALT
process of testing a product by subjecting it to aggravated operating conditions (stress, strain, temperatures, voltage, vibration rate, pressure etc.), thus in excess of nominal service parameters, in an effort to uncover faults and modes of failure in a short period of time to define explicitly its full lifetime

Note 1 to entry: This is thus a test for fast-forwarding time, and it is a destructive test in principle. Usually, it is conducted applying the more relevant failure stresses (this means that they are previously identified), aggravating normal conditions of use.

For example, if a manufacturer/supplier wishes to guarantee a product for e.g. 10 years, by ALT it can verify what is the risk to be accounted for, without waiting 10 years or more. This type of test is particularly relevant for components or systems that are to be introduced to the market.

3.1.2
accelerated stress test
AST
process of applying high levels of stress for a short period of time to a device under test (DUT) assuming it will exhibit the same degradation mechanisms as it would in a longer period of time at lower stress levels

Note 1 to entry: This is thus a test for assessing degradation generated by stress. Although it is non-destructive in principle, the device could also be damaged, but certainly less than during ALT. It is not finalised to the determination of the lifetime, but at the determination/study of potential long-term failure mechanisms and possible mitigation strategies. It is a test more suitable for research and development (R&D), because it is useful e.g. for verifying what the effect is of specific operating conditions on the test object or the system, with a view to developing better materials and components.

3.2   Abbreviated terms and symbols

3.2.1   Abbreviated terms

ACL	anode catalyst layer	LSCF	lanthanum strontium cobalt ferrite
ALT	accelerated lifetime test	MEA	membrane-electrode assembly
ASR	area-specific resistance	MPL	microporous layer
AST	accelerated stress test	NEDO	New Energy Development Organization
BET	Brunner-Emmett-Teller	OCV	open-circuit voltage
BoP	balance-of-plant	OEM	original equipment manufacturer
BPP	bipolar plate	PEFC	polymer electrolyte fuel cell
CCM	catalyst-coated membrane	PEM	proton exchange membrane
CL	catalyst layer	R&D	research and development
CV	cyclic voltammetry	RDE	rotating disc electrode
DUT	device under test	RH	relative humidity
ECSA	electrochemically active surface area	SEM	scanning electron microscopy
EIS	electrochemical impedance spectroscopy	SOC	solid oxide cell
EDX	energy dispersive X-ray analysis	SOFC	solid oxide fuel cell
EPMA	electron probe microanalysis	STP	standard temperature and pressure
FCTT	United States Fuel Cell Technical Team	TEM	transmission electron microscopy
FCCJ	fuel Cell Commercialization Conference of Japan	TPB	triple-phase boundary
FIB-SEM	focused ion beam SEM	XPS	X-ray photoelectron spectroscopy
GDL	gas diffusion layer	XRD	X-ray diffraction
HRTEM	high-resolution transmission electron microscopy	XRF	X-ray fluorescence
ICP	inductively coupled plasma		

3.2.2   Symbols

ASR	area specific resistance	$\Omega \cdot m^2$, $\Omega \cdot cm^2$
J	current density	A/m^2, A/cm^2
$\Delta J/\Delta t$	rate of current density variation	$A/m^2 s$
$q_{\dot{v}}$	rate of variation of the gas volumetric flow rate	m^3/s^2
T	temperature	°C, K
x	undefined parameter	-
$\Delta x/\Delta t$	rate of change of x	s^{-1}
$\Delta \alpha_r$	relative degradation rate	-

NOTE 1   To express the rate of change of a certain parameter x, a dot above the parameter symbol can be used. For instance, $\dot{T}$ can be used to express the temperature ramp with a unit of K/s, $\dot{q}_v$ used to express the speed of variation of the gas flow rate with a unit of $(m^3/s)/s$. Alternatively, $(\Delta x/\Delta t)$ can be used. For instance, the temperature ramp can be expressed by $(\Delta T/\Delta t)$ and the speed of current density variation can be expressed by $(\Delta J/\Delta t)$.

NOTE 2   The quantity $\Delta \alpha$ chosen for calculating the degradation rate can be the cell or stack voltage, current, power density or ASR, each with their own unit.

4   Outlook of a possible standard/specification on accelerated tests

From a supplier's point of view, a standard to define test methods for estimating the lifetime of the product (ALT) is of highest importance, also in view of engendering the required confidence to formulate product guarantees, which in turn are crucial for investors and project developers to market innovative solutions. After having defined the correct end application, accelerated tests are expected to predict lifetime and the influence of correlated events (freeze start, air-air-start, starvation, cell potential reversal, cycling, etc.). A standard for these tests would define the test and measurement methods, the testing protocols (e.g. kinds and ranges of stress to be applied, etc.) and provide a clear indication of the degradation mechanisms that the application of such procedures would accelerate. The definition of certain test parameters can be given (e.g. air and H2 quality) to be able to compare different solutions by a common test procedure.

In the case of ALTs, it is important to quantify the degree of acceleration that the tests would lead to. In the case of ASTs, used for R&D, the aim of these tests would be to provide a benchmark for comparison between different products/components/materials as to the response to a specific degradation mechanism or event. In order to predict the influence of single events on lifetime, further systematic tests can reveal, by knowing type and number/frequency of the defined events and the nominal operational profile of the tested system, the resulting lifetime in "real-world" conditions (not in accelerated tests).

Possibly, indications can be given as to the number of test repetitions required for reliability, so that the standard could be used also for "certification" by accredited bodies.

In the rest of this document, reference will be made to accelerated tests generically, without specifying ALT or AST, since each accelerated testing procedure can be declined as addressing a specific degradation mechanism (AST) or estimated lifetime (ALT) depending on the parameter and protocol definitions. This differentiation will be the task of a subsequent work on a Technical Specification or International Standard.

5   Project review and suggested liaisons

Certain fuel cell manufacturers have developed internal correlations between accelerated tests and full-scale duty cycles, but these results tend to be treated as trade secrets because of their commercial value. There are a number of available documents or liaisons that can be useful in the development of a possible standard on accelerated testing.

A liaison is suggested with the US Department of Energy (US DoE) project on accelerated tests within a framework project called HYDROGEN, which sees the collaboration of players from France, USA and Japan. Accelerated testing protocols for PEFCs, specifically for heavy duty vehicle applications, are being developed in a collaboration between M2FCT (USA), IMMORTAL (EU), and FC-PLATFORM (Japan) with original equipment manufacturers (OEMs): sharing of drive cycles from the 21st Century Truck Partnership (21CTP) as well as system modelling efforts. Also, a group "Baselining, MEA testing and protocols" will explore MEA testing from 1 cm^2 cells (Japan), 5 cm^2 and 50 cm^2 cells (USA) to short stacks (EU) for better understanding a scaling of performance and durability from small differential cells to operating stacks.

Besides one accelerating stress factor representative of a single degradation mechanism, combined effects on the durability are recognized to be significant. For example, a chemical/mechanical protocol has been proposed to examine the combined effects of humidity and radicals' formation on the durability of polymer electrolyte membranes [1][1]. A new "highly accelerated stress test" for the combined chemical mechanical degradation has been proposed by General Motors and is currently utilized by 3M [2]. The M2FCT project has also announced that they will employ this protocol.

The U.S. DRIVE Fuel Cell Tech Team Cell Component Accelerated Stress Test and Polarization Curve Protocols for PEM Fuel Cells use the existing profiles for automotive accelerated testing, though it is unclear how widely these protocols are used in industry. Some aspects of these profiles are considered unsatisfactory, leading to development of internal protocols.

In the Japanese New Energy Development Organization (NEDO) Project on durability and reliability of SOFC stacks (2013-2019), there are numerous data which are related to accelerated stress testing. Among them, the following tests were conducted as accelerated stress testing on stacks, and these tests are the main candidates for accelerated stress testing:

1) intentionally adding of impurities to the electrodes in single cells (such as Cr-vapor, S-vapor, and Si-vapours, etc.);
2) thermal cycling of SOFC stacks,
3) high fuel utilization test at constant current density in the stacks (up to 85 % U_f),
4) load cycling tests with constant fuel flow in the stack.

For polymer electrolyte fuel cell (PEFC) test protocols, a publication is available by NEDO (December, 2012).

The Chinese standard GB/T 38914-2020: "Evaluation method for lifetime of proton exchange membrane fuel cell stack in vehicle application". is already defined but it has strong and generally unproven assumptions. The strongest assumptions are (1) linear independence of degradation triggered by separate events, i.e. assuming no path dependency, (2) linear extrapolation allowed up to user defined degradation limit (e. g. 60 mV voltage loss at nominal load).

A correlation with monitoring and diagnostic tools installed in real stacks/systems could be useful (see EU-funded projects INSIGHT/RUBY/REFLEX for instance).

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[1]   Numbers in square brackets refer to the Bibliography.

In the Bibliography, a number of useful website resources [3] to [6], publications on measurement procedures [7] to [14], relevant scientific papers [15] to [28] and standards [29] to [44] is given, as well as an indication of European [45] to [52], North-American [53] to [58] and Japanese [57] and [60] projects that have contributed to the state of the art on accelerated testing.

6   Intrinsic degradation mechanisms and their effects

In the following, an overview is given of those degradation mechanisms that characterise long-term lifetime limitation, and which it would be desirable to accelerate in order to assess their effect over very long operation times in significantly shorter testing times. The focus is on intrinsic mechanisms, i.e. those mechanisms that are unavoidable and related to the "natural" deactivation of a component due to the occurring conditions of use. Extrinsic mechanisms (poisoning, mechanical shocks etc.) are important causes for lifetime limitation but are not strictly necessary to be accelerated.

Given that:

– an AST correlates to a specific benchmark (materials, application area, etc.),

– a sensitivity analysis is needed to determine the window of acceptance of the effects of a given accelerating stressor,

the Quantification methods in Table 1 consider the operating regime for which each are applicable. The tables below (Table 1 for PEFC technology first, Table 2 for SOC technology afterwards) are intended to help correlate:

1) the identification of the degradation problem,

2) a possible simplification of it in terms of cause (the most important stressor),

3) what to measure to verify the effectiveness of this simplification,

4) how to evaluate the accuracy of the simplification.

As pointed out in the introduction, there are difficulties to specify one single stress factor for each degradation mechanism. For example, the process fabrication temperature (firing temperature) of SOCs and materials combination can affect the degradation factors presented in the table. Also, the operation mode (in particular for reversible operation as described in IEC 62282-8-101) will lead to different degradation mechanisms even with the same cell configuration. It is important that these effects are considered and included when considering the "window" of the accessible stressors.

As indicated in Table 1 and Table 2, the effect of the degradation mechanisms can be qualified by post analyses methods. However, these are out of scope of the IEC TC 105 standards. Test procedures rely on electrochemical measurements, such as I-V characteristics, long-term potentiostatic or galvanostatic measurements, current interruption methods, and/or electrochemical impedance spectroscopy (EIS) measurements.

Table 1 – PEFC degradation phenomena, governing parameters and diagnostics

Degradation mechanism (primary cause)	Stressors	Effects and consequences	Quantification methods
GDL carbon corrosion	Gas switching on cathode Temperature Cell hydration Cell overpotential Cathode high potential (caused by reverse current) Anode high potential (cell reversal due to fuel starvation, caused by anode stoichiometry < 1)	Decrease in hydrophobicity Increase in gas transport resistance Increase in ohmic resistance	GDL or MPL transport properties (in-situ?) Limitations such as transport resistance, protonic resistance (by EIS) Cyclic voltammetry (effective catalyst surface area) Current mapping SEM analysis, also contact angle measurement as hydrophobic behaviour can be affected, also porosity measurements
Cathode catalyst layer carbon corrosion	Cathode high potential (Caused by reverse current, (anode flooding, H2 starvation if anode stoichiometry < 1), such as during start-up, with H2/Air front) Gas switching on cathode Temperature High relative humidity	Contribution to catalyst particles modification: catalyst particle modification → detachment of catalyst particles, agglomeration of catalyst particles, dealloying (Pt-alloy) Contact loss leading to increase in protonic and electronic resistance Thinning of the catalyst layer Change in porosity	Cyclic voltammetry (ECSA losses and modification of electrochemical properties vs. PtOx formation/reduction) Limitations such as transport resistance, protonic resistance (by EIS) Current mapping SEM and TEM analysis CO2 exhaust measurement Raman spectroscopy
Anode catalyst layer carbon corrosion (cell reversal)	High potential (→avoid a mixture of stressors like membrane drying during the test!) Anode high potential (caused by anode stoichiometry < 1)	Support oxidation Loss of carbon support of the ACL → reduced anode ECSA, evolution of anode microstructure	ECSA, SEM post-mortem, Anode catalyst composition / where can alloy components be found after test? Raman or XPS for post-mortem carbon morphology characterization Cyclic voltammetry (ECSA losses and modification of electrochemical properties vs. PtOx formation/reduction) Limitations such as transport resistance, protonic resistance (by EIS) Current mapping SEM and TEM analysis

Degradation mechanism (primary cause)	Stressors	Effects and consequences	Quantification methods
Anode corrosion (cell reversal tolerance)	High potential (→avoid a mixture of stressors like membrane drying during the test)	Pt-oxidation, oxidation/dissolution of other metals	Cyclic voltammetry (effective catalyst surface area) Current mapping SEM analysis distribution throughout the cell / Ir-Ir/Ru-mapping; Pt-concentration by ICP
Catalyst de-alloying / selective corrosion, structure changes due to dealloying Multi-metallic Pt based catalysts electrochemical Ostwald ripening and oxidation / dissolution	High potential causing oxidation / dissolution Local flooding Potential cycling Temperature Cell hydration	Surface area loss Dissolution of smaller particles Modification of particles surface composition Contamination of ionomer (membrane and catalyst layer) by metallic cations Pt precipitates within the ionomer	Cyclic voltammetry (ECSA losses and modification of electrochemical properties vs. PtOx formation/reduction) Advanced electron microscopy (SEM, TEM, EDX), effluent analysis (ICP) for alloy components, metal mapping in MEA. HRTEM of particles with element mapping. ECSA can increase by corrosion of alloys
Platinum oxidation/ dissolution/ agglomeration/ Electrochemical Ostwald ripening	High potential Potential cycling (for PtOx and dissolution based mechanisms) Temperature (High T > 80°C) High humidity (wet or flooding conditions) Cell hydration	Surface area loss (all modes) Dissolution of smaller particles Pt precipitates within the ionomer	ECSA assessed by cyclic voltammetry Charge transfer resistance assessed by EIS Particles size distribution, catalyst and catalyst layers structure modifications by advanced electron microscopy (SEM, TEM, EDX)
Ionomer degradation for the membrane and the catalyst layers (chemical stability losses)	Cell potential including OCV Temperature Relative humidity (low RH or T and RH cycles for the membrane) Cell hydration O2 permeation through membrane (triggers H2O2 formation on anode – chemical stress on ionomer and membrane on anode side) Impurities (Ions) from stack (BPP) or bench for membrane chemical attack (scope?)	Mass transport property changes (water uptake, wetting) Proton conductivity losses Ionomer distribution within the membrane or the CLs Gas leakage through the membrane	Mechanical or electrochemical leak test to assess permeation (H2 crossover by voltammetry) High frequency resistance and CL protonic conductivity by EIS Product water conductivity (fluoride emission) Membrane or CL structure modifications by advanced electron microscopy (XPS for structural changes in the ionomer?)
Membrane cracking (mechanical degradation including relationship between cathode layer cracking and membrane failure; migration of CeOx))	Wet-dry cycles Temperature Stack mechanical compression (uneven)	Gas leakage through the membrane	Mechanical or electrochemical leak test Product water conductivity SEM analysis Mechanical properties of membrane

Degradation mechanism (primary cause)	Stressors	Effects and consequences	Quantification methods
Metallic bipolar plate degradation (base material and coating)	High potential	Increase in contact resistance, delamination of coating, poisoning of MEA	Resistance measurements (in- and ex-situ), effluent analysis, also post-mortem MEA-analysis for metal ions, SEM/X-EDS/XPS for coating characterization
Graphite bipolar plate degradation	High potential	Loss of carbon	SEM, contact angle (?), Raman, increase in roughness, change in surface structure
Sealant degradation	pH, high potential, fluoride concentration, contact to membrane	Disintegration of elastomer, contamination of other components by degradation products	SEM (elastomer surface), EDX, XPS (composition of elastomer, elemental mapping over the cell, dependent on elastomer)

Table 2 – SOC Degradation phenomena, governing parameters and diagnostics

Degradation mechanism (primary cause)	Stressors	Effects and consequences	Quantification methods
Nickel agglomeration + Ni migration	Temperature Thermal cycling Cell polarization Steam content	Change in microstructures in Ni-oxide cermet Loss of TPB active area Loss of Ni particles connection	Microstructural characterization techniques like SEM analysis and more accurate techniques if needed ASR measurements I-V characteristics
Ni re-oxidation	Steam content (low H_2 partial pressure) Fuel starvation Redox cycling	Change in microstructures in Ni-oxide cermet Loss of TPB active area Ni(OH) or NiO formation	SEM analysis ASR measurements I-V characteristics
Ni evaporation	Steam content	Change in microstructures in Ni-oxide cermet Loss of TPB active area Ni(OH) or NiO formation	ASR measurements I-V characteristics
LSCF demixing	Polarization Current density Oxygen partial pressure Humid air	Change in microstructures Active surface reaction area reduced Sr segregation	SEM analysis ASR measurements I-V characteristics

Degradation mechanism (primary cause)	Stressors	Effects and consequences	Quantification methods
Phase transformation (at ZrO_2 based electrolyte)	Polarization Temperature	Cubic → Tetragonal phase transformation Ni dissolution will facilitate the Tetra formation	SEM analysis Current interruption Raman spectroscopy
Interface reaction/diffusion Formation of resistive layers	Temperature Polarization Current density	Change in microstructures Formation of insulating phase (e.g. $SrZrO_3$)	EIS analysis SEM analysis ASR measurements I-V characteristics Current interruption
Crack/Interface delamination, exfoliation	Temperature Thermal cycling Mechanical pressure Oxygen partial pressure	Loss of contact surface, which induces an increase in ohmic resistance	EIS analysis SEM analysis
Contact loss	Temperature Thermal cycling Mechanical pressure Oxygen partial pressure	Loss of contact surface, which induces an increase in ohmic resistance	EIS analysis, SEM analysis
Interconnect oxidation	Temperature Atmosphere (enriched air, pure O_2, steam content)	Formation of an oxide phase that increases ohmic resistance	SEM analysis
Chromium evaporation	Temperature, pressure, Atmosphere (especially steam content)	At the interconnect, the presence of chromium in the metal substrate can lead to volatile oxide, oxy-hydroxide and/or hydroxide formation that could release Cr species through evaporation that will subsequently poison the cell (formation of secondary phases and change in composition of the ceramic)	SEM, TEM, Raman spectroscopy, X-ray diffraction, EDX
Tightness loss	Flow rates / pressure in stack Delta P between fuel and air chambers	Mixing of fuel and air in the stack that can combust, or loss of fuel out of the stack, decreasing the stack performance	OCV, ratio between inlet and outlet flow rates, T measurement, SEM analysis

7 Quantification methods for determining (accelerated) degradation

7.1 General

In order to correlate the degradation effects under specific accelerated testing conditions with those under nominal conditions, and thereby make a statement as to the expected lifetime in nominal conditions based on accelerated test results, it is important to quantify the effects of degradation in both conditions and find a way to correlate them. There are a number of techniques available for degradation quantification, some of which are in-operando (measurements during testing) and some of which are post-test. These are listed below. Note that these techniques just describe diagnostic methods but do not give indications on testing conditions that could be valid for accelerating degradation in a way that can be extrapolated to regular degradation.

7.2 In-operando diagnostic methods

Valid for PEFC and SOFC:

– Determine voltage increase (electrolysis) or decrease (fuel cell) over time, if operating at constant current (or current decrease over time if operating at constant voltage), associated with a given accelerating stressor or a combination of stressors. It is useful to monitor other parameters during such tests as well, e.g. temperature, ratio between inlet and outlet flow rates.

– In addition or instead of an i-V curve, it can be an option to use a design-of-experiment (DoE) approach, changing different parameters (current density, temperature, pressure, RH, stoichiometry etc.) to find effects that could not be visible in a standard i-V curve operation. Model-based data analysis will give the influence of each parameter.

– The method of current interruption can be useful for measuring instantaneous performance with accuracy. This method can be applied to distinguish the ohmic and electrode polarization separately for practical stacks. Anode and cathode polarization can be identified through the dependence of stack response as a function of partial pressures of gases at the fuel and oxygen electrodes (H_2, H_2O, O_2).

– Also the start of the mass transfer limitation region, under different conditions, is an indication for a change in properties (i.e. electrochemically active surface area (ECSA) or hydrophobicity in PEFC).

Valid for SOFC only:

– Determine the evolution of the area-specific resistance (ASR, from EIS or based on the current-voltage curve, see e.g. IEC 62282-8-101), which can be compared to the ASR evolution at nominal operating conditions. To this effect, record i-V curves and take EIS measurements (for calculation of the ohmic resistance, mass and charge transfer resistances, polarisation resistance and total resistance increase) regularly during the ageing test, but without affecting too much the long-term testing profile. For example, EIS measurements are taken around the current density at which the test sample is operated for the test, and i-V curves are carried out so that they do not lead to excessive cycling. For reference, perform a durability test at defined condition, for at least 2 000 h, and calculate ASR degradation. The procedure is repeated in accelerated testing conditions and the ASR evolution rates are compared.

– Define a (set of) reference or standard condition(s) of operation determining a "state-of-health" of the tested sample. For example, cell/stack response at OCV conditions can be considered a "state-of-health", which also avoids polarisation effects (in case of SOC only; in PEFC, hydrogen crossover can cause polarisation at the cathode). Then, carry out a reference durability test at conditions that are different to the "state-of-health" conditions. Record i-V curves and take EIS measurements before and after a durability test in the "state-of-health" conditions and compare the difference. This gives you an assessment of the intrinsic change in state of health caused by the intervening durability test. An accelerated test that is accurate is expected to generate the same change in state of health as the reference durability test in "regular" operation (but in a shorter period of time).

7.3 Mathematical transfer function formulation method

Modelling activity can help in identifying the correlation between degradation effects and stressing parameters. This can indeed improve the standardization of experimental protocols and their application to different component technologies. The use of models to reproduce degradation mechanisms and physical phenomena in detail can also help in reducing time and costs for the characterization of such correlations, reducing the need for extensive experimental campaigns. Moreover, establishing the statistical significance of reproduced phenomena could also consolidate the validity range within which the degradation rate estimation falls; this approach addresses industrial needs, reducing the problem of features fluctuation of components due to manufacturing processes.

Mathematical models capable of representing the degradation phenomena at different levels can be combined for the identification of the stressor-degradation correlation. Although the models needed will strongly depend on component features (mainly related to different manufacturer specifications, manufacturing processes, physical characteristics, different materials, etc.), defining a generalized approach is fundamental to provide a robust and repeatable methodology that can be used by different bodies. An example of such a methodology has been presented in [27]. In this work the attention was oriented towards Ni agglomeration degradation effects on SOFC operation, but a general approach in eight consecutive steps was proposed:

– Step 1: identify the degradation mechanism(s) and design models of the affected parameters at microscale level;

– Step 2: identify the affected mesoscale parameters and design related models;

– Step 3: design a simplified performance model that can embed the aforementioned models at different scales;

– Step 4: link the models through a multiscale approach;

– Step 5: apply a parametric analysis to assess the influence of the operating conditions on degradation rate;

– Step 6: identify the accelerating stress factors;

– Step 7: design mathematical transfer functions to correlate accelerating factors and degradation rate;

– Step 8: develop quantitative guidelines for lifetime estimation and accelerated test protocols design.

As remarked before, the models that can be used depend on operating mechanisms and component features, but the proposed methodology can be used as a generalized "check-list" for a modelling approach to accelerated test protocols standardization.

7.4 Post-test methods

Post-operation analysis can determine the physical/material/chemical state of the tested sample. For an accelerated test to be valid, this state is expected to correlate to that of a sample operated under nominal conditions, defined as the reference and representative of in-the-field ageing. In Table 3, there is a brief description of methods and corresponding properties that can be determined:

**Table 3 – Examples of diagnostic methods for the measurement
of key degradation properties**

Diagnostic method	Properties measured	PEM	SOC
Structural			
Optical inspection	Leaks, oxidation	OK	OK
SEM-/SEM-EDS/TEM	Layer compositions and thicknesses	OK	OK
SEM/EDS/EPMA	Catalyst layer loadings and thicknesses	OK	OK
XPS/XRD/XRF	Surface and bulk chemical composition	OK	OK
Raman spectroscopy	Phase transformation of Zirconia electrolyte	n.a.	OK
FIB-SEM	Reconstruction of electrode morphology	OK	OK
Profilometry and Interferometry	Thickness and thickness variation	OK	OK
Mechanical testing	Tensile strength test for membrane, compression set for GDL	OK	n.a.
Catalyst & kinetics			
TEM/EDS	Catalyst particle size and size distribution, support micro-structure	OK	OK
ICP	Catalyst particle composition	OK	OK
N_2 adsorption/BET	Catalyst support surface area	OK	OK
RDE & voltammetry	Catalyst activity at low overpotential and electrochemical surface area	OK	OK
Small differential single cells	Electrochemical analyses (incl. all in-situ methods) of MEA samples from large size, field-operated MEAs	OK	n.a.
Floating electrode	Catalyst activity at high overpotentials	OK	OK
XRD	Crystal structure and average crystallite size	OK	OK
Electronic charge transport			
4-probe impedance	Electrical conductivity	OK	OK
Contact resistance	Contact resistance	OK	OK
Mass transport			
Mercury porosimetry	Porosity, pore-size distribution, tortuosity	OK	OK
N_2 adsorption/BET	Surface area, small pore structure	OK	OK
Dynamic vapour sorption	Water uptake / swelling properties of membranes	OK	OK
Gas analysis	H_2, N_2, O_2 permeability	OK	OK
Water intrusion Contact angle	Hydrophobicity (difficult for a GDL ripped off the MEA)	OK	n.a.
n.a. stands for "not applicable".			

Post-test analysis is critical to assess the heterogeneity of the ageing from cell to cell and across the length of the cell. The quantification of heterogeneity is critical for multiple ageing modes, especially for PEFC Pt surface area loss, membrane failure, and bipolar plate impedance rise. A fully implemented segmented cell with an extensive sensor strategy could shift the emphasis to in-operando diagnostics, but the full understanding would still require post-test analysis.

Good quality, replicable post-test analysis is therefore considered helpful for benchmarking the real-world state that aims to be approximated by accelerated tests, but providing guidelines/procedures for post-test analysis is out of scope of TC 105. If necessary, they can be shown in an appendix or annex.

However, a mature accelerated testing procedure allows to determine the end-of-test state of health from in-situ cell/stack testing only without the need for extensive post-test analytics. In order to use appropriate testing conditions (e.g. voltage limits) and to interpret the results correctly (e.g. cyclic voltammetry (CV) data), it is important that materials of the sample be known (e. g. Pt vs. Pt-alloy). Today's established accelerated test procedures (e.g. DOE catalyst tests = voltage cycling under H2/N2) are well suited for material screening but do not allow for a direct correlation of the number of accelerated test cycles with a load profile test in a cell/stack.

Another useful approach could be to carry out characterization tests (ASR measurement, EIS, etc.) on samples taken from in-field-operated stacks. If it is possible to disassemble and extract usable samples from such stacks, this would provide valuable insight into the response of a sample that is already aged in the field, provided enough information from the in-field operation conditions is available.

8 Approaches to accelerated testing

8.1 Internal reference test and accelerated test validation

In order to adequately off-set the effects of different test facilities carrying out accelerated tests, it is important to always create an internal reference test for each test item. This test is carried out on baseline components which form a comprehensive benchmark for the evaluations under accelerated test conditions.

8.2 Single-component accelerated tests

For the purpose of selecting or developing a specific component, it is sufficient to carry out accelerated tests on this component alone, but it is important to also take into account what the effects would be in a stack environment. A window of acceptance can be defined where stress tests on a single component can be considered representative of conditions in real long-time operation.

As validation of a single-component accelerated test, a single long-term durability test of the component in stack environment can provide a benchmark for the single-component accelerated test.

Single components can be tested both for the understanding of ageing mechanisms in accelerated stress tests (e.g. for PEFC stack components such as the CCM, MEA, GDL, BPP, gaskets) or for evaluation of lifetime and failure modes (in accelerated lifetime tests for components such as MEA, BPP and unitary BoP components).

For PEFC, there is a possibility for defining a standard cell set-up structure (flow field for gas and coolant, or alternatively well-defined thermal boundary conditions) and operation (e.g. coolant flow, stoichiometry, relative humidity, pressure, temperature) to have a unique benchmark on which to base the internal reference test, and which would also allow comparability of test data between different entities (customer/supplier, public institutions). This approach is being pursued in TC 105/WG 106 for performance testing and could be considered in accelerated testing as well.

In solid oxide cells/stacks, the wide variety of cell-stack structures, such as planar, flat-tubular, and tubular, make it difficult to define such a standard, therefore only prescriptions on the test conditions (gas flow, temperature, current density, etc.) are feasible. This approach is being pursued in the EU harmonization activities regarding high-temperature electrolysers [30].

Furthermore, where accelerated testing is used for design development, or blind-test selection of components, only ranking will be used so there is no need for an "absolute" reference.

8.3 Stack environment and system influence: combining degradation mechanisms within an accelerated test

Accelerated test protocols addressing single mechanisms (or partly combined for one single component like the membrane) exist for sub-scale tests, similar to existing accelerated test protocols defined by US DoE, FCTT, NEDO, FCCJ. It is useful to include the influence of the system level into the definition of test procedures: so that e.g. gas flow, water management, and thermal management strategies are addressed by the test procedure (e.g. perform test at two different temperatures or different sets of combined conditions of *T/RH/p*).

In new protocols, an application-oriented approach would be interesting, thus extending the scope to multiple mechanisms occurring at one or more components concomitantly to replicate at least partly the real ageing situation. This could mean a standard protocol to be considered for each application (e.g. passenger car or commercial vehicle, etc.) particularly when going from core components level to cell or stack scale. Tests considering assemblies would focus on lifetime estimation (i.e. accelerated lifetime tests on e.g. short-stack, full-stack, module and system) rather than studying single ageing mechanisms.

In SOFCs, utilization of high steam content can trigger multiple degradation mechanisms, especially if combined with varying H_2/H_2O ratios (at the negative electrode side). The direction of polarization can also be used to emulate SOFC and solid oxide electrolysis cell (SOEC) conditions at the same time.

In this regard, it is essential to define a window of acceptance where a given stressor accelerates a specific mode of degradation sufficiently well without affecting other degradation effects in a way that makes the test not representative of real conditions. A number of degradation effects that could be grouped in a viable way (to be further considered):

PEFC:

–   ionomer degradation + membrane degradation,

–   platinum dissolution + catalyst de-alloying,

–   catalyst particles dissolution + carbon support corrosion,

–   cathode catalyst degradation + membrane degradation,

–   anode corrosion + membrane degradation,

–   catalyst de-alloying + ionomer/membrane contamination,

–   bipolar plate coating corrosion + membrane degradation,

–   GDL degradation + catalyst degradation (related to hydrophobicity losses or to carbon corrosion).

SOFC:

–   Ni agglomeration and migration,

–   interconnect oxidation and contact loss,

–   interconnect oxidation and Cr poisoning,

–   LSCF demixing and phase transformation,

–   delamination and contact loss.

8.4 Product duty cycle

Besides taking into consideration the effects of an accelerated test and the possible convolution of degradation mechanisms accelerated, the accelerated test needs also to be representative of a specific product duty cycle, imposed by the application and characterized by:

– size and frequency of potential transients,

– operating current density or load profiles,

– frequency and type of start-up/shutdown cycles (e.g. air-air starts),

– ranges or usage profiles of operating conditions (e.g. temperature, pressures, humidity).

To cover different possible product duty cycles and their specific conditions, it is unavoidable to define and perform accelerated tests within a range of conditions:

– e.g. different temperatures, or different sets of conditions,

– several different potential ranges or load profiles to replicate potential cycling,

– generate a design curve showing relationship between degradation and stressor.

These are then defined for each accelerated test that represents a specific duty cycle, to allow a more effective translation of accelerated test results to the actual duty cycle results.

Where the product duty cycle is determined by the manufacturer, it could be described that the cell/stacks should be pre-conditioned (ageing) by a method specified by the manufacturer before the actual accelerated test.

8.5 Combining accelerated tests to an accelerated test programme

What is missing in current protocols are combined ageing tests. Combined ageing (e.g. a "pseudo-real" drive cycle test) for multiple mechanisms is very important and currently not defined. The individual composition of several operating blocks is possible, but with a suggestion for standard profiles (like for a passenger car vs. a commercial vehicle). Such an approach has been explored in the ID-FAST project [47].

Examples for building blocks of a PEFC test programme are expected to represent the end use application to appropriately characterize the degradation expected. For example, combining phases of end use operation, such as:

– rated power,

– idling current,

– start and stop,

– open circuit voltage.

Specific usage cycles (cars, heavy duty vehicles, stationary, …) could be based on a further definition of conditions for these 4 tests.

Examples for building blocks of an "accelerated drive cycle test":

– load cycling blocks (e.g. lower power and/or high power) including possibly idle and start-up steps,

– idle blocks at high voltage for short periods (e.g. 0,85, 0,80 or OCV for 1 s or 15 s or 60 s),

– start-up blocks representative of different real situations (e.g. Air/Air or H_2/H_2 with true H_2/Air front on anode or cathode sides).

Full accelerated test programmes can also include specific conditions or combined tests addressing chemical and/or mechanical membrane ageing with, e.g.:

– relative humidity cycles + OCV,

– relative humidity cycles + anode/cathode pressure difference.

Accelerated testing blocks could be arranged as necessary to represent a specific application (e.g. a passenger car vs. a commercial vehicle).

It is important in this regard to verify whether the simple superposition or juxtaposition of stressors (accelerated tests) leads to an allowable estimate of combined degradation. The order of test blocks will also have an influence to be considered.

Examples of accelerated test programmes for SOC technology are still under development. An approach has been envisaged in the AD ASTRA project [45].

9   Accelerated testing environment

The suggested lowest level for a meaningful accelerated test is single cell components/layers, but at full size. For practical application, it is important to use the stack (at least a short stack of few cells) for stress testing to simulate the real application. Accelerated test procedures are somewhat different for each level. For example, it could make sense to use voltage control to perform load-unload testing for single cells, whereas it is much more intuitive to use current/power control for stacks.

Gas flow control can be another important factor to determine the stack performance. The cell-stack geometry and shape can be different in the SOC case (such as flat-tubular, tubular, planar and some others) and the specific flow-field plates and designs for the PEFC case. A change of the cell/stack shape will affect the gas flow and induced stress for core components and performance degradation, which can be considered for accelerated test protocols.

Developing a test profile that is independent of cell/stack geometry would allow given accelerated test profiles to be applied to any test cell, full-sized cell, or full stack. The challenge is that cell flow-field and MEA design can have an impact on response of cells to different (accelerated) test conditions (e.g. drier reactants at cell inlet can accelerate ionomer degradation locally vs. more hydrated reactants towards outlets after reactants have absorbed product water). A baseline protocol can be provided so that accelerated test characteristics can be normalized – within certain limitations – between different cell designs. It is therefore important to dedicate more development work to identifying the ranges within which the aggravation of testing conditions (leading to accelerated degradation):

– is representative of the targeted application and product duty cycle in real-world operation,

– does not alter significantly the interdependency or eminence of intrinsic degradation mechanisms,

– leads to an increase of degradation that can be correlated in a quantifiable way with the degradation in real-world operating conditions,

– leads to a profile of degradation that allows to predict durability in real-world conditions,

– can be applied to cells/stacks of different sizes, geometries, assembly structures, materials,

– can be combined (contemporaneously or in sequence) to evaluate different characteristics or operation modes.

Once there is more certainty as regards the applicability of defined aggravated conditions according to the points above, there will be a better base to work on for the normalization of these test conditions in procedures fit for IEC standardization.

10 Conclusions

An accelerated test is expected to be within some hundreds or a thousand hours and be able to predict (in the case of an accelerated lifetime test) the lifetime over respectively 8 000 hours to 40 000 hours (5 years) at least, depending on targeted applications.

It is in any case important to have appropriate industrial benchmark data to refer to when defining an accelerated test to avoid addressing non-relevant mechanisms.

In order to be applicable to the many different geometries and configurations of cell/stack technology, it is therefore suggested to define an accelerated testing approach (a consistent methodology), which defines the stressor parameters to work with for a given component/cell/stack and degradation mechanisms and identifies the physical indicators to be measured and monitored.

The approach thus identifies the required data from a real-world case that are the reference objective for the given accelerated test.

It is necessary to define a window of acceptance for stressors to be appropriate, so that the triggering of mechanisms/effects that are not the objective of the test (which is likely to be unavoidable) is not too influential. Within this window of acceptance, the effects of the accelerating stress factor can be called representative of a single degradation mechanism, even if other intrinsic mechanisms are (marginally) affected.

Finally, the consistent methodology is expected to provide the means to correlate the long-term degradation effects from a given real-world ageing case with the required test conditions for a meaningful accelerated test. This could be through a combination of post-test measurements and numerical or analytical correlations between the real-world operating conditions and ageing data or at least final performance level (that are known) and the required accelerated test conditions and ageing data to replicate the real-world end-of-life state.

Defining a generalized transfer function can be difficult. Thus, a method on how to determine a transfer function would be sufficient and a great advancement: to our knowledge, no such 'ready to use' method is available yet.

Therefore, at the current state of knowledge, more development work is needed to identifying the ranges within which the aggravation of testing conditions (leading to accelerated degradation):

– is representative of the targeted application and product duty cycle in real-world operation,
– does not alter significantly the interdependency or eminence of intrinsic degradation mechanisms,
– leads to an increase of degradation that can be correlated in a quantifiable way with the degradation in real-world operating conditions,
– leads to a profile of degradation that allows to predict durability in real-world conditions,
– can be applied to cells/stacks of different sizes, geometries, assembly structures, materials,
– can be combined (contemporaneously or in sequence) to evaluate different characteristics or operation modes.

Important questions that remain outstanding during the investigation are:

–   How can we distinguish and clarify the concentration of degradation effects (in order to compact the test cycle) vs. degradation effect amplification (e.g. by increased temperature)?

–   How can we clarify/specify the method to define the transfer function from real life to accelerated test AST or ALT?

–   How can we clarify/specify the method to characterise in-situ and post-mortem criteria and especially validation criteria?

Once there is more certainty as regards the applicability of defined aggravated conditions according to the points above, there will be a better base to work on for the normalization of these test conditions in procedures fit for IEC standardization.

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