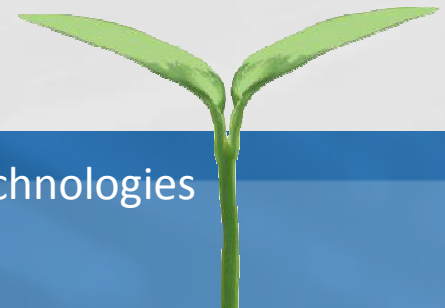


Button-cell-size test cells of SOFC and components; possibilities and limitation, with reference to the NorECs ProboStat™ and FCMix

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NorECs Norwegian Electro Ceramics AS

International Symposium on Diagnostic Tools for Fuel Cells Technologies
June 23-24, 2009, Trondheim, Norway



NorECs Norwegian Electro Ceramics AS

Who are we?

- **NorECs** is a small company that designs and manufactures equipment for high-temperature electrochemical research.
- **Our aim** is to commercialize a broader range of scientific equipment, materials, and applications derived from Norwegian university research.
- **Our focus** is electroceramics, their properties, and characterization at high temperatures and under controlled atmospheres.

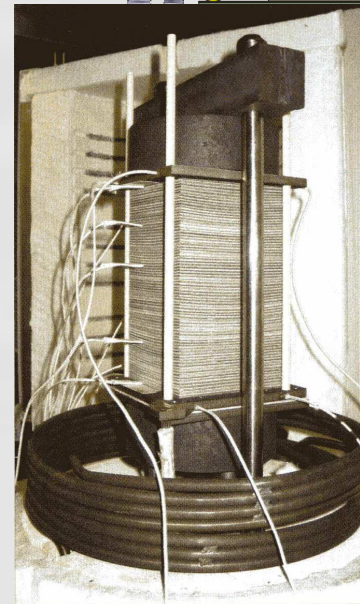
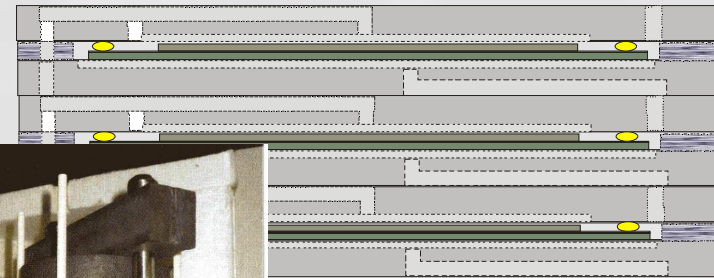
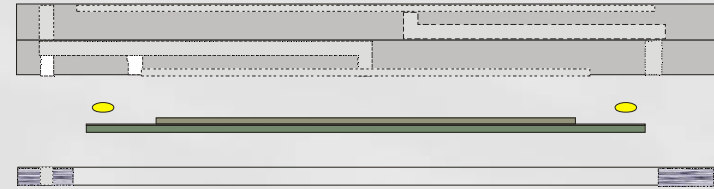
Outline

- Fuel cell testing; Stacks and button cells
- The ProboStat™ sample holder system
- Button Fuel Cell test with the ProboStat™
- Feeding Fuel and Oxidant; FCMix
- Button-cell test using the ProboStat and FCMix
- Conclusions



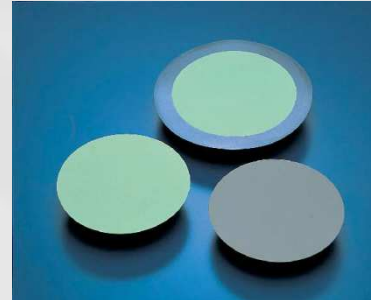
SOFC characterisation challenges

- Stacks
 - Sealing
 - Current collection
 - Thermal management
 - Water handling
 - Fuel utilisation
 - Cycling – thermal, red-ox
 - Interconnect behaviours
 - Contamination; Cr, Si...



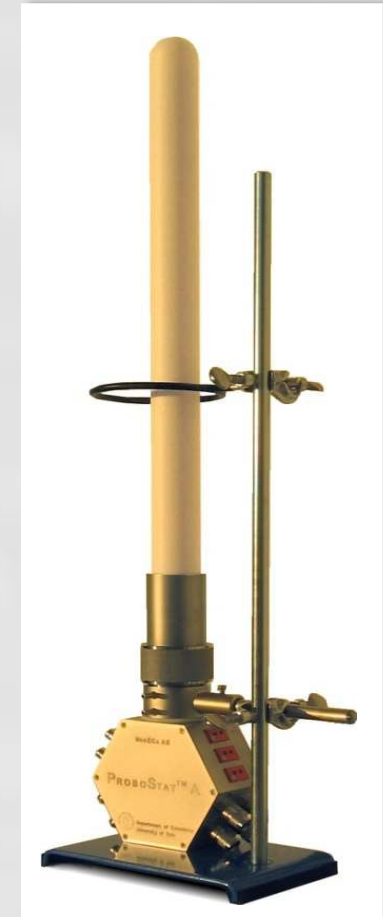
SOFC characterisation challenges

- Button cells
 - Sealing
 - Current collection
 - Fuel and air supply
 - Cycling; thermal, red-ox
 - Contamination (Si...)
 - Water handling (?)
 - Safety (?)
 - Relevance (!)



SOFC based on LaGaO_3
(Mitsubishi Materials Corporation)

ProboStat™ 



ProboStat™

ProboStat™ is a versatile sample holder for measurements of electrical properties, transport parameters, and kinetics of materials, solid/gas interfaces and electrodes at high temperature under controlled atmospheres.



Running experiment with the ProboStat™. (UiO)

Brief specification

- **Samples:**
 - up to 24 mm disk
 - up to 50 mm long bar
- **Temperature:**
 - long term < 1400 °C,
 - short term < 1600 °C.
- **Atmospheres:**
 - Oxidizing, inert, reducing
 - Wet or dry
 - Single or dual
- **Overpressure:** ca. 20 atm (with MA253 SS outer tube) at 900 °C
- **Vacuum:** ca. 1×10^{-2} mbar
- **Maximum current:** 5 A
- **Maximum voltage:**
 - 42 V
 - 10 kV (upon request)
- **Overall length:** 75 cm
- **Outer tube diameter:** 40 mm

ProboStat™

One cell; many accessories, methods, properties

Material properties and applicable methods:

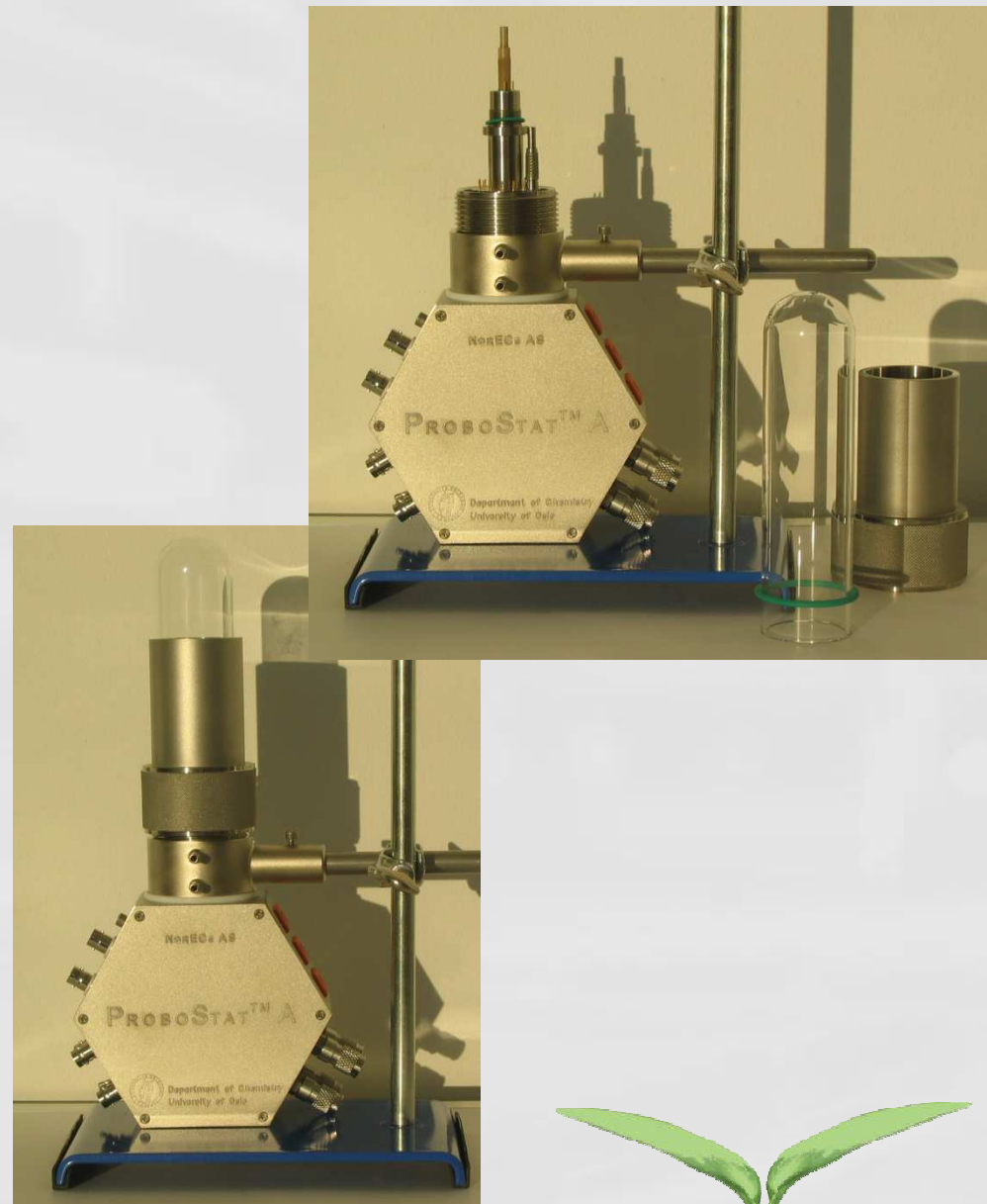
- Conductivity vs. T , pO_2 , pH_2O , ect.
- DC, AC, impedance spectroscopy
- Dielectric properties, loss, ect.
- Disk, van der Pauw, and bar geometries
- 2, 3, and 4 electrodes
- Ionic transport number
- Proton transport number
- H/D isotope effect
- Seebeck coefficient
- Annealing or sintering under controlled atmospheres
- I-V characteristics
- Fuel cell component and single cell testing
- Electrode kinetics
- Electrochemical pumping, gas permeation and electrocatalysis with gas analysis (e.g. GC or MC) on outlets
- Sensor testing
- Poling of ferroelectrics possible with the high-voltage (10 kV) version



ProboStat™

Base unit configuration

- 16 electrical feedthroughs
 - 6 for electrodes,
 - 4 for shields,
 - 3×2 for thermocouples;
- 4 gas inlets/outlets (brass or steel);
- 3 thermocouple sockets;
- 6 individual BNC connectors;
- Water cooling or heating;
- Grounding and shielding switches;
- Sockets for sample support tube, enclosing tube, gas supply tubes, spring load.



ProboStat™

Base unit configuration

Electrical feedthroughs and socket for sample support tube



Thermocouple sockets

gas inlets/outlets for inner and outer chambers



BNC connectors



Gas inlet and outlet for inner chamber

Gas inlet and outlet for outer chamber

Water cooling / heating



Shields and grounding switches

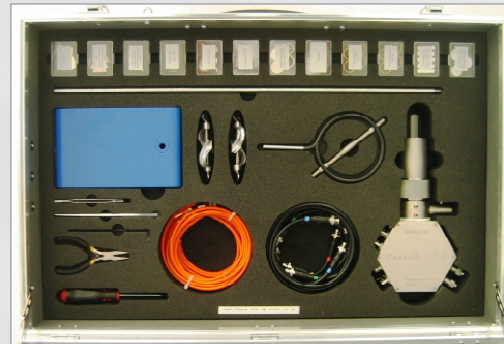


ProboStat™

Base unit accessories

- Support tube assemblies in 5 standard sizes for disk and bar samples;
- Gas supply tubes;
- Spring-load assemblies;
- Thermocouples;
- Electrode contacts;
- Grounding;
- Internal shielding;
- Sockets for HT tube for sample support, enclosures and gases.

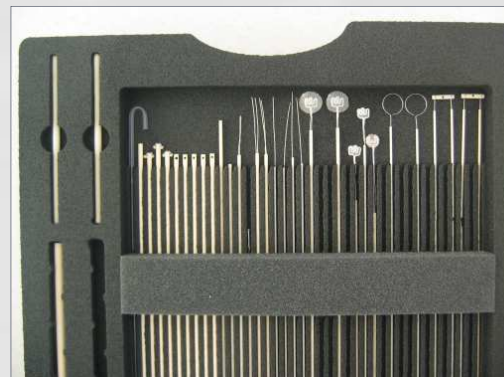
Extensively furnished system



Base unit, BNC and thermocouple compensation cables, small parts.



Outer tubes and support tube assemblies.

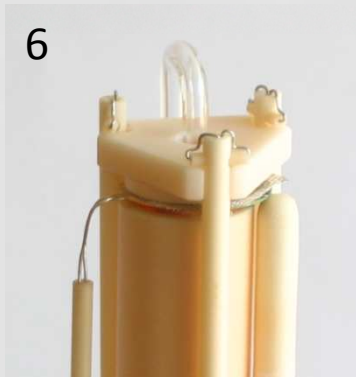
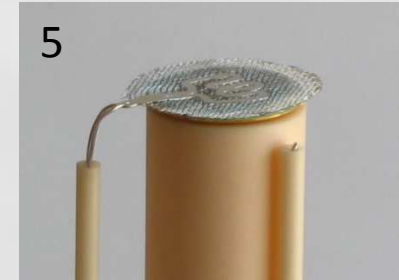
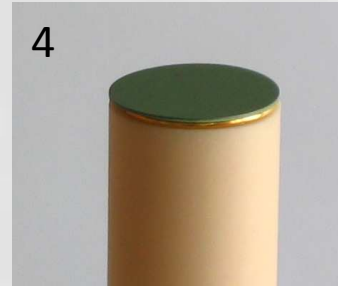
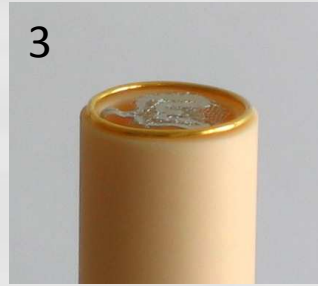
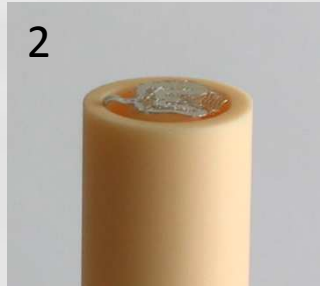
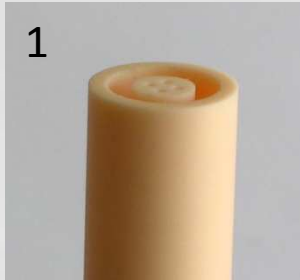


Gas supply tubes, electrodes, thermocouples, spring-load system parts.

ProboStat™

SOFC test setup

“Fortunately there are now a number of commercially available test fixtures, such as the ProboStat, that conform to the basic design parameters of the apparatus described above...” www.fuelcellmaterials.com



Assembling for SOFC test

1. Sample support tube and multi-bore inner gas tube.
2. Inner Pt two wire “hand” electrode contact with Pt net.
3. Gold sealing ring.
4. ASC2 button fuel cell from InDEC B.V.
5. Outer Pt two wire “hand” electrode contact with Pt net, thermocouple (S-type).
6. Spring-load system, outer quartz gas tube, and thermocouple protection cap.

Feeding Fuel and Oxidant

FCMix

- FCMix: simple & safe gas mixer for tests of small fuel cells
- Supply and wetting of fuel and oxidant with fine control of flow and overpressure.
- Three digital flowmeters calibrated for e.g. H₂, Ar and air.
- Routable inert gas allows soft and safe start.
- Two simple gas humidifiers with overpressure safeguard and regulation.
- Flow rate, e.g. 50 ml/min.
- 19" rack mount or benchtop
- Alternative to expensive and sometimes overdimensioned FC test stations that work with too rough overpressure controls.



FCMix



ProGasMix is a versatile gas mixer for simultaneous control of (pO_2+pH_2O) over entire pO_2 range from oxidizing to reducing conditions. ProGasMix can blend up to 3 gases in 2 different mixtures + control humidity.

SOFC test

with ProboStat™ and FCMix

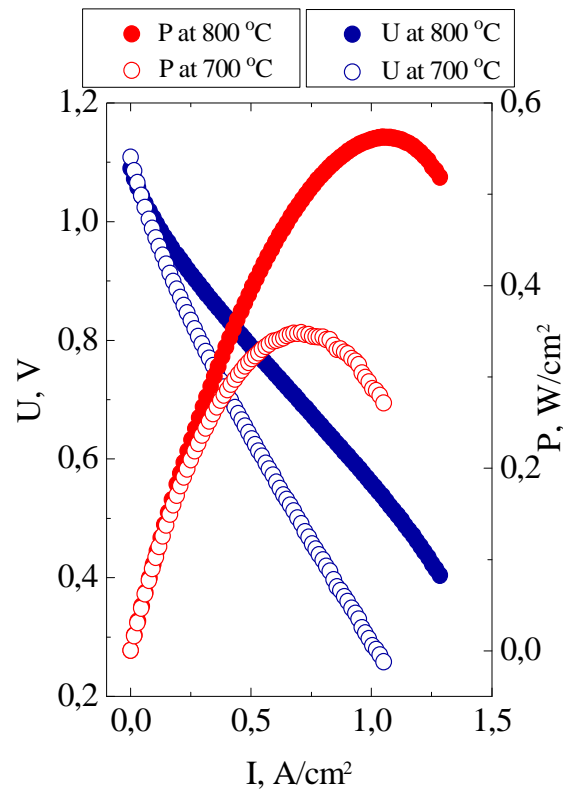


Fig.1. Fuel cell performance for:
wet H₂, Pt | ASC2 | Pt, dry air

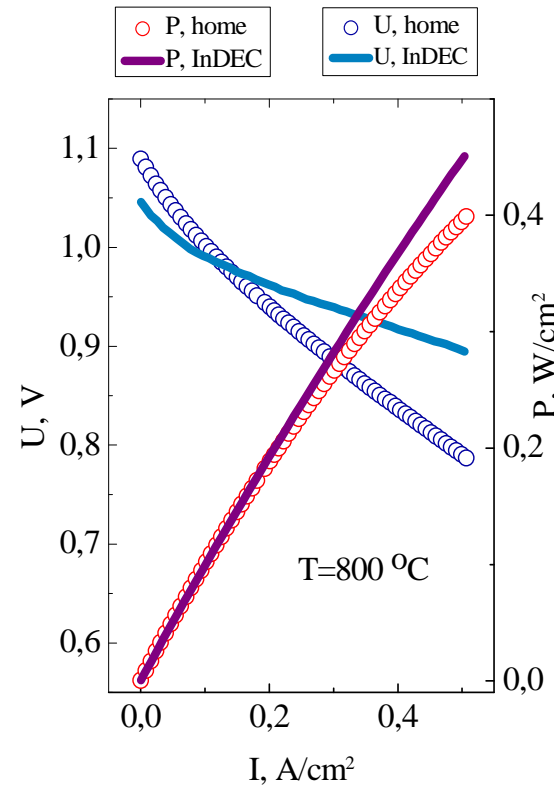


Fig.2. Fuel cell performances obtained with
the ProboStat™ and from InDEC.

ASC2 (from InDEC):
anode: porous NiO/8YSZ
electrolyte: dense 8YSZ
cathode: porous LSCF

Fuel: wet H₂;
Oxidant: dry air;
Gas flow rate 30 ml/min.

Solartron 1287
CorrWare + CorrView

Sealing:
Gold gasket
OCV_{theor} = 1.102 V
OCV_{exp} = 1.091 V

Conclusions

- The ProboStat™ is a commercially available sample holder system for the electrochemical characterizations at high-temperatures and under controlled atmospheres.
- The ProboStat™ excels in easy exchange of samples and electrodes, and versatility for many tests of materials and electrodes including SOFC research.
- Fuel and oxidant may be fed with the FCMix – a simple gas mixer for the SOFC research.
- Some elements of future research for diagnostics
 - In-situ spectroscopy & diffraction
 - FCs in the HT ESEM or ETEM

