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Advanced Diagnostics Tools and Analysis Methodologies in Solid Oxide Fuel Cells

24. June 2009 | J. Malzbender, P. Batfalsky, L. Blum, S. M. Groß, V.A.C. Haanappel, N. H. Menzler, A. Neumann, V. Shemet, R.W. Steinbrech, I.C. Vinke

International Symposium on Diagnostics Tools for Fuel Cell Technologies, Trondheim 2009

- Role of “Advanced Diagnostics Tools and Methodologies”
- Post operation methodology
- Post operational analysis methods
- Post operation analysis procedure
- Example G-Design stack
- Improved design

Role of “Advanced Diagnostics Tools and Methodologies”

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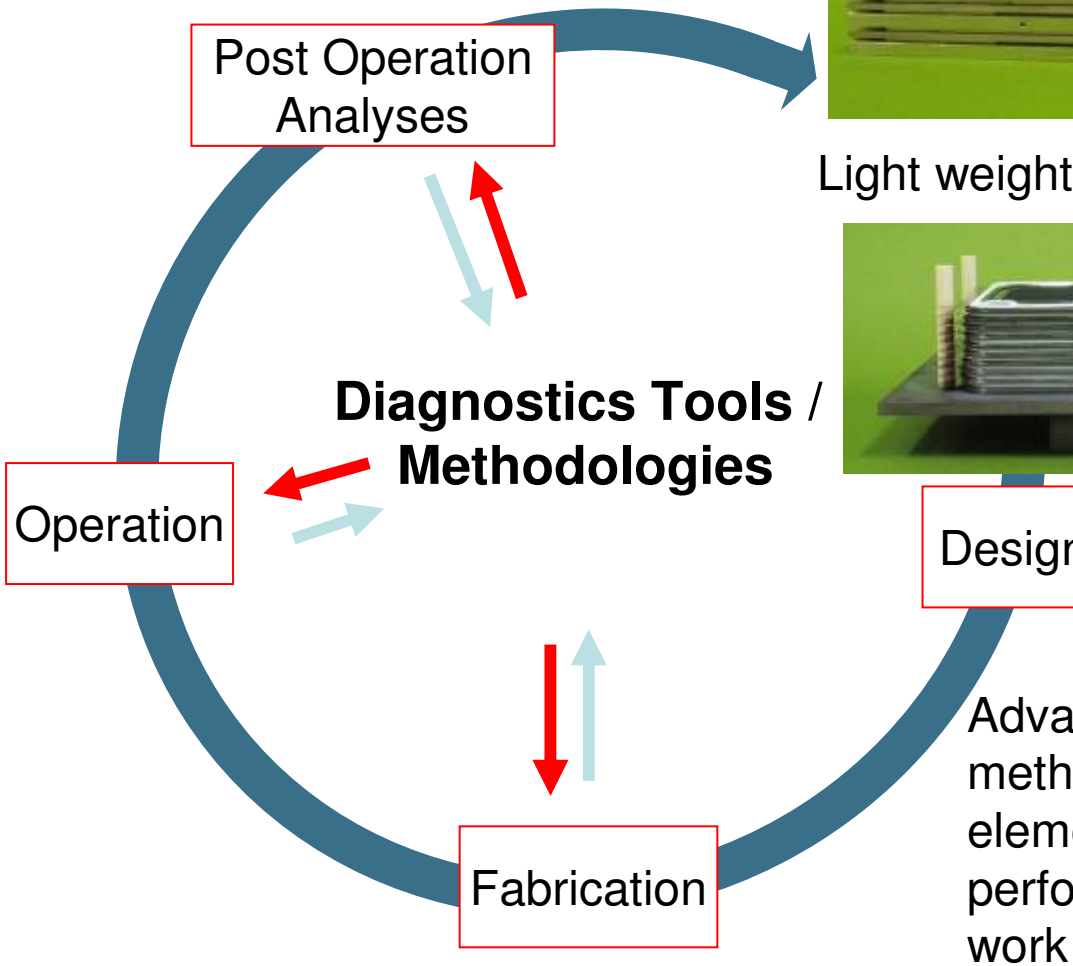
Short stack for SOFC development

Light weight stack (APU application)



Advanced characterization methods are an essential element to understand the stack performance within the frame-work of a systematic testing

Post Operation Methodologie

- Disassembling with selected experts depending on expected degradation mechanism. Experience in synthesis, production, interaction, thermo-mechanics, corrosion, thermo-chemistry, sealants, stack / system operation, single cell testing, microscopy, SEM
- 108 dissections from 8.2002 to 9.2008.
- Electro-chemical results and irregular events are considered.
- A digital photographic image is taken of every stack plane.
- Unusual observations are investigated microscopically during disassembling.
- After dismantling more detailed SEM (TEM) investigation are carried out.
- Every stack opening is discussed in a subsequent meeting, suggesting further detailed follow up work.
- Reports are passed on to selected members of the SOFC development team.
- Selected results are presented to the entire SOFC team in semi-annual meetings.

Post Operational Analysis Methods

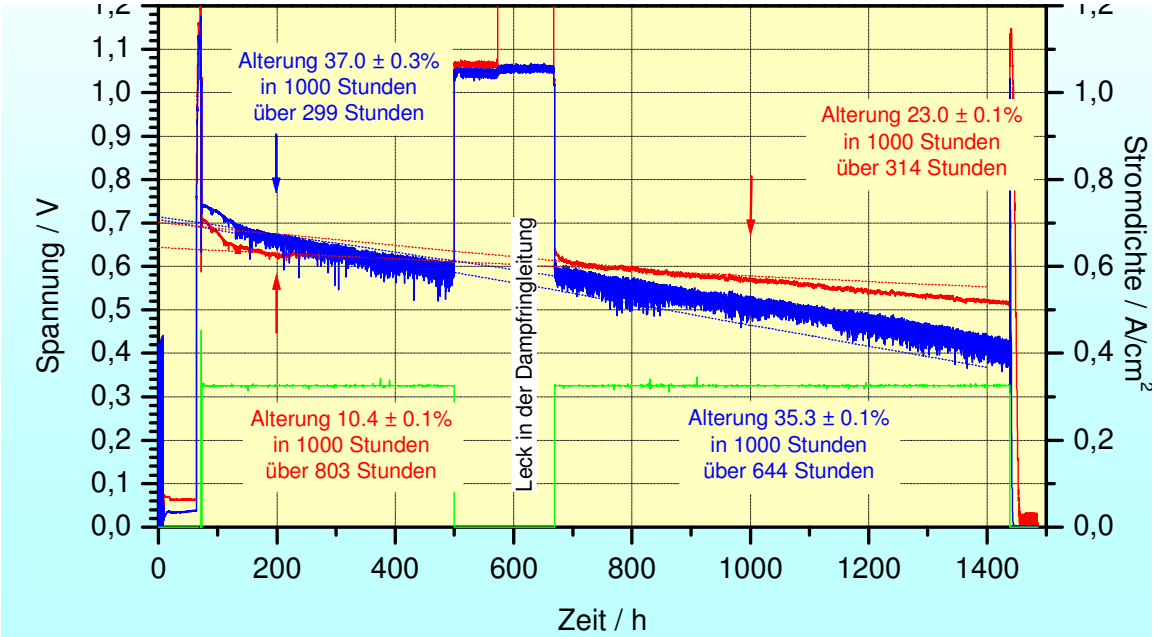
Materialography & Image analysis	Macroscopic changes, color
SEM and EDX	Microscopic / structural changes, qualitative chemical analysis,
Wet chemical analysis	quantitative, coarse localized
SIMS	quantitative, localized
Thermography, computer tomography	short circuit localization, porosity
XRD	structural changes
TEM	local changes, interfaces, reactions
Leakage, liquid dye inspection	localization of leakages

Post operation analysis procedure
Example G-Design stack (G1002-04)

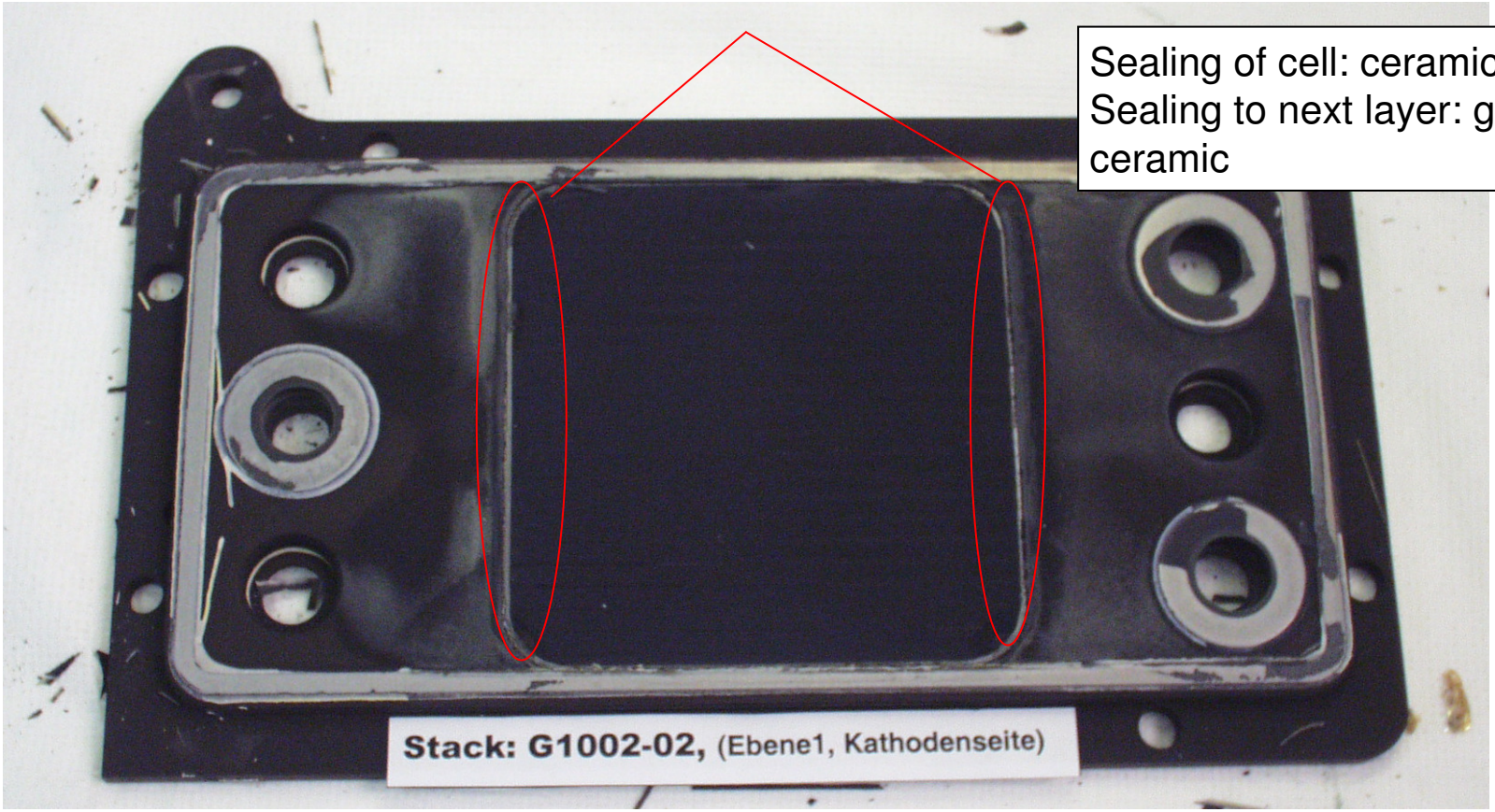
Investigation of degradation and failure by comparison of electrochemical results with stack dismantling results

G-Design problems :

- **low power output**
- **high degradation rate**

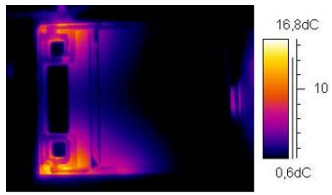


Ceramic glue shows partly red coloring and bubbles.
In addition a short circuiting to the next cassette was detected.

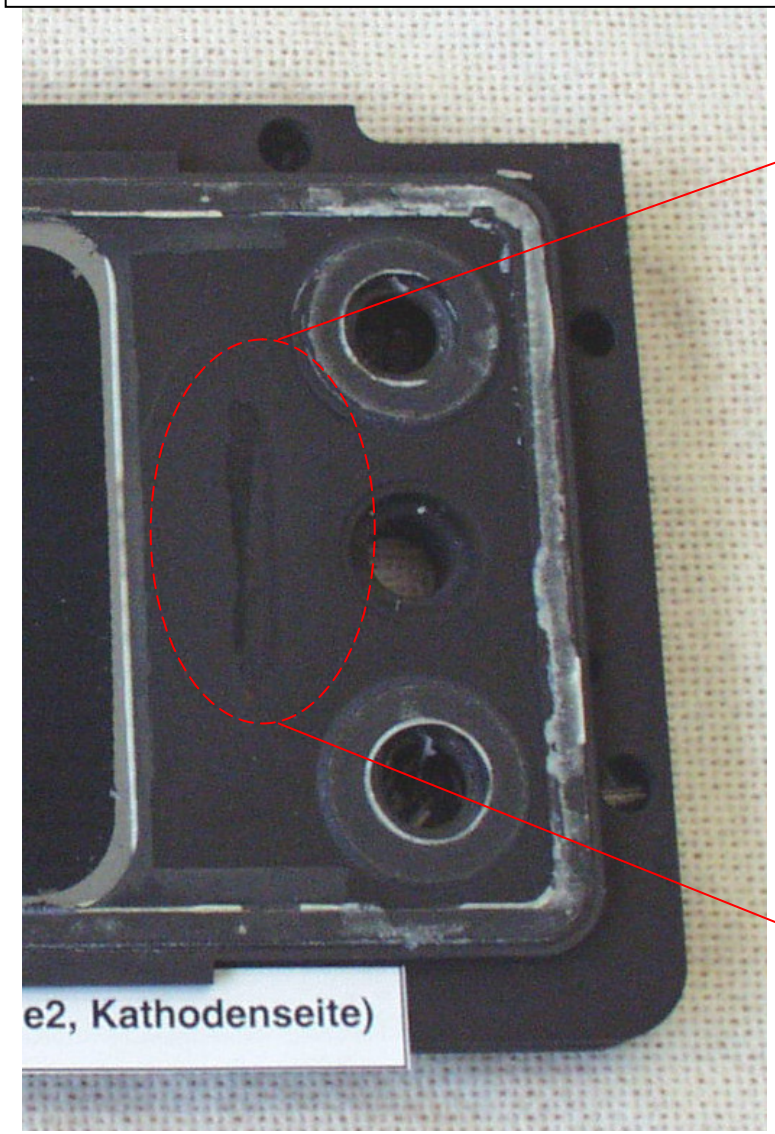


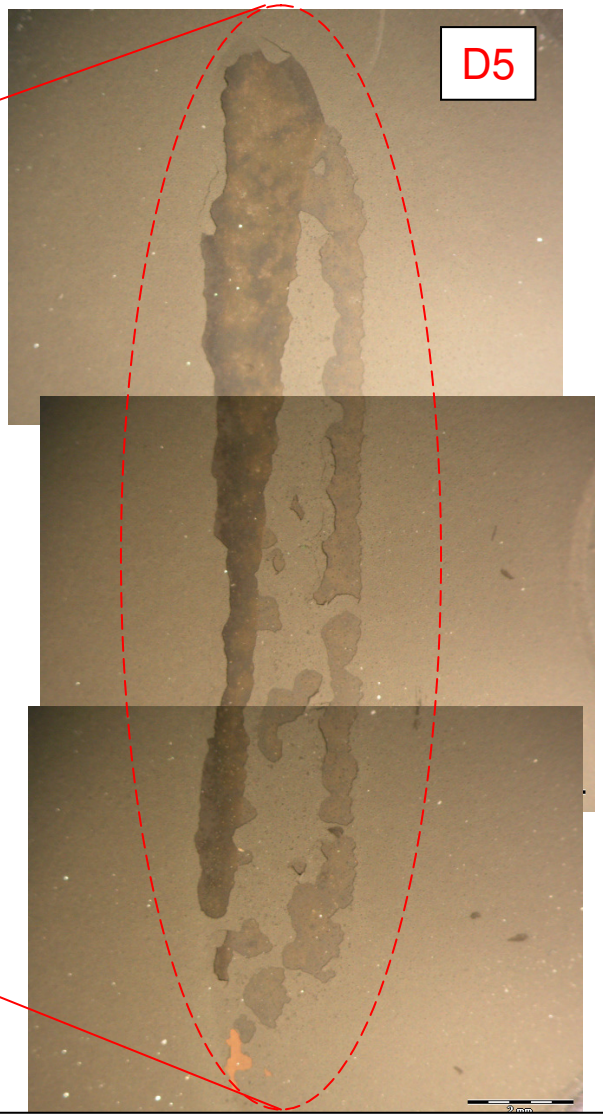
Sealing of cell: ceramic glue
Sealing to next layer: glass-ceramic

Before further dismantling took place the origin of the short circuiting was investigated using infrared camera imaging (thermography).



Origin of the short circuiting was a deformation of the manifold.

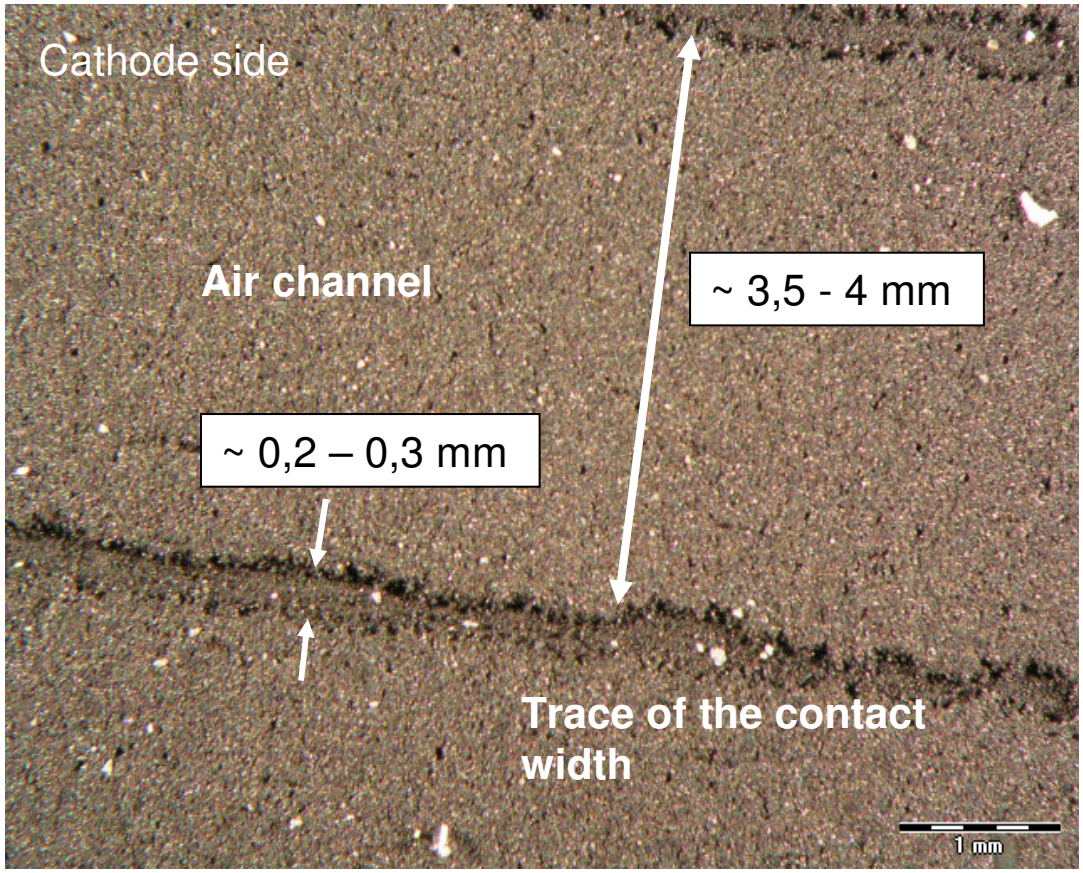




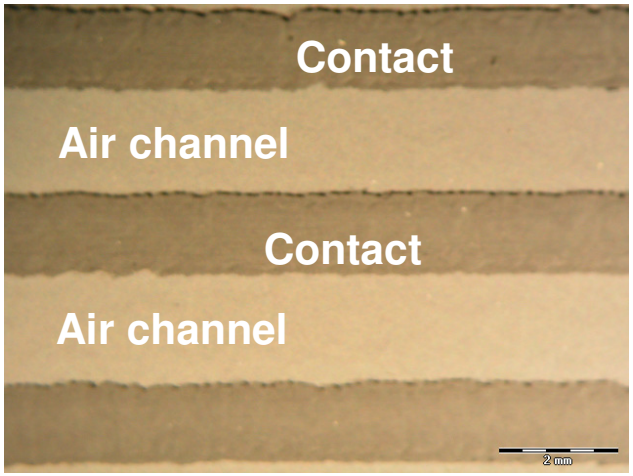
Mechanically damaged contact layer

Origin of low power output

Stack G1002-3



Standard F2060-1

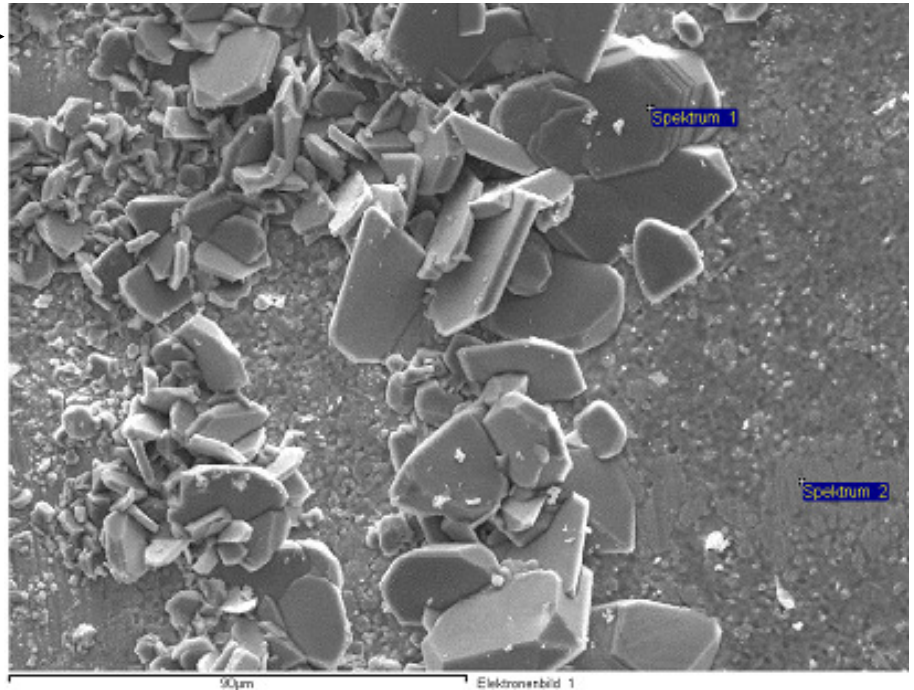


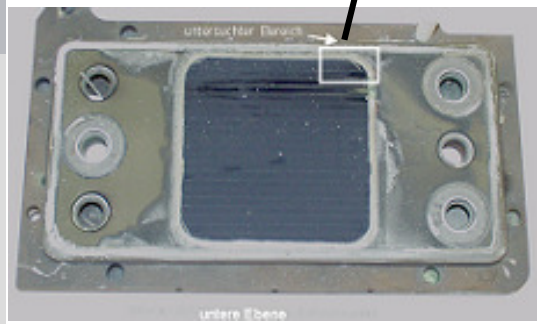
Small contact width compared to standard design

High degradation rate

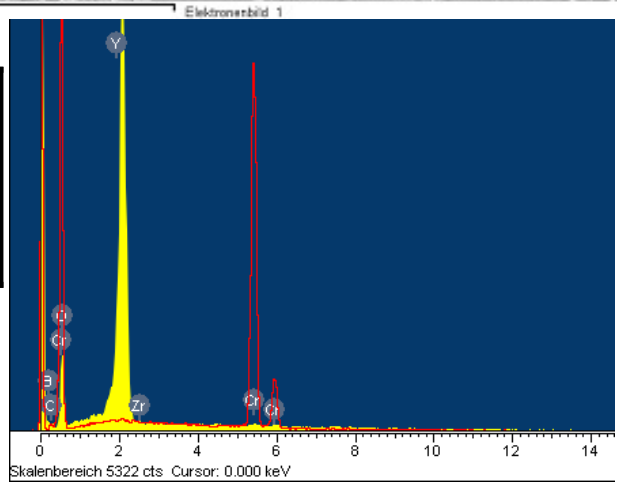
Formation of Cr_2O_3 on the electrolyte →

Edge of the cell near the sealant



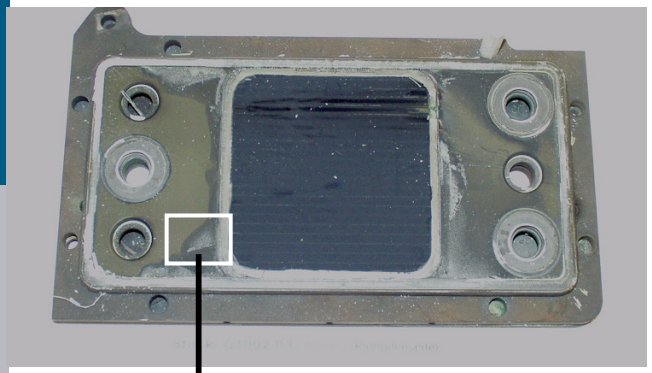


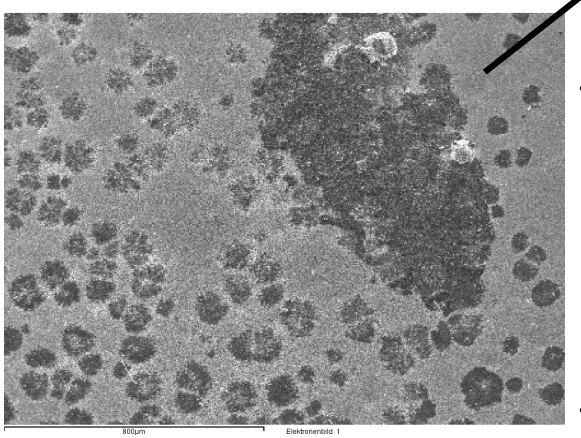
Element	Mass %	Atom %	Comp. %	Form
Cr K	68.4	40	100	Cr_2O_3
O	31.6	60		

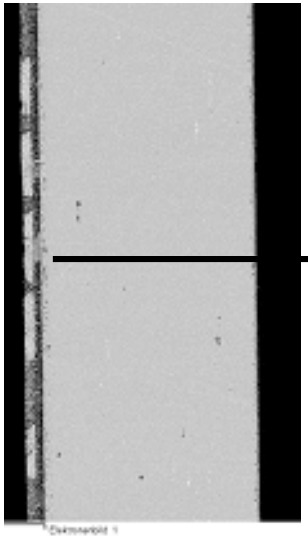


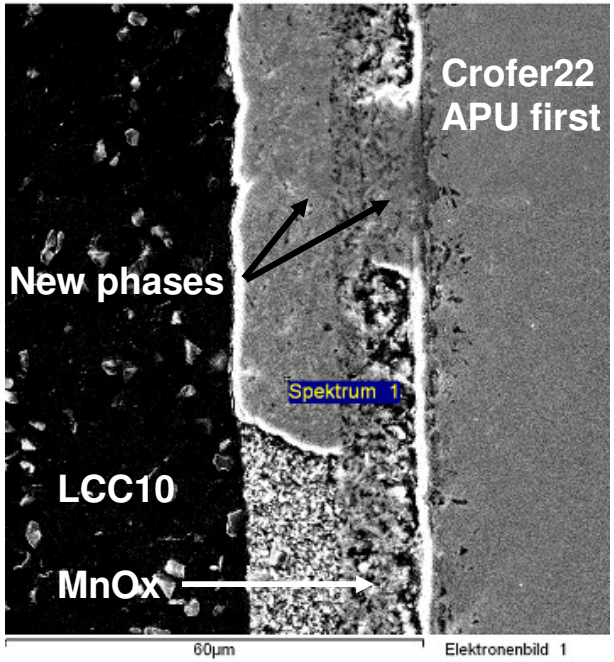
High degradation rate

Modified contact layer

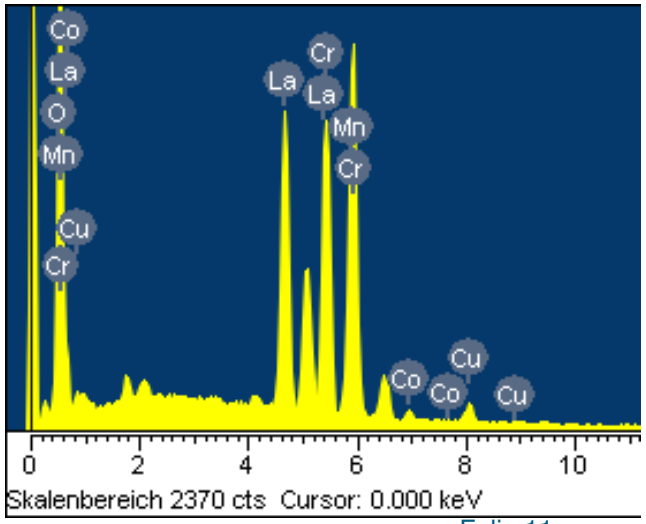




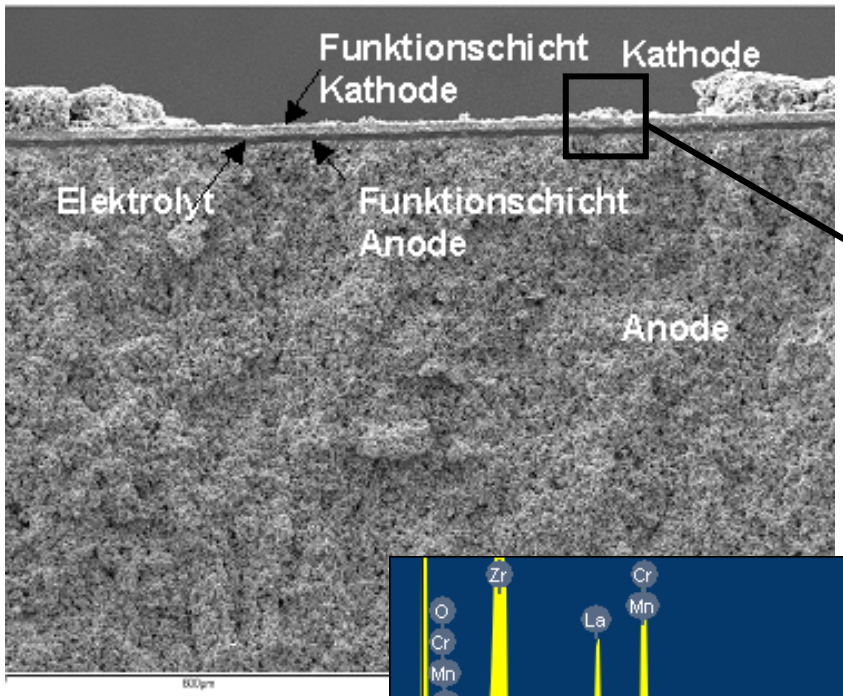


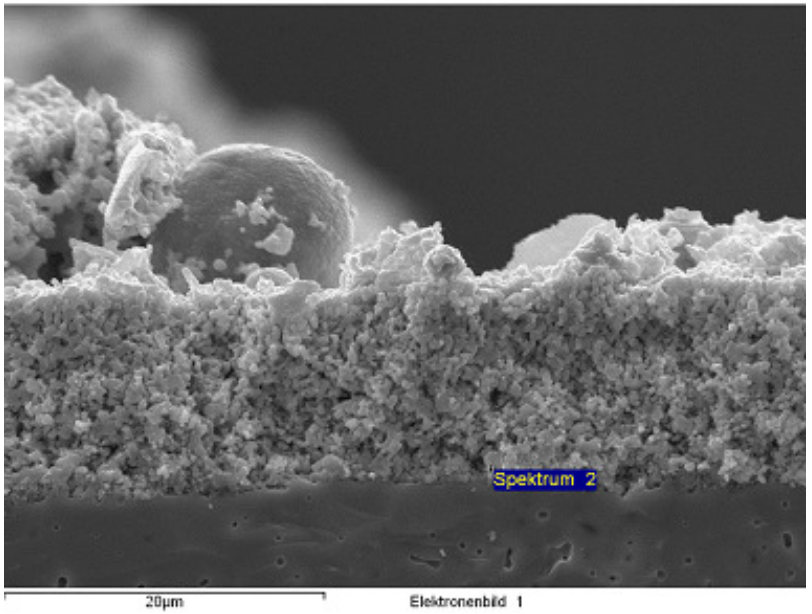


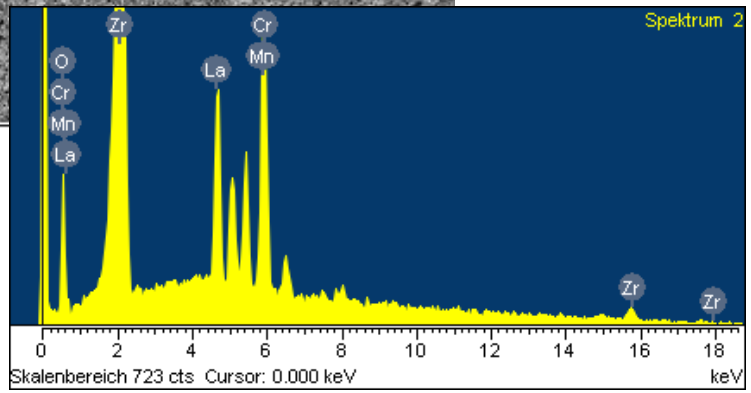
Element	Mass %	Atom %	Comp. %	Form
Cr K	14.5	11.8	21.3	Cr_2O_3
Mn K	25.4	19.5	32.8	MnO
Co K	0.97	0.69	1.2	CoO
Cu K	2.8	1.8	3.5	CuO
La L	35.2	10.7	41.3	La_2O_3
O	21.2	55.6		



Chromia composites near the three phase boundary







Element	Mass %	Atom %	Comp. %	Form
Cr K	4.5	3.7	6.5	Cr_2O_3
Mn K	14.6	11.3	18.9	MnO
Zr L	37.7	17.6	50.9	ZrO_2
La L	20.2	6.2	23.7	La_2O_3
O	23.0	61.3		

Problem:

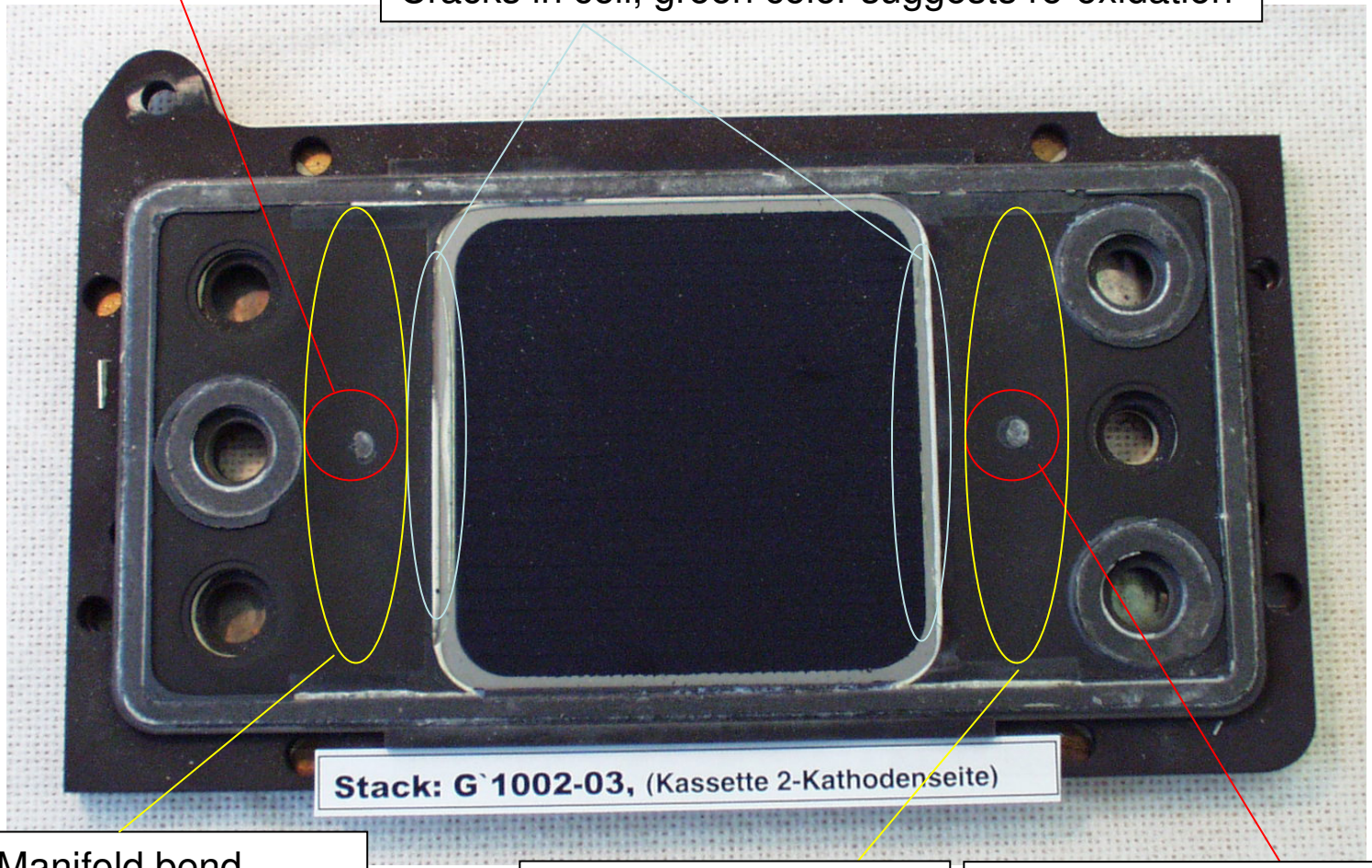
- Short circuit due to local upwards bending of the manifold (creep effect)
- Cr_2O_3 reaction products (from gas phase) were detected on the surface of the electrolyte
- New reaction products were detected in the contact layer (influence of the ceramic glue)
- Chromia composites were found near the three-phase boundary which might be associated with the high degradation

Solution:

- Application glass-ceramic support point
- Substitution of ceramic glue by glass – ceramic sealant
- Components of the ceramic glue could be confirmed, a follow up stack in the same design with glass – ceramic sealant had a degradation of (2,6-2,8)% / 1000h compared to (23-35%).

Application point of glass-ceramic support point

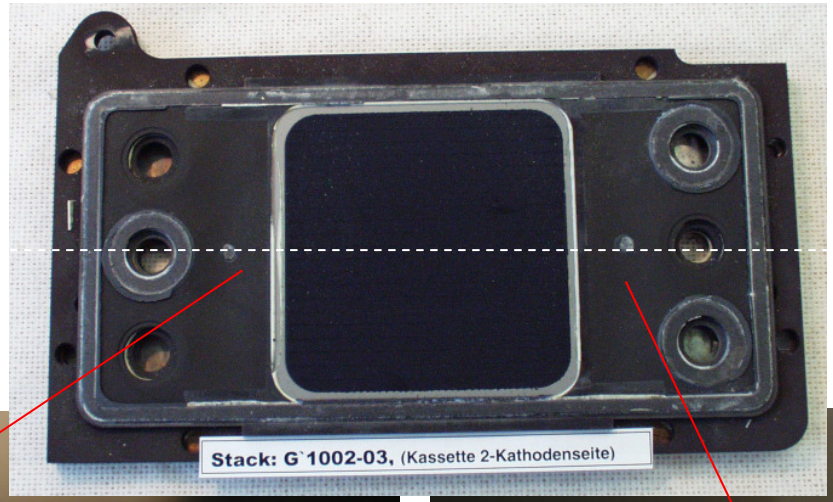
Cracks in cell, green color suggests re-oxidation



Manifold bend towards anode side

Manifold bend towards anode side

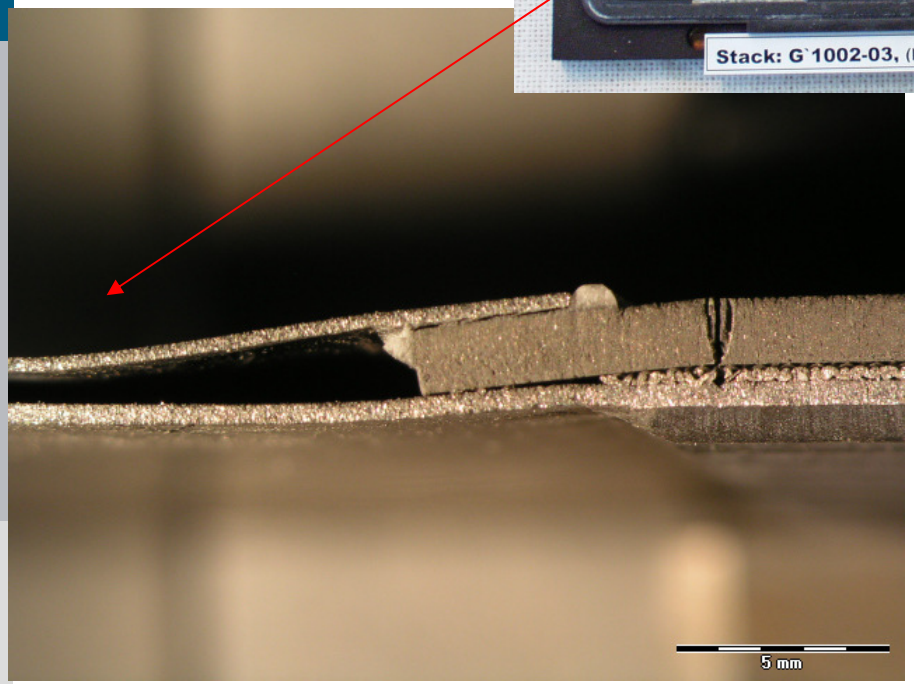
Application point of glass-ceramic support point

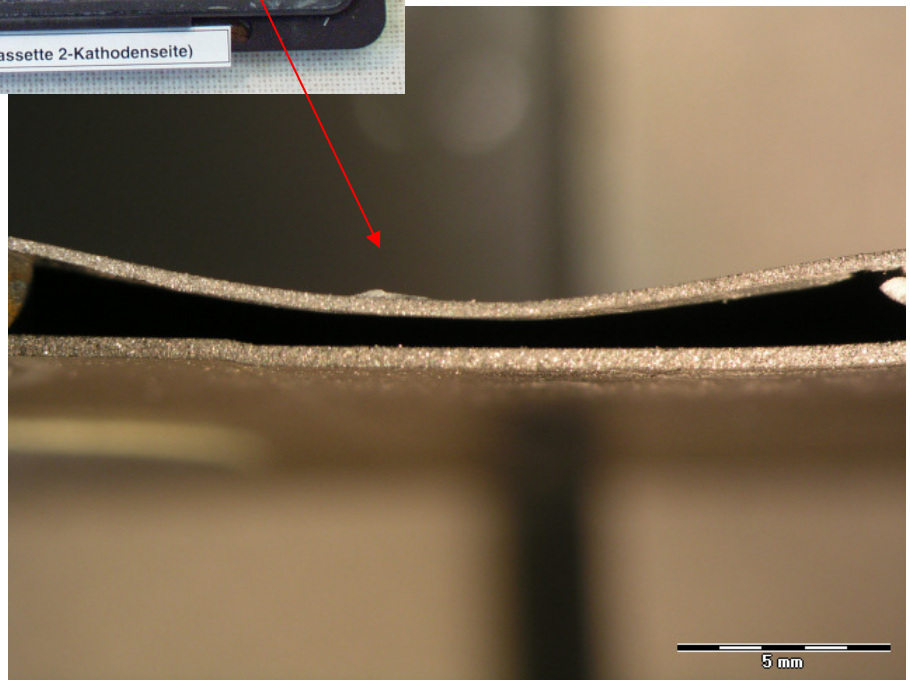


Cut

Air inlet

Air outlet

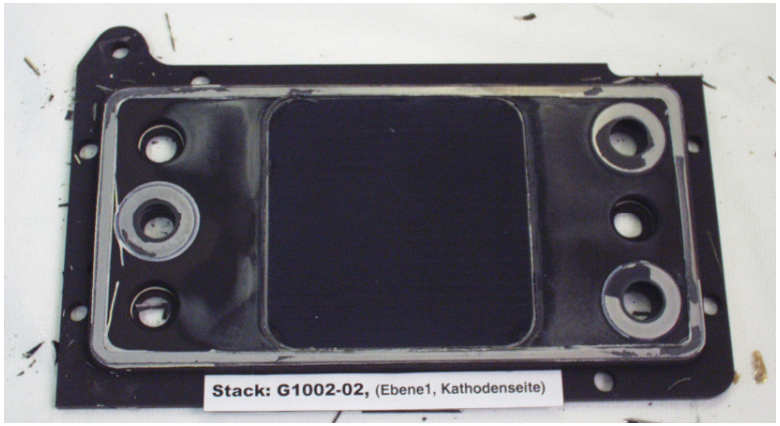


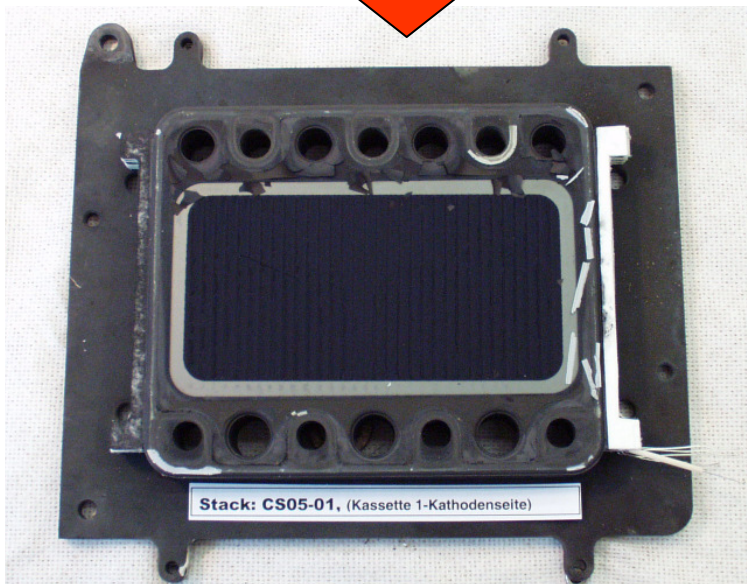


Large manifold made from thin metal sheets is not geometrically stable at high temperatures

Results are short circuit or cell fracture

Improved Design



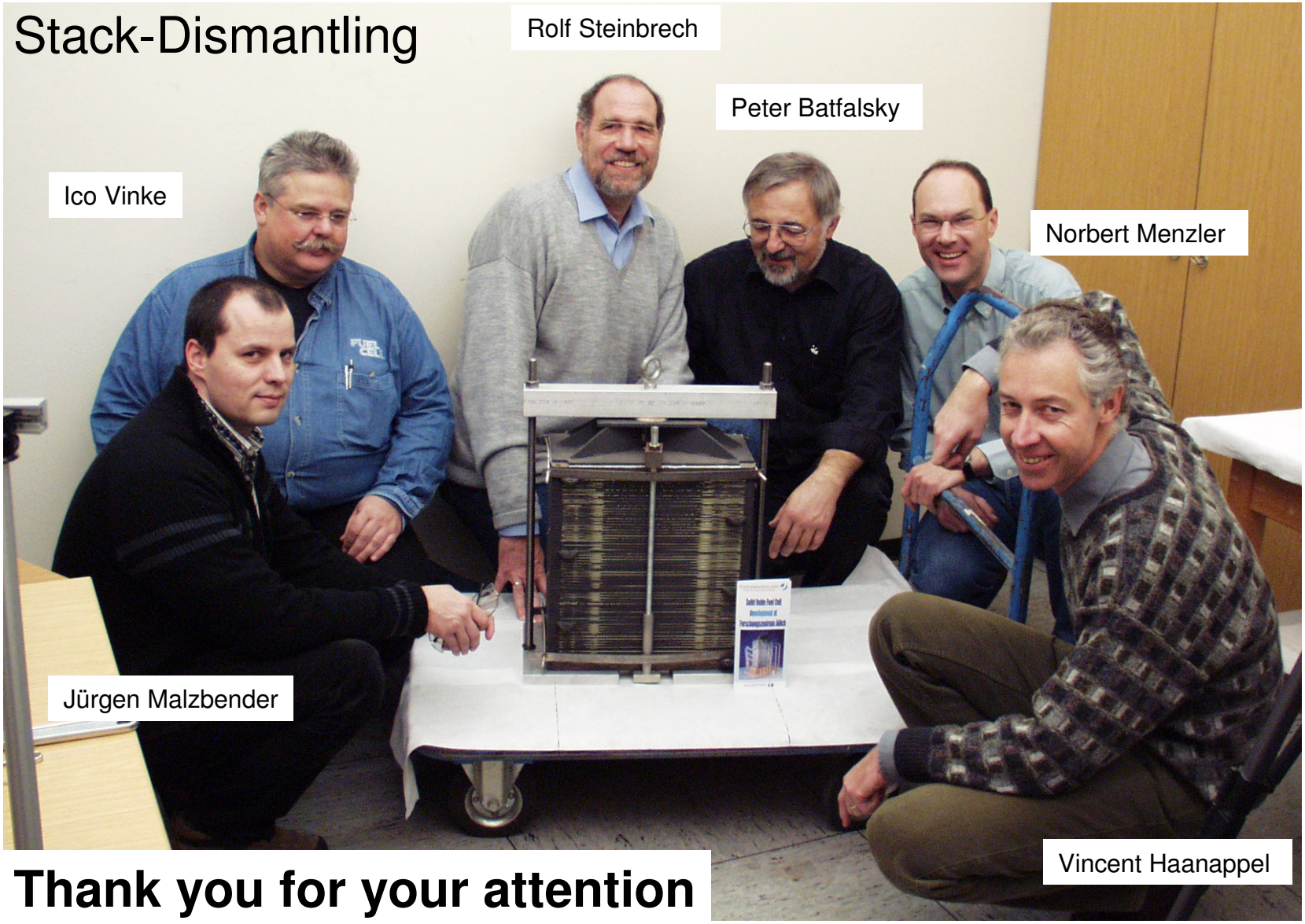


Using a combination of metallic and glass-ceramic sealants.

Significantly reduced size of unsupported manifold.

In addition asymmetric cell to permit smaller in-plane gradient on thermal cycling.

Stack-Dismantling



Thank you for your attention