

# Elektrochemische Oberflächentechnologie – vom modernen Lötverfahren bis zum Chrom(VI)-freien Korrosionsschutz.

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## Overview

- 1 Introduction to CEST
- 2 Soldering technology for microstructured components
- 3 Structured chromium
- 4 Chromium-free passivations on steel strip

*... linking academia to industry*

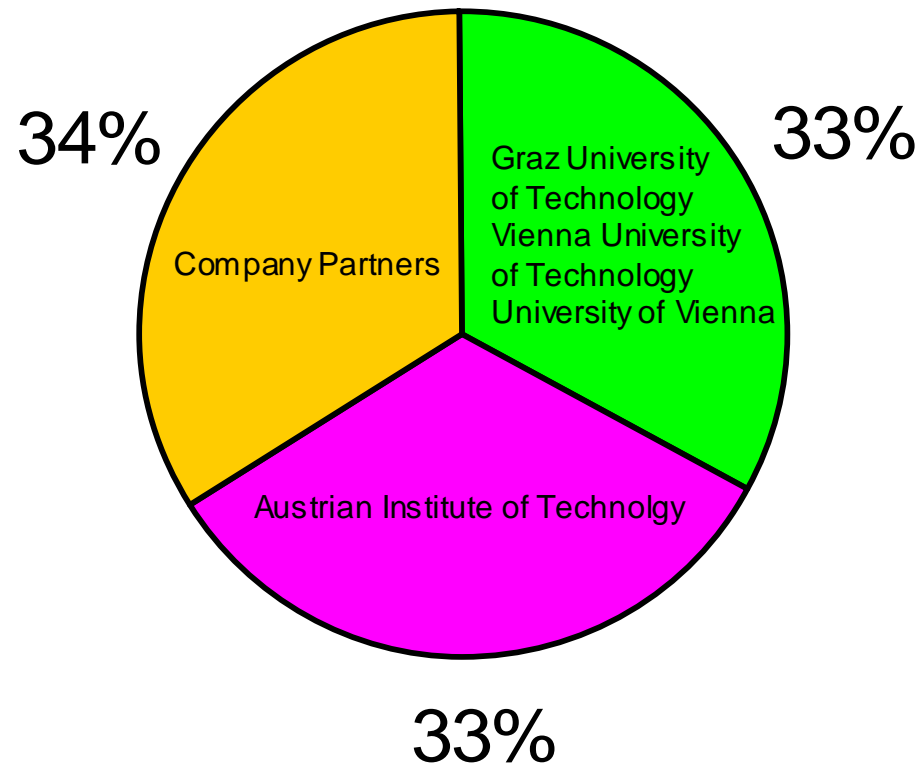


CEST Kompetenzzentrum für elektrochemische Oberflächentechnologie GmbH

# CEST Facts and Figures

Founded 2008 as K1 Centre within Austria's COMET Programme  
(successor of *Kplus* ECHEM 2000-2007)

CEST Ownership:



## COMET K1 Centres

### **K1-Centres**

The objective of K1 centres is the initiation of high-quality research defined jointly by science and industry with a medium-term to long-term perspective. K1 centres implement top-level research with a focus on scientific and technological developments to qualify for the markets of the future.

*Number: approximately 15, public funding: 40 – 55%, project duration: 7 years*

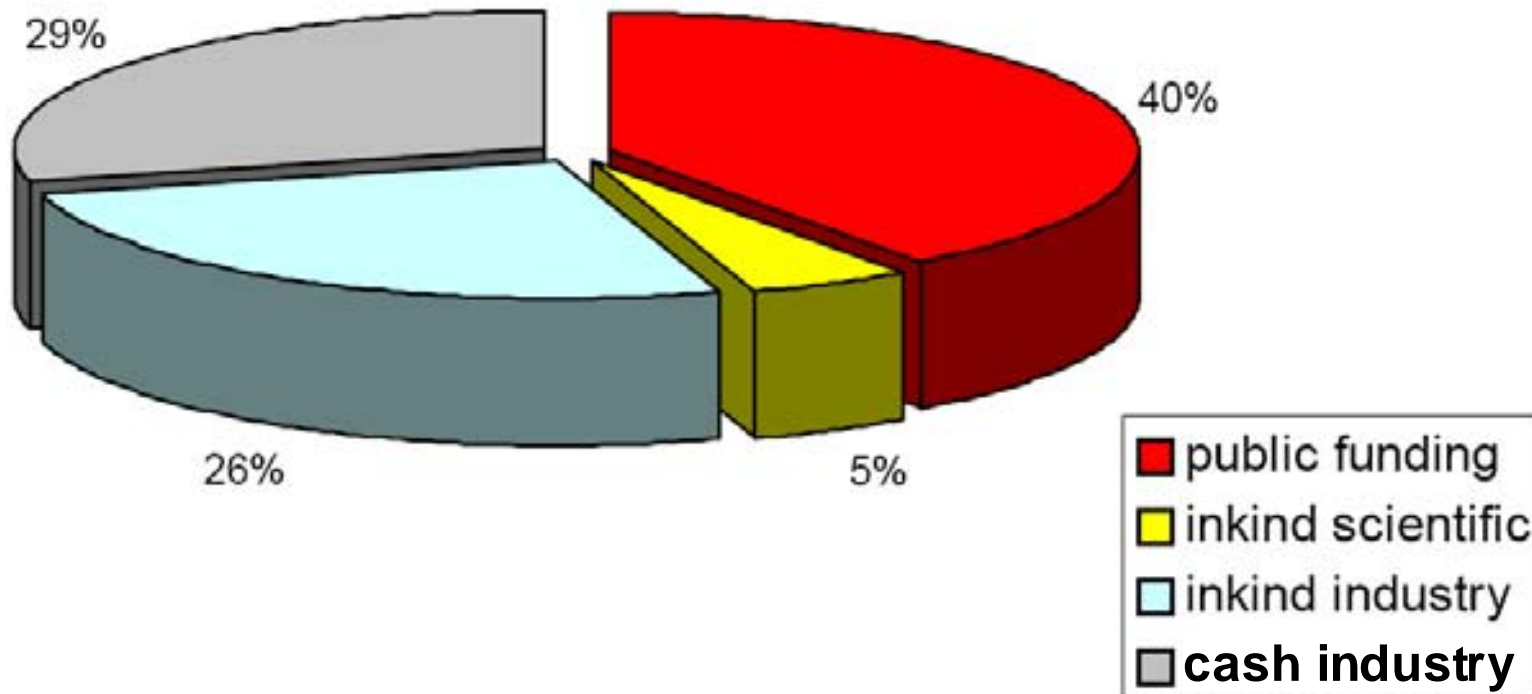
### **General criteria (apply to all COMET projects)**

- Research programme defined jointly by science and industry
- Research competence and connection to science
- Relevance of implementation in industry
- Quality of collaboration between science and industry
- Management and implementation

### **Criteria for centres (additional for K1 and K2-Centres)**

- Bundling of players to strengthen synergies
- Human resources development
- International integration

## K1 CEST Funding



K1 CEST COMET Budget: € 22.5 Mio. for 2008-2011

Public funding: 26,5 % FFG (BMVIT & BMWFJ)  
13,5 % State of Lower Austria

# CEST Profile

## Service (COMET & Non-COMET):

- Research, development and analysis
- Optimisation of processes and production
- Feasibility studies
- Development of instruments
- Projects

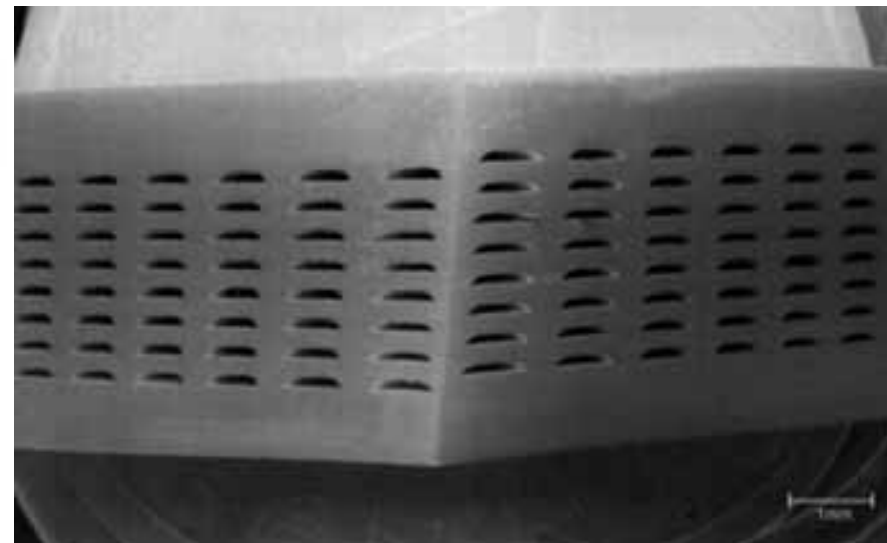
## Currently 30 COMET projects organised in the five Research Areas

1. Electrodeposition and dissolution
2. Corrosion research
3. Process control
4. Modelling and simulation
5. Surface analytics and electrochemical *in situ* techniques

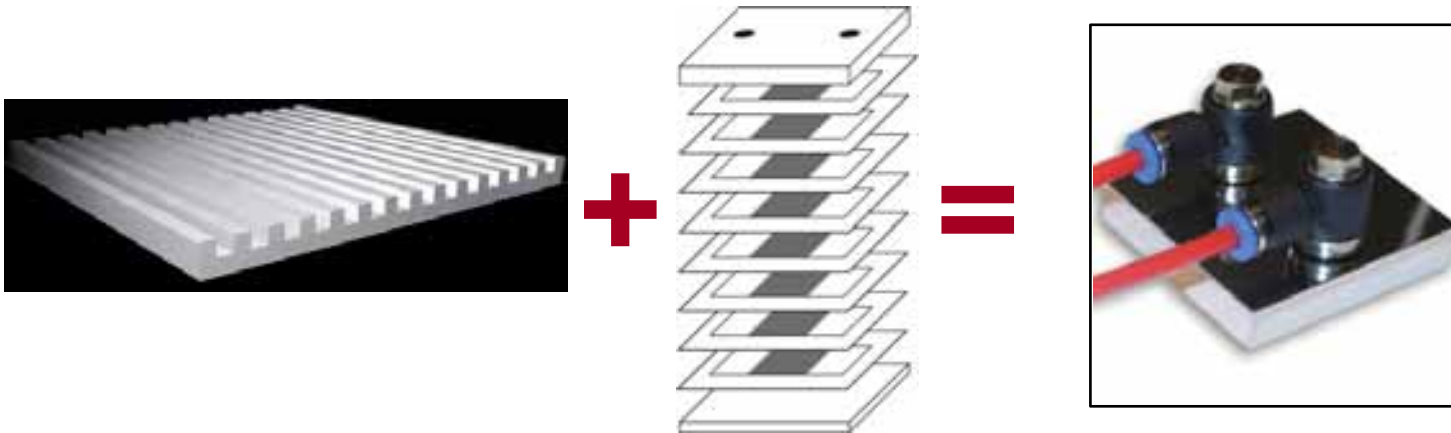
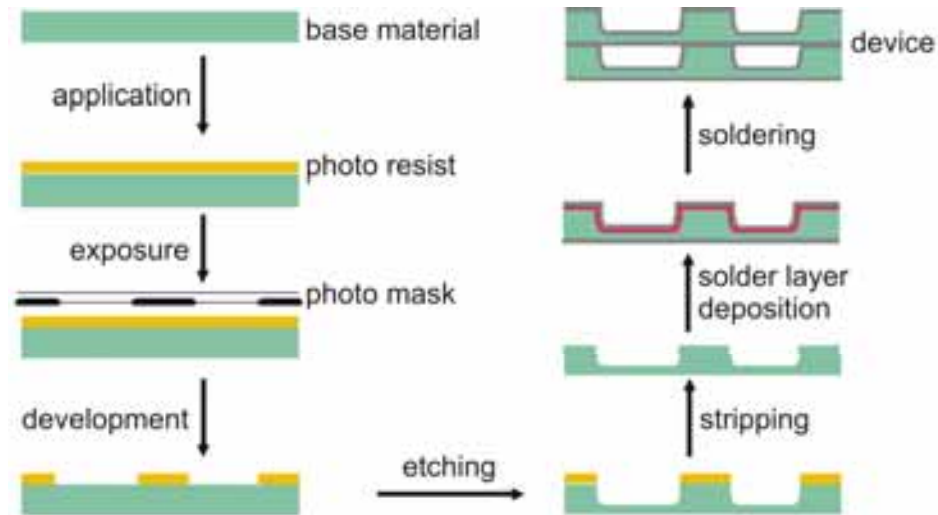
## Main topics of research:

- Advanced functional layers
- Replacement of Cr-VI in surface technology
- Electrochemical surface technology in non-aqueous electrolytes
- Local electrochemical *in situ* techniques

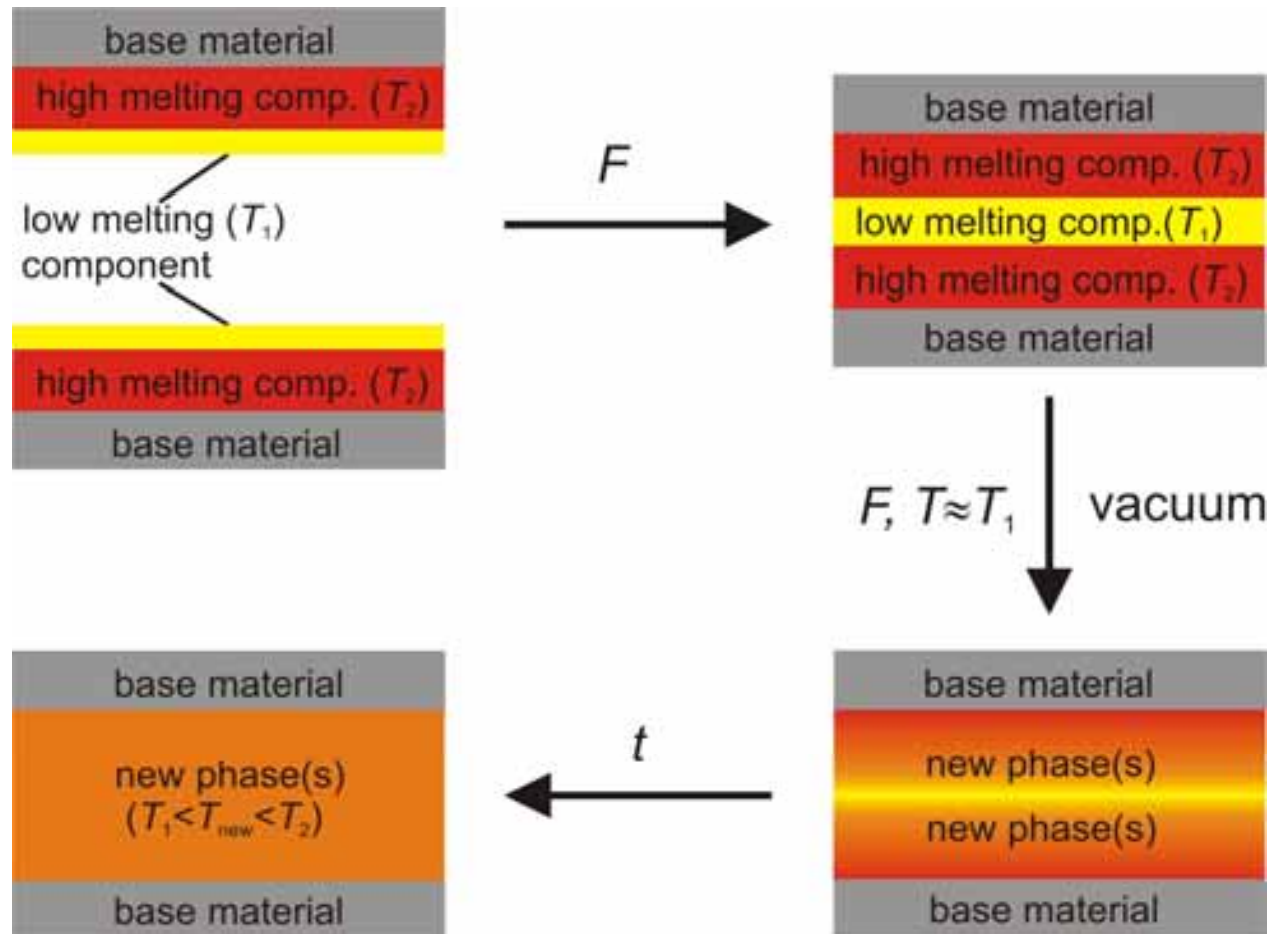
# Microstructured Components



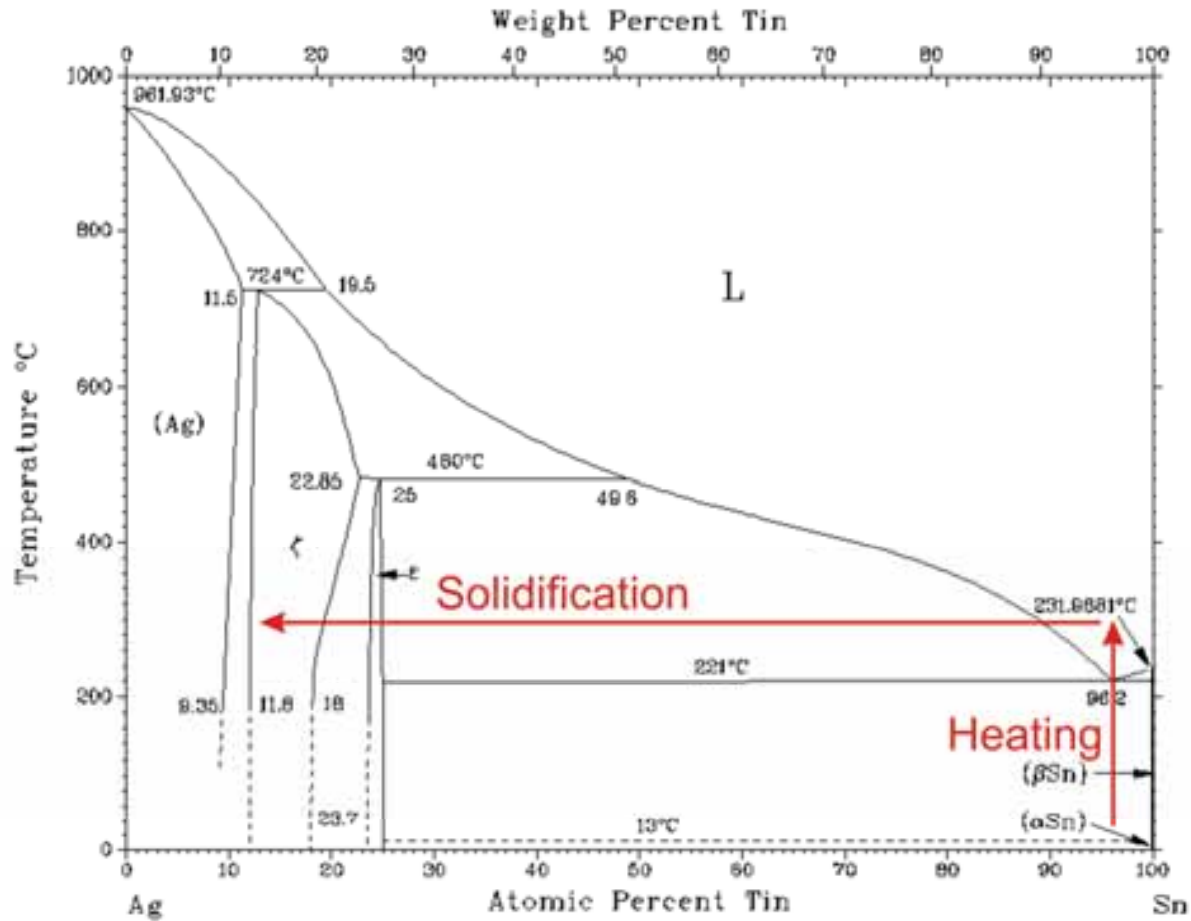
# Process suitable for mass production of $\mu$ -reactors



# Isothermal diffusion soldering (IDS)



# IDS: The Ag/Sn system



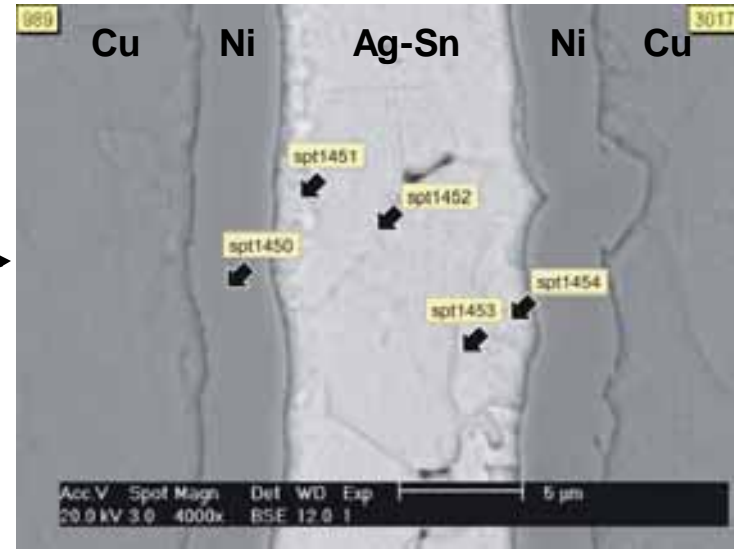
# IDS with electrodeposited layers



250 °C  
30 min

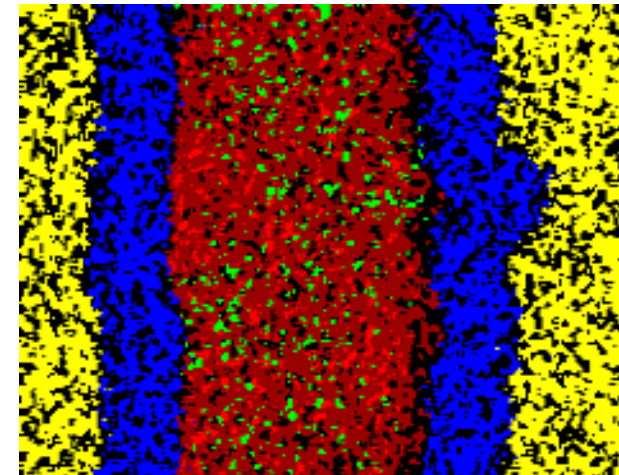


$10^{-5}$  mbar  
5 N/mm<sup>2</sup>



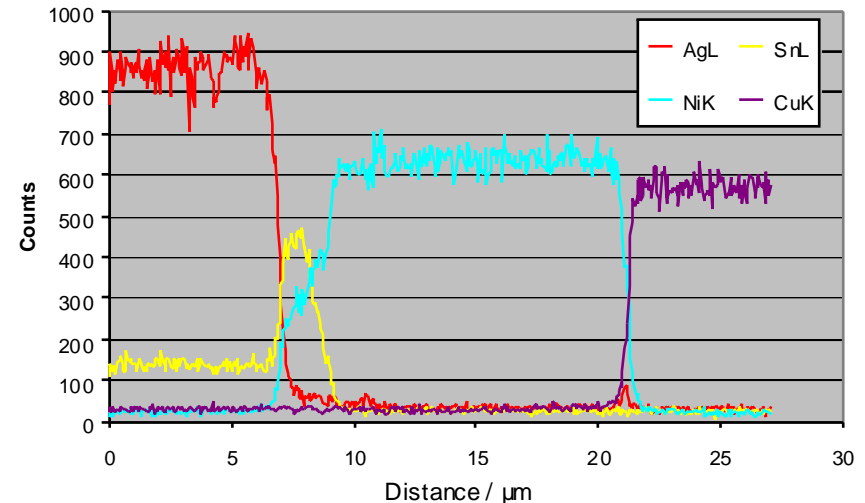
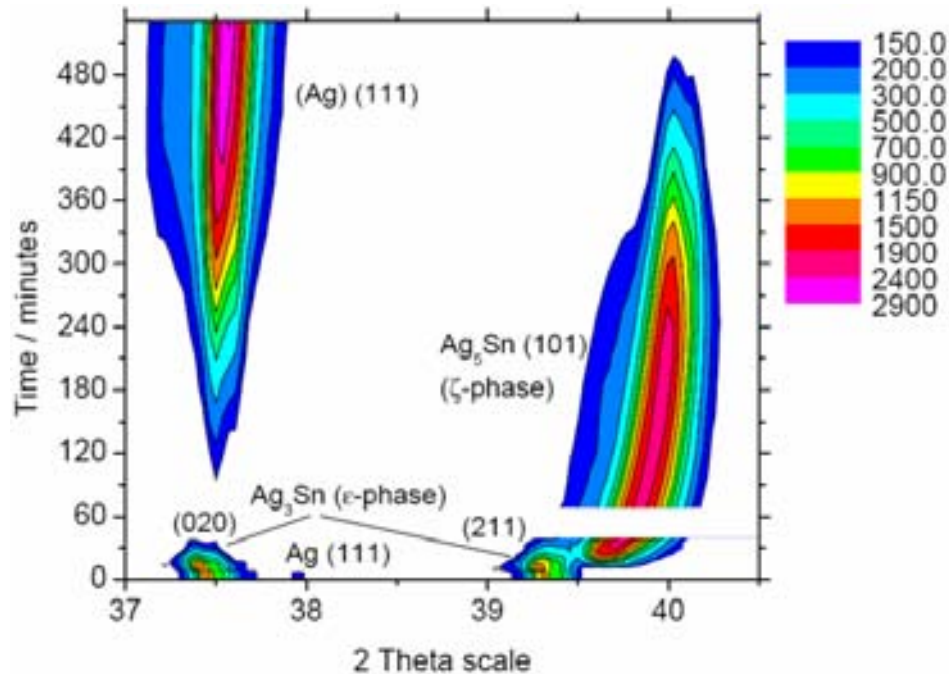
Microstructured copper sheet electroplated with 4 μm Ni, 5 μm Ag and 1.5 μm Sn.

Red: Ag  
Green: Sn



Element	spt1450	spt1451	spt1452	spt1453	spt1454
Ag		86.05	75.51	81.08	84.17
Sn		6.54	24.49	12.04	5.53
Ni	97.28	4.28		3.44	6.30
Cu	2.72	3.13		3.44	4.00

# IDS: The stability of the Ni „barrier“

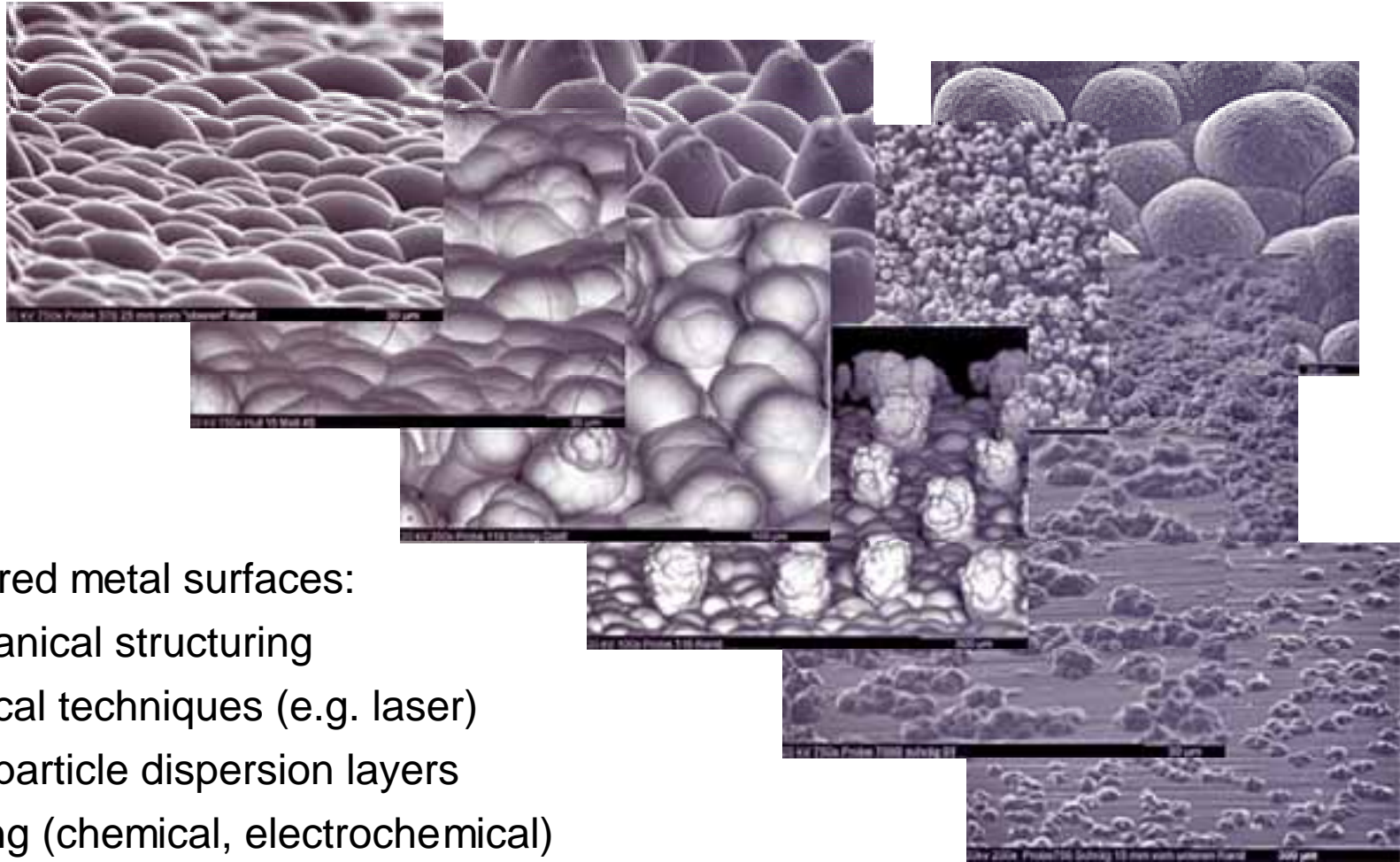


X-ray diffractograms of copper foil electroplated with 5  $\mu m$  of nickel, 4  $\mu m$  of silver and 1  $\mu m$  of tin recorded under Ar at 320 °C.

EDX line scan of a cross-section of a copper foil electroplated with 10  $\mu m$  of Ni, 4  $\mu m$  of Ag and 2  $\mu m$  of Sn after heating at 320 °C for 9 h under Ar.

B. Gollas, J. H. Albering, K. Schmut, V. Pointner,  
R. Herber, J. Etzkorn, *Intermetallics* 16 (2008) 962–968

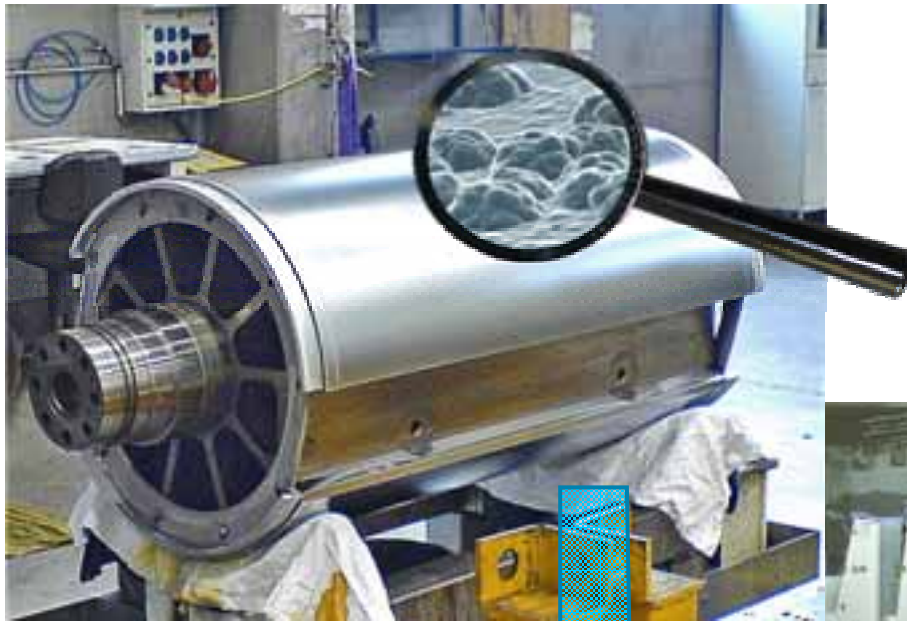
# Structured chrome



Structured metal surfaces:

- Mechanical structuring
- Physical techniques (e.g. laser)
- Hard particle dispersion layers
- Etching (chemical, electrochemical)

# Structured chrome



**R&D-Project:**  
KBA (Printing machines)  
Voestalpine  
(Temper rolling steel)

## Further applications:

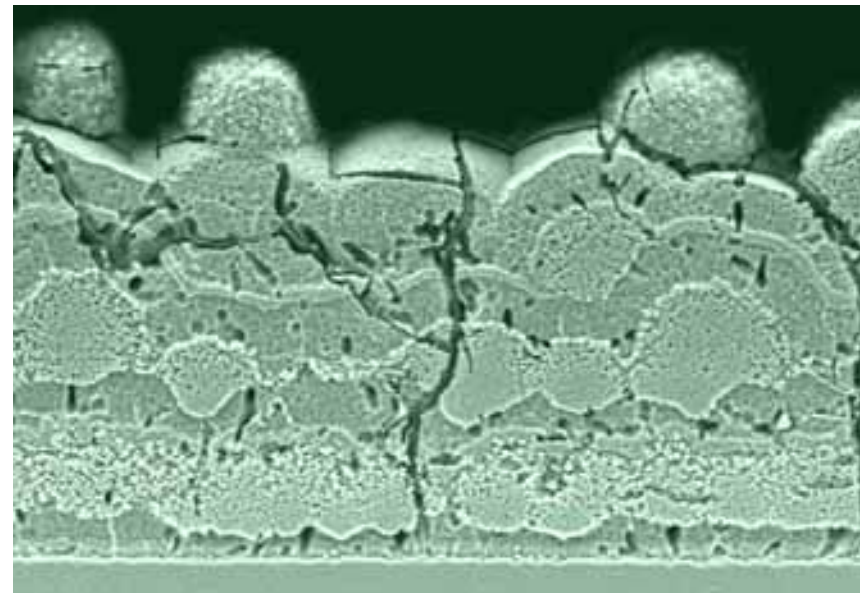
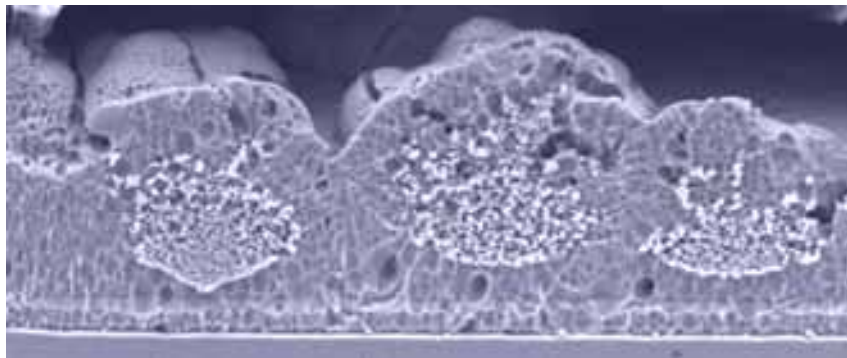
Embossing

Surfaces with novel tribological properties



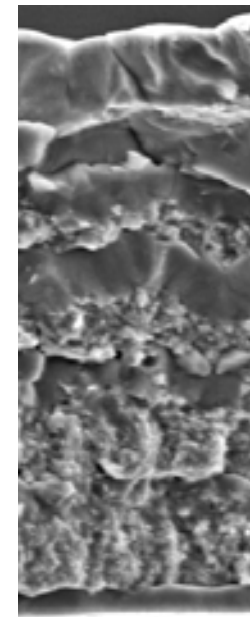
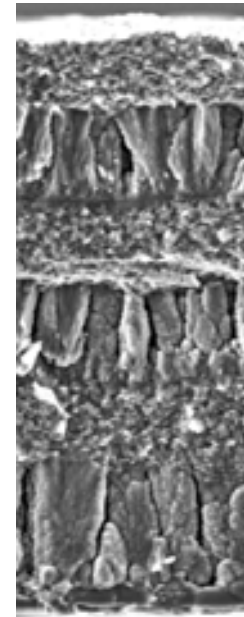
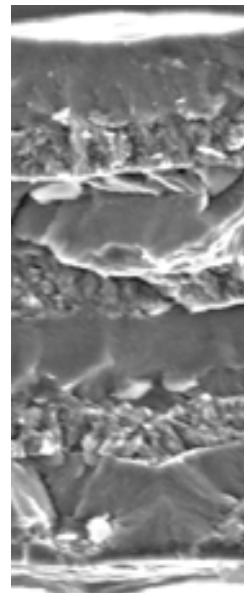
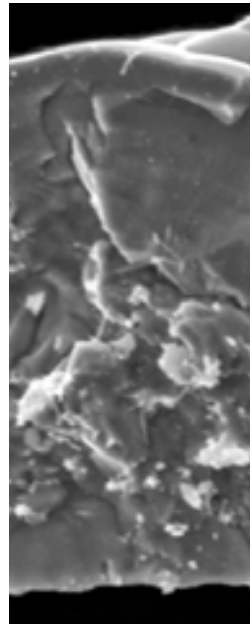
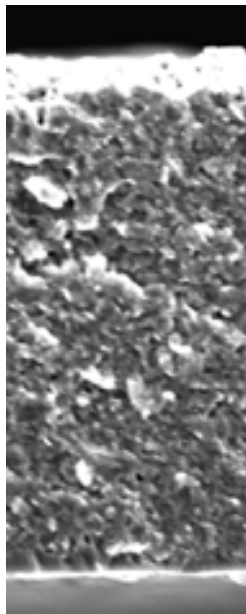
## Structured chrome

- “Surface design” through understanding/ variation of the deposition parameters
- Hardness > 1000 HV
- Same crystal structure, at least 2 different morphologies
- New technique: thermal control



## Structured chrome

- Control of properties: adhesion, hardness, internal stress
  - Sandwich structures (low crack and/or stress) at constant current
  - Gradient layers
  - Exact knowledge of working window required
- ⇒ deposition parameters ↔ properties

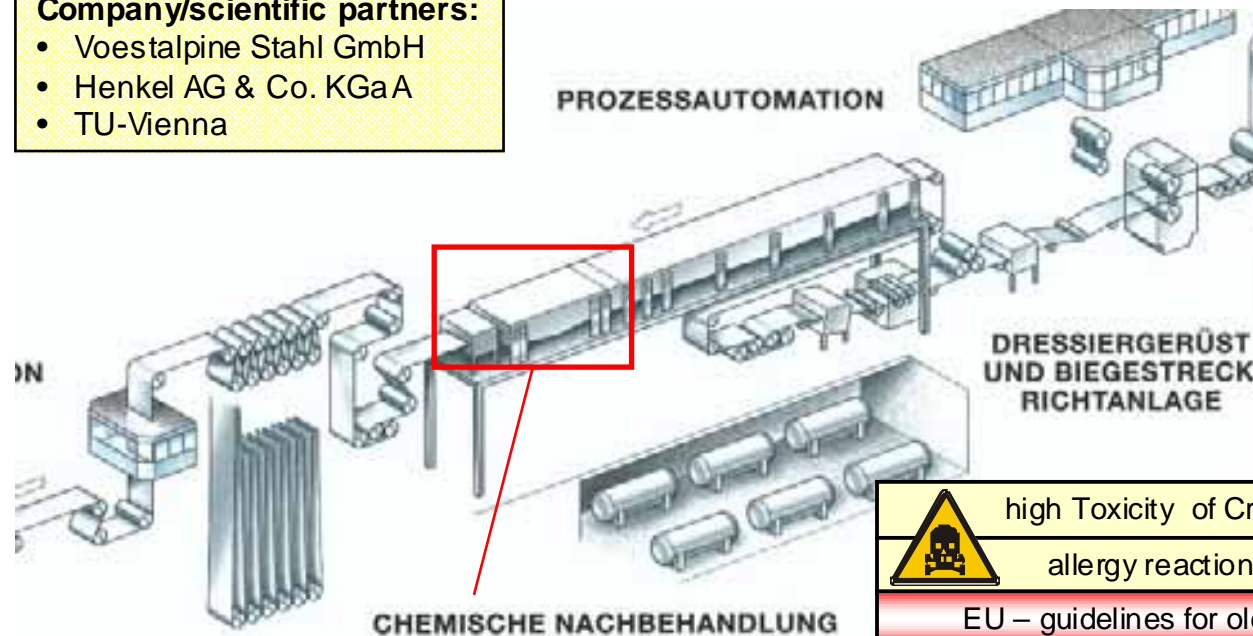



# Cr-free passivation on steel strip

2005 changeover of entire product range at **voestalpine** to the new chromium-free technology!

**Company/scientific partners:**

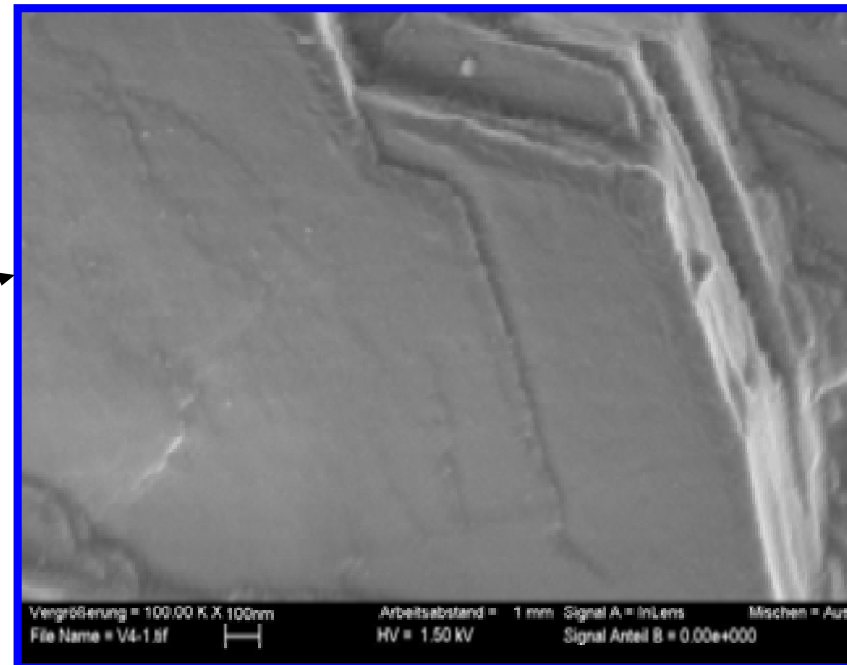
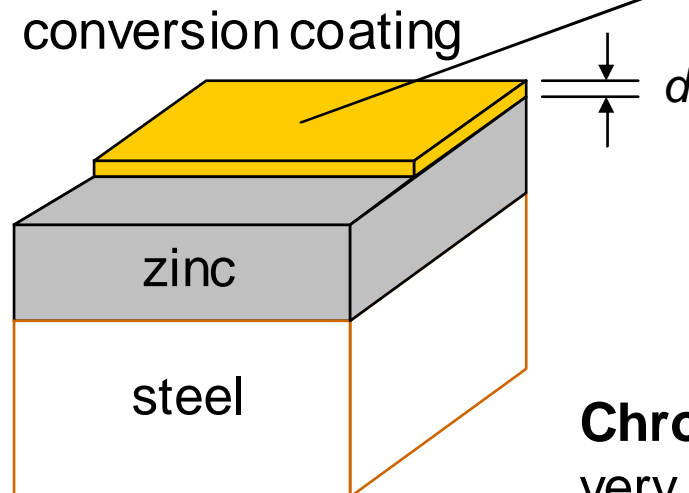
- Voestalpine Stahl GmbH
- Henkel AG & Co. KGaA
- TU-Vienna



	high Toxicity of Cr (VI) - compounds
	allergy reactions, carcinogenic
	EU – guidelines for old vehicles / July 2003
restrictive handling of Pb,Hg, Cd and <b>Cr(VI)</b> (max. 0 g Cr (VI) p. vehicle)	

## Conventional Cr(VI) passivation

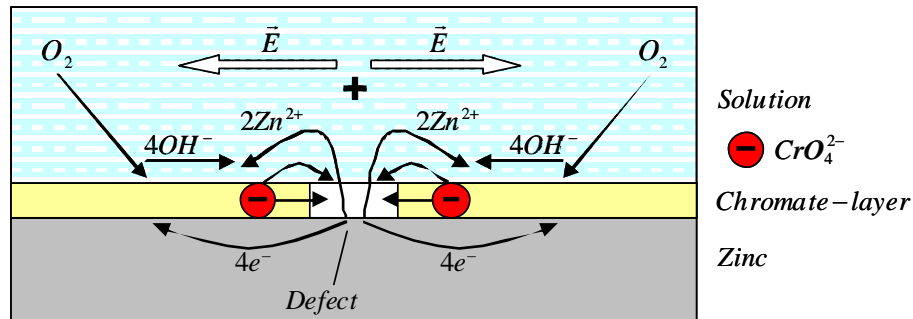
Surface loading 4-40 mg Cr/m<sup>2</sup>  
 $d = 20$  to  $120$  nm



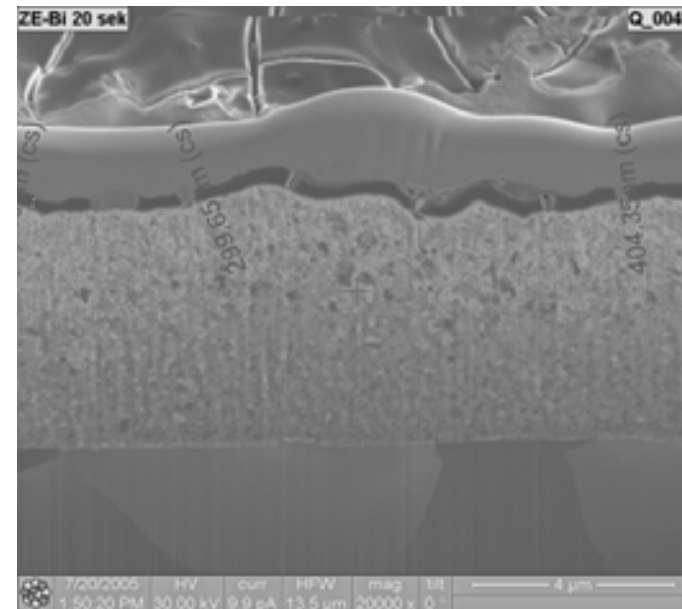
**Chromate Conversion Coatings (CCC)** are very thin amorphous (gel) layers, and highly protective against white rust formation.

## Key properties of Cr(VI) CCC's

- Inhibition of the ORR (oxygen reduction reaction)
- pH-stability
- Self healing



Model for the self healing properties of chromate layers.



Structure of a CCC. Cross sectional view, prepared by FIB

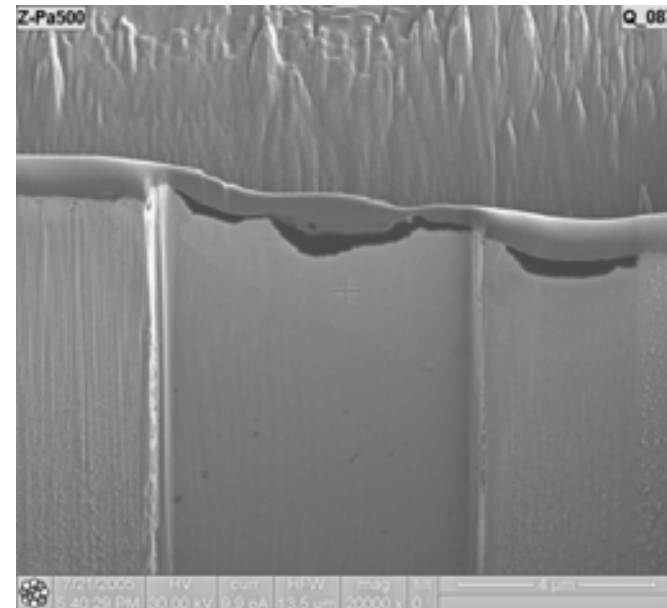
Fleischanderl, M.; Sagl, M.; Pernkopf, W.; Faflek, G.; Kronberger, H.; Besenhard, J. O.; Nauer, G. E., GDCh-Monographie (2005), 206-214

## Examples for chrome-free surface treatments

### Passerite™ 5004

Developed in ECHEM/CEST projects

- 👍 Excellent barrier on galvanized steel
- 👍 Test results compare favourably with Cr(VI) passivation
- 👎 No self healing
- 👉 Search for inhibitors with self healing properties
  - anionic inhibitors
  - nano container



Structure of Passerite 5004. Cross sectional view, prepared by FIB

# Interested?

CompanyPartners
Andritz AG, Vienna, A
AT&S Austria Technologie und Systemtechnik AG; Leoben, A
Atotech Deutschland GmbH, Berlin, D
Alz o Nobel Surface Chemistry AB, Stenungsund, S
Banner GmbH, Linz, A
Collini Holding AG, Hohenems, A
Doka Industries, Amstetten, A
EADS Deutschland GmbH, München, D
Fronius International GmbH, Wels, A
Glanzstoff Austria GmbH, St. Pölten, A
Hauser GmbH, Linz, A
Henkel KgaA, Düsseldorf, D
Infineon Technologies Austria AG, Villach, A
INGENIA Krantechnik GmbH, St. Florian, A
Gewa Wärmetechnische Anlagen GmbH, Lehden, D
OKA-TECH GmbH, Hochemmingen, D
Verzinkerei Herzlake GmbH & Co. KG, Herlake, D
Maschinenfabrik KBA-Mödling AG, Mödling, A
Schleifring und Apparatebau GmbH, Fürstenfeldbruck, D
Umicore Galvanotechnik GmbH, Schwäbisch-Gmünd, D
voestalpine Stahl GmbH, Linz, A
Wietersdorfer & Peggauer Zementwerke GmbH, Klein St. Paul, A

+ more than 20 national & international scientific Partners

Currently 67 staff  
(including: 25 PhD/Master students)

Join us!

## CEST Upcoming Events 2009

- See us at the *ECS Fall Meeting*, Vienna, 4-9 October 2009
- CEST Monthly Seminar on *Topics in Applied Electrochemistry*  
9 October 2009, 9.30-10.30, Wiener Neustadt:  
Donald R. Sadoway, Massachusetts Institute of Technology, Department of Materials Science and Engineering, Cambridge, USA:  
"Non-aqueous electrolytes for storage of electrical energy and electrolytic production of metal"
- Round Table Discussion  
*The future of ionic liquids in electrochemical surface technology*  
9 October 2009, 15.00-18.00, Wiener Neustadt
- Details: <http://www.cest.at/>