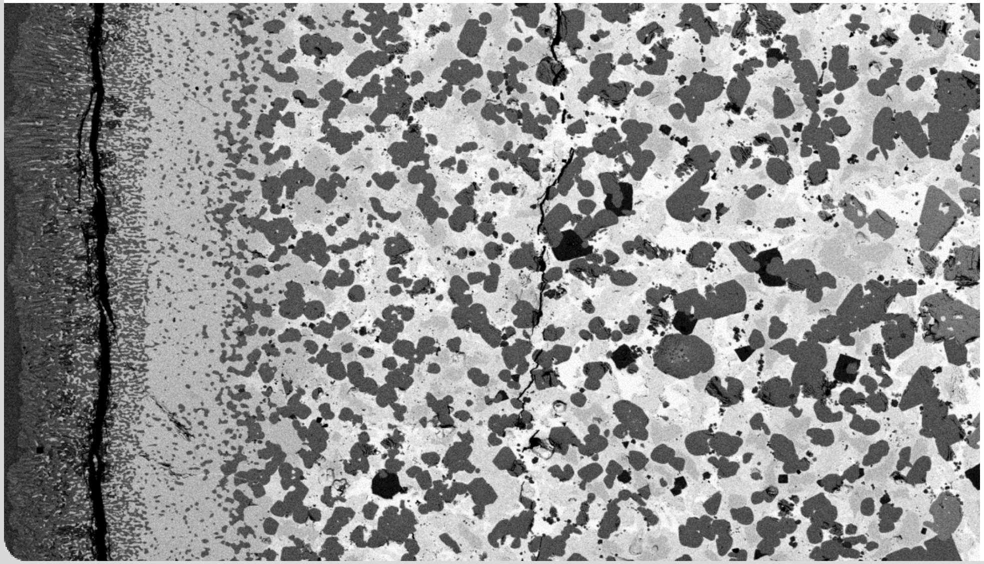
Evaluation of liquid tin corrosion on austenitic steels as well as nickel-based alloys and first tests on possible protective surface layers at high temperature

Thomas Emmerich, Carsten Schroer

KARLSRUHE INSTITUTE OF TECHNOLOGY – INSTITUTE FOR APPLIED MATERIALS



Overview

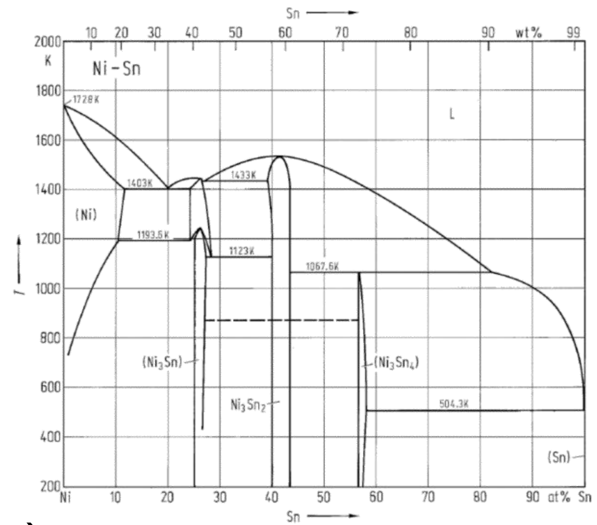
- Introduction
  - Liquid tin
  - Motivation
- Evaluation of liquid tin corrosion
  - Tested materials
  - Setup and procedure of corrosion experiments
  - Analysis of liquid tin corrosion
- Development of protective surface layers
  - Properties and production of surface layers
  - Analysis of layer behaviour in liquid Sn

Introduction

- Application of liquid Sn as heat transfer medium
  - Large liquid temperature range 232 – 2620 °C
  - Allows high heat flux
  - Not volatile or toxic

J. Pacio et al., Sol. Energy 93 (2013) 11–22.

- Corrosion of metallic materials
  - Solution of alloying elements
  - Formation of intermetallic phases with Sn (stannides)



B. Predel, Ni-Sn (Nickel-Tin), in: O. Madelung , Ni-Np – Pt-Zr. Landolt-Börnstein - Group IV Physical Chemistry. 5I, Springer-Verlag, Berlin/Heidelberg, 1998, 1–4

- Compatible materials
  - Rhenium, tungsten, quartz-glass, ceramics, graphite

R.N. Lyon, Liquid-metals Handbook, U.S. Government Printing Office, 1950.
E.L. Reed, J Am Ceram Soc 37 (1954), 146–153.
H. Shimotake et al., T Am Nucl Soc 10 (1965), 141–146.

- Alternative
  - Protective surface layers on steels or Ni-based alloys

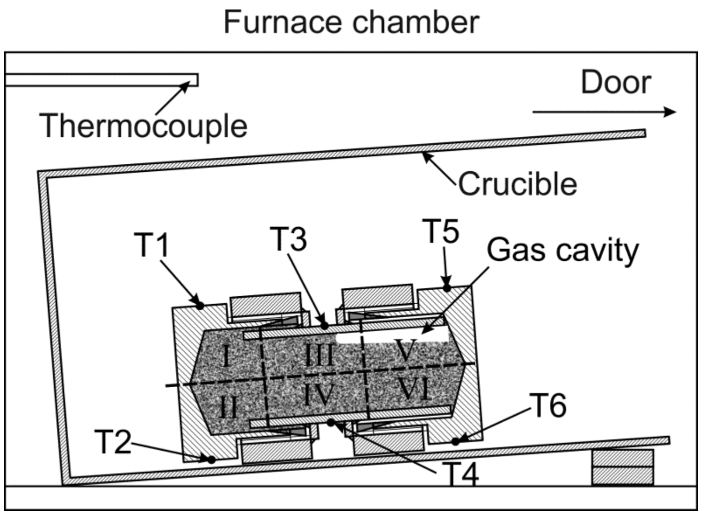
Evaluation of liquid tin corrosion
Tested materials

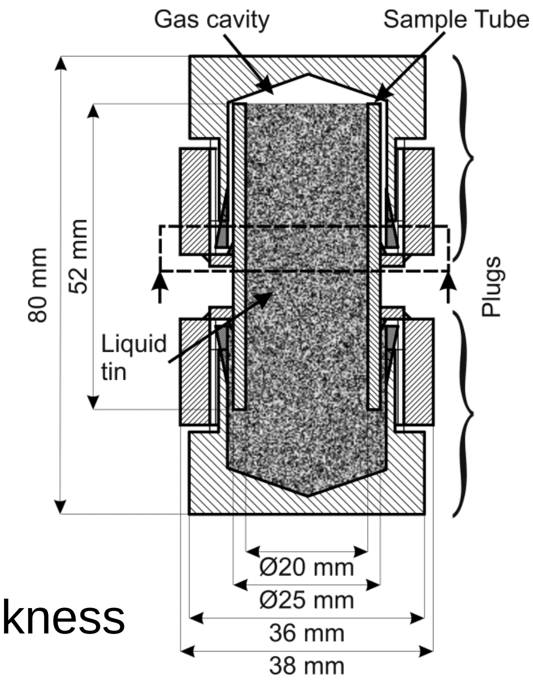
- Austenitic steels (1.4301, 1.4571) at 500 and 700 °C
- Ni-based alloys (2.4650, 2.4663) at 700 and 1000 °C

Material	Al	C	Co	Cr	Fe	Mo	Si	Ti	Ni
1.4301	-	≤ 0.07	-	17.5-19.5	Bal.	-	-	-	8-10.5
1.4571	-	≤ 0.08	-	16.5-18.5	Bal.	2-2.5	-	5x C≤ Ti≤ 0.7	10.5-13.5
2.4650	0.45	0.05	19.8	20	0.44	5.9	0.09	2.1	Bal.
2.4663	0.99	0.06	11.7	21.97	1.09	8.53	0.13	0.41	Bal.

Evaluation of liquid tin corrosion

Setup and procedure of the corrosion experiments

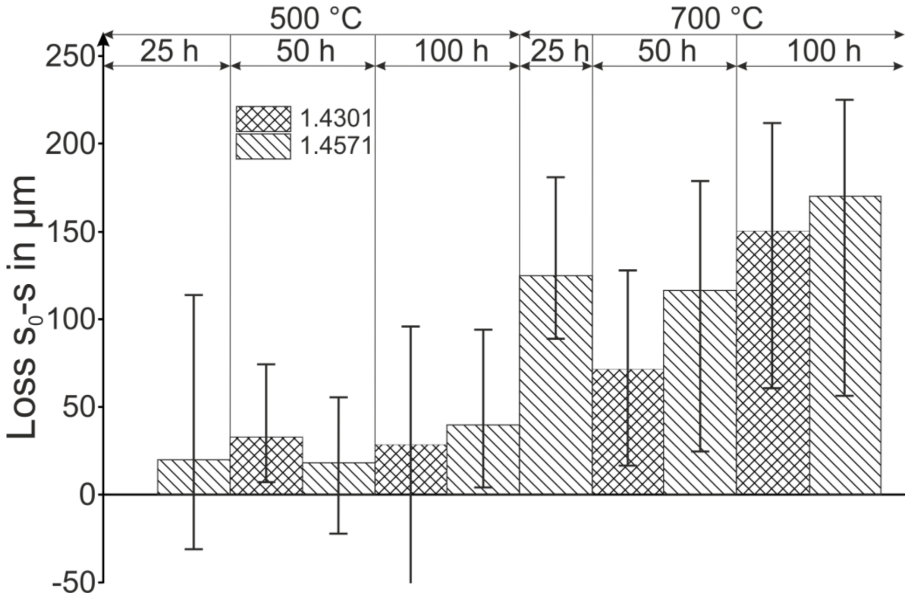


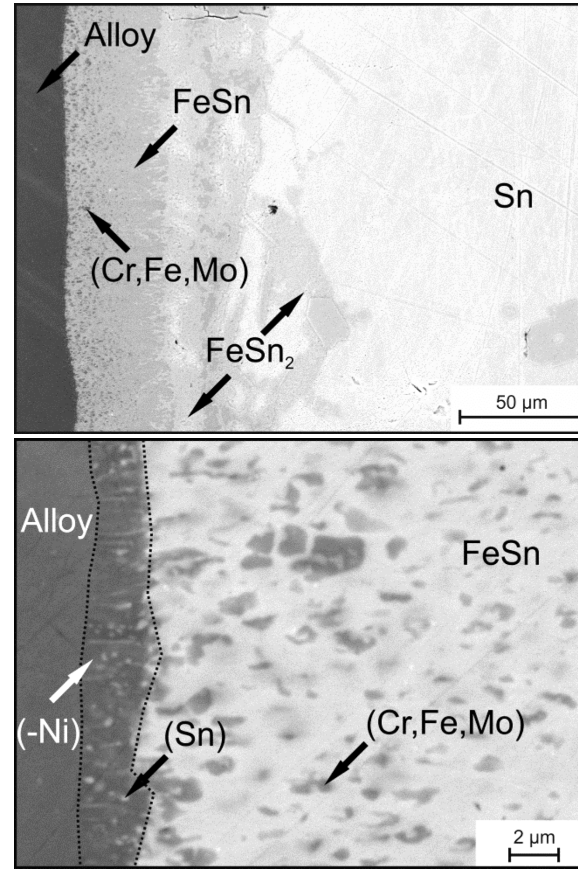


- ❑ Preparation
  - ❑ Determination of the average wall-thickness
  - ❑ Filling in Ar-atmosphere
- ❑ Testing
  - ❑ Exposure at 500, 700 and 1000 °C for 25, 50 und 100 h
  - ❑ Measurement of the temperature distribution
- ❑ Post-test analysis
  - ❑ OM, SEM, EDX

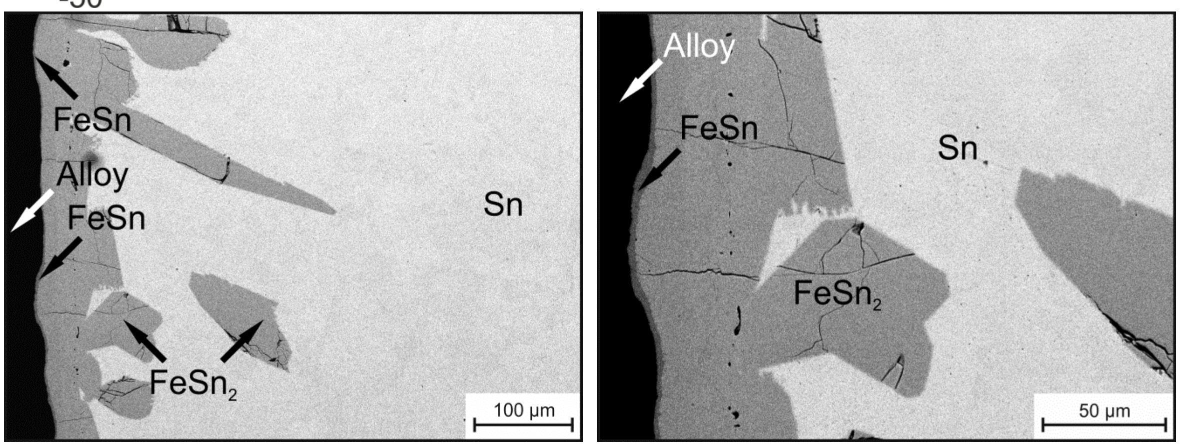
Evaluation of liquid tin corrosion

Steels after corrosion experiments





1.4571 at 700 °C for 25 h



1.4301 at 500 °C for 100 h

Evaluation of liquid tin corrosion

Ni-based alloys after corrosion experiments

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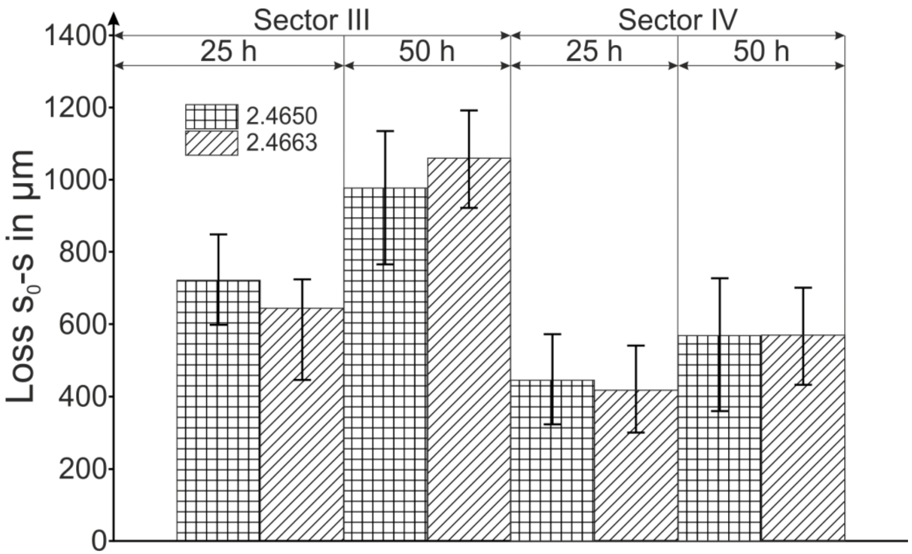
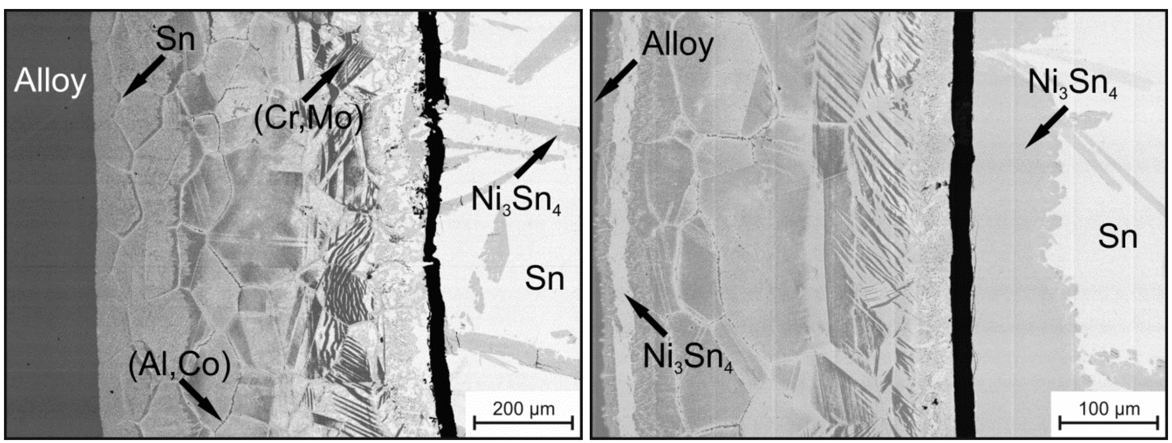
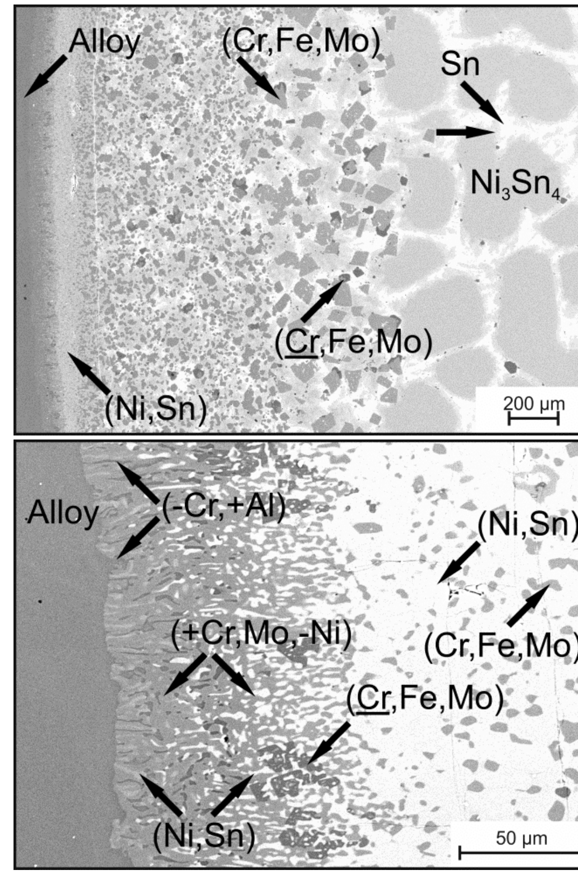




2.4663 at 700 °C for 25 h



2.4663 at 1000 °C for 25 h

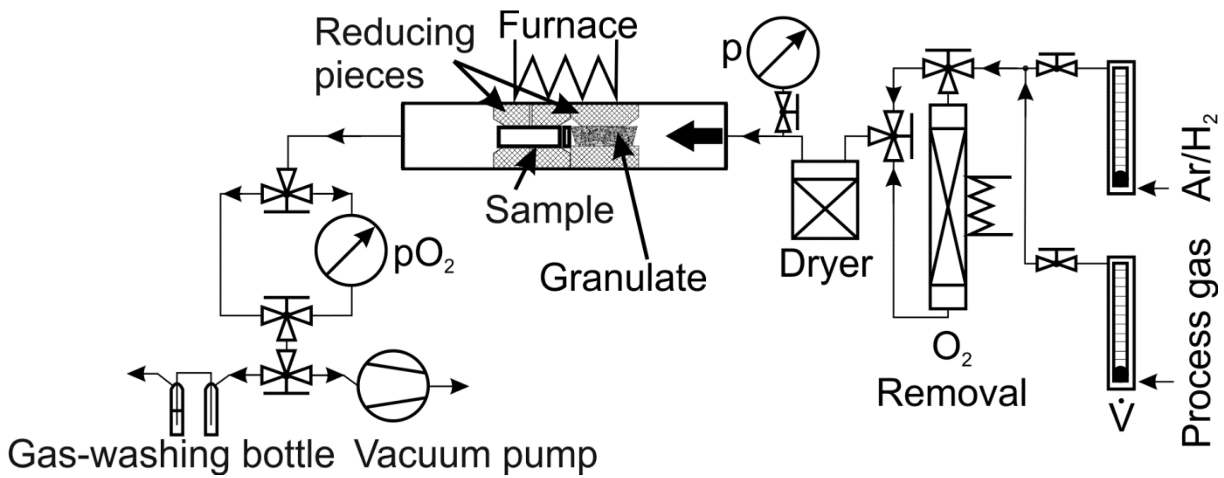
Evaluation of liquid tin corrosion

Conclusions from corrosion experiments in liquid tin

- Solution based corrosion
  - High solubility of Ni causes selective leaching of Ni and higher material losses of Ni-based alloys than of steels
  - Less soluble Cr, Fe and Mo form α-, σ- or similar phases
- Growth of stannides
  - Precipitate from locally saturated melt
  - Re-precipitation of solutes leads to further material consumption
  - Solid state diffusion through layers allows corrosion to continue
- Consequences
  - Dense protective surface layers necessary
  - Short grace periods especially at 1000 °C
  - Precise corrosion monitoring necessary

Development of protective surface layers

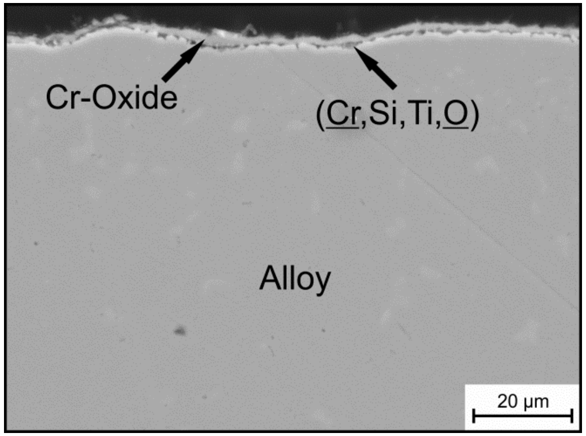
Properties and production of surface layers

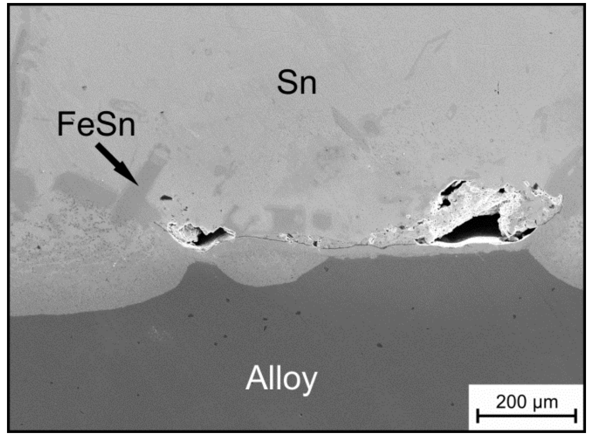


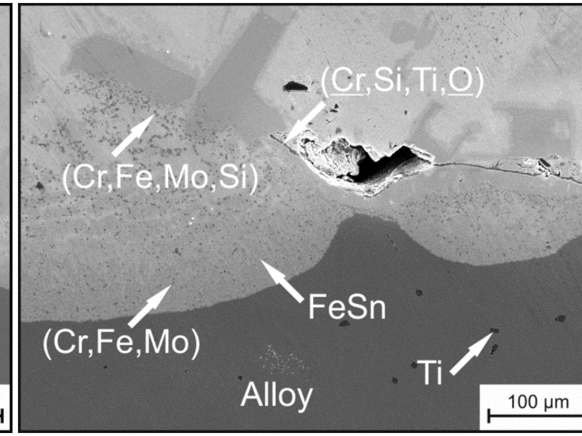
Carbides	Oxides	Nitrides
❑ C shows very low solubility in liquid tin	❑ O is soluble in liquid tin ❑ Short term protection	❑ N shows very low solubility in liquid tin
❑ Low pressure carburisation ❑ At 1000 °C in propane ❑ p ≤ 10 mbar, t – 8 h ❑ PVD: TiC	❑ High temperature gas oxidation ❑ At 800 °C in flowing Ar 6.0 ❑ t – 48 h	❑ High temperature gas nitration ❑ At 800 °C in flowing N_2 6.0 ❑ t – 192 h ❑ PVD: TiN

Development of protective surface layers

Corrosion experiments on oxide layers

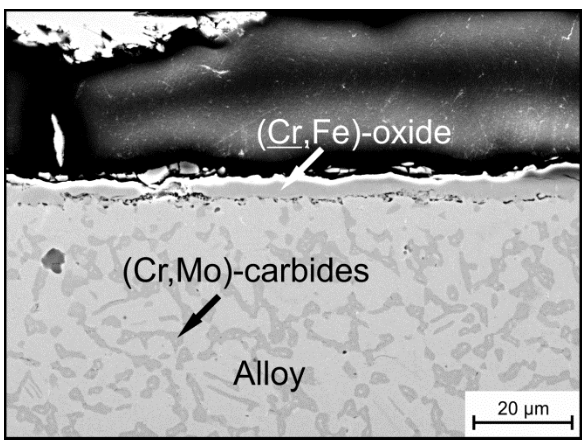


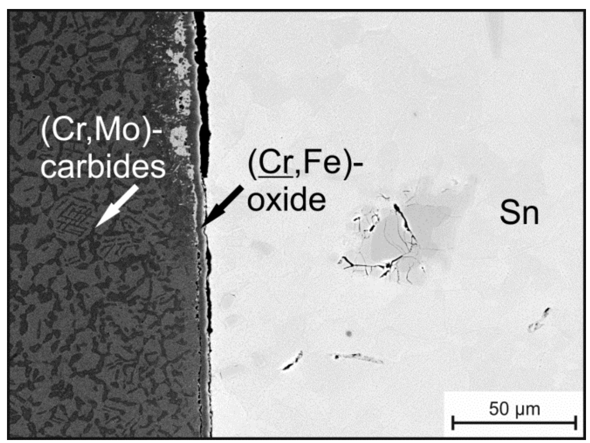


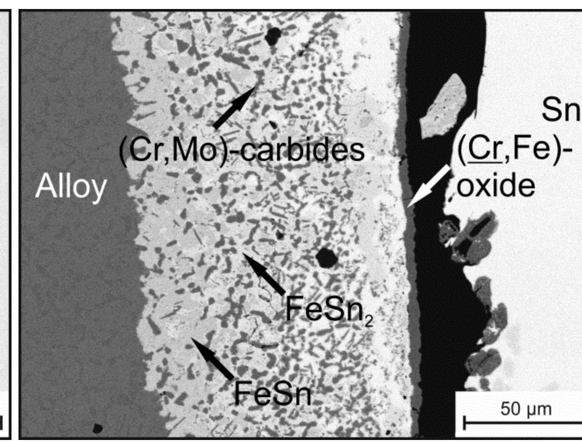


Oxidised

1.4571 at 700 °C for 100 h





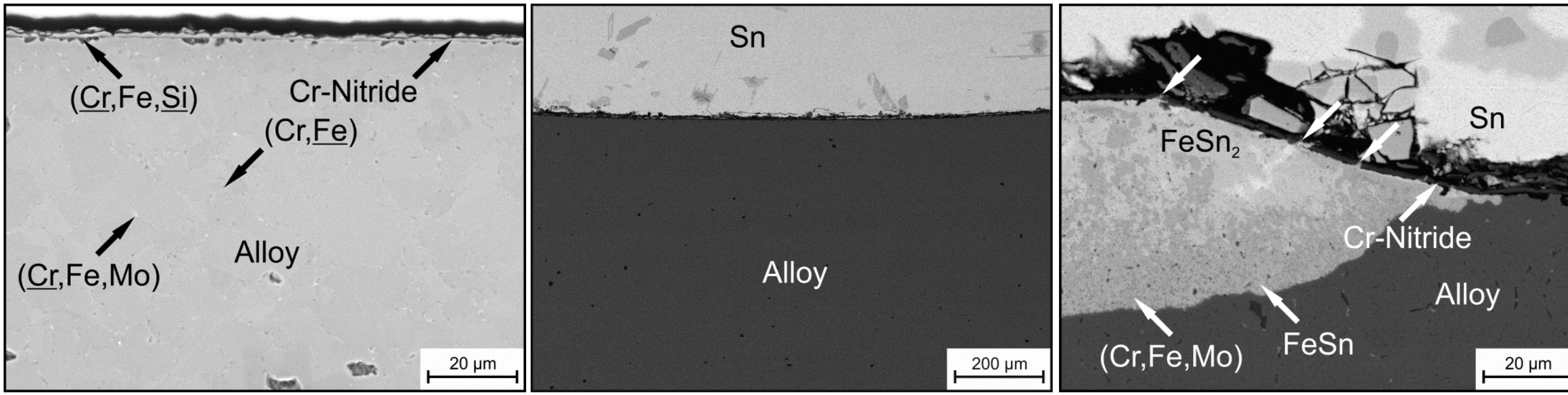


Carburised

1.4571 at 700 °C for 100 h

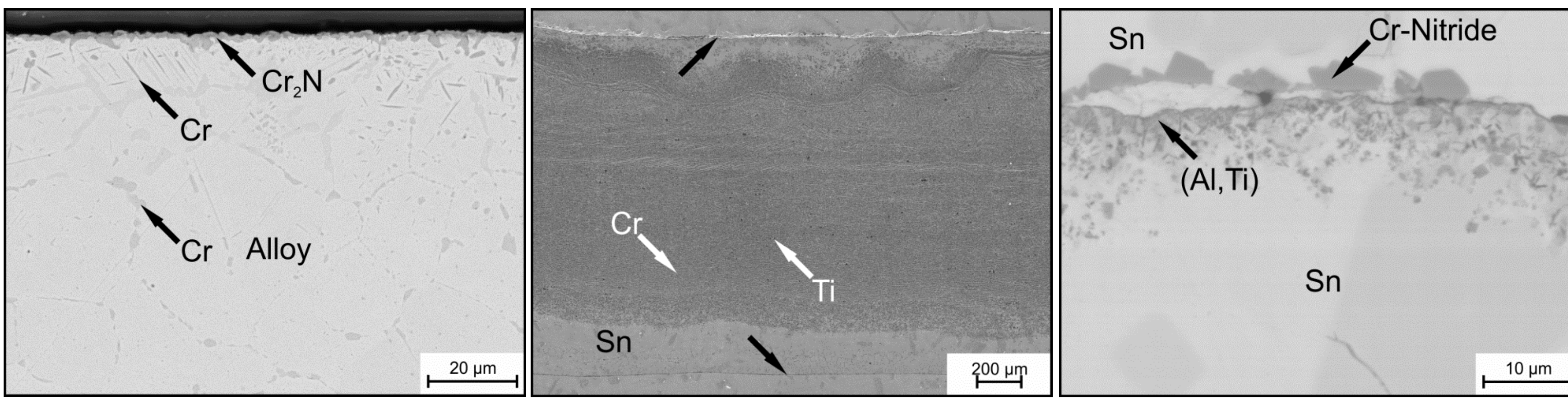
Development of protective surface layers

Corrosion experiments on nitride layers



Nitrided

1.4571 at 700 °C for 100 h



Nitrided

2.4642 at 700 °C for 50 h

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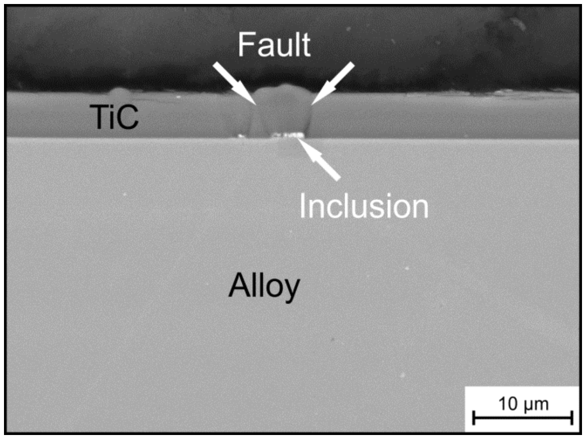
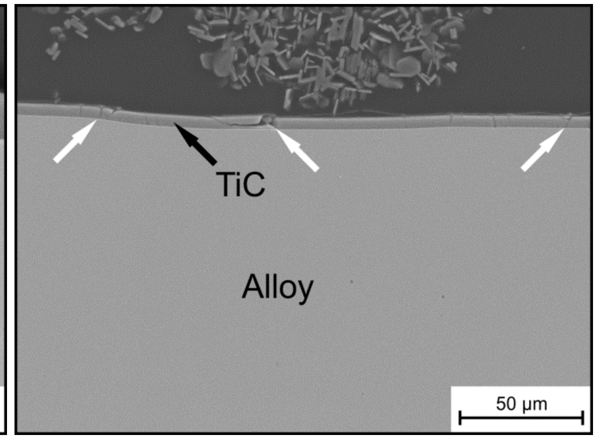
Development of protective surface layers

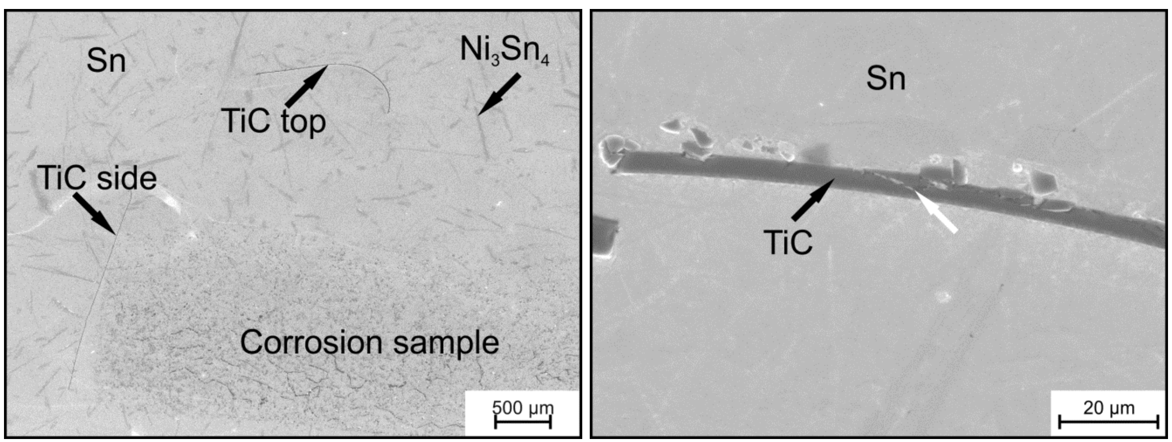
Corrosion experiments on PVD layers



PVD



700 °C for 50 h in Ar



2.4642 at 700 °C for 50 h

Development of protective surface layers

Conclusions from corrosion experiments on surface layers

- Oxides
  - Dissolution of thin Cr-oxide layers underline limited stability
  - Thicker layers necessary for protection against liquid tin
- Carbides
  - Carbides show stability against liquid tin
  - No reduction of corrosion as network
  - Deposition as complete carbide layers required
- Nitrides
  - Formed continuous layers and protected wide sample areas
  - Penetration of liquid tin only through cracks in the layer
  - Increase of chemical stability by higher alloying content of Al or Ti than Cr
- Most promising approach
  - Preferably nitrides for thermo-chemical layer formation
  - Compensation of different thermo-mechanical properties for deposited coatings by functionally graded layers
  - Layers of chemical stable compounds like Al- or Ti-nitrides to prevent transformation by alloying elements

Thank you for your attention!

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