Karlsruher Institut für Technologie

Challenges to Developing W-Based Materials for Fusion Applications

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Electro-chemical processing for tungsten fabrication and joining by layer deposition

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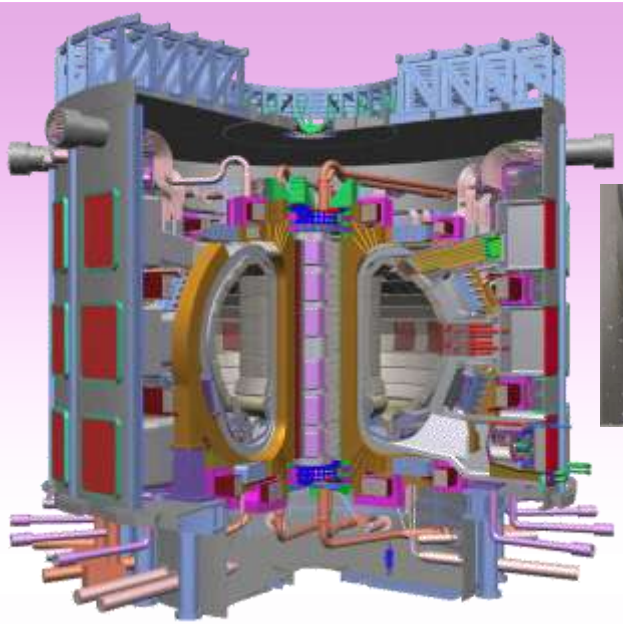
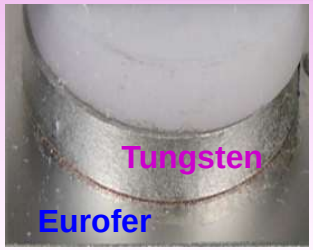
Outline:

- Motivation
- Challenges in W handling
- Machining by electro-chemical dissolution (ECM)
- Layer deposition / joining from aqueous systems
- Innovative deposition from aprotic electrolytes
- Conclusions

Motivation for Electro-Chemical Processing



Divertor development



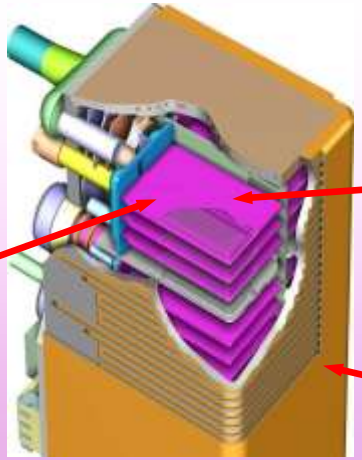
Application:

- **Machining**
- **Joining**
- **Composites**

W, Cu
Steel, SS

Activation control
Cleaning of EDM processed BU's

Blanket development

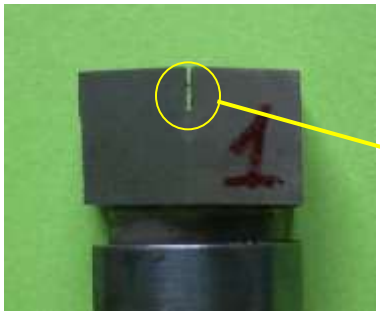


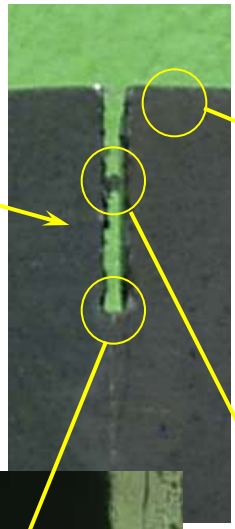
T-permeation and corrosion barriers
Al, Er, W-based

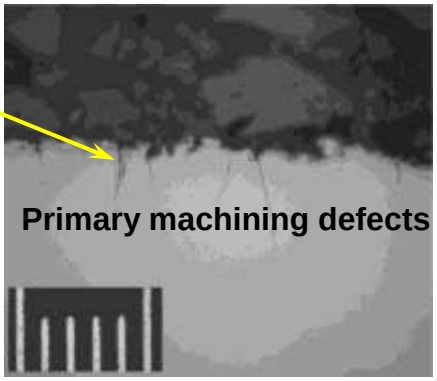
FW coating
W-scales
Oxidation protection Al, Si, Y

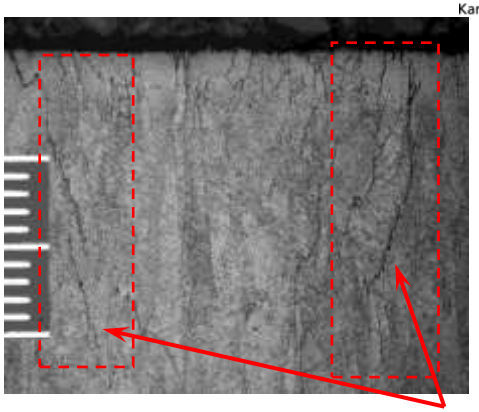
Motivation for Electro-Chemical Processing of W

Typical defects and failures of W parts

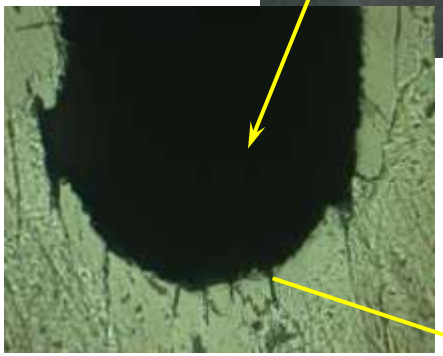


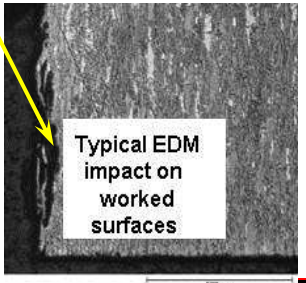


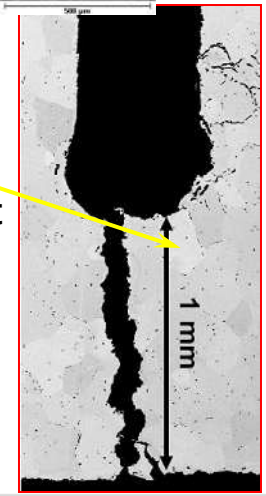


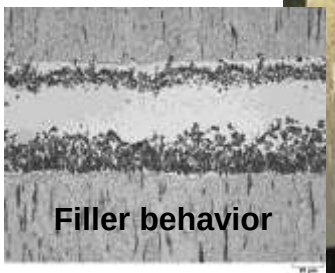


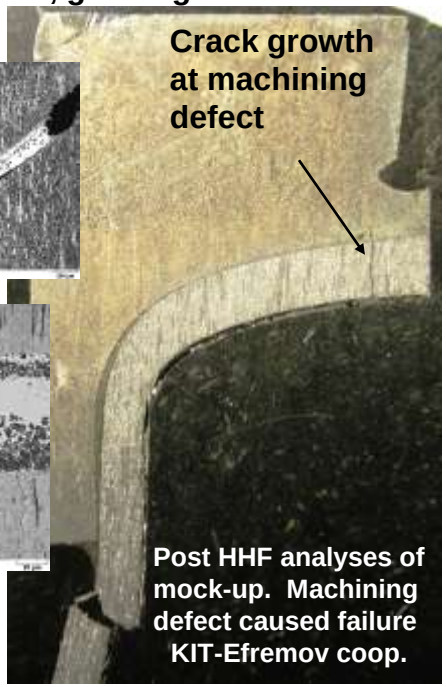
Secondary failures, growing under HHF load







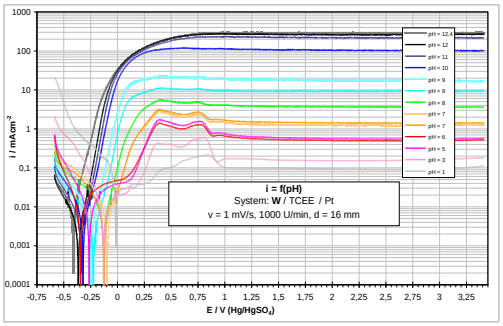




HHF results defined needs for development in W-processing and joining

- Smooth surfaces
- No defects from machining
- No sharp edges
- Adapted fillers for brazing

Electro-Chemical Reactions



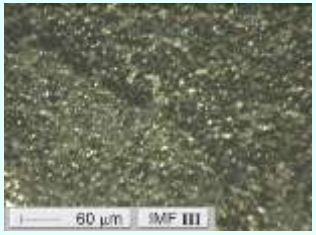
Anodic reaction
W-dissolution

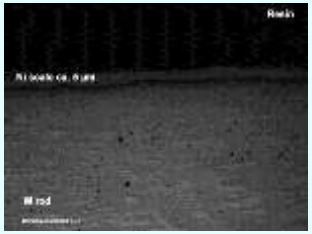
Cathodic reaction
Deposition

Basic investigation

Application

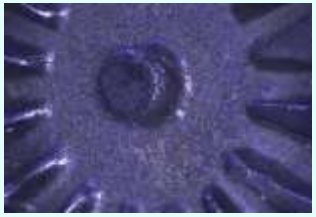
Surface treatment
Electro-polishing
S-ECM

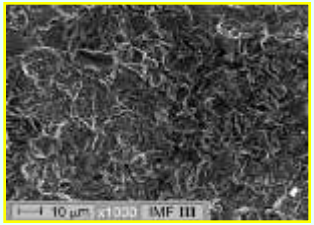




Layer deposition
brazing e.g. by Ni, Cu, Pd
protic

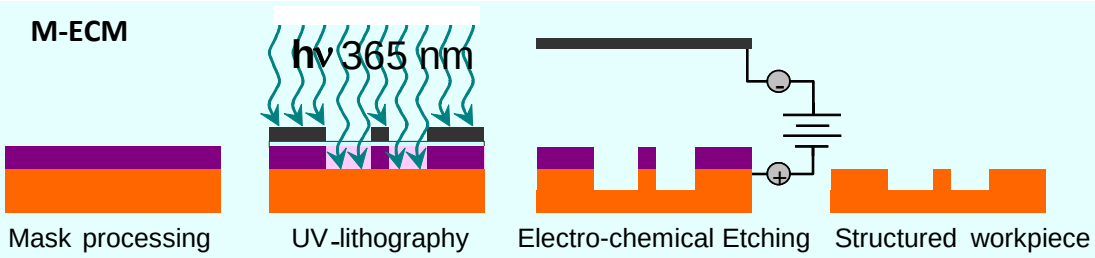
Machining
ECM
C-ECM, M-ECM





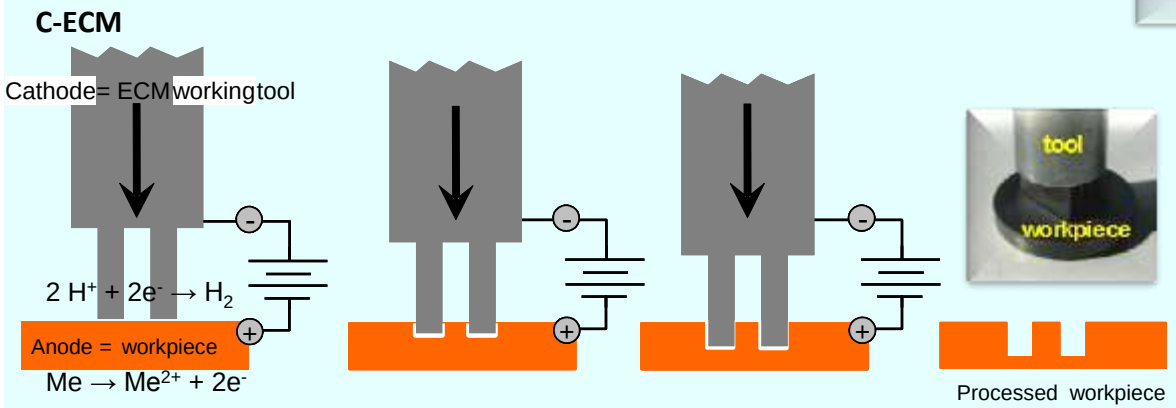
Deposition
aprotic
e.g. W or Ta

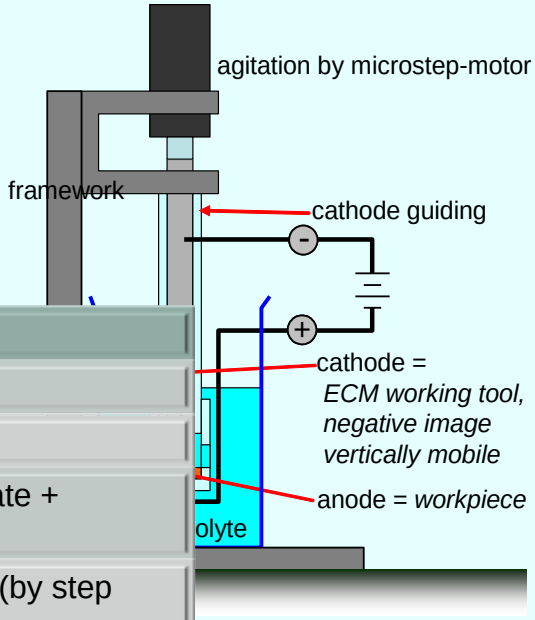
ECM for 3-D structuring



ECM Requirements
- Electrolyte development
- M-ECM: anode mask process
- C-ECM: cathode tool

ECM Advantages
- No cracks by ECM process
- Surface polishing
- residue-free metal removal





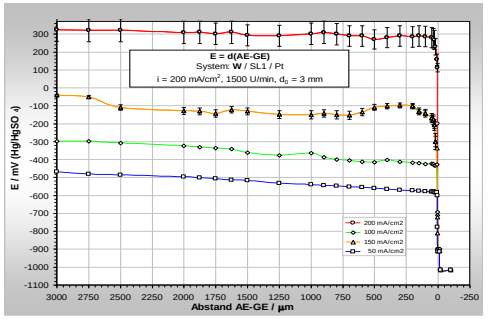
MAIN DIFFERING FEATURES		
	M-ECM	C-ECM
Technique	Conventional installation	Complex facility
Parameters	Current x time = charge	Charge + distance + steprate + convection
Cathode	passive counter electrode	active shaping component (by step motor)
Tool design	2-dim. mask (positive)	3-dim. electrode (negative)
Transformation	+2-dim g +3-dim	-3-dim g +3-dim

W machining – impact on quality

Parameter dependencies

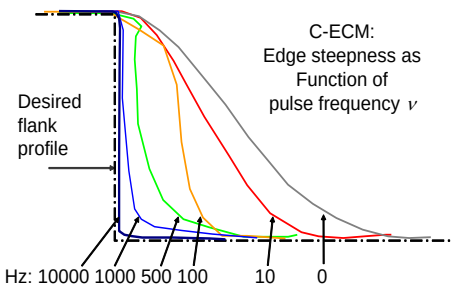
Smaller distance

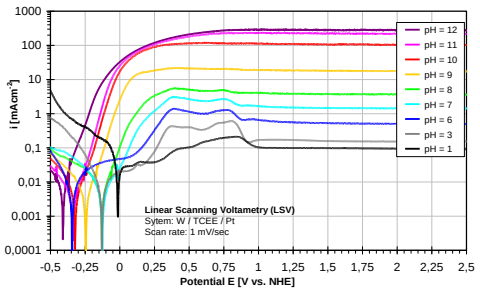
tool – work piece



Quality improvement

Higher frequency of pulsed DC power



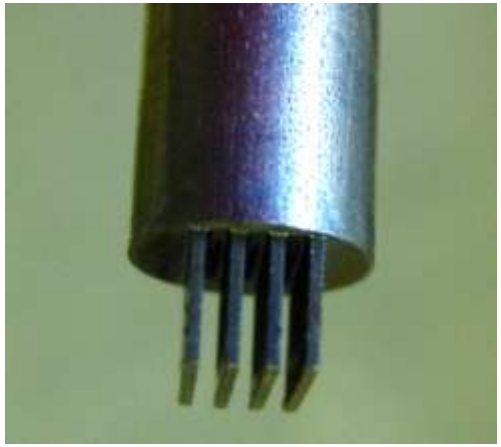


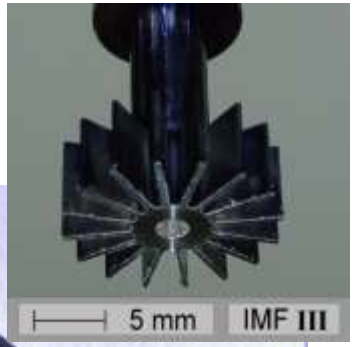
Mobility control / reduction

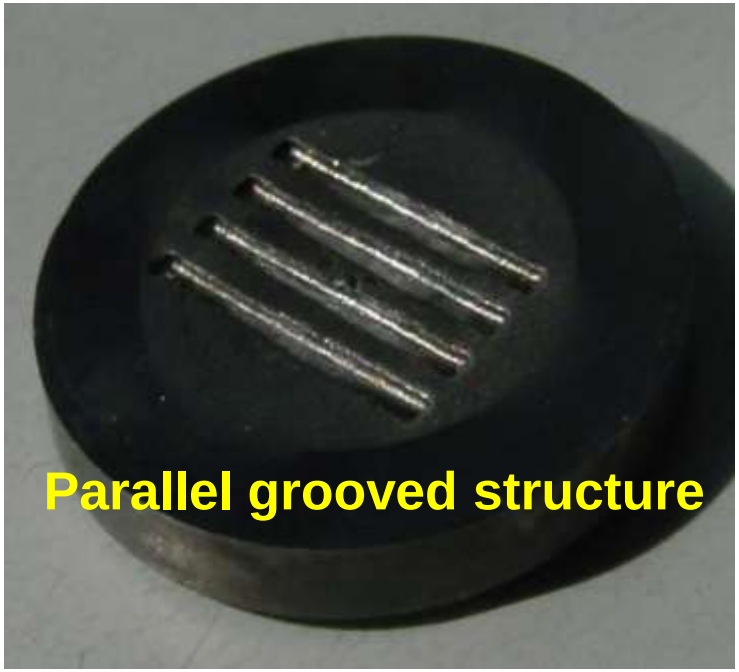
Conductivity of electrolyte

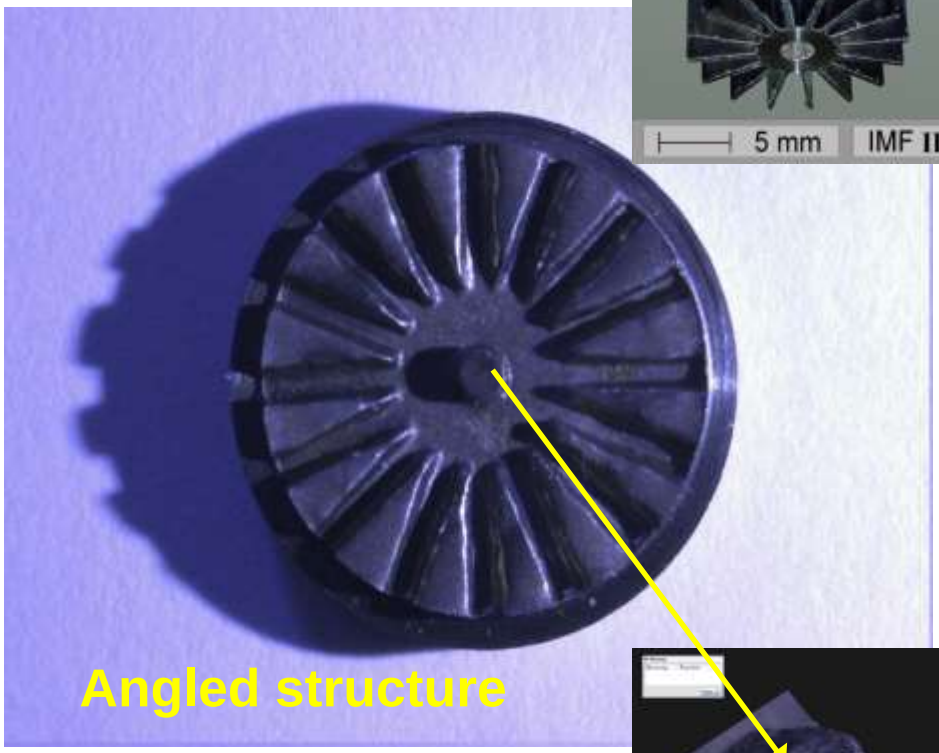
Tungsten by C-ECM
(Anodic dissolution)

C-ECM demonstrator cathodes

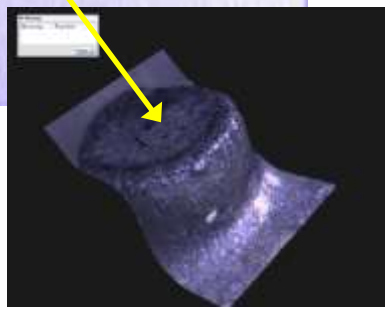






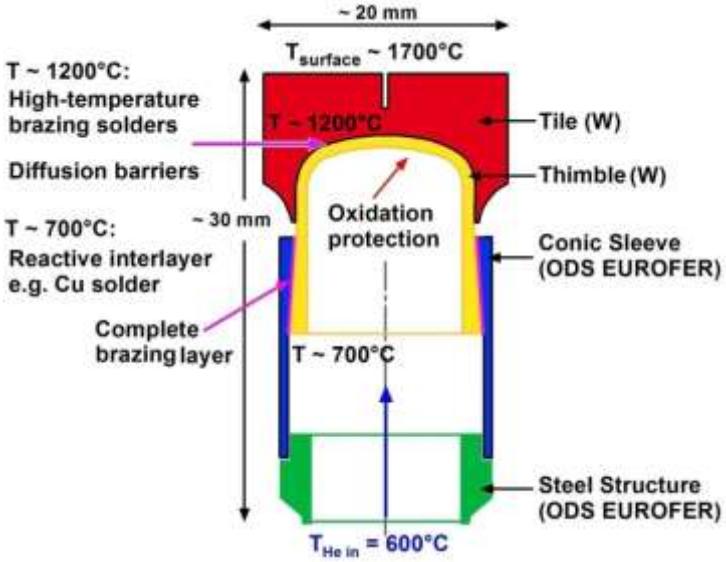


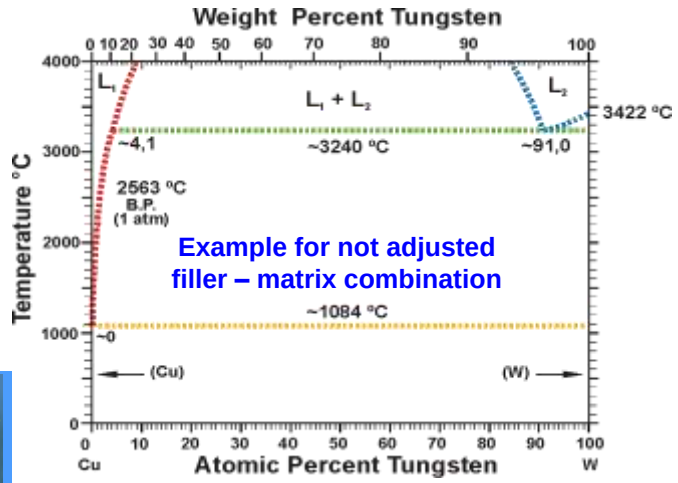
Generated W-structures



Functional layers by cathodic deposition

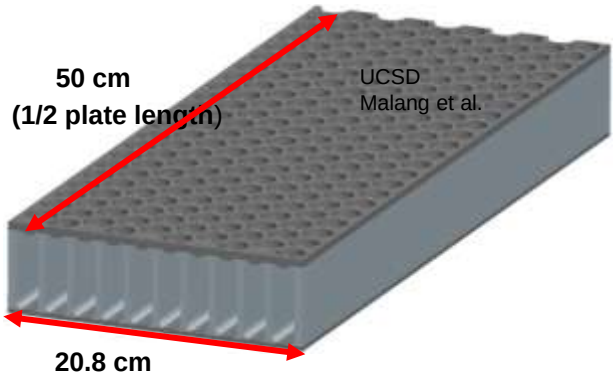
Requirements:





Brazing lacks in the past

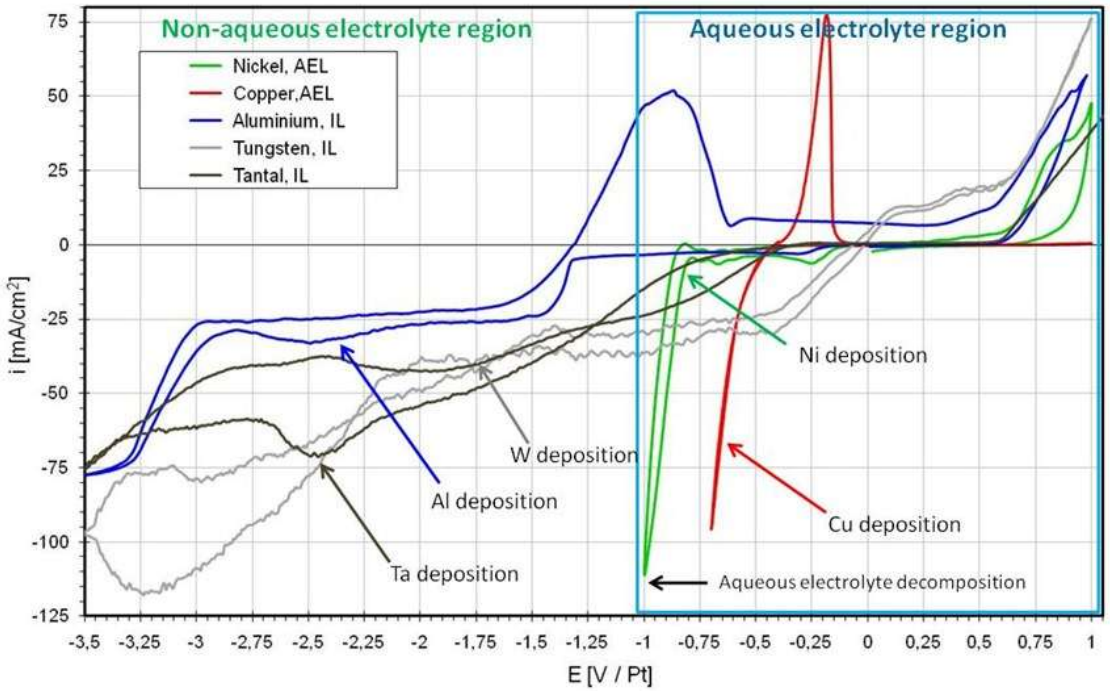
- No wetting
- Incomplete wetting
- Not adapted filler composition
- Embrittlement



Development paths in EC:

Oxidation protection by scales e.g. Ta, Si, Cr, Y, Zr, La, Al, Eu
Diffusion barriers e.g. W, Ta, V, Cr, (Ni), Fe
Fillers based on e.g. Ti, V, Pd, Zr, (Ni), Cu, Fe

Electro-chemistry for coating development

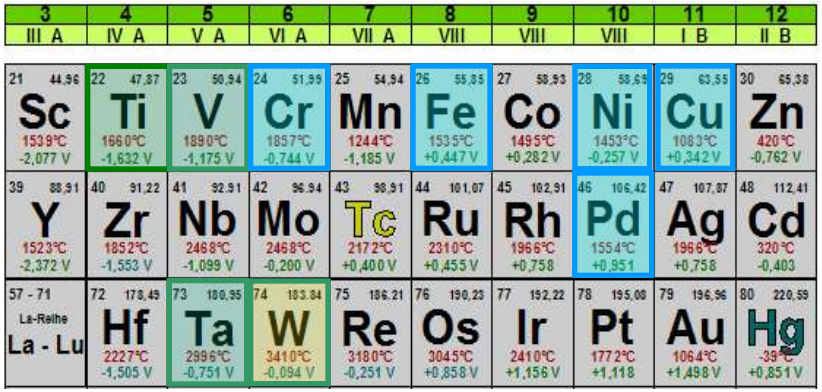


EC measurements of protic and aprotic metal deposition systems

Organic electrolytes:

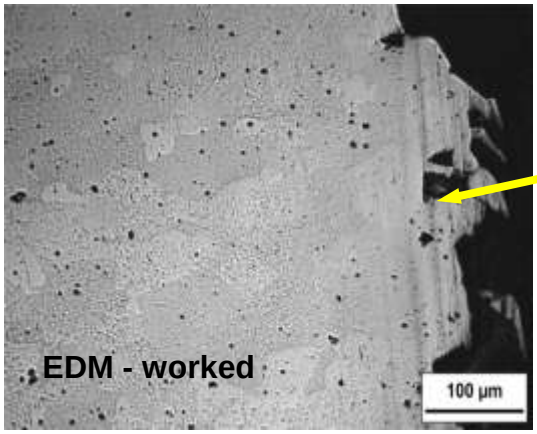
EMIM-Cl (Ethyl-Methyl-Imidazolium-Cl)
PC (Propylencarbonat)

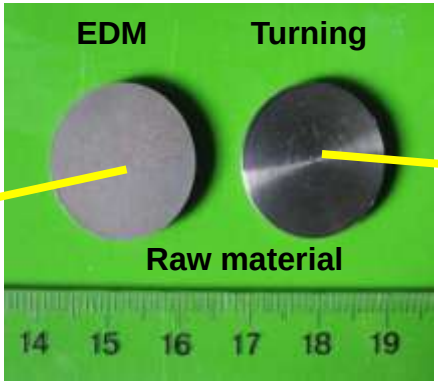
Elements with relevance to joining and electro-chemical deposition

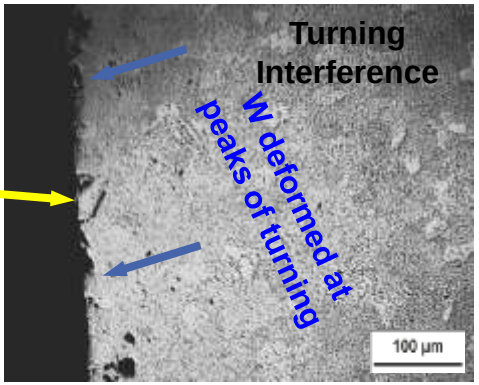


Coatings from aqueous systems

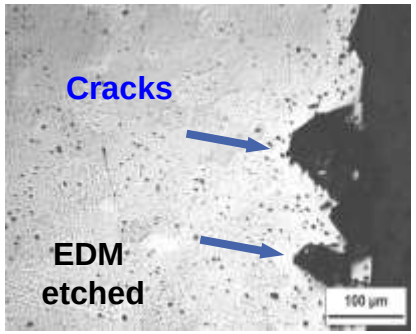
Surface activation



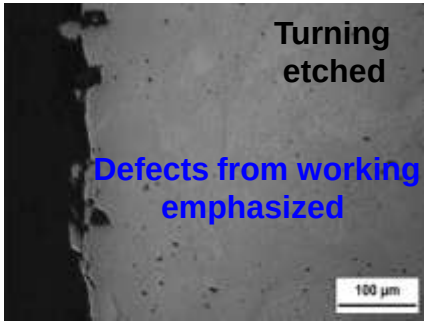


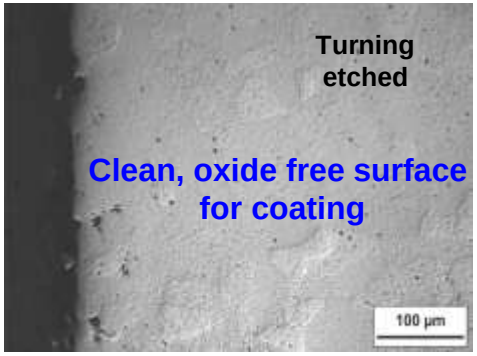


Surface activation by



$K_3[Fe(CN)_6]$ * KOH	small roughing
HNO_3 (conc.) * HF	high temp.
KOH	polishing, low adherence
NaOH * $KMnO_4$	HCl cleaning
HCl * H_3C-COOH	roughing
NH_3 * $NaNO_3$	polishing, current

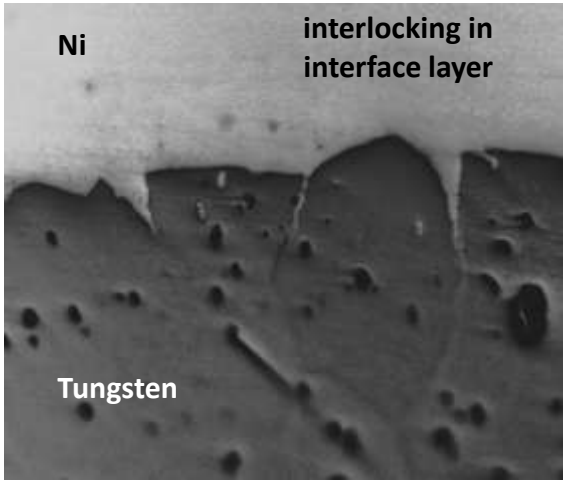




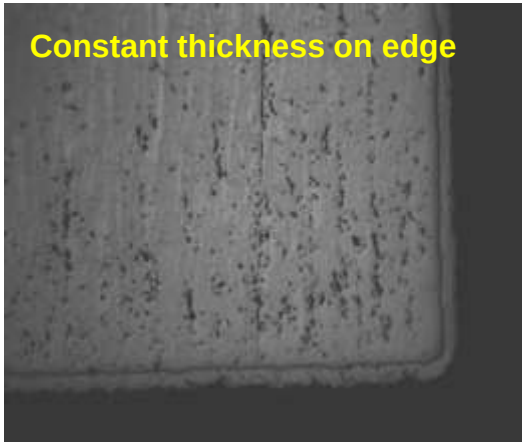
Coatings from aqueous systems

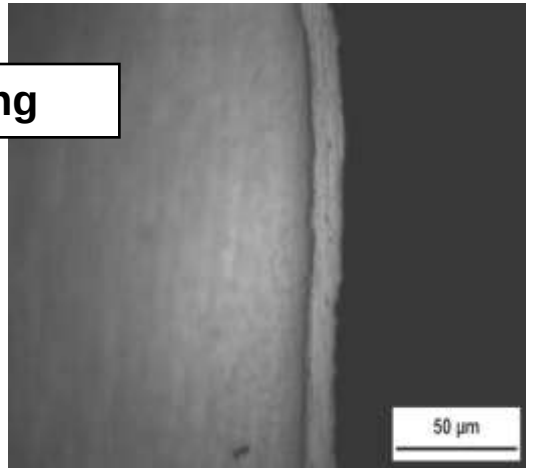
Advantage of homogeneous layers

EDM worked part

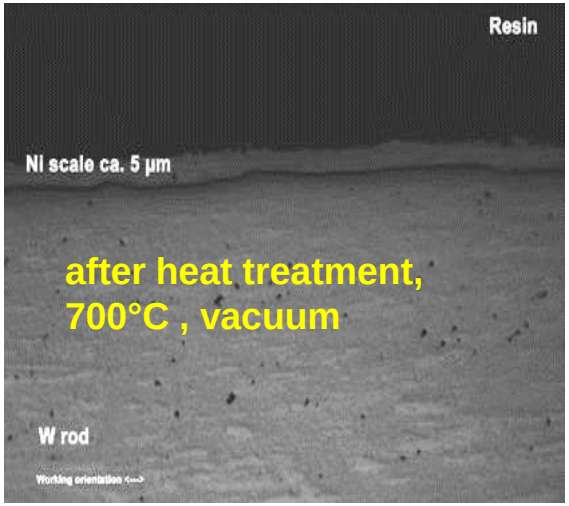


Parts by turning





EDM initiated cracks contrasted



Electrolyte: 1.3 M Nickel sulfamate $Ni(SO_3NH_2)_2$
T = 52 °C, pH = 3.5, i = 10 mA/cm², D = 12 µm/h

W –steel joining with Ni and Cu coatings from aqueous systems

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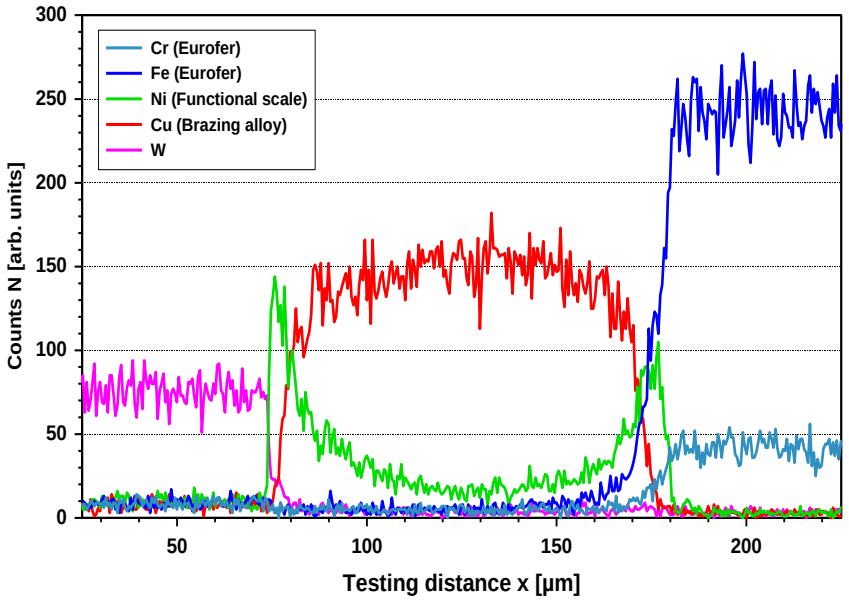
Functions:

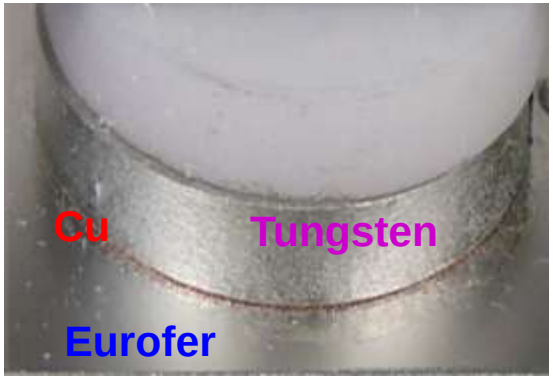
W – Ni interlayer as alloying support

Ni – Eurofer surface activation

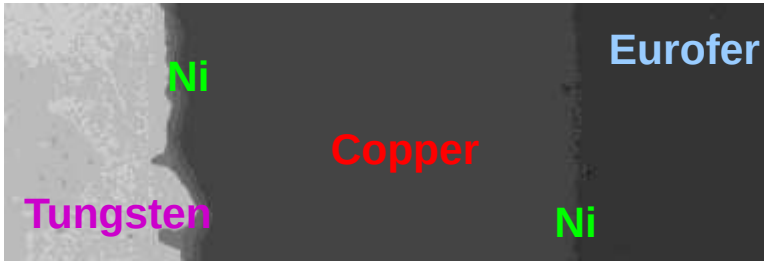
Cu filler metal

Brazing by Ni – Cu alloying



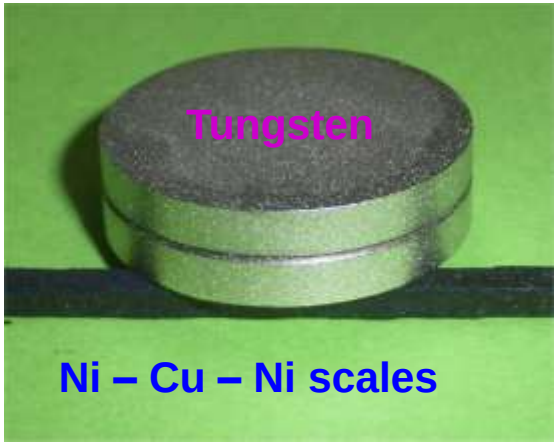


Brazed at 1100°C

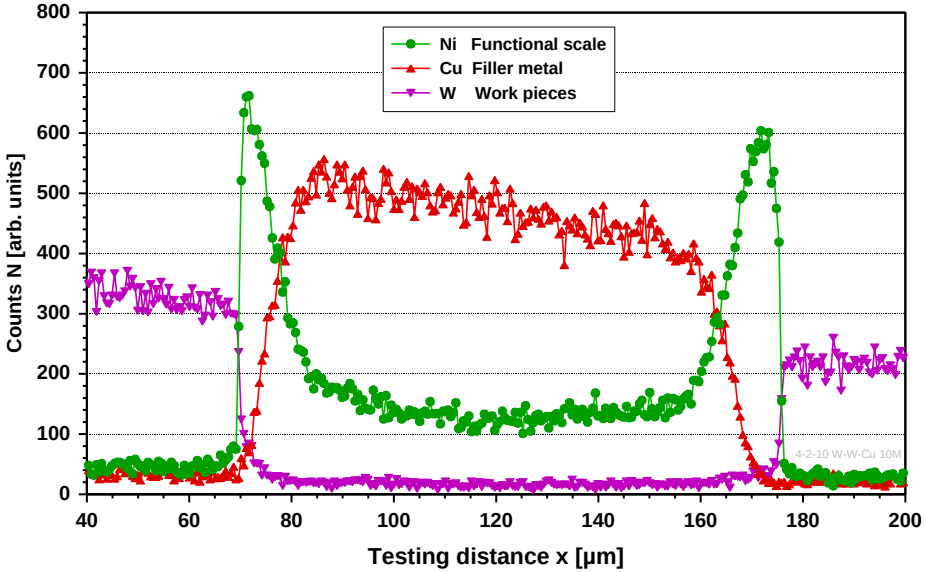


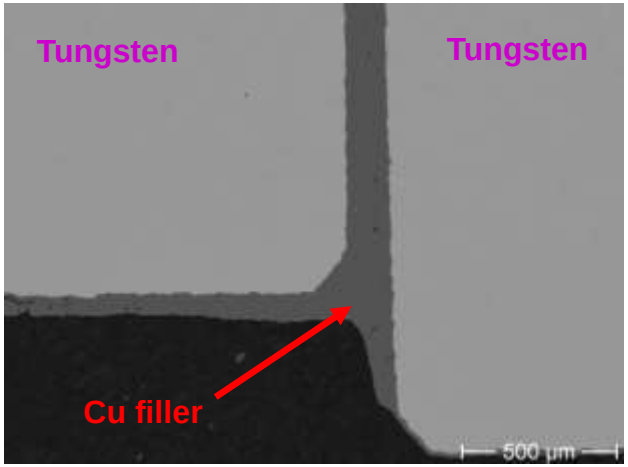
W – steel joint

W – W brazing with Ni and Cu coatings



Brazed at 1100°C

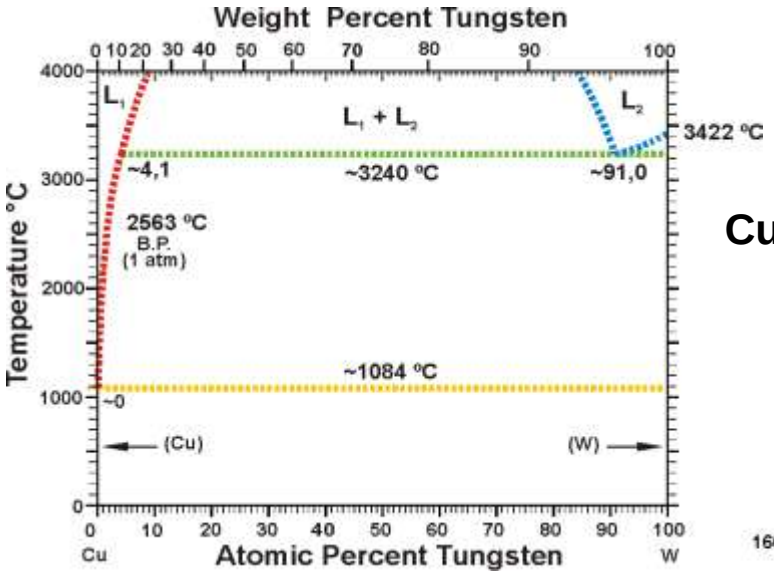




W – W joint

Coatings from aqueous systems

Joining of W-W by Cu and Pd



No miscibility

Direct reactive joining of W-W

not possible by Cu

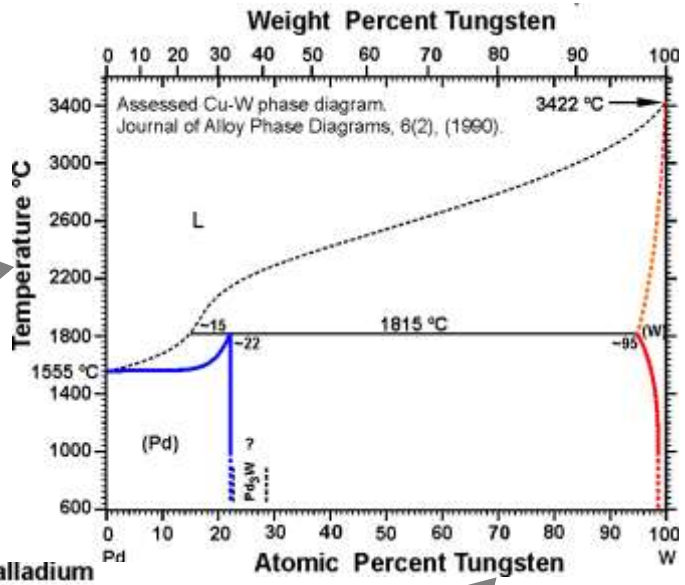
⇒Functional layer
Pd required

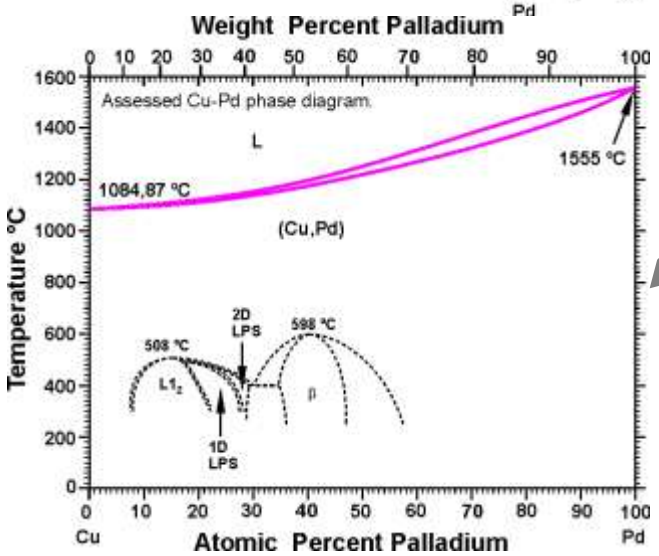
W - Pd - Cu

Cu+W

Pd-W

Pd-Cu





Cu is appropriate as filler metal due to fully miscibility with Pd

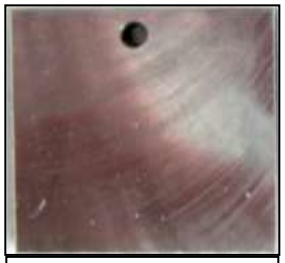
Coatings from aqueous electrolyte systems

Pd on W as reactive interlayer for Cu brazing

Electrolyte testing

Cu substrate

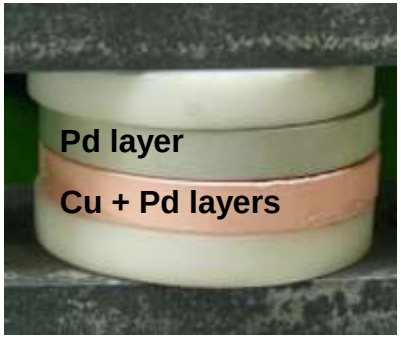
Pd deposited on Cu



W after chemical conditioning

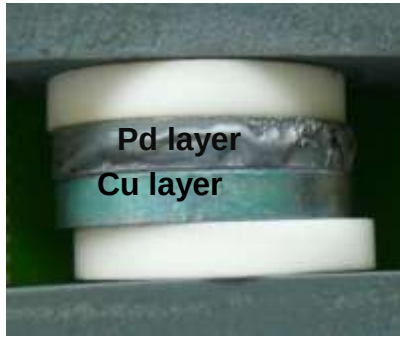
W coated by Pd + Cu

W - Pd - Cu + W - Pd



As deposited on W

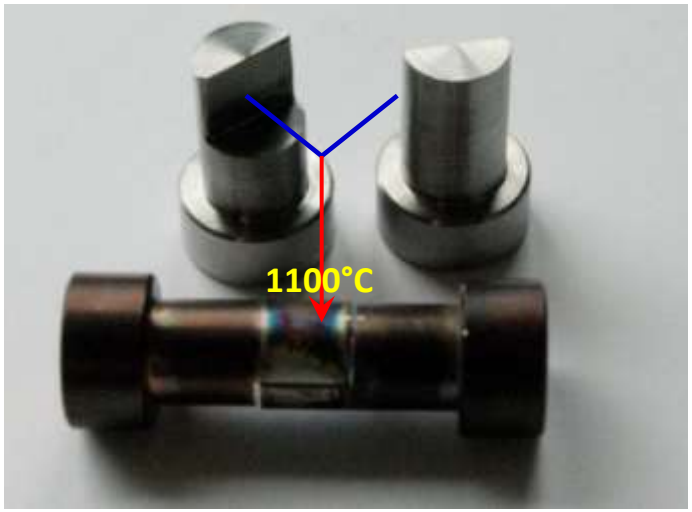
T = 1100°C, t = 10 min in Ar
+ cooling in air

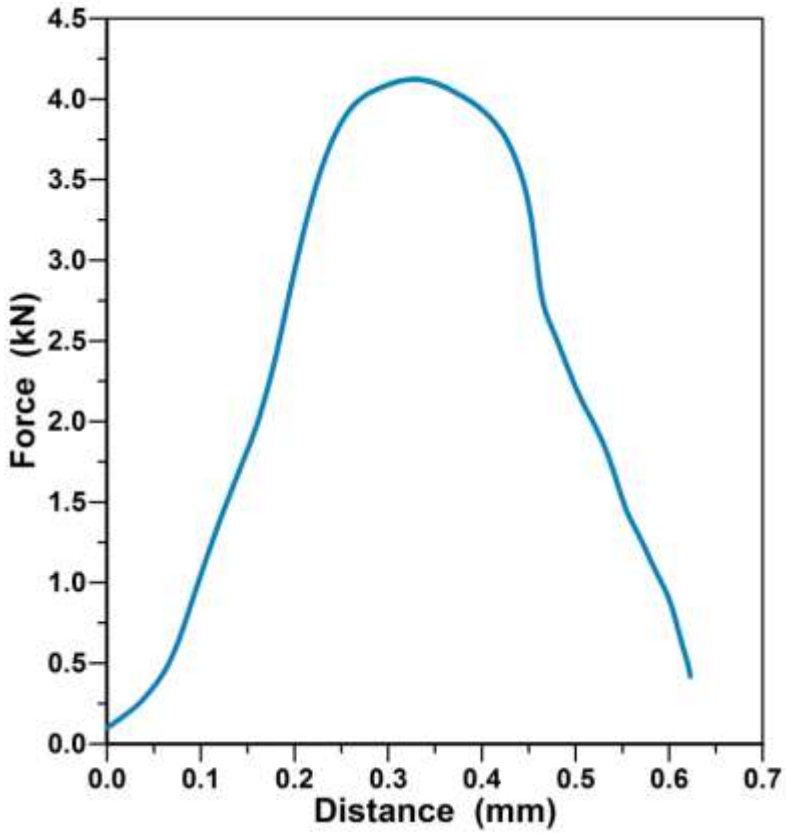


After brazing

Mechanical characterization of joints from aqueous electrolytes

Shearing strength tests of brazed specimen





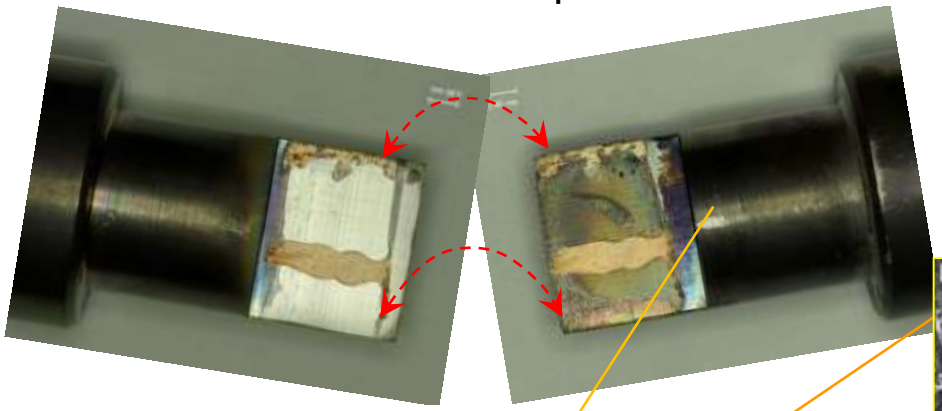
First shear tests show applicability of test method, but further development and optimization is needed

Mechanical characterization of joints from aqueous electrolytes

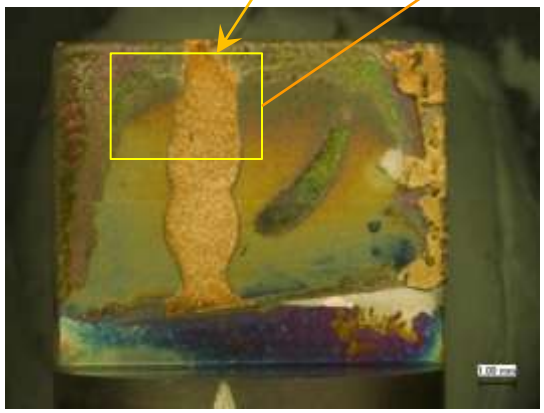
Shearing strength tests of brazed specimen

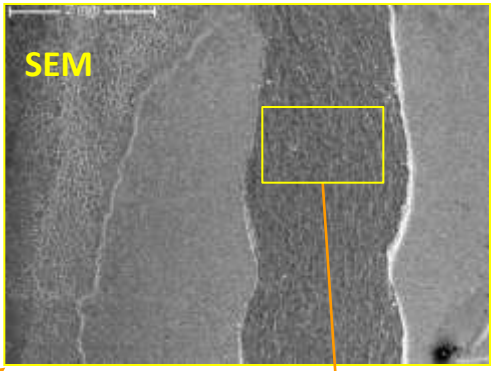
<u>Planned:</u> Eurofer-Pd-Cu-Pd-Eurofer

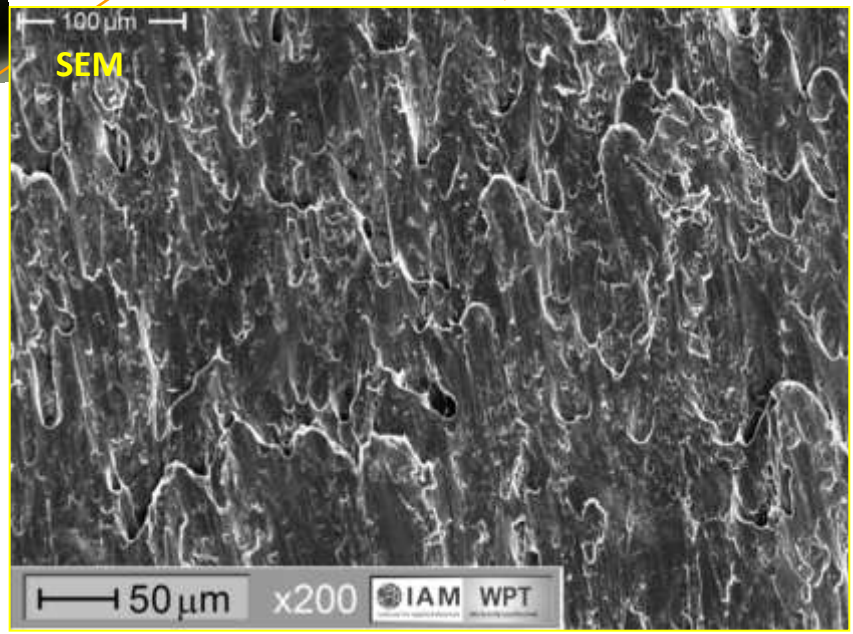
Preliminary specimen as model system steel-Ni-Cu-Ni-steel for test qualification



Specimen after test, optical overview







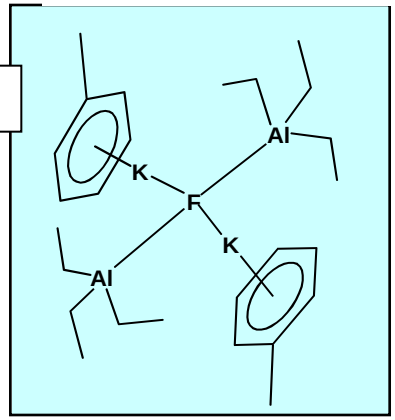
Brazing material breaks amongst the brazed Cu bulk

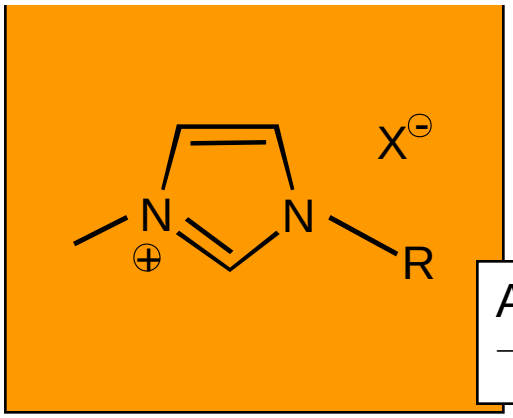
Deposition from aprotic systems

Properties of organic aprotic electrolyte systems

Solvens	Toluol, Xylol Diisopropylether	Quarternay Amin salts e.g. Ethylimidazolium chloride
Ionic solubility of solvens	no	yes
Al-carrier system	$KF{\cdot}2\,Al(R)_3$ $R = C_nH_{2n+1}$ mit n = 2-6	$AlCl_3$ **WCl_6**
Temperatures	100°C	RT ... 200°C
Reactivity - Water	extremly high	modest
Reactivity - Air	extremly high	low
Reactivity - Temperature	modest	stable up to 300°C
Toxicology / biodegrability	aromates: ++/---	amines: -/+
Max. conductivity [mS/cm]	19,5	22

Only Al

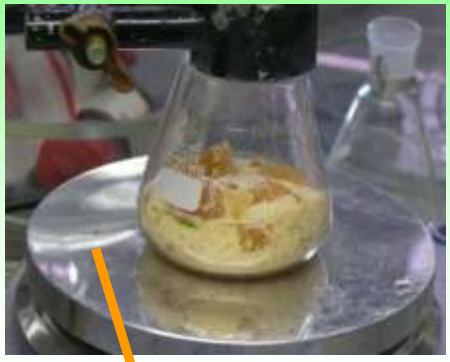




Al^{3+} + 3 Cl^- or W^{6+} + 6Cl^-
→ EMIM-$AlCl_4$ or EMIM-WCl_6

Ionic liquids as electrolyte for W deposition
- Ethylmethylimidazolium-chloride + WCl_6

EMIM-Cl, RT



IL melting

EMIM-Cl, 80°C

Mixing
IL + salt

EMIM-Cl + Salt

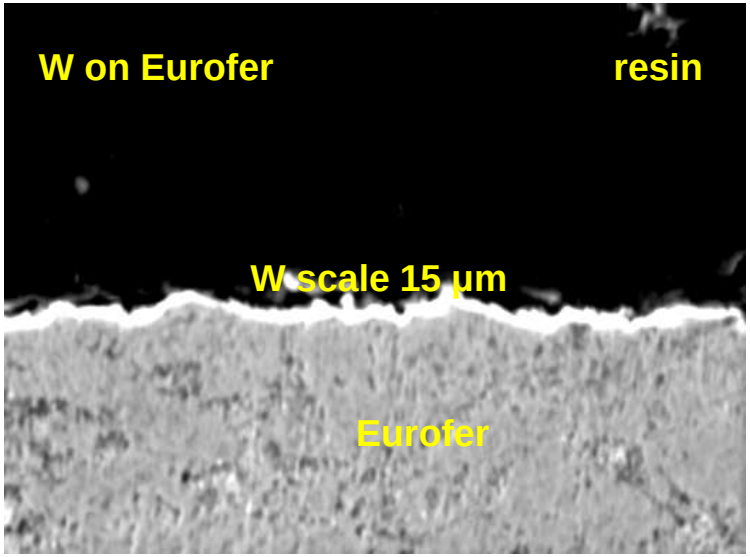
Deposition

EMIM-Cl + Salt + DC

Scale

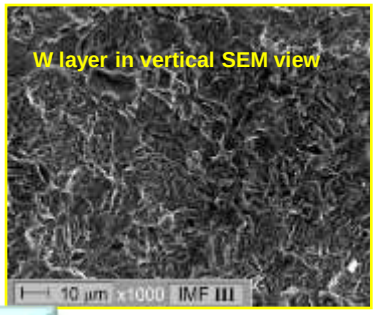
Deposited metal

Coatings from organic electrolytes (IL)



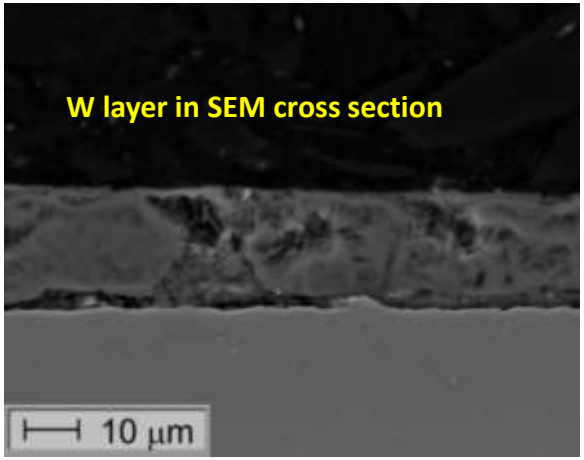
Tungsten layer on Eurofer steel

Deposited at 120°C
Electrolyte (IL) EMIN-Cl + WCl_6



Development needs for W, Ta ..

- Evaluation of solvent / salt compatibility
- Analyses of current modes
- Analyses of electrolyte composition
- Adhesion
- Morphology



Conclusions

Electro-Chemical Machining (ECM) of tungsten

- ECM is a tool to machine W successfully without chemical passivation
- Quality improvement realized by short DC pulses and gap control
- Demonstrators fabricated by C-ECM with 10 kHz pulse generator

Electro-chemical deposition for joining and functional scales

Aqueous systems:

- Successfully performed W – W and W-steel joining by deposited Cu, Ni
- Multilayer deposition on W parts under execution to increase operation temperature
- Substitution of model metal Ni successfully demonstrated by Pd plating
- Mechanical shear tests showed well adherence of filler at bulk

Organic systems:

- IL systems applicable for deposition of refractory metals