



Metso MeChanICs Mid-term Review 27.9.2012

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Background

Personal info and Metso Materials Technology

- Janne Nurminen, Born 1969
 - Engineer 1996 (Masters in materials science)
 - PhD 2008 (Laser cladding technology)
 - At Metso from 2.1.2008
- **1980's**: Rauma-Repola's materials technology unit at the Lokomo steel factory
 - MIR I&II submersibles built and delivered by Rauma Oceanics
- **1993**: Rauma Materials Technology Oy
- **1999**: Rauma/Valmet ->Metso Oyj
- **2000**: Metso Powdermet Oy
- **2007**: Metso Materials Technology
- **2011**: Engineered Materials and Components together with Lokomo Foundry
- **2012**: Metso Materials Technology disbanded



Secondment plan

- WP1:

- Study of influence of HIP diffusion bonding process on grain size and mechanical properties of OFE copper and the resulting surface roughness on diamond turned discs.

- WP2:

- Work on innovative possibilities for enhancement of joining through HIP diffusion bonding process and study roughness requirements for ensuring good bonding quality including potential half-notch reduction (outer wall - iris wall).

- WP3:

- Study of feasibility of HIPing a CLIC AS disc from powder.
 - Characterization of chemical, mechanical, electrical and microstructural properties of HIPed material.
 - Study of possible encapsulations for near-net-shape HIPing

- WP4:

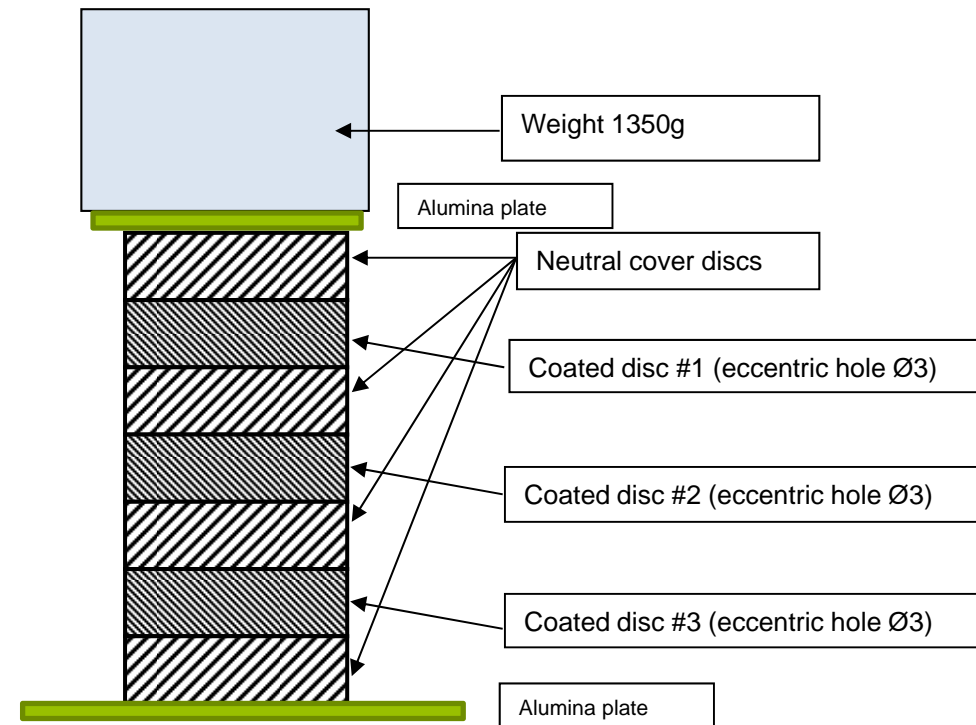
- Study of brazing of parts pre-coated with enabling layers.

Introduction to WP4

- Problem to be solved: geometrically difficult joint prevents the use of Cu-Au foil normally used for brazing
- Reference to prior tests EDMS 1141453 and 1108977
- The normal foil thickness used is approx. 55 μm which is the target brazing material thickness before the heat cycle
- The choice of deposition method was electrodeposition that can provide this thickness feasibly but:
 - Only pure metals are easily deposited which means individual Cu- and Au-layers to form the brazing alloy
 - Diffusion is needed to bring the brazing material to melting range
 - Gold diffuses very fast into copper and, hence, a nickel layer was deposited to prevent gold diffusion out from the joint area.

Sample setup

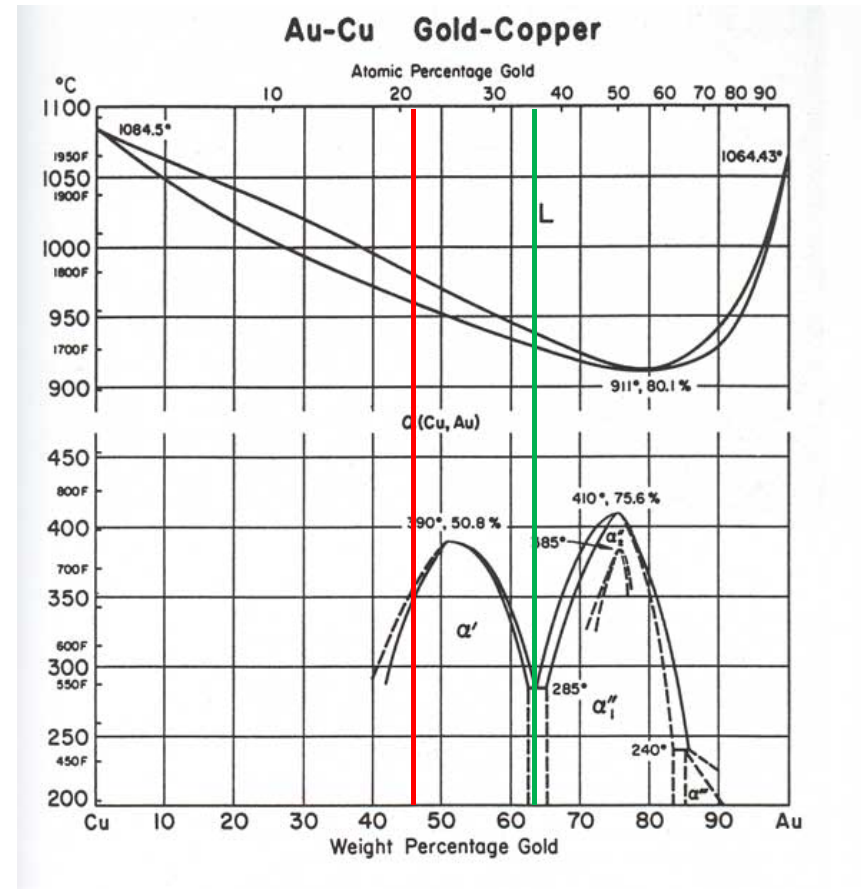
- Three layered copper discs and four neutral cover discs were cleaned, pickled and stacked in the high vacuum furnace Torvac at VTT, Finland.
- The dimensions of the discs were $\text{Ø}40 \times 15\text{mm}$.
- The composition is 63 wt. % gold if only the coatings are calculated
- The composition is 46 wt. % gold if the same $25\mu\text{m}$ of Cu is calculated from the other joint wall (good initial contact and diffusion between the walls).



Brazing material

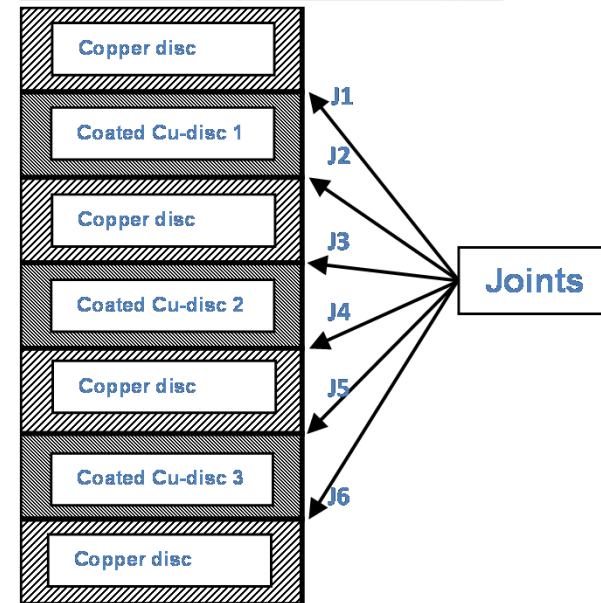
- The **composition** is 63 wt. % gold if only the coatings are calculated
- The **composition** is 46 wt. % gold if the same 25 μ m of Cu is calculated from the other joint wall.
- Electrodepositions were done in CERN.

	Set 1	Set 2	Set 3
Ni diffusion barrier	10	10	10
0 th Cu layer	25	20	17.5
1 st regular Au layer	20	10	5
1 st regular Cu layer		5	2.5
2 nd regular Au layer		10	5
2 nd regular Cu layer			2.5
3 rd regular Au layer			5
3 rd regular Cu layer			2.5
4 th regular Au layer			5
Total thickness	55	55	55



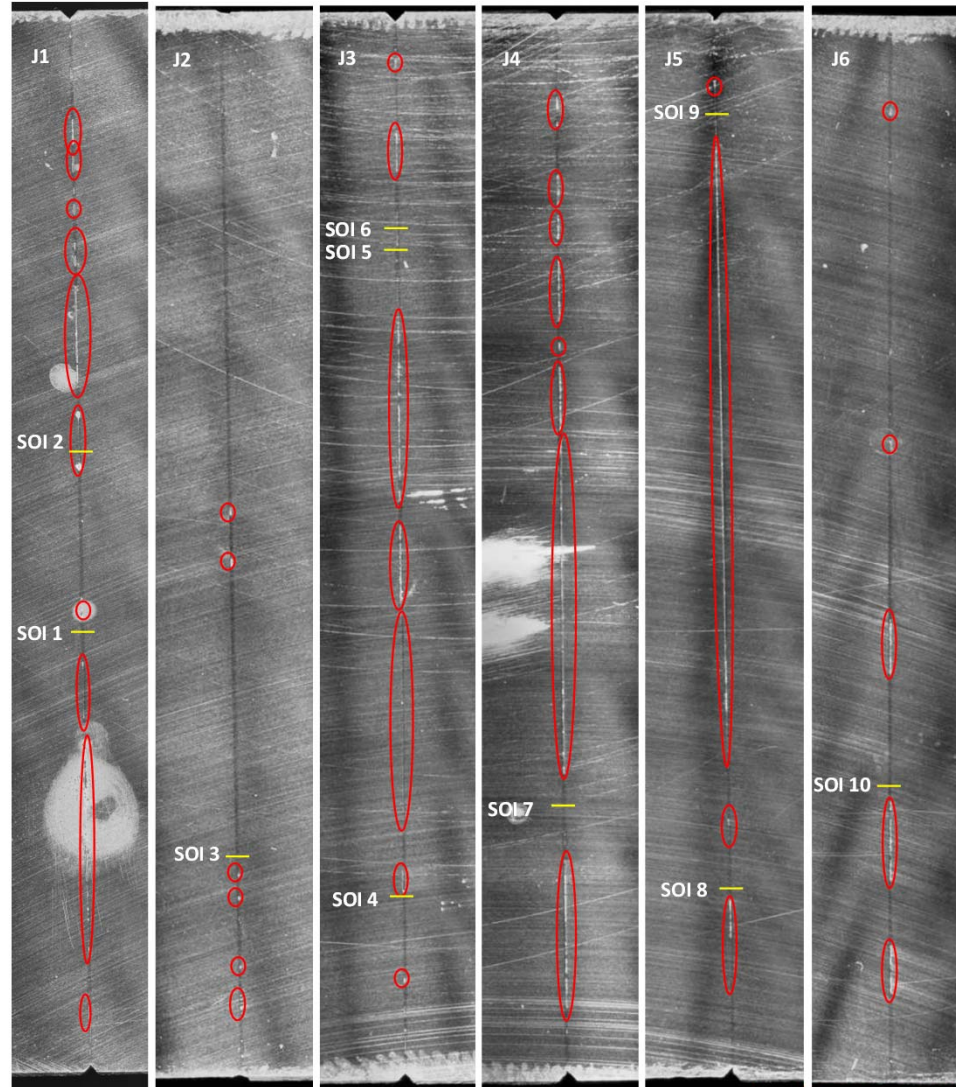
Brazing program

- Ramp 20 - 950°C at the rate of 20°C/min
- Hold one hour
- Ramp to 1000°C at 100°C/h
- Hold 10 minutes
- Cooling free with furnace
- The vacuum level in the furnace was $2 \times 10^{-4} - 7 \times 10^{-5}$ mbar
- The alumina plates under the stack and steel weights were broken during the heat cycle.
- Investigations:
 - The stack was cut longitudinally by wire electroerosion.
 - Light and SEM-microscopy to study the brazing quality
 - EDS-analysis to investigate the diffusion behaviour of brazing materials



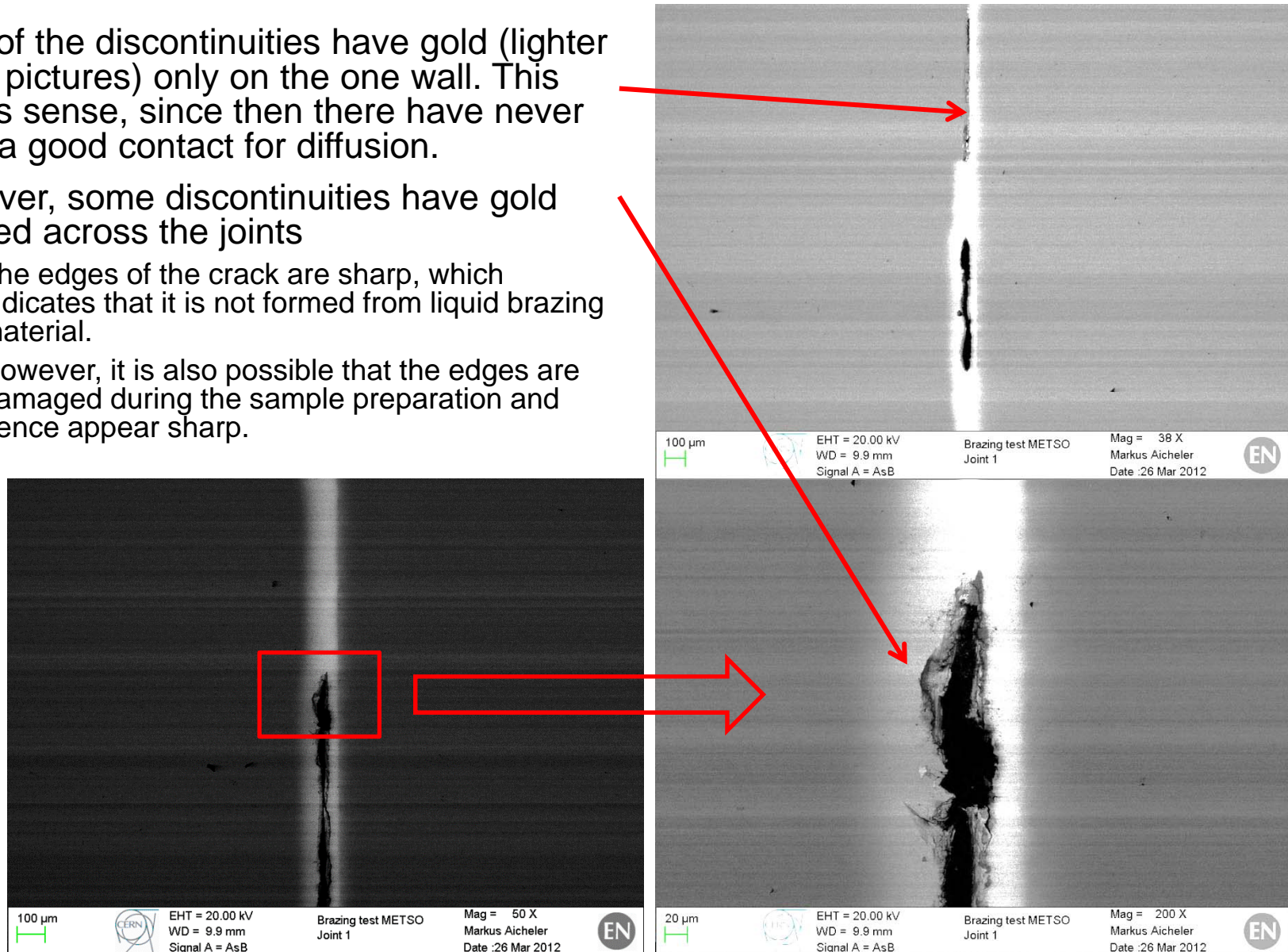
Results

- Joints 1-6 are shown
- Red markings show the places with (gaps)
- SOI 1-10 are sites of interest where EDS-analysis were done



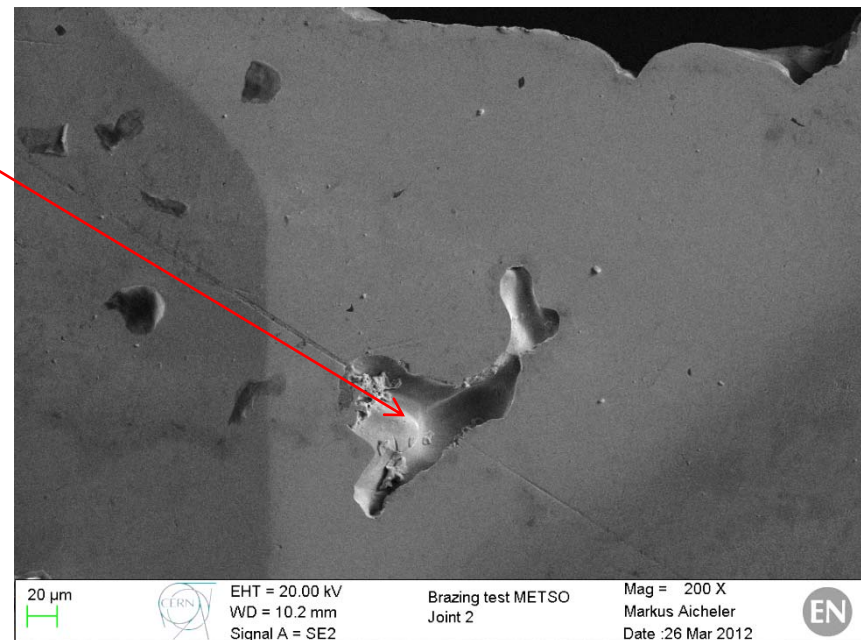
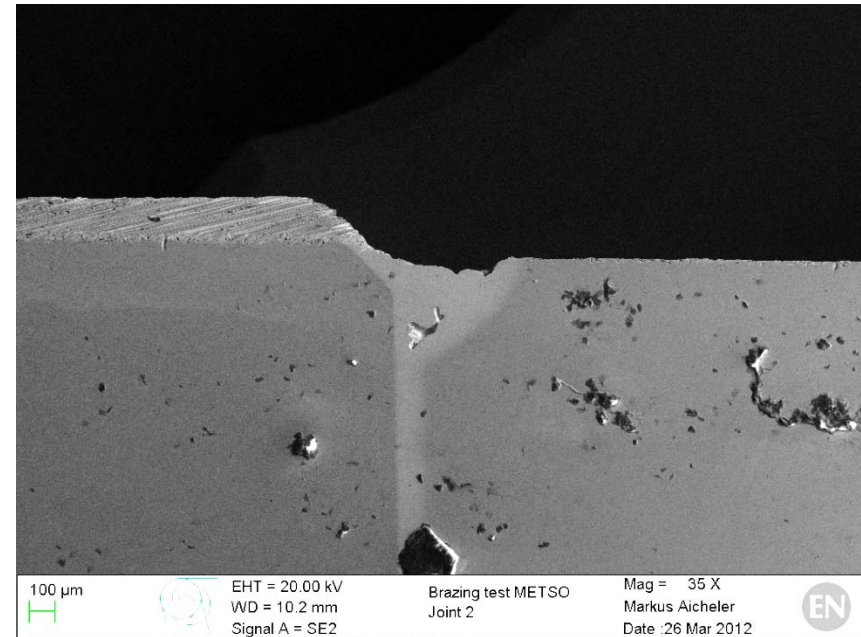
Diffusion around the discontinuities

- Most of the discontinuities have gold (lighter in the pictures) only on the one wall. This makes sense, since then there have never been a good contact for diffusion.
- However, some discontinuities have gold diffused across the joints
 - The edges of the crack are sharp, which indicates that it is not formed from liquid brazing material.
 - However, it is also possible that the edges are damaged during the sample preparation and hence appear sharp.

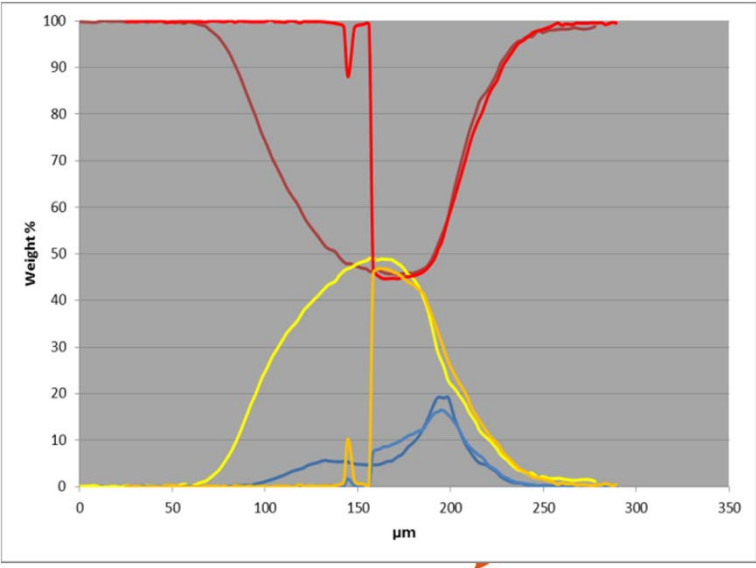
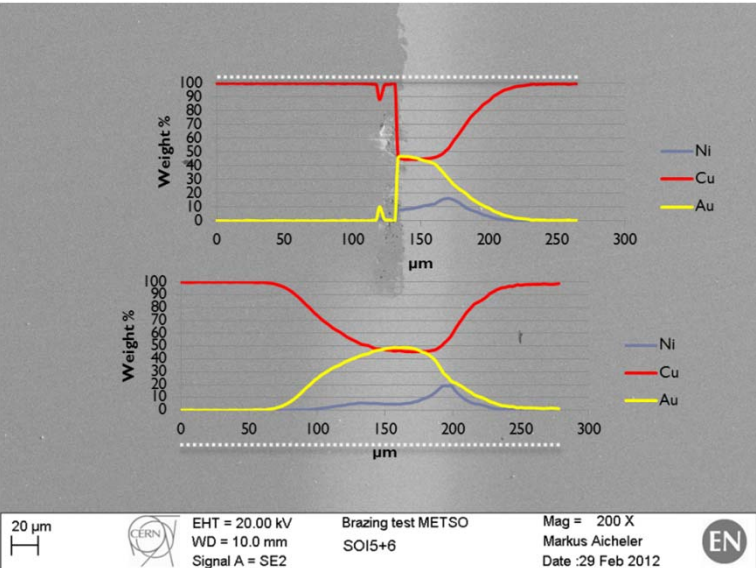
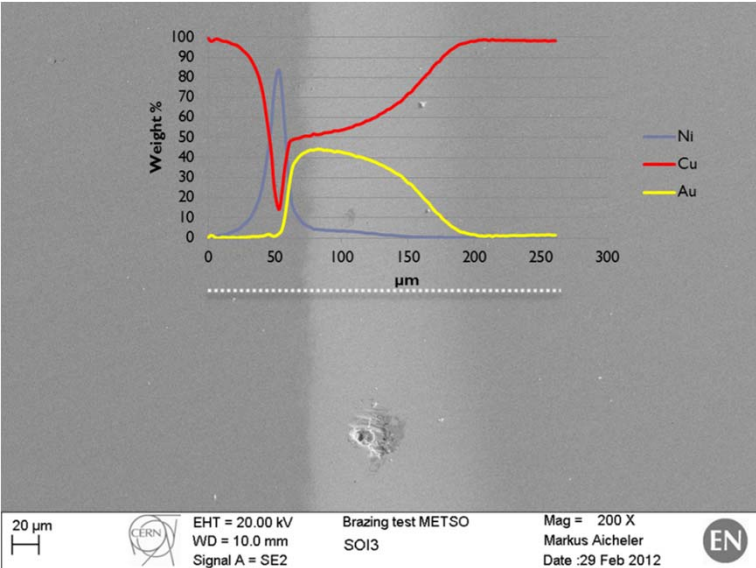
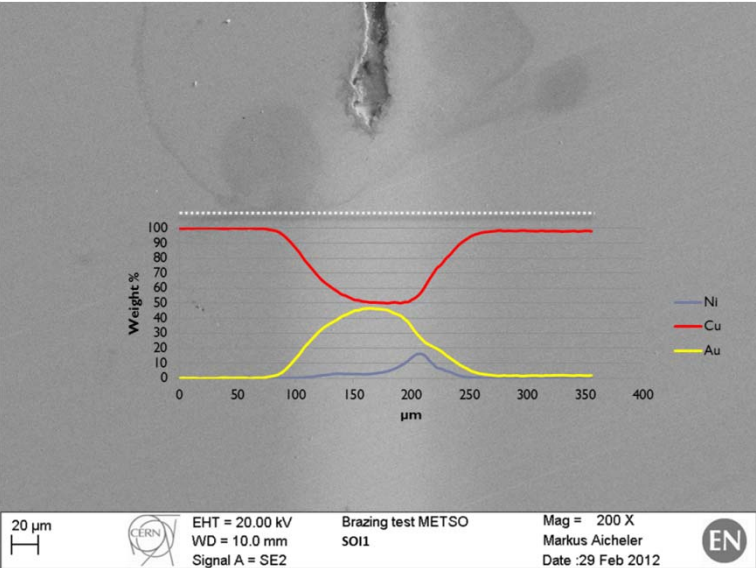


Liquid phases exist?

- Considering whether there never been any liquid brazing material:
 - in the joints 2 and 6 the chamfers are filled with once molten brazing alloy.
 - In joint 2 there were also a shrinkage cavity found confirming this
- Those are also the best joints indicating that it is possible that the bad joints failed simple because there were not enough brazing material for the joints

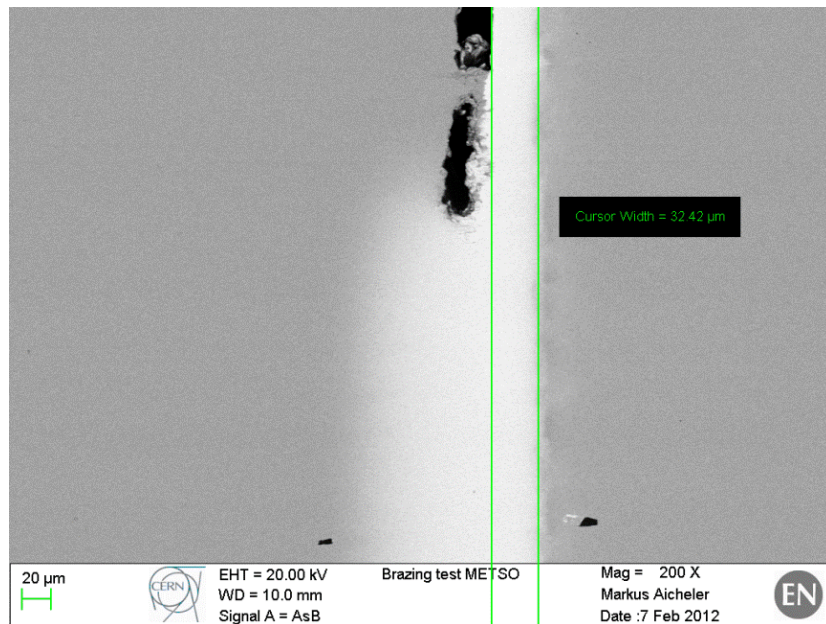


EDS-scans

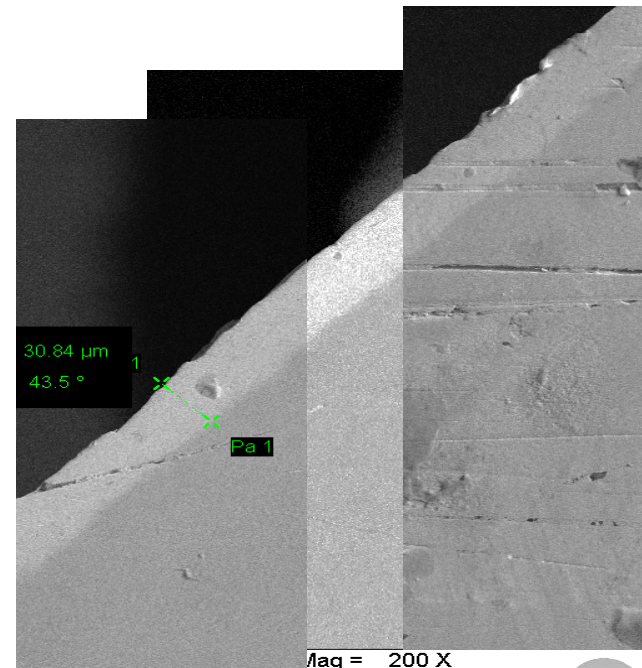


Thickness of the coatings

- Thickness of the coating system was measured by SEM.
 - Thickness of the coatings (diffused gold, no Ni-layer) is around 30 μm .
- Compiled from 3 images of coated discs 1, 2 and 3 gives perfect match on coating thickness of all set-ups. (measured from the outer rim of the sample).



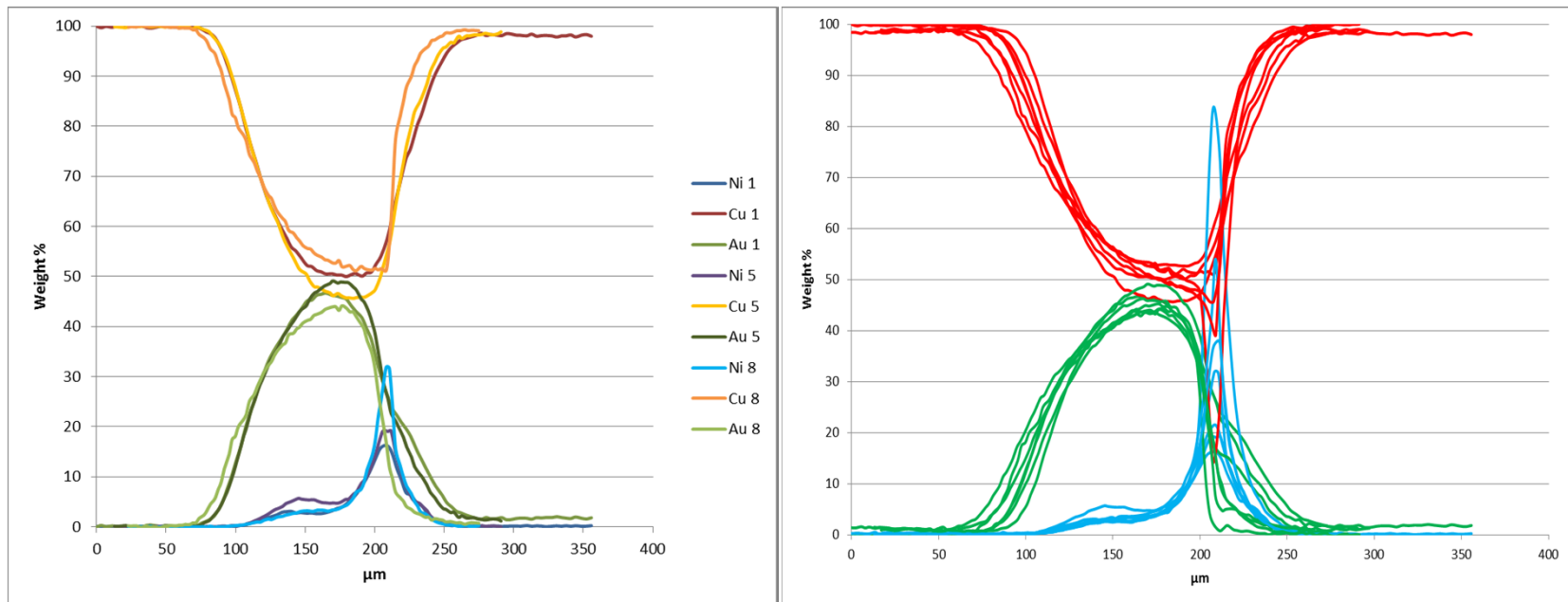
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Comparisons J1, J5, J8 and all together

- 3 scans from 3 configurations (coatings) were put in one graph (left) to see whether there is differences between diffusion of the coatings
- All well diffused measurements were put to one graph (right) to show that the differences in Au-Cu distributions are minor in spite of large differences in deposited Ni-layer.



Findings on diffusion system

- If one makes simple calculations considering diffusion length of Au in Cu and/or Cu in Au it is found that in 950°C the mean diffusion length for one hour is:
 - 42 µms for Au in Cu
 - 98 µms for Cu in Au
 - This is more than enough for even up the composition in coating layers
- During temperature levelling, part of the coating system should liquefy and form a contact to the other joint wall
 - After the joint is closed then there is no need to hold the temperature at 950°C and it can make things worse
 - Probably we still need the 1000°C peak temperature to make the brazing material to melt again and flow to fill the possible gaps.
- It would be more feasible to choose parameters that produce only one melting step
- Gold diffused and mixed with copper as expected and formed a brazing alloy – Au-distribution width was 100...150 µm
- The resulting diffusion of gold was similar with all coating systems
- The peak gold content in brazed joints were 45-50 wt. % resulting in melting point about 30...40°C lower than the peak heat treatment temperature of 1000°C, 10 min
 - With the final gold distribution that would mean about 50 µm of melted material (Au > 40 wt. %)
- The measured Au-Cu layer at the outer wall of the discs and at the non-diffused areas were around 30µm

Conclutions and next steps

- It can not be concluded definitely if electrodeposited nickel/gold/copper system can produce metallurgically sound joints with used coating thicknesses:
 - There were good joint areas and bad ones
 - Treatment temperature program, surface quality need to be optimised to produce sound joints
- This study did not reveal any benefits of depositing Au/Cu in thinner layers
- The effect of Ni layer could be clearly seen by EDS-scans.
- Some liquid phase has been formed during the brazing cycle
- Hyphotesis is that the joining can succeed with thicker electrodeposited layers
- New brazing tests are planned

Personal experiences during the project

- Professional:

- There were great deal of subtle things to learn and get perspective: working methods, management, scientific equipment, databases etc.
- French, although 6 months was not enough.
- New connections and international experience

- Every day life:

- Bureaucracy considering apartments, bank accounts, insurances etc. are challenging
- Weather, food, environment and recreational possibilities are excellent.
- Very nice people far from home that are willing to make new frinds!

- Future impact:

- Due to disclosure of MMT, there is probably no benefit from my internship when working in Metso
- Considering career outside of Metso – there would possibly be some benefit from the internship if applying a job on research and development.