

A study on production of bimetallic raw materials for the CLIC accelerating structures. Status report.

Why bimetallic raw material?

Materials foreseen

Techniques and prototypes

Cast copper

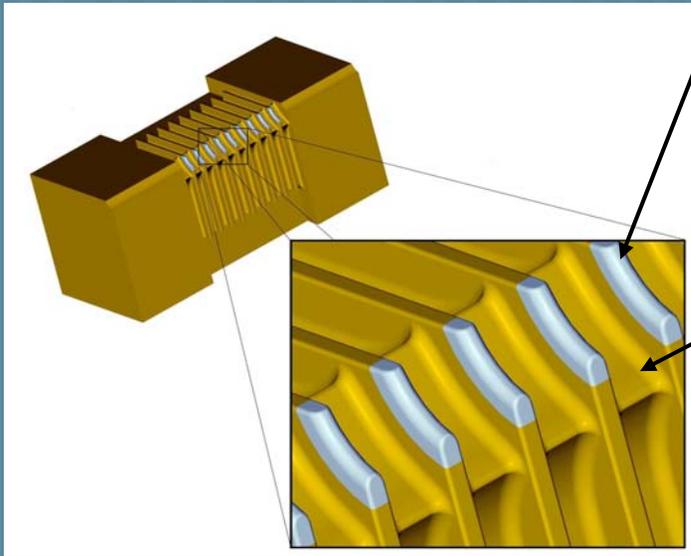
Coextrusion

HIP diffusion bonding

Explosion bonding

Perspectives

Why bimetallic raw material



- Regions of high electric field:
 - breakdown and field emission
 - pure Mo (or alternative refractory & low P_{vap} alloy)
- Regions of high pulsed currents:
 - Cyclic thermal stresses, possible fatigue damage at the surface
 - CuZr alloy UNS C15000 (or alternative copper alloy with best match of conductivity and fatigue resistance)
- Tiny inserts and extreme geometrical tolerances required → machine from raw bimetallic

Remind on materials foreseen

- Pure Molybdenum
 - Possible fragilisation if recrystallised (recrystallisation may occur at temperatures above 900 °C depending on time exposure and previous cold work)
- CuZr, UNS C15000
 - Solution annealing at 900 °C to 925 °C, followed of fast cooling
 - Artificial ageing 450 °C to 550 °C
 - The best (mechanical strength) temper states are obtained by addition of cold work before or/and after ageing.

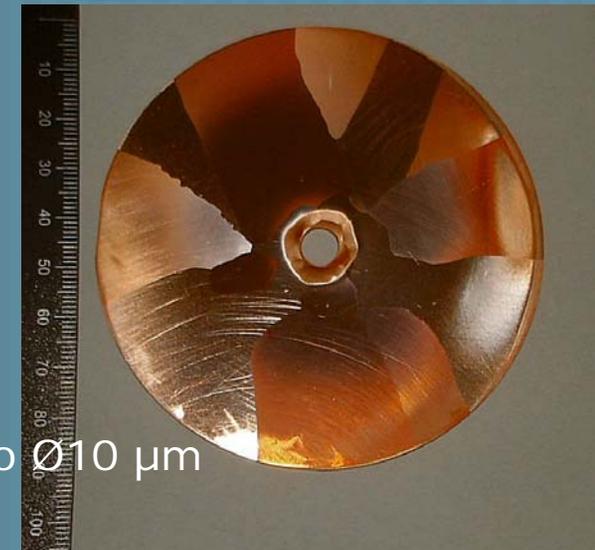
Co-extrusion (Lutch /RU)

- Co-extrusion with interlayer: Cu or CuZr/X/Mo
- Conditions
 - Inside evacuated stainless steel can
 - $< 600\text{ }^{\circ}\text{C}$
 - 75% Mo section reduction
- Preliminary sample of Cu/Ni/Cu produced
- Results of shear test are unsatisfactory
 - ✗ Shear strength of only 27 MPa
 - ✗ Delamination at the Cu/Ni and Ni/Mo interfaces



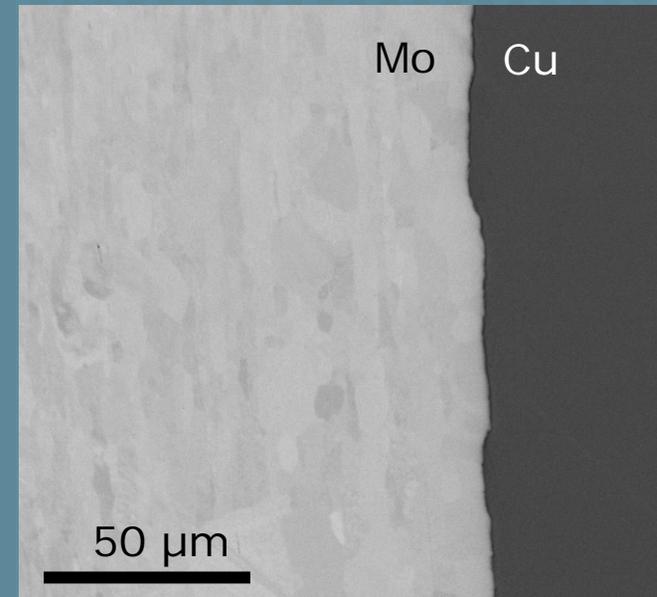
Vacuum casting (H.C. Starck /DE)

- Vacuum casting of Cu-OF (or CuZr?) around a solid Mo insert.
- Preliminary sample Cu-Mo: \varnothing 90 mm x 300 mm with \varnothing 8 mm Mo insert.
- Results partially satisfactory:
 - Interface
 - ✓ Free of defects
 - ✓ Depth of Cu diffusion inside Mo $14 \mu\text{m}$
 - ✓ Shear strength $180 \pm 13 \text{ MPa}$
 - ✓ 40 % shear in CuZr, 60 % debonding
 - ✓ Pull strength $123 \pm 10 \text{ MPa}$
 - ✓ Fracture on the Cu side
 - Cu matrix
 - ✗ macro grains, one grain along the thickness!
 - Low overall porosity (0.92 %), but pores up to $\varnothing 10 \mu\text{m}$



Vacuum casting

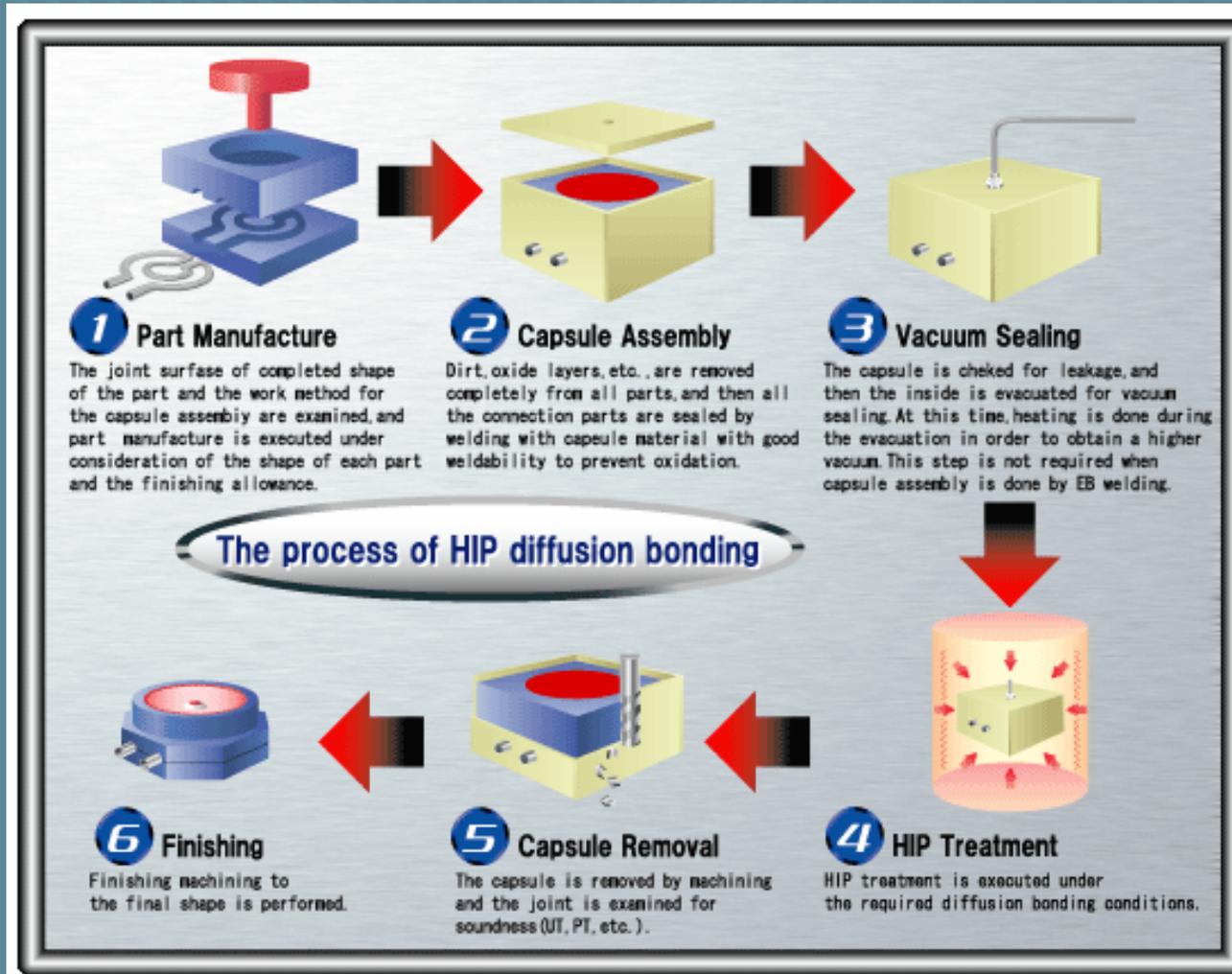
- Results partially satisfactory (Cont.):
 - Mo insert
 - ✓ From hardness and metallography:
No (major?) recrystallization



- Concerns related to the cast microstructure
 - Anisotropy of the matrix due to giant grains
 - Pores size incompatible with aimed dimensional tolerances (may be red out by post HIPing)
 - Chemical segregation is suspected to happen when casting CuZr alloy instead of pure Cu, that may impair effectiveness of thermal treatments and isotropy

HIP diffusion bonding (Metso /FI). Remind of technique.

- S.Sgobba CLIC Meetings of 5/11/2004 and 10/06/2005.
(Courtesy of METSO)



HIP diffusion bonding. Historic of prototypes

- Insert 99.97 %Mo, matrix 99.85Cu-0.15Zr UNS C15000
- 1st piece: Ø50 mm x 100 mm, insert Ø5 mm
 - From extruded CuZr bar
 - Only characterization (See CLIC meeting 10/06/2005)
- 2nd piece: Ø87 mm x 300 mm, insert Ø8.6 mm
 - From forged CuZr bar for functional diameter
 - Improved hole drilling (gun drill)
 - Improved cleanliness of interfaces before insertion of Mo bar
 - Shorter stay at HIP (6 h vs. 10 h), but lower cooling speed (0.16 °C/s vs 0.056 °C/s)
 - Portion for characterization
 - See presentations of H. Belle to the METSO-CERN meeting 8/02/2006 and EST-MME seminar 24/02/06
 - Portion for Machining test (HDS6)
 - Portion for bi-metallic HDS11 prototype



HIP diffusion bonding. Historic outcome of 1st piece

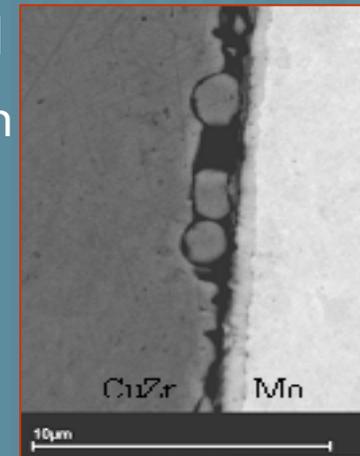
- 1st piece
- Summary and open questions (CLIC meeting 10/06/2005) :
 - “ ...
 - HIP quenching results in a temper state that requires further solution annealing to be susceptible to age
 - Ageing after further solution annealing is effective ...
 - ...
 - The interface shows (locally?) good interdiffusion and high shear strength
 - EDM resulted in a separation of the matrix and the insert (due to the process or to locally poor adhesion?) ...”

HIP diffusion bonding. 2nd piece aims

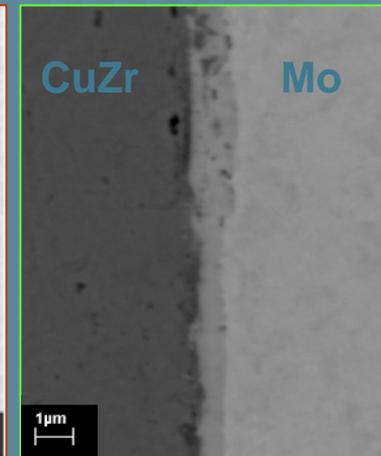
- Tests of 2nd piece aimed to:
 - Verify bond soundness (as received following HIP-quench and after thermal treatments)
 - Characterise matrix and insert with the larger geometry and shorter HIP time
 - After solution annealing, optimise ageing temperature for mechanical strength and electrical conductivity
 - Solution annealing (900 °C, 1 h + water quench)
 - Three alternative ageing T (550 °C, 500 °C and 450 °C, 3 h)
- And following the debonding due to solution annealing...
 - New campaign on the way for optimisation of alternative direct treatment after HIP-quench.

HIP diffusion bonding. 2nd piece results

- Bond microstructure as HIP-quenched
 - ☑ Some defects at the interface found on 1st piece not observed here
 - Intermetallic layer at the interface
 - Three characteristic regions (with and without precipitates) in the CuZr side
 - ☑ Diffusion of Cu into Mo along 16 μm .

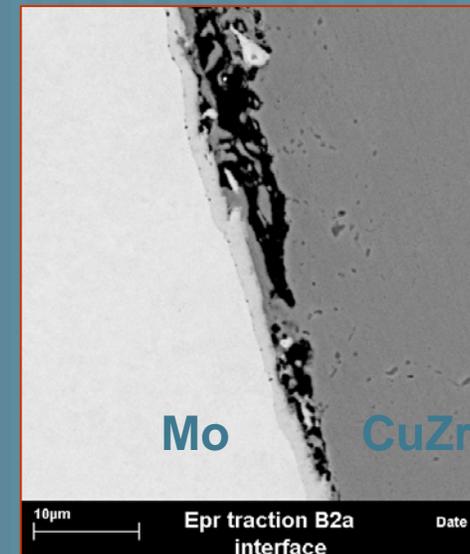


1st



2nd

- Bond microstructure after solution annealing (900 °C 1 h + water quench)
 - ☒ Some cracks



2nd after
solution
annealing

HIP diffusion bonding. 2nd piece results

- Bond strength as HIP-quenched
 - ☑ Relatively high shear and pull strength
 - ☑ 30 % shear in CuZr, 70 % debonding
 - ☑ Pull fracture partially on Mo and after yield of C
 - ☒ 1 over 4 pull specimens detached at preload
- Bond strength after solution annealing and a
 - ☒ Low shear strength
 - ☒ Systematic detachment of pull test specimens
- ➔ Quench at the end of solution annealing rises strength

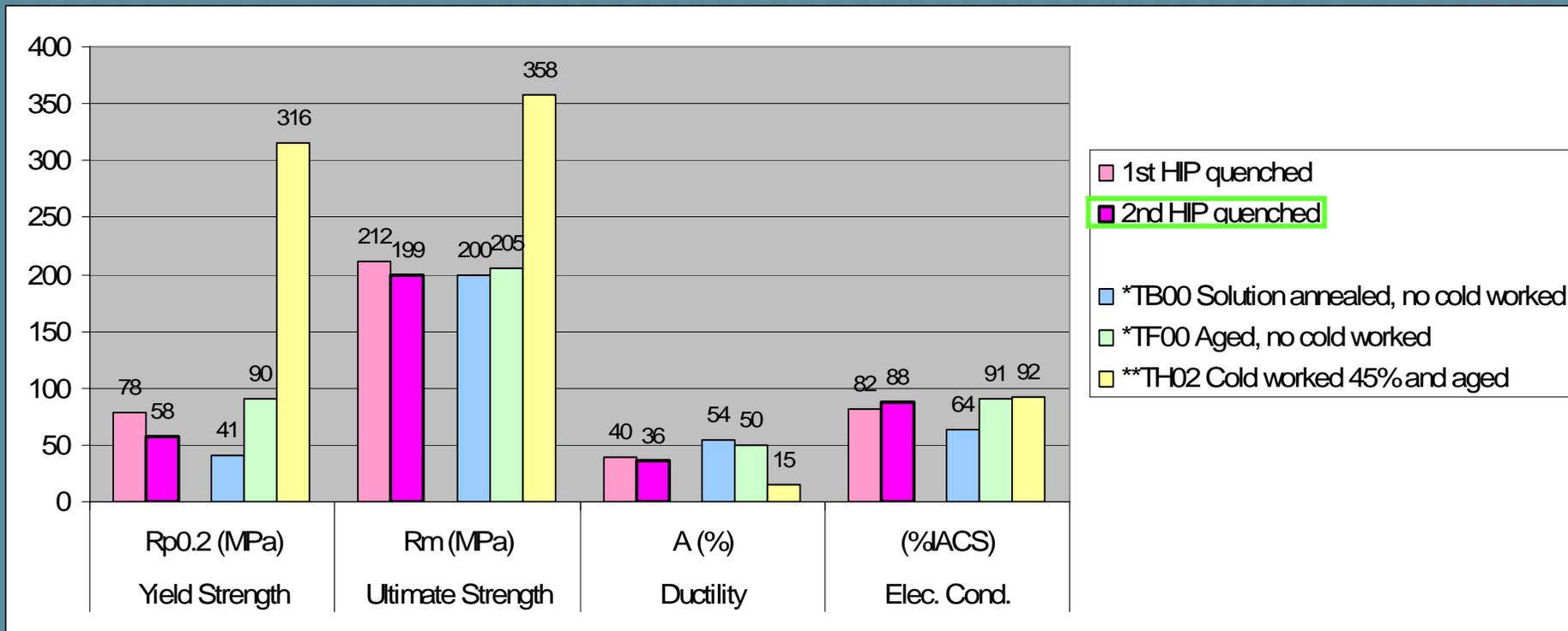


(From 900 °C to RT the free shrinkage of CuZr is about 1% higher than Mo)

| | 1 st | | | 2 nd | | | | |
|----------------------|-----------------|----------------|-----------------------------|-----------------------|----------------|----------------------|--------------|--------------|
| | HIP | HIP +Sol. Ann. | HIP +Sol. Ann. +Aged 550 °C | HIP | HIP +Sol. Ann. | HIP +Sol. Ann. +Aged | | |
| | | | | | | 550 °C | 500 °C | 450 °C |
| Shear strength (MPa) | 206 | | | 165 ±9 | - | 28 ±18 | - | - |
| Pull strength (MPa) | - | 1/1 Detached | 1/1 Detached | 175 ±18 ¼ detached | 1/1 detached | 1/1 detached | 1/1 detached | 1/1 detached |

HIP diffusion bonding. 2nd piece results

- Tensile and electrical properties of CuZr matrix as HIP-quenched compared to reference temper states of UNS C15000

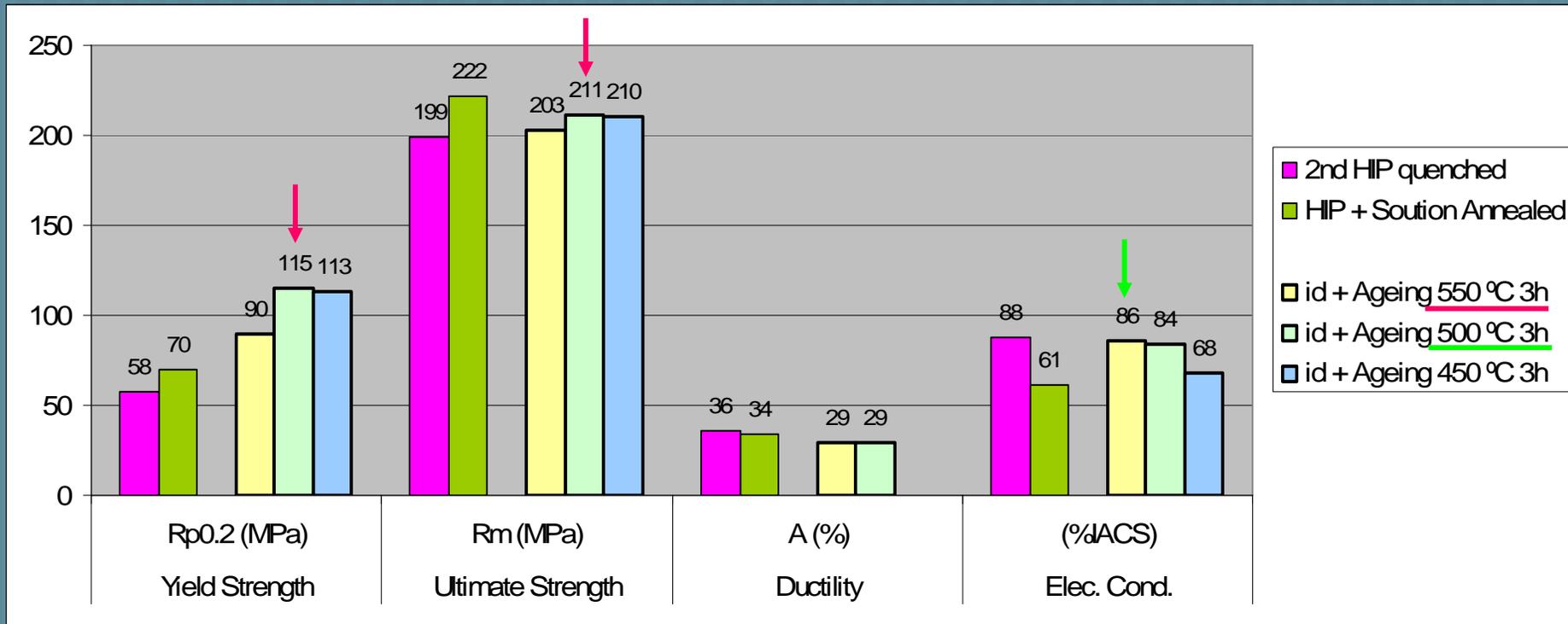


* ASM Haandbook

** Outokumpu bar used so far for fatigue tests

HIP diffusion bonding. 2nd piece results

- Solution treatment resulted in fragilisation of the interface, nevertheless optimisation study of ageing temperature yields:



HIP diffusion bonding. Conclusions on 2nd piece tests

- HIP diffusion bonding may have high bond strength but there is a doubt about homogeneity
- Bond strength is systematically weakened by water quenching after solution treatment
- An alternative treatment to improve the as HIP state (avoiding quench) should be optimized
- Higher mechanical strength than for pure copper can be obtained, but far from those obtained with temper states that include cold working.

HIP diffusion bonding. 2nd piece results

- From 1st piece tests: HIP-quench cooling is too slow to be effective as solution treatment
- From 2nd piece tests: adding a solution treatment after HIP yields to interface fragilisation.
- Based on literature and own data (see my presentation to the METSO-CERN meeting 8/02/2006), a way to slightly increase strength and electrical conductivity is a direct treatment after HIP
- A study for optimisation of direct treatment after HIP-(slow)quench is now in progress.

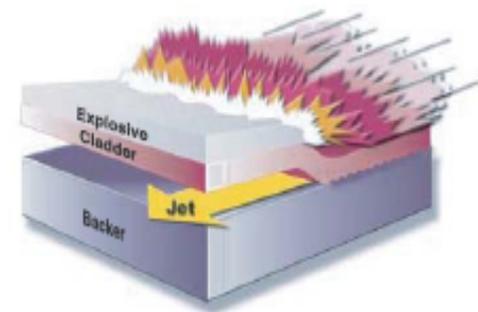
HIP diffusion bonding. Perspectives

- CERN: optimisation of direct heat treatment
- METSO:
 - Understanding and solution of the inhomogeneous adherence problem
 - Further ways of mechanical strength improvement compared to be competitive with cold worked tempers (Cold Isostatic Pressure (CIP) cycle after HIP, explosion after HIP, ...)
 - Have to formulate a technico-financial proposition to consolidate the HIP diffusion bonding way.

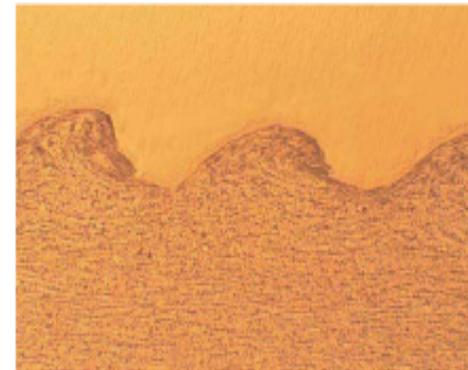
Explosion bonding. Technique

- “Solid state welding process that is used for the metallurgical joining of dissimilar metals. The process uses the forces of controlled detonations to accelerate one metal plate into another creating an atomic bond... is considered a cold-welding process which allows metals to be joined without losing their pre-bonded properties.”

1. Metals' surfaces are ground and fixtured parallel.
2. Special formulated explosive powder is placed on the cladder surface.
3. Detonation front travels uniformly across the cladder surface from the initiator.
4. Cladding metal collides with backer at a specific velocity and impact angle.
5. Momentum exchange causes a thin layer of the mating surfaces to be spalled away as a jet.
6. Jet carries spalled metal and oxides from the surfaces ahead of the collision point.
7. Thin layer of “Micro-fusion” 10^{-6} inch thick is formed at the characteristic wavy weld line.
8. Force of several million psi forces metals into intimate contact while metallurgical weld solidifies across the complete surface.
9. Speed of the explosive detonation does not allow time for bulk heating of metals.
10. Detaclad® process assures that the backer materials retain specified physical properties and the cladding material retains the specified corrosion resistance properties.



Explosion Welding



Photomicrograph of a typical explosion weld

<http://www.dynamicmaterials.com>

Explosion bonding. Historic of prototypes (Minsk /BL)

■ 1st two pieces

- Mo and Cu procured by Minsk
- Flat and grooved pieces



■ Characterisation

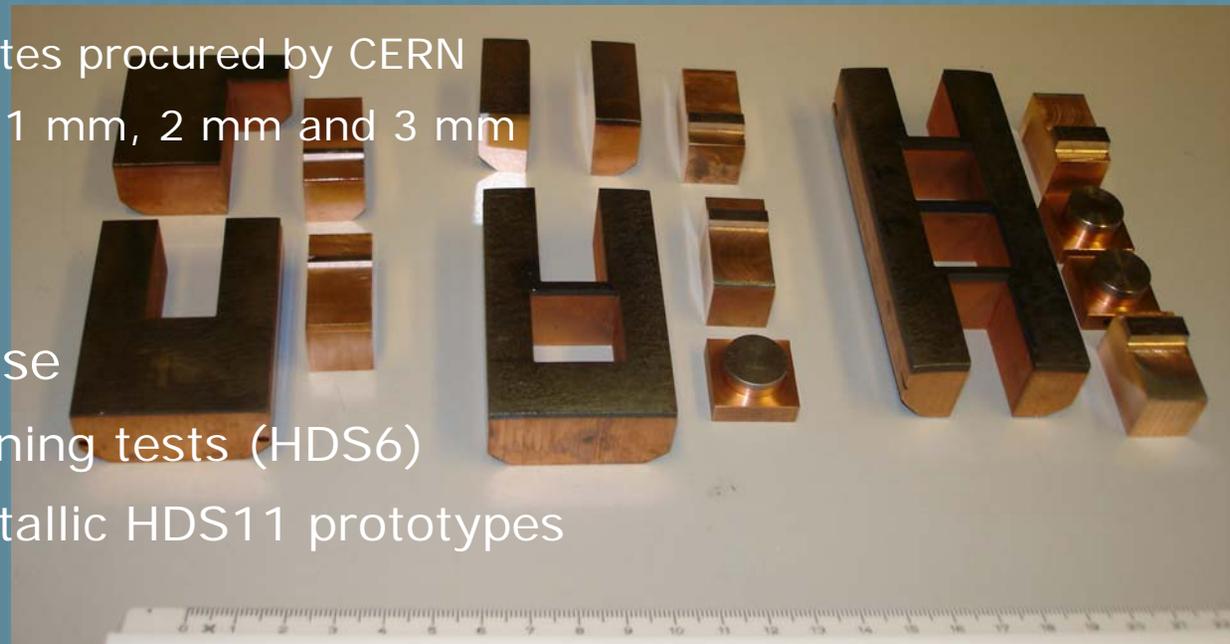
■ 2nd three+1 pieces

- Mo and CuZr plates procured by CERN
- Mo thicknesses: 1 mm, 2 mm and 3 mm
- Only flat pieces

■ Characterisation

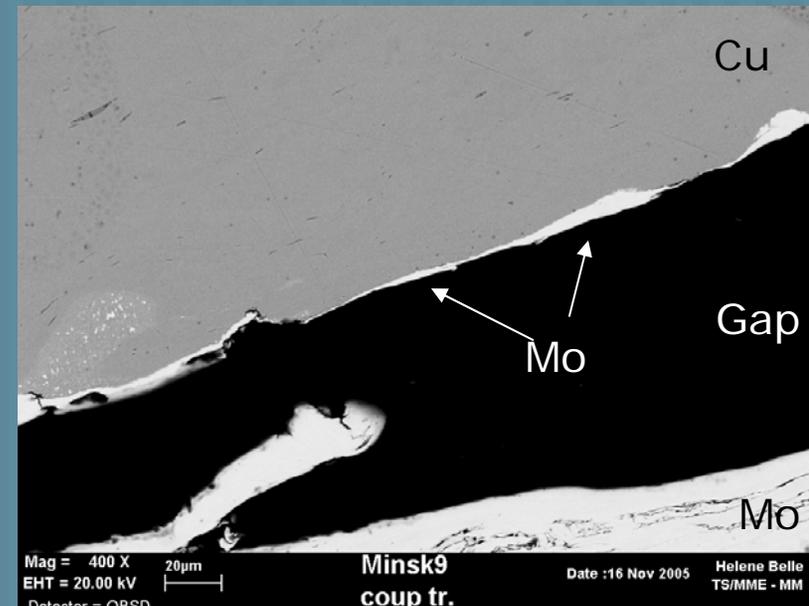
■ Proposal for 3rd phase

- Portions for Machining tests (HDS6)
- Portions for bi-metallic HDS11 prototypes



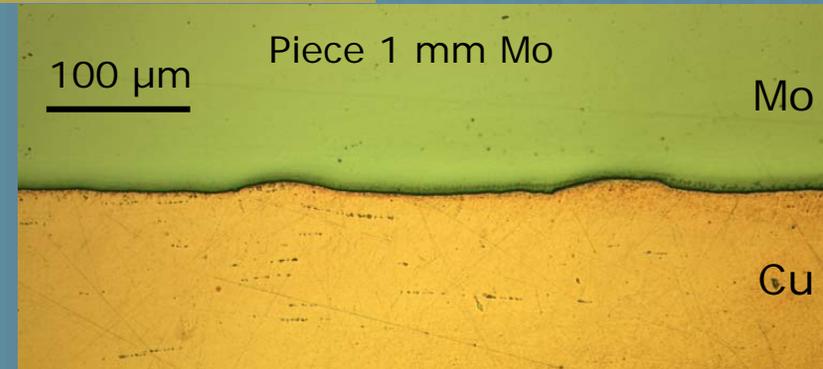
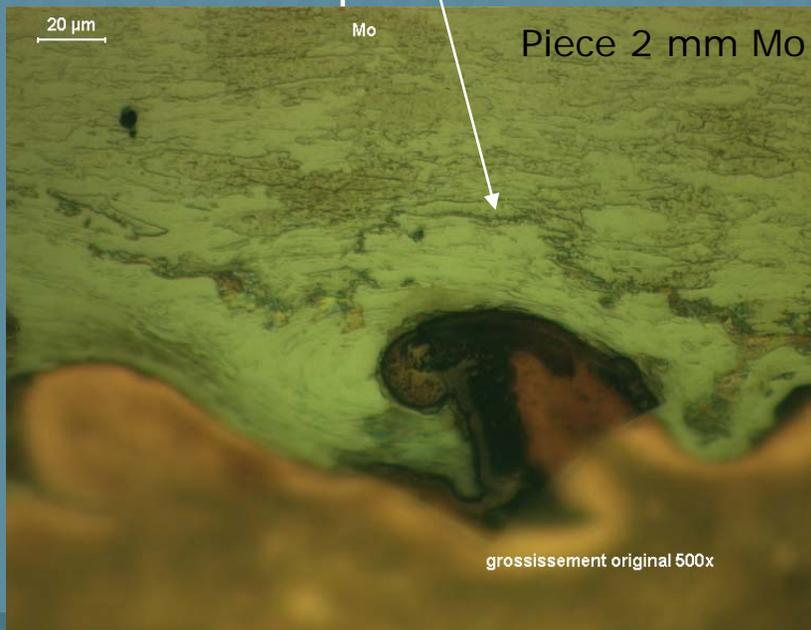
Explosion bonding. 1st pieces results

- Bond microstructure
 - Flat piece free of defects
 - Grooved piece delaminated by cracks on the Mo side
- Bond strength
 - Limited material for test
 - Shear strength >84 MPa (probably more because experimental difficulties)
 - Fracture on the Mo side, no debonding.



Explosion bonding. 2nd pieces results

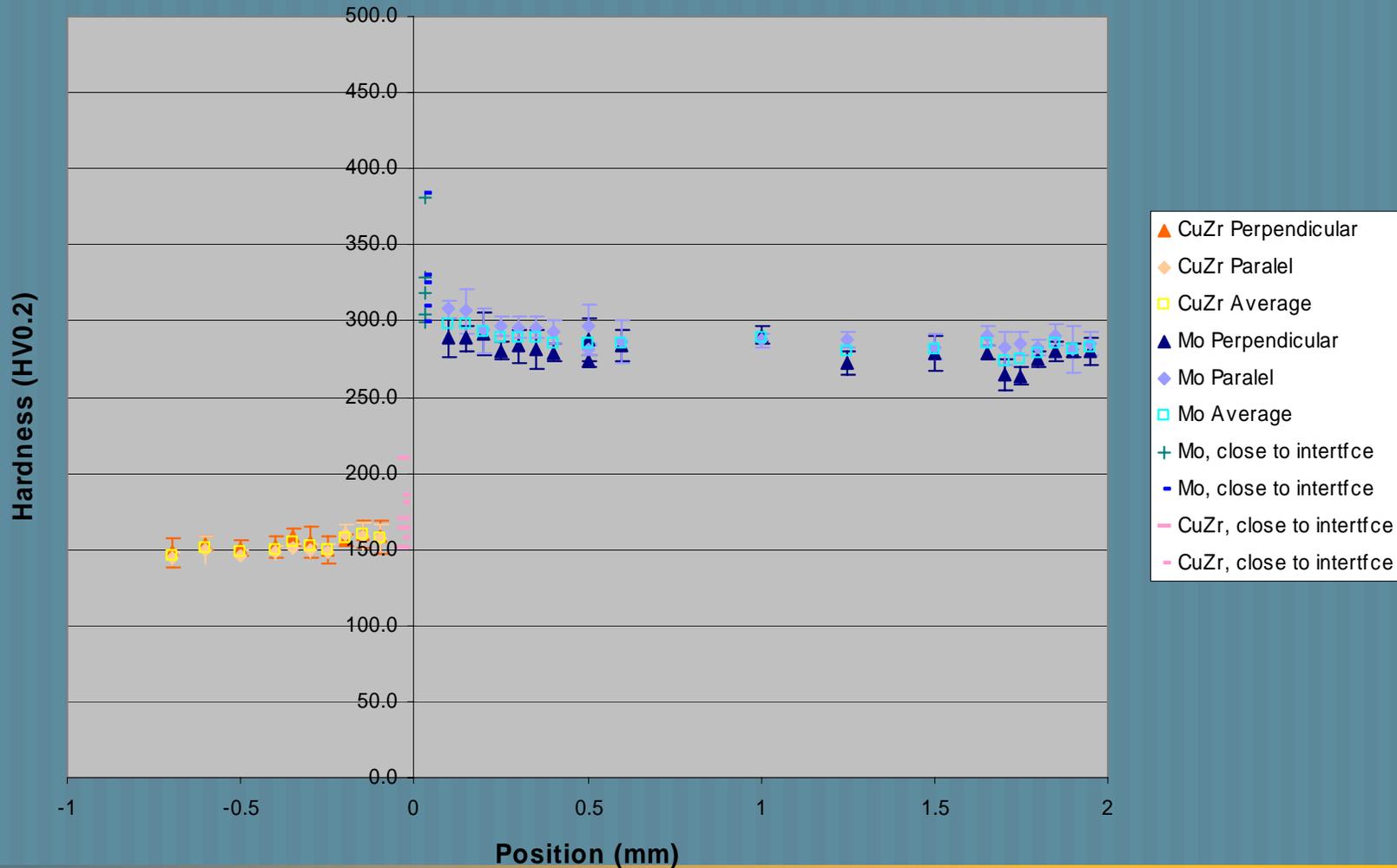
- Microstructural observation
 - Different wave amplitude
 - No voids or cracks at the interface
 - No cracks on the Mo
 - Irregular line at approx 30 μm of the interface visible only on etched sample



Explosion bonding. 2nd pieces results

■ Micro hardness profiles

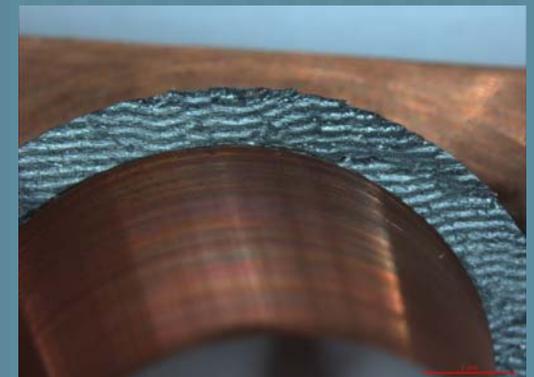
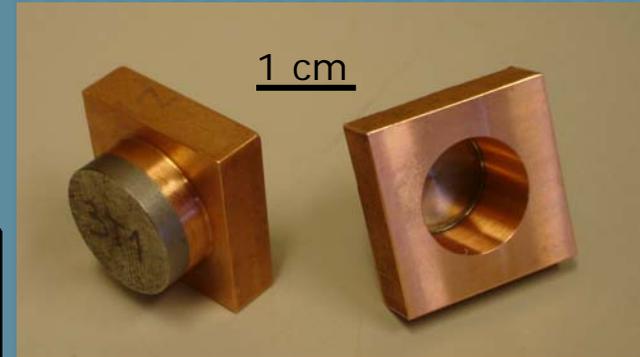
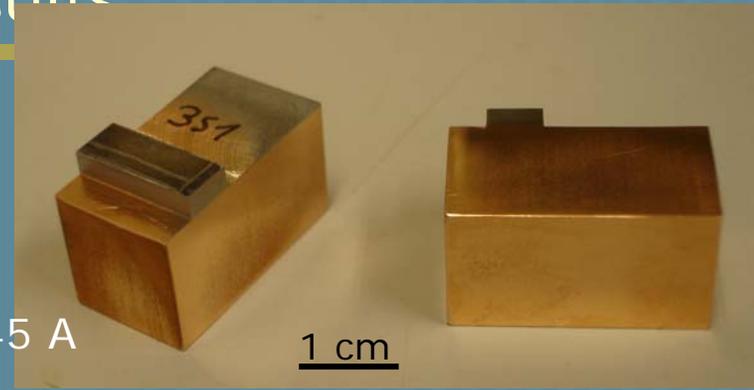
Sample 2 mm Mo



Explosion bonding. 2nd pieces results

■ Bond strength

- Tests adapted from standards
 - Shear test: ASTM B 898 modified
 - Pull test: Ram Tensile Test MIL-J-24445 A modified
- Comparatively high strength values
- Fracture always on the Mo close to the interface showing a wavy aspect



| | 1 st | 2 nd | | |
|---------------------------|-----------------|-----------------|------------|----------------|
| | | 1 mm | 2 mm | 3 mm |
| Shear strength up (MPa) | 110 ±19 | 261 | 309 | 225 |
| Shear strength down (MPa) | | 249 | 227 | 200 |
| Pull strength (MPa) | - | - | 77 | 121 ±72 |

Explosion bonding. Conclusions on 2nd piece tests

- Explosion bonding shows promising
 - Comparatively high bond strength values
 - The CuZr to bond can be in any temper state, including cold worked and aged, and needs no further heavy thermal treatments
- Possible presence of a weakened region on the Mo close to the interface
- Change from flat to locally cylindrical geometry needs more development
- Possible problems of flatness on larger pieces

Comparison HIP-diffusion and explosion bonding

| | <u>HIP diffusion bonding</u> | <u>Explosion bonding</u> |
|--------------------------|---|---|
| Bond performance | Good as HIPed | Good |
| Shear strength | 165 +-9 MPa | 309-227 MPa |
| Pull strength | 175 +-18 MPa | 77 MPa |
| Defects at the interface | Not as HIPed, but homogeneity to be improved | Not |
| Machining | ? | ? |
| CuZr Matrix performance | Temper states limited, no cold work, may be no proper ageing. | Any temper state, including cold worked and aged |
| Tensile strength max. | 211 MPa | 358 MPa |
| Need of post treatments | Yes, to get limited improvement | No |
| Geometry | Cylindrical | Flat |
| Large scale production | Not much scale factor | Possibly scalable to production of large pieces, would need special production of flat CuZr |