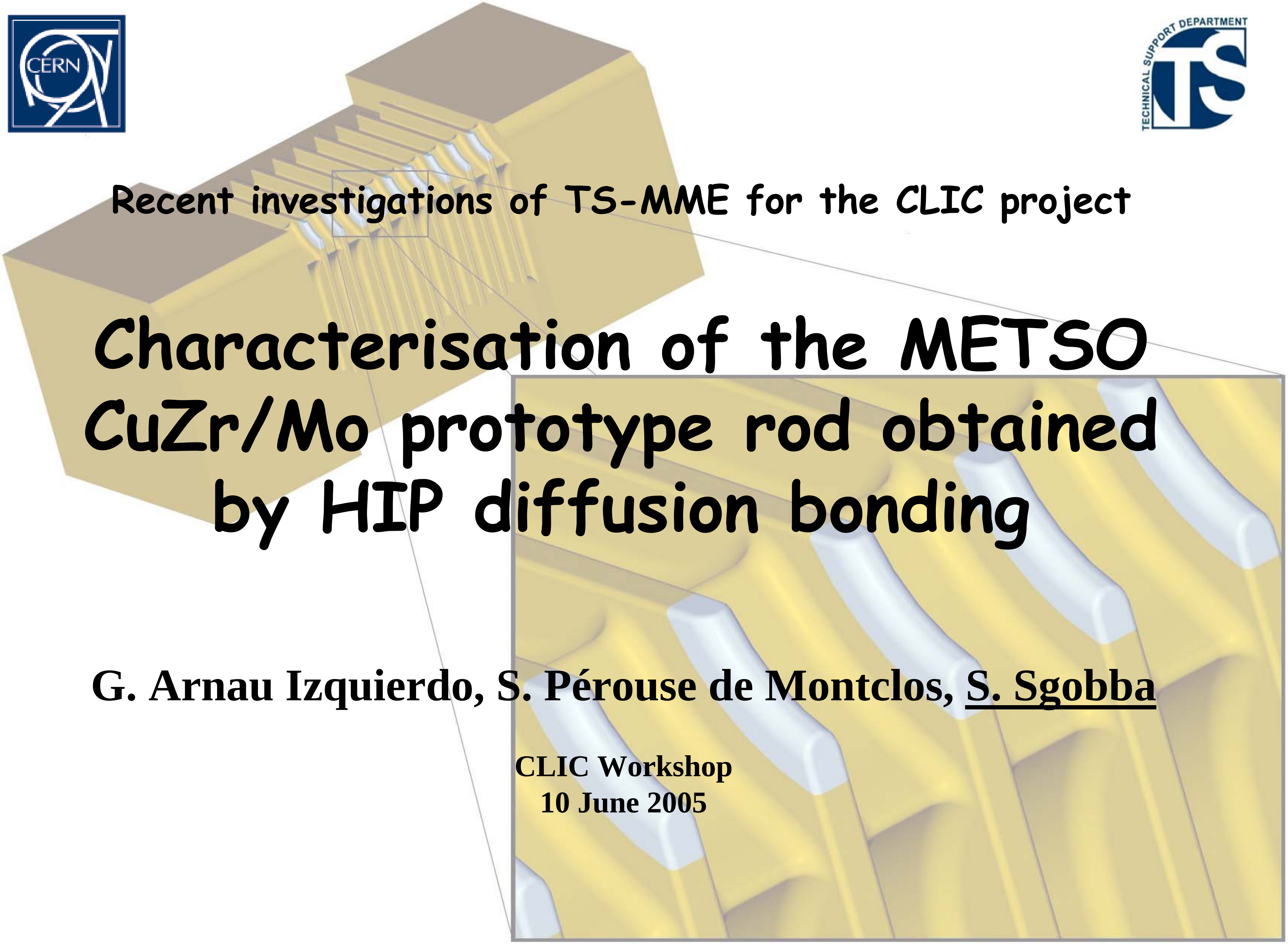

Recent investigations of TS-MME for the CLIC project

Characterisation of the METSO CuZr/Mo prototype rod obtained by HIP diffusion bonding

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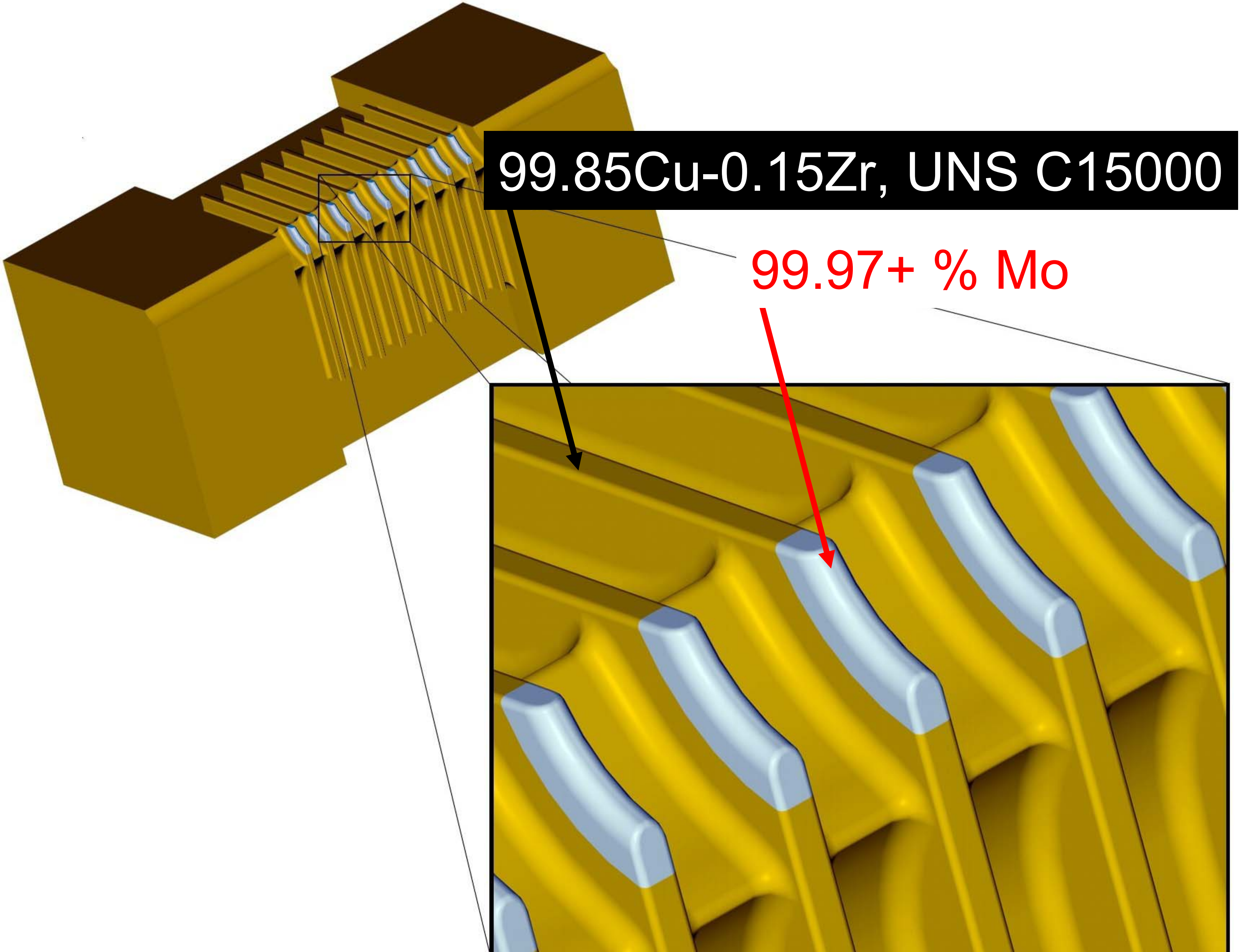
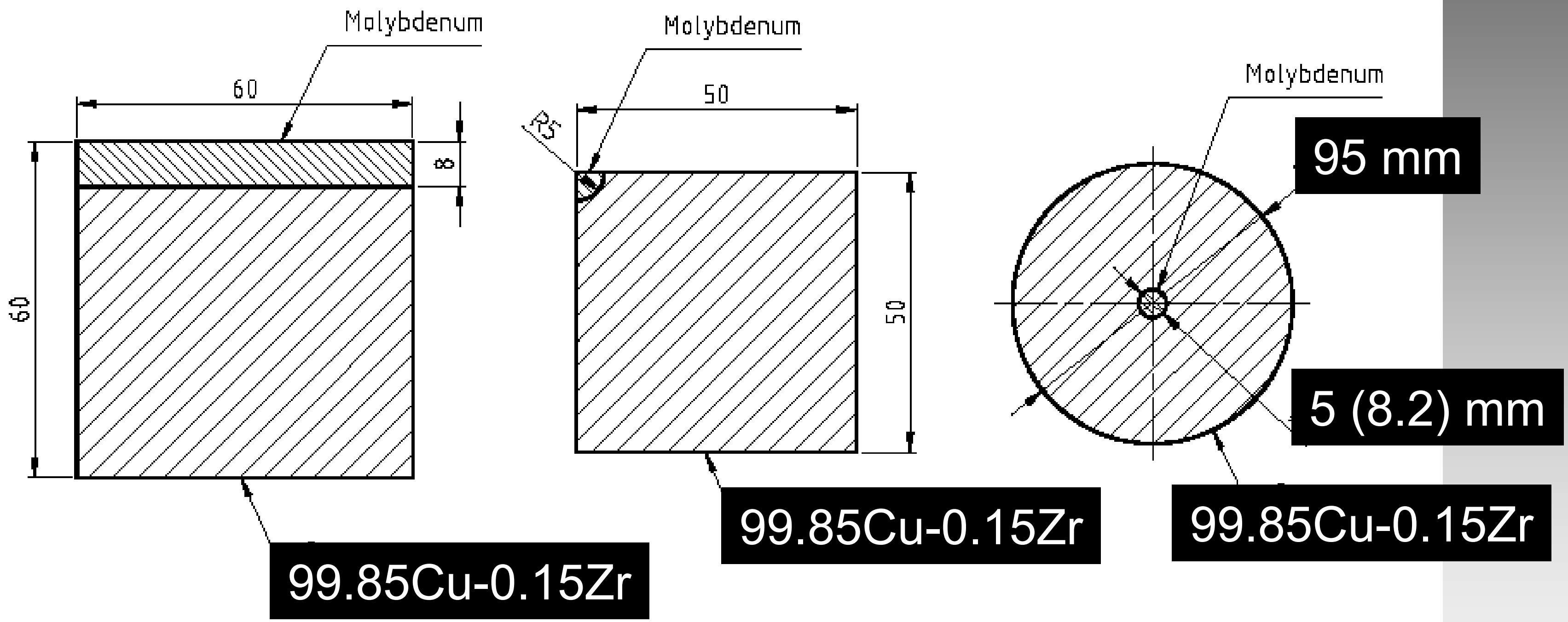
CLIC Workshop
10 June 2005

Outline

1. Remind, HIPing applied to diffusion bonding
2. Microoptical observations, CuZr matrix and Mo insert
   Grain size
3. Mechanical properties, CuZr matrix and Mo insert
   a. Hardness and microhardness profiles
   b. Tensile properties of the CuZr matrix
4. Strength and characterisation of the interface
   a. Shear strength
   b. (Tensile strength)
   c. Soundness of the interface
   d. Diffusion profile, precipitation of intermetallic phases
5. Evolution of HIP-quenched CuZr with further thermal treatments
6. Summary and open questions


99.85Cu-0.15Zr, UNS C15000
99.97+ % Mo

Aimed structure



The Mo + 99.85Cu-0.15Zr system : 1) C15000 alloy

Typical heat treatment:

◊ Solution annealing ⇒ Nominal T, t = 900 to 925 °C, 5 to 30 min, fast cooling

◊ (In reality HIP quenching from 900 °C, 10 h (!), cooling 20 °C/min)

◊ Artificial aging (aged only) ⇒ T, t = 550 °C, 3 h

The Mo + 99.85Cu-0.15Zr system :
1) C15000 alloy

Table 18 Typical mechanical properties of C15000

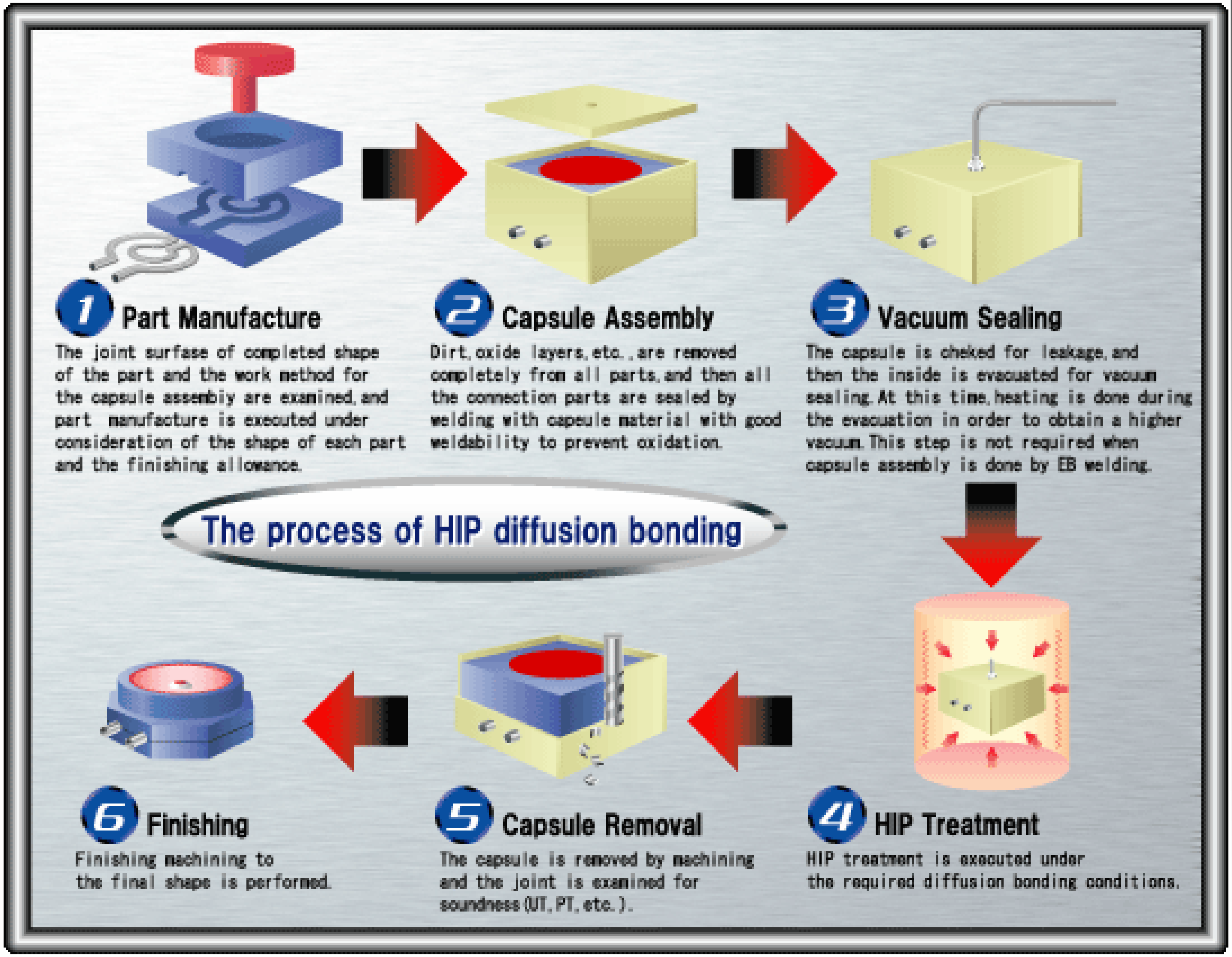
Section size mm	Section size in.	Cold work, %, after: Solution treating(c)	Cold work, %, after: Aging(d)	Tensile strength MPa	Tensile strength ksi	Yield strength(a) MPa	Yield strength(a) ksi	Elongation(b), %
Rod								
5	0.20	...	76	430	62	385	56	8
6	0.25	10(e)	...	285	41	250	36	34
9.5	0.37	80	44	470	68	440	64	11
13	0.50	56	47	460	67	435	63	15
16	0.62	61	31	440	64	430	62	15
19	0.75	50	34	435	63	420	61	15
22	0.87	48	52	430	62	415	60	15
25	1.0	48	47	430	62	415	60	15
32	1.25	32	17	430	60	400	58	18
Wire								
1	(0.04)	...	98(f)	525	76	495	72	1.5
2.3	(0.09)	...	62(f)	495	72	470	68	3
		0	...	200	29	40	6	54
		...	0	205	30	90	13	49
6	(0.25)	0(e)(g)	...	255	37	75	11	50
13	(0.50)	30(e)	...	365	53	340	49	23

a) At 0.5% extension under load. (b) In 50 mm or 2 in. (c) At 900 to 925 °C (1650 to 1695 °F). (d) For 1 h or nore at 400 to 425 °C (750 to 795 °F). (e) Mill annealed. (f) Solution treated, cold worked the stated mount, then aged. (g) OS025 temper.

Strength of zirconium-copper depends primarily on cold working. Aging results in some increase in strength, and an increase in electrical conductivity

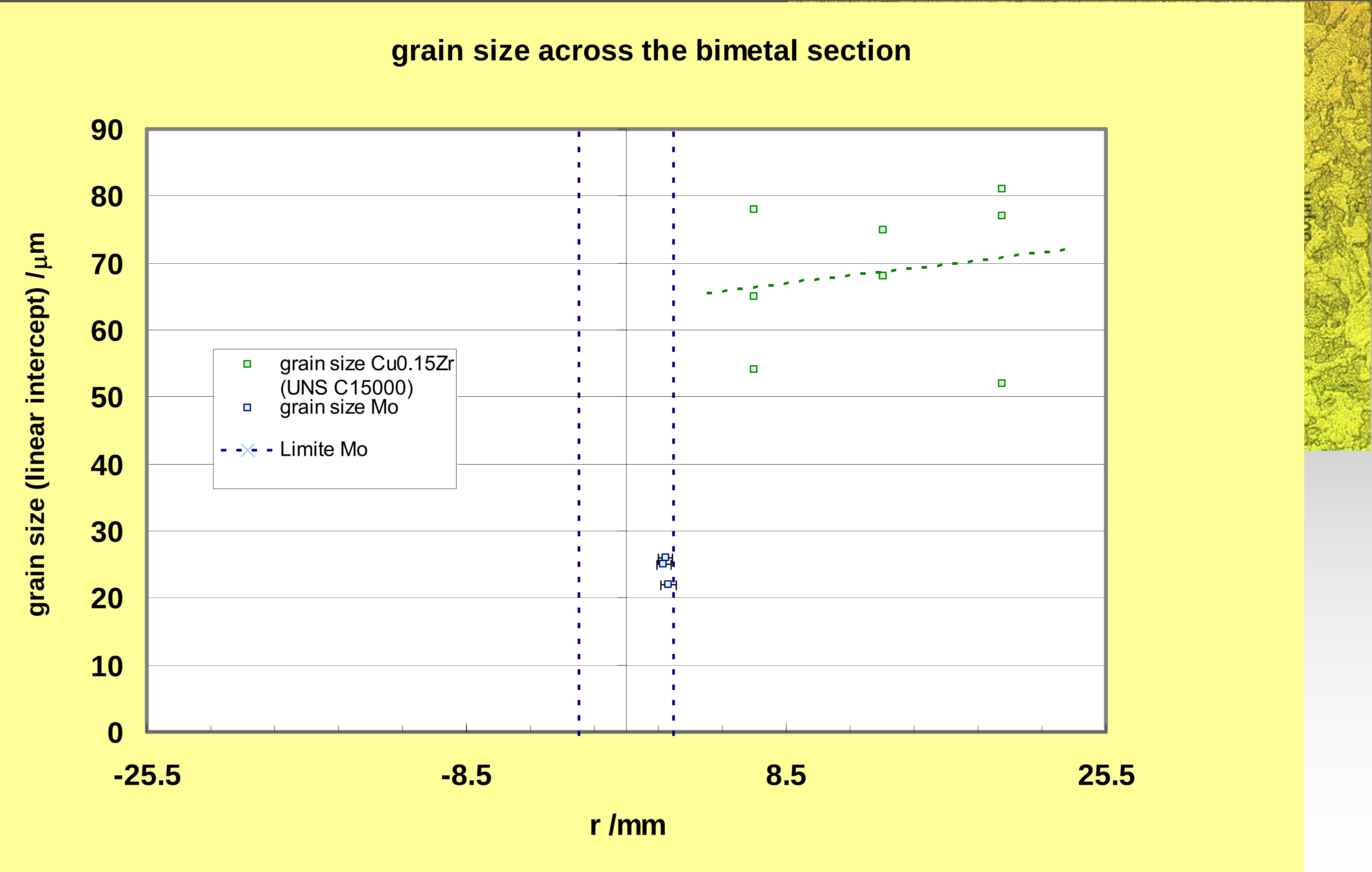
Solution-treating temperature	Amount of cold work, %	Aging Temperature	Aging Time, h	Tensile strength	Yield strength	Elongation, %	Hardness, HRB	Electrical conductivity, % IACS
°C		°C		MPa	MPa			
900	20	475	1	310	260	25	48	85 min
900	80	425	1	425	380	12	64	85 min
980	None	. . .	. . .	200	41	54	. . .	64
980	20	. . .	. . .	270	250	26	37	64
980	80	. . .	. . .	440	420	19	73	64
980	None	500	3	205	90	51	. . .	87
980	None	550	3	205	90	49	. . .	95
980	20	400	3	330	260	31	50	80
980	20	450	3	330	275	28	57	92
980	85	400	3	495	440	24	79	85
980	85	450	3	470	425	23	74	91

Diffusion bonding by HIP (courtesy of Metso)



Bimetallic structures

Microoptical observations, CuZr matrix and Mo insert



Microoptical observations, CuZr matrix and Mo insert

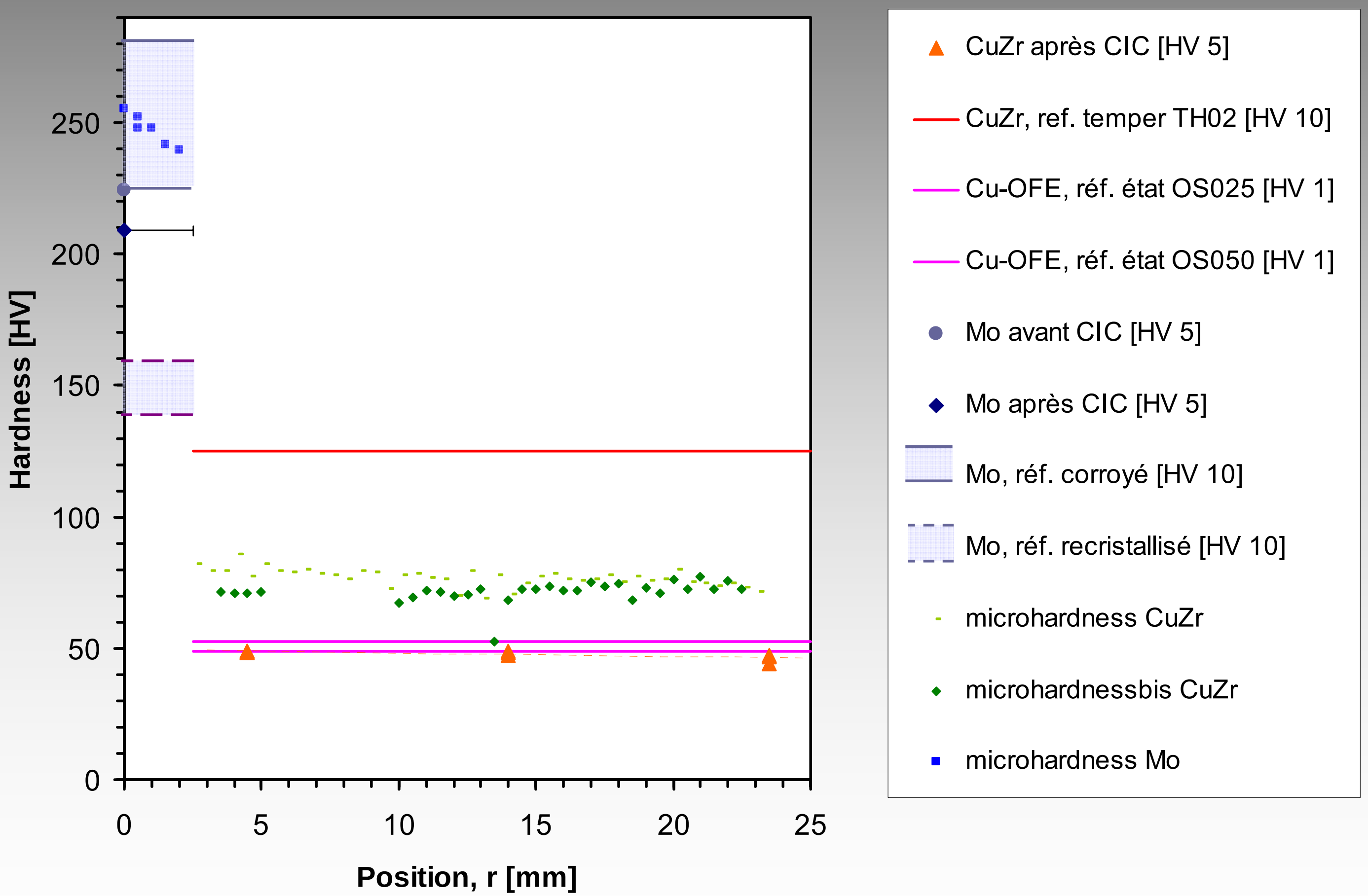
❑ As-received state (HIP-quenched):

- Cu-Zr matrix: grain size, approximately 70 μm
- Increasing from centre to periphery?

- Mo insert: cross-sectional grain size, approximately 25 μm

- Presence of an envelope adhering poorly to the Cu-Zr matrix

Mechanical properties, hardness and microhardness profiles

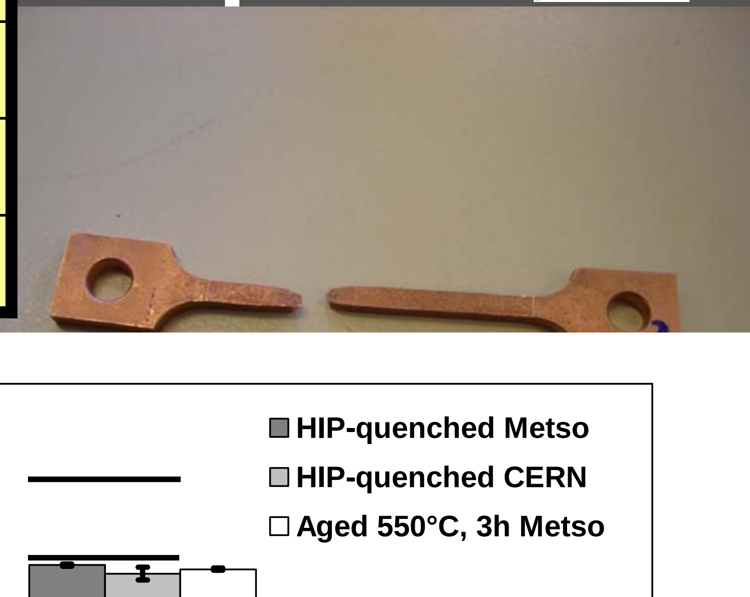


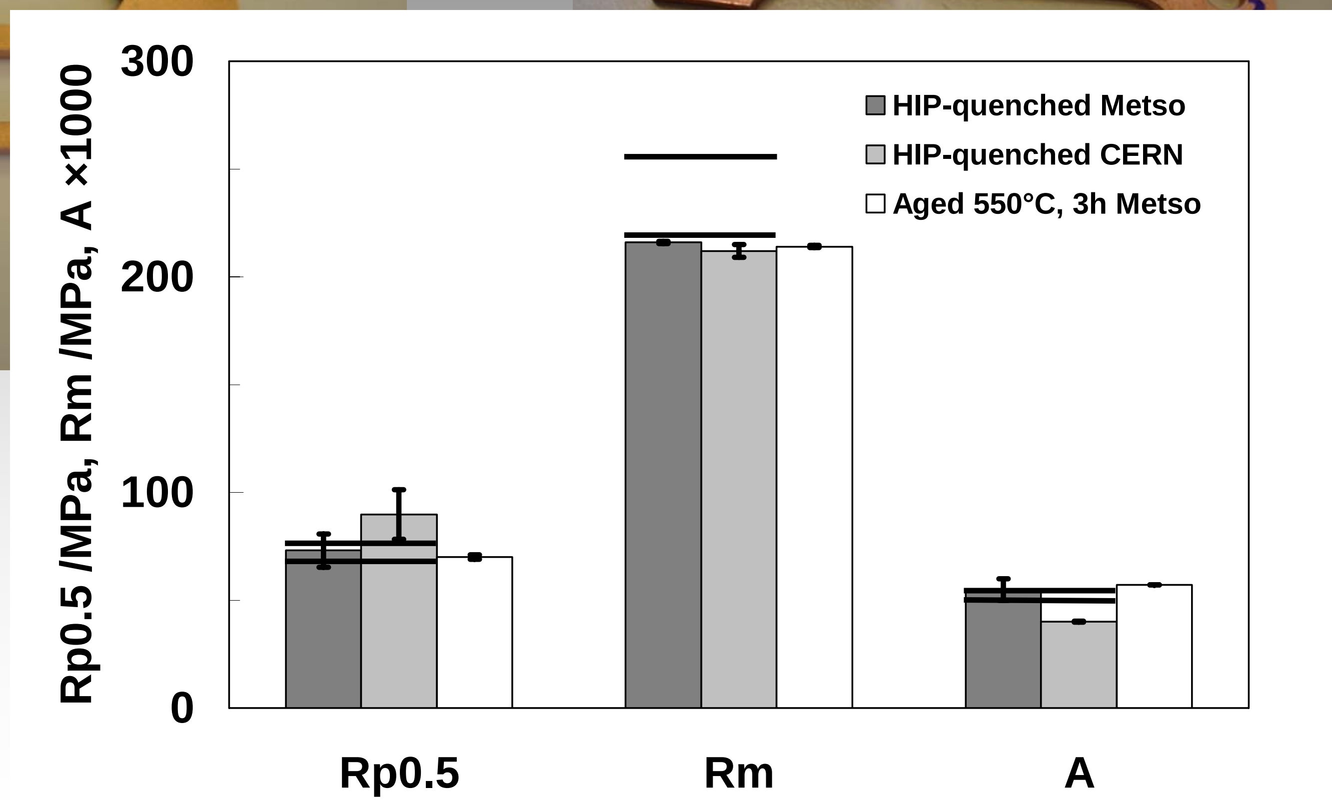
HIP-quenched Cu-Zr, compares to fine grained, annealed Cu-OFE

Mo is not significantly softened by the HIP-cycle. Light softening occurs progressively with time at T

… test … temper

Sample	Rp0.2 /MPa	Rp0.5 /MPa	Rm /MPa	A /%
HIP-quenched Metso	64	73	216	55
OFE/OFCu, OS050	-	*69*	*220*	*45*
C15000, 6 mm wire, mill annealed OS025	-	*75*	*255*	*50*
HIP-quenched CERN	79	90	212	40
Aged 550°C, 3h Metso	60	70	214	57





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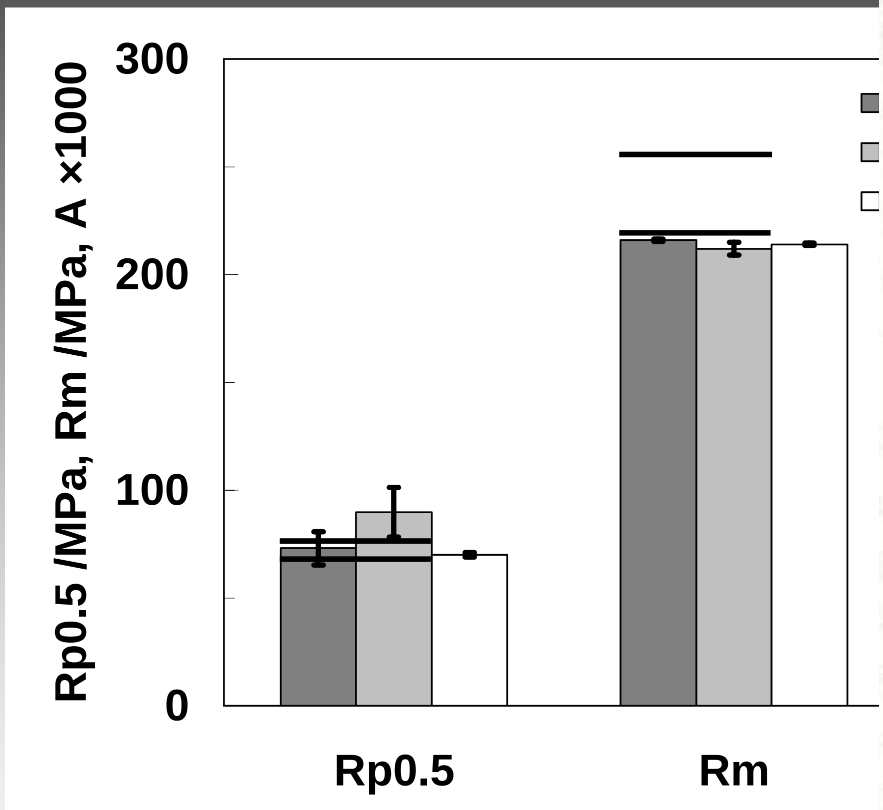


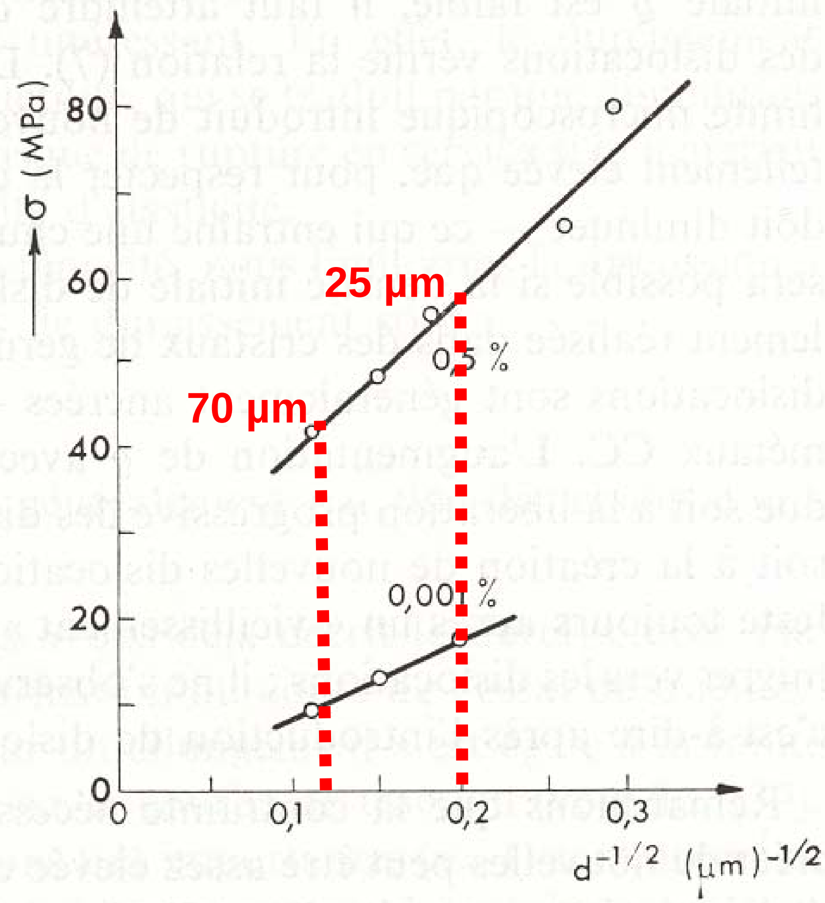


FIG. 201. — *Variation de la limite d'élasticité conventionnelle du cuivre* (pour des déformations de 0,001 et 0,5 p. 100 *en fonction de la dimension des grains* (d = diamètre en microns).

Hall-Petch relationship for copper with grain sizes of 1 mm–3 μm (Kozlov, 2002):

$\sigma = \sigma_0 + k \cdot d^{-1/2}$, $k = 0.14 \pm 0.05$ MPa·m$^{1/2}$

Δσ (25 μm .. 70 μm) ~ 7 MPa .. 15 MPa

Mechanical properties, tensile properties of the CuZr matrix

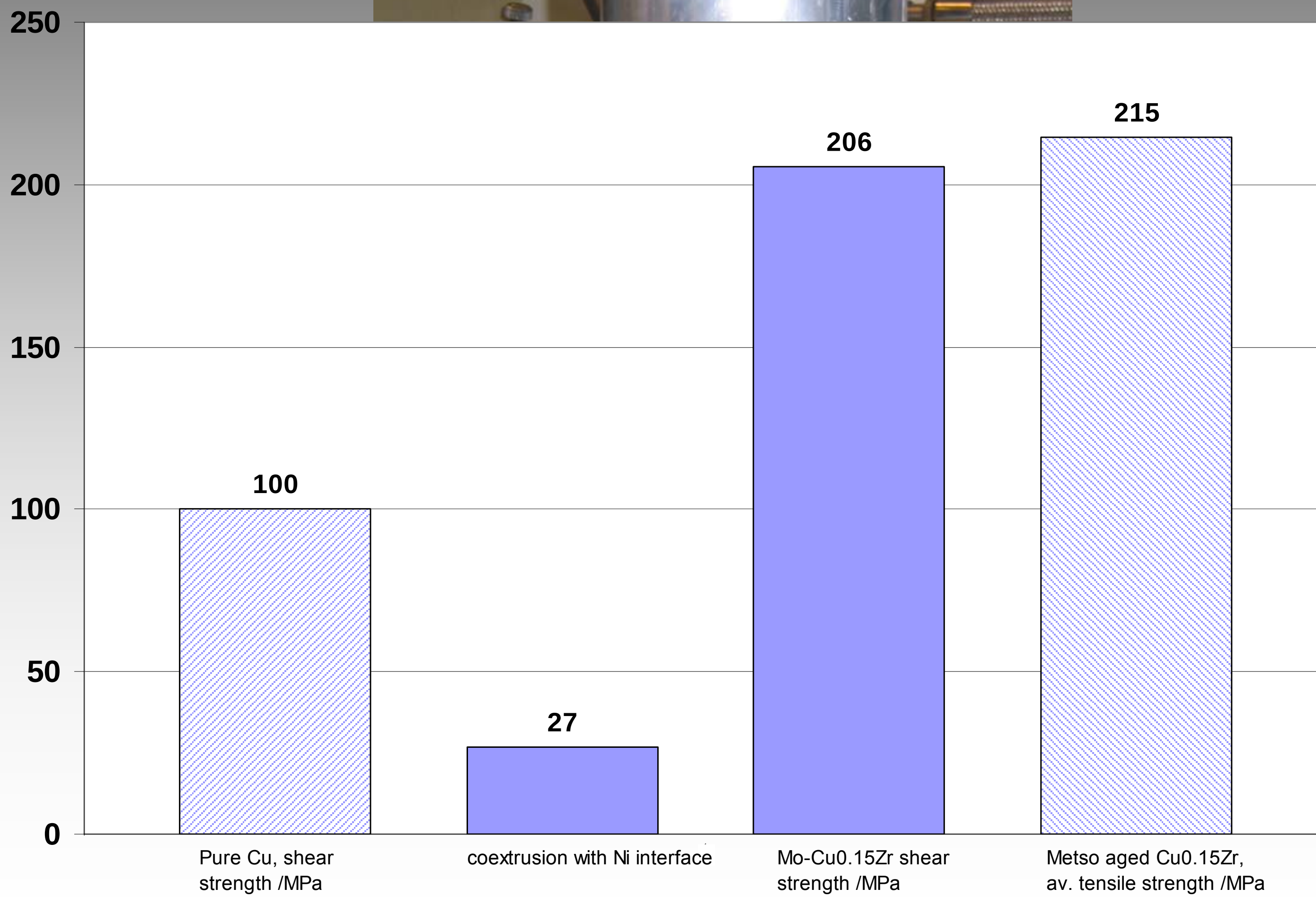
❑ As-received state (HIP-quenched):

- Metso and CERN results are consistent
- The HIP-quenched state is not susceptible to directly harden following an ageing treatment

Alliage	Etat		Résistance traction [MPa]	Limite élastique [MPa]	Allongement [%]	Cond. électrique [%IACS]
C15000	OS025	(recuit, grain < 25 µm)	255	75	50	
	TB00	(mis en solution)	200	41	54	64
	TF00	(TB00 + vieilli)	205	90	50	87 - 95
	TH02	(TB00 + écroui ½dur + vieilli)	358	316	15	
	TH04	(TB00 + écroui dur + vieilli)	470	425	23	91
Cu-OFE	OS025	(recuit, grain < 25 µm)	235	79	55	100
	OS050	(recuit, grain < 50 µm)	220	69	55	100

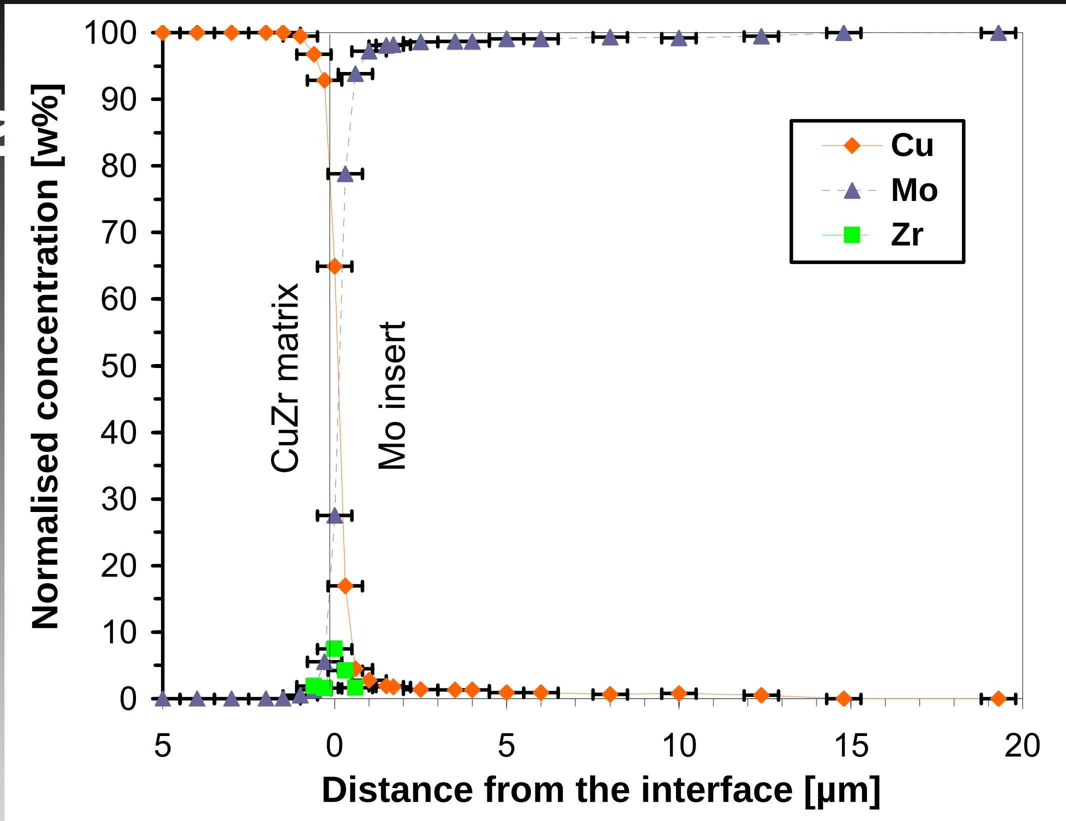
Strength and characterization of the interface

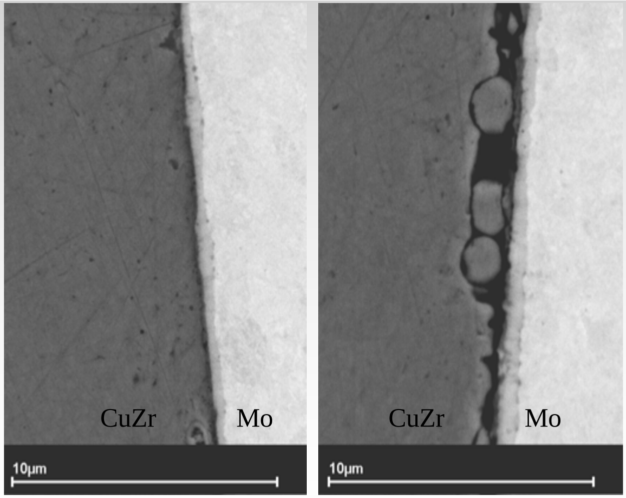
Shear test, values in MPa



Shear strength compares with measured tensile strength of HIP-quenched CuZr

Scanning Ele





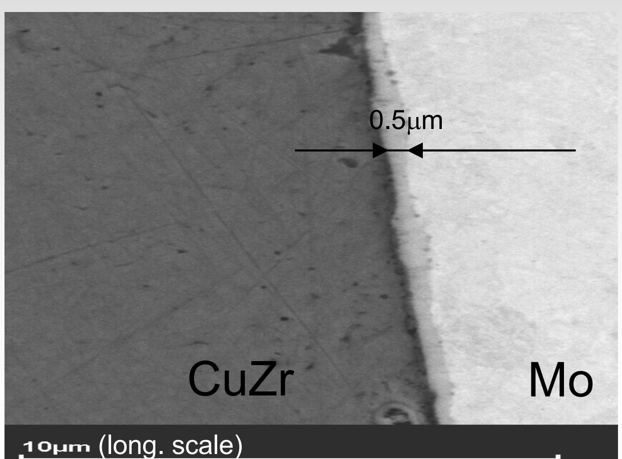
Strength and characterization of the interface

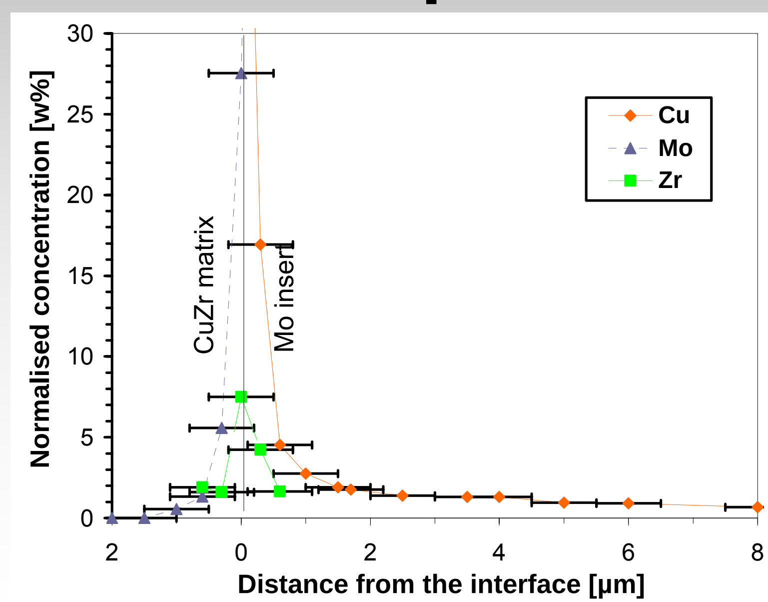
A typical diffusion length:
$x^2 = D(T) \cdot t$
where $D(T) = D_0 \exp(-Q/RT)$

and D(T) is the interdiffusion coefficient at the temperature T, Q is the activation energy and R is the universal gas constant

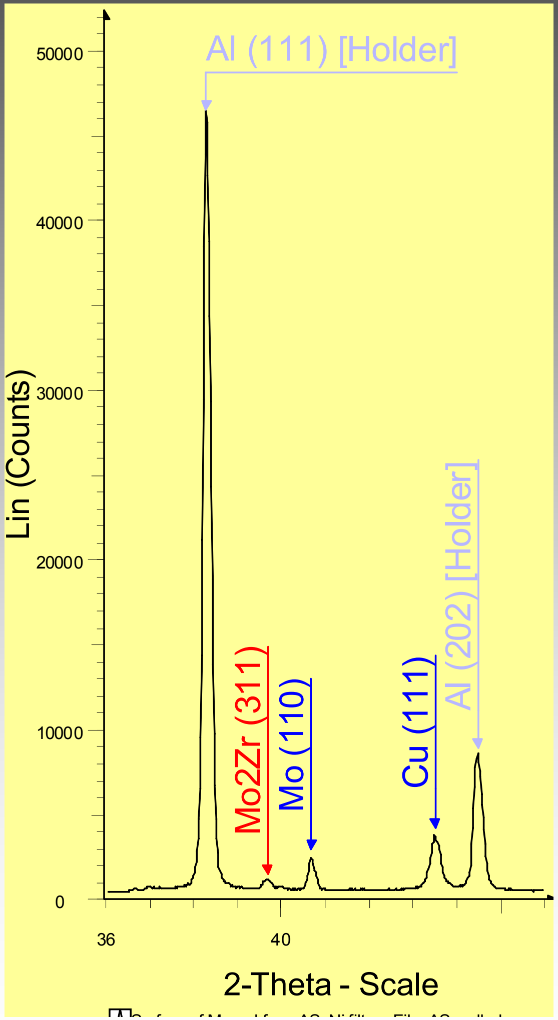
$Q = 146$ kJ mol^{-1}
$D_0 = 2.82\ 10^{-10}\ m^2\ s^{-1}$

x = 1.78 µm, in agreement with the measured profile





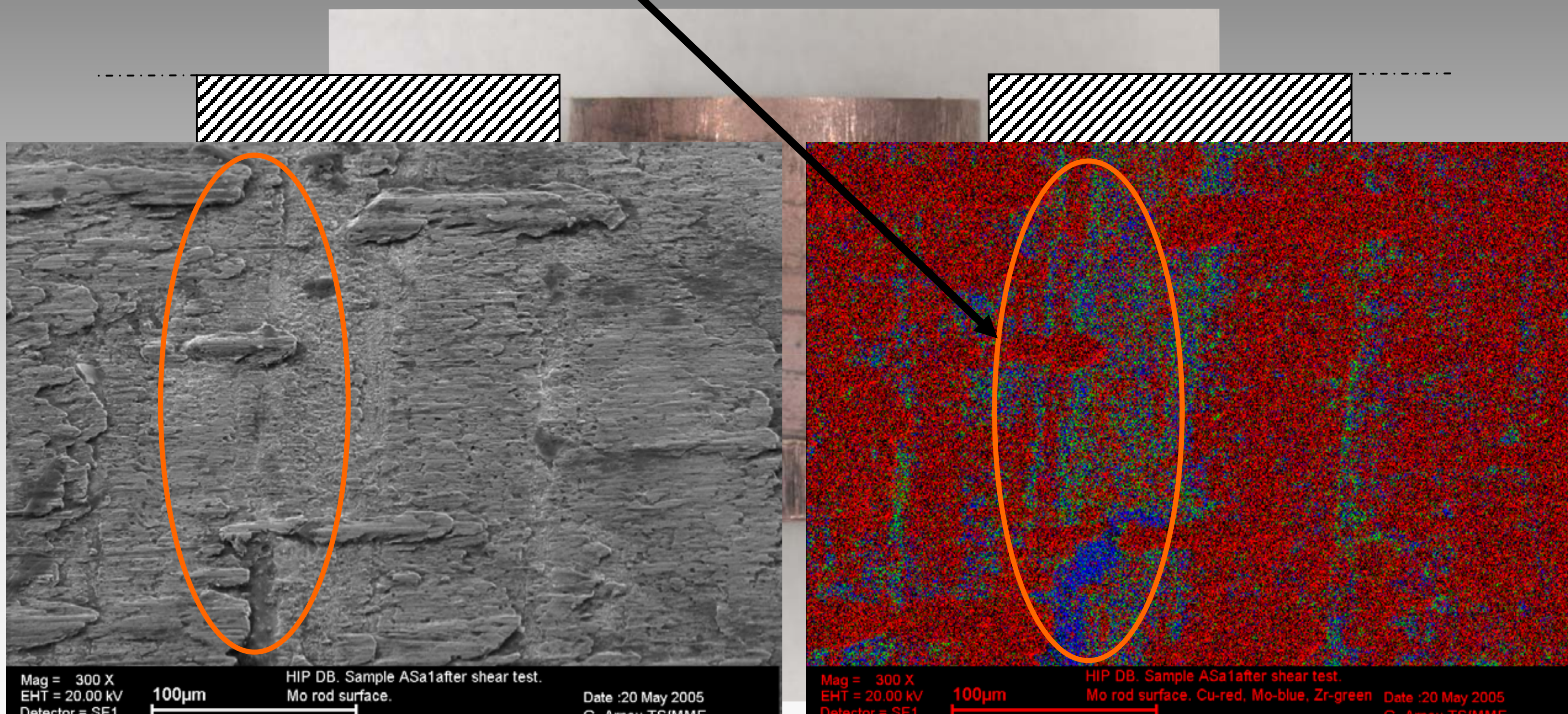
X-Ray diffraction



Strength and characterization of the interface

Precipitation of intermetallic phases

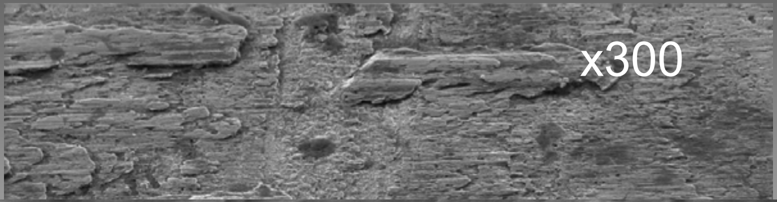
Mo and Zr are associated and found at the same locations.

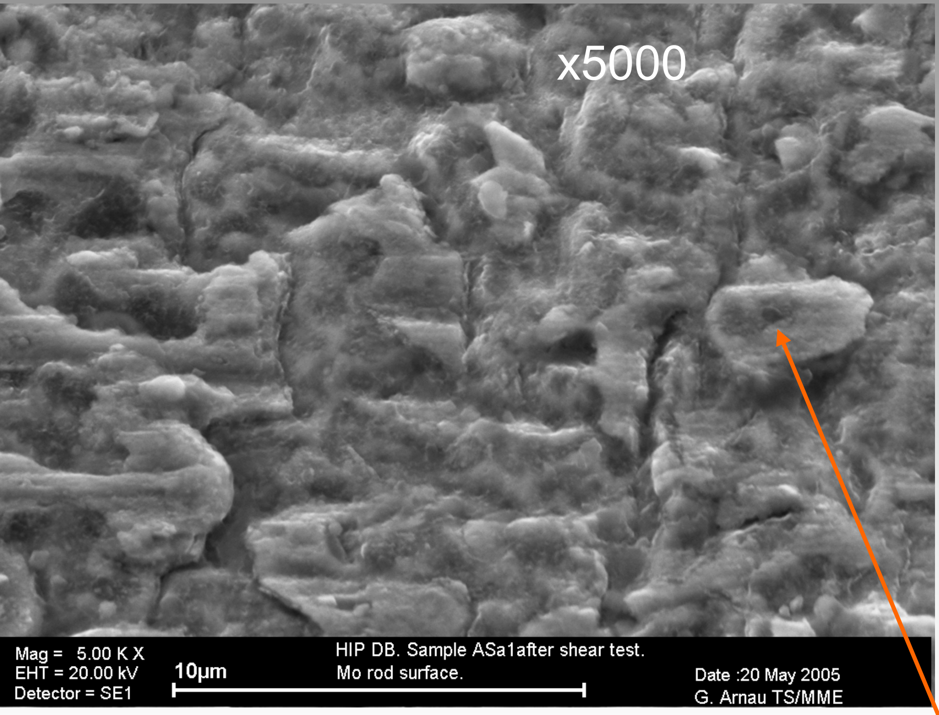


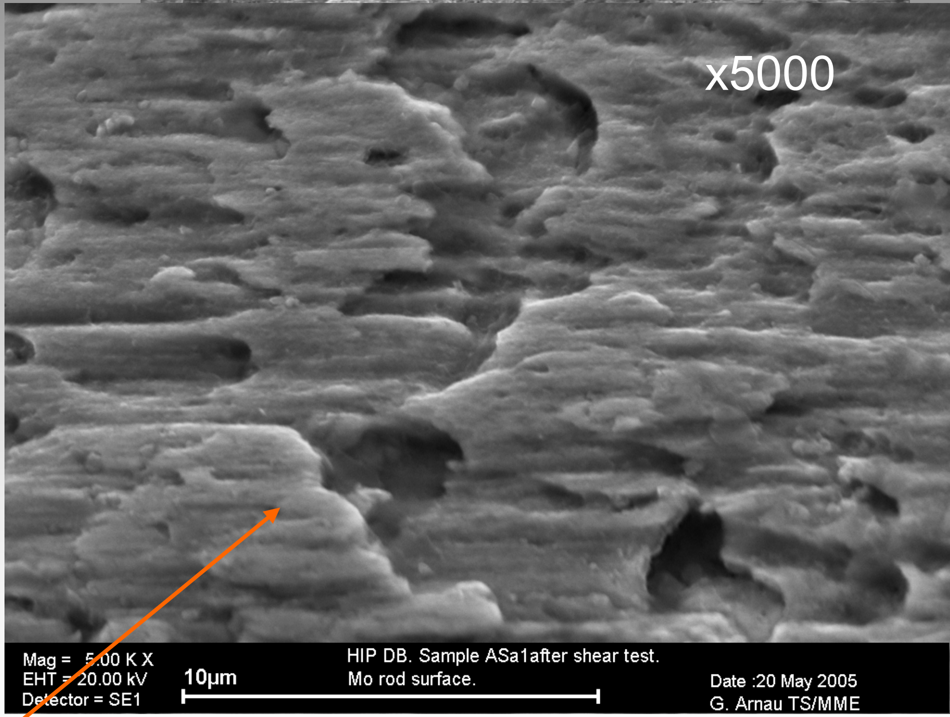
Shear surface as from a SEM observation and distribution map of Cu (red), Mo (blue) et Zr (green)

Strength and characterization of the interface

Topographical aspect of the sheared interface

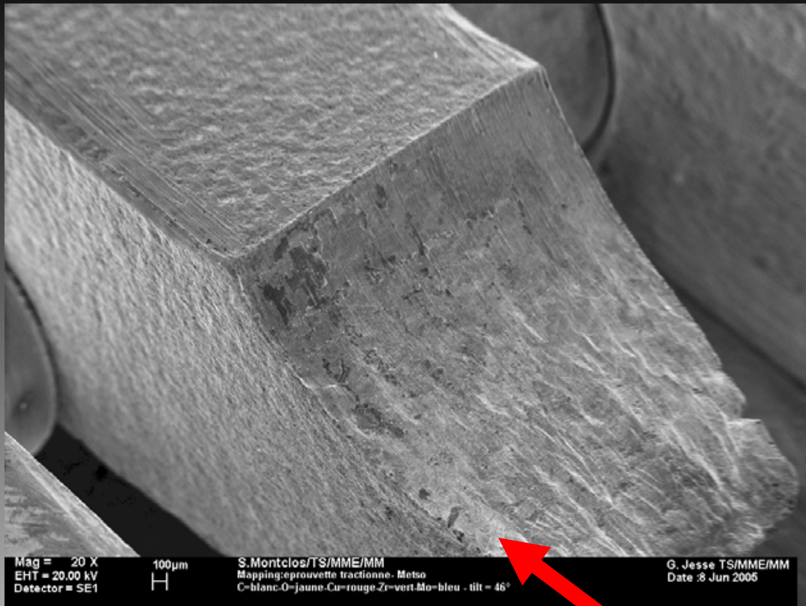


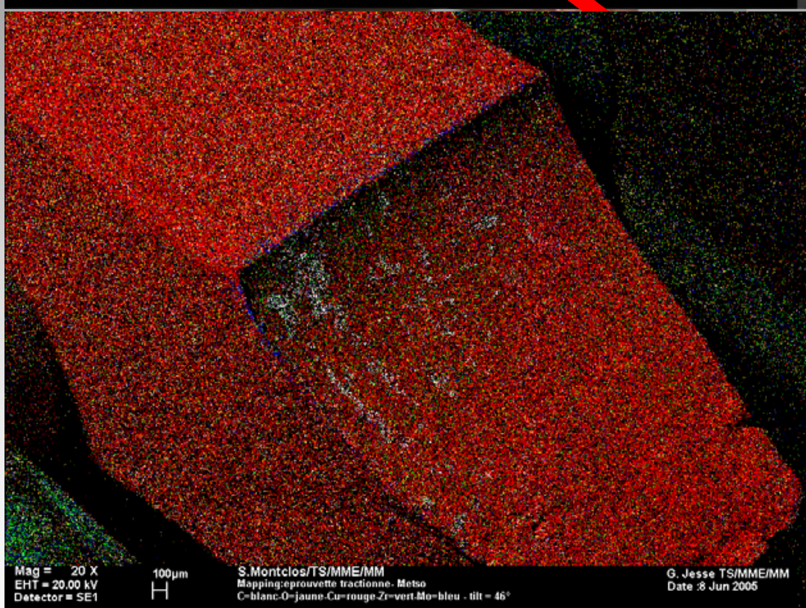


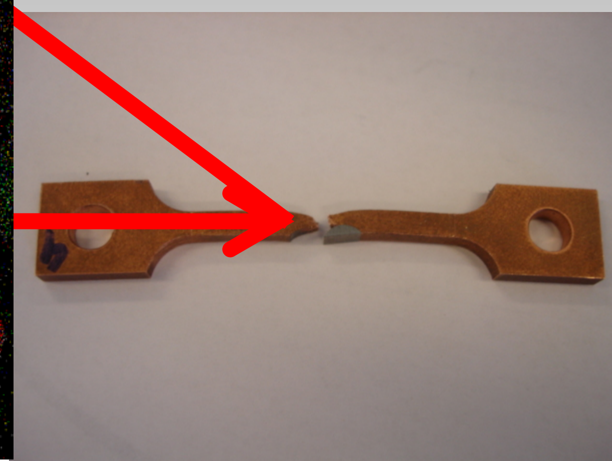


Sheared-off interface

d characterisation
face, tensile tests







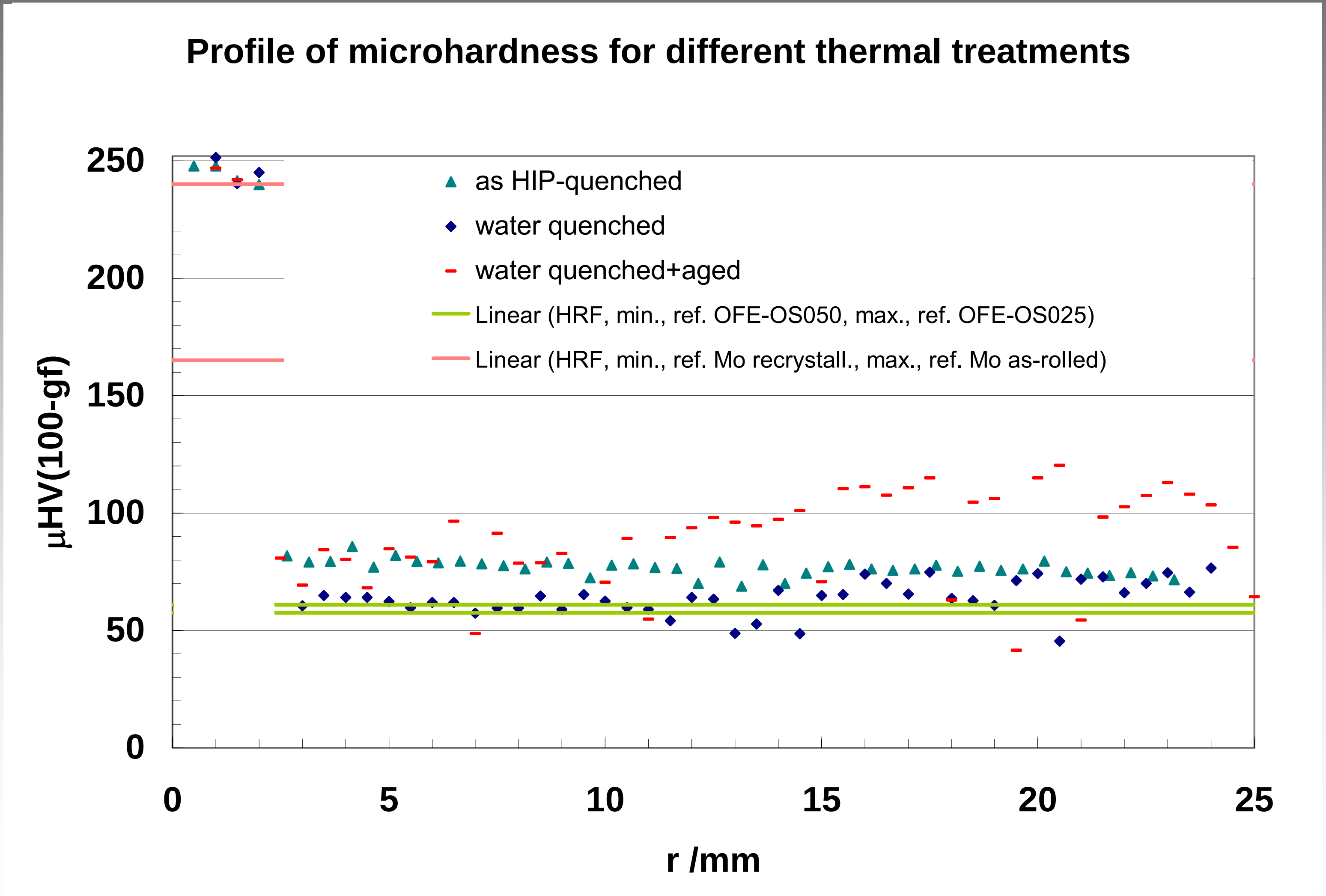
Estimated (bulk as HIP-quenched CuZr):
Rp0.2 = 85.3 (79) MPa
Rm = 233 (212) MPa

Strength and characterization of the interface

❑ CuZr-Mo interface

- Despite the presence of an intermetallic phase Mo_2Zr…
- The shear strength of the bimetal interface is higher than the one of the CuZr matrix
- Interdiffusion has occurred as foreseen from diffusion parameters of the Mo-Cu system
- Locally cavities are observed (not expected following a HIP process)
- EDM resulted in one case in a separation of the two phases

Evolution of HIP-quenched CuZr with further thermal treatments



Evolution of HIP-quenched CuZr with further thermal treatments

❑ Mo

- No influence on hardness of an additional solution annealing and of further low temperature ageing treatments

❑ CuZr

- HIP-quenched and solution annealed states are comparable in hardness
- Artificial ageing (550 °C, 3h) is effective after further solution annealing
- Hardness increases from less than 50 HV (annealed states) to approximately 65 HV (solution annealed + aged state)
- For the aged state, increase of microhardness from centre to periphery (Zr depletion)?

Summary and open questions

1. HIP quenching results in a temper state that requires further solution annealing to be susceptible to age

2. Ageing after further solution annealing is effective (more or less depending on the position?)

3. Depletion of Zr due to diffusion toward the insert-matrix boundary?

4. The interface shows (locally?) good interdiffusion and high shear strength

5. EDM resulted in a separation of the matrix and the insert (due to the process or to locally poor adhesion?)

6. Presence of a Cu envelope?