



## Materials testing & lessons learned for Crab Cavities

Adrià Gallifa Terricabras CERN EN-MME, on behalf of WP4

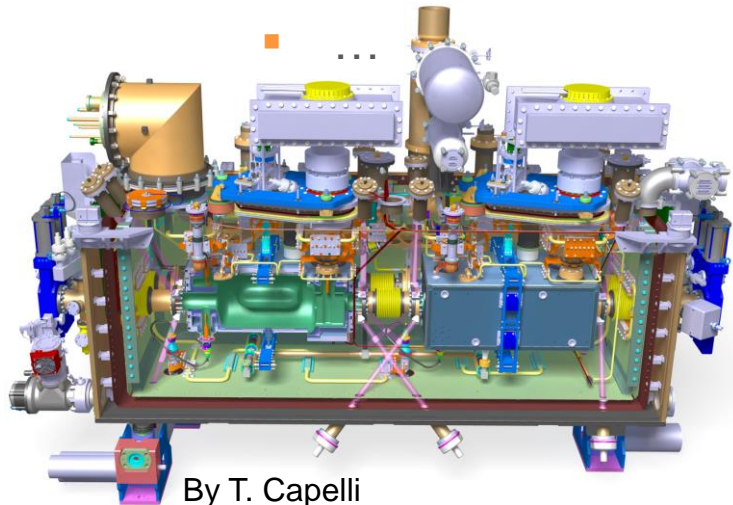
12th HL-LHC Collaboration Meeting · Uppsala · 19 - 22 September 2022



# It's a tight relationship...

## Materials

- Nb RRR300
  - Ti alloys
  - Cu alloys
- Stainless steel
- Cryophy, mumetal
- Ceramics ( $\text{Al}_2\text{O}_3$ )
  - Filler metals



## Components

- Bare cavities
- Brazed SS-Nb extremities
- Tuning system
- 2<sup>nd</sup> beam pipe
- He tank
- Ti-SS transitions
- Outer Vacuum Vessel
- Bellows
- HOMs and antenna couplers
- Cold & warm magnetic shield
- Coaxial lines
- RF feedthroughs
- Gaskets
- Fasteners
- ...



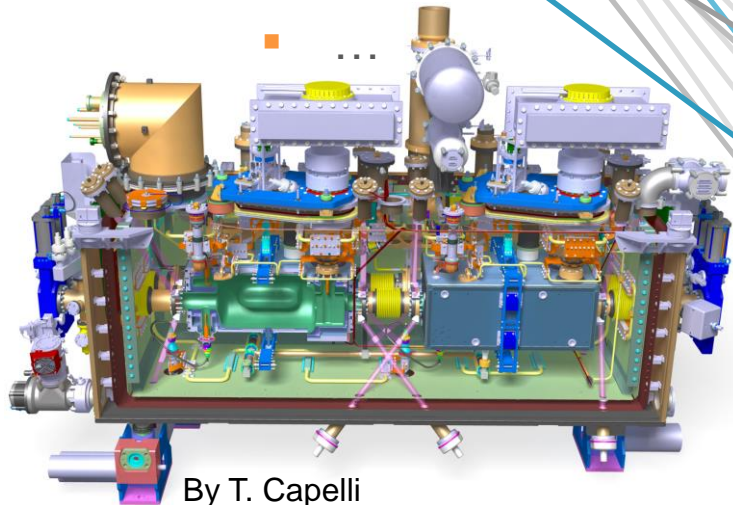
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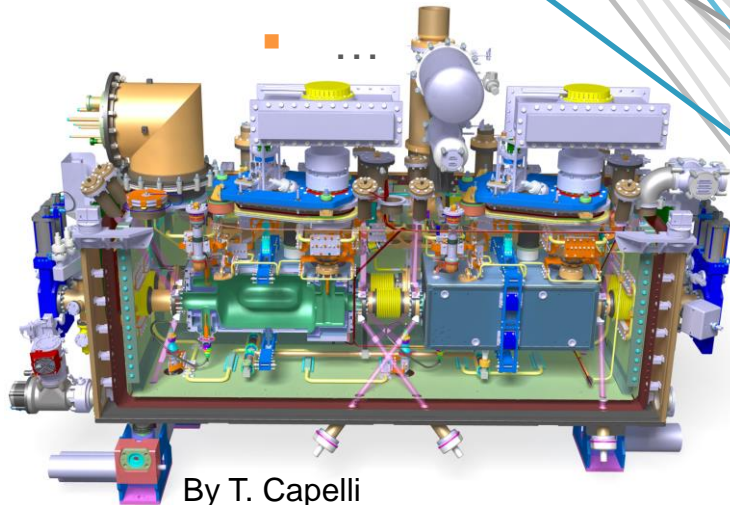


By T. Capelli

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## Manufacturing process

- Cutting
- Deep drawing
- Machining
- Grinding
- Bending
- EB welding
- TIG welding
- Vacuum brazing
- Heat treatments
- Surface treatments
- ...

# Our 'holy' documents

## Engineering specifications for Dressed Crab Cavities (EDMS 1389669) / Cryomodule (EDMS 2043014) & sub-components

- Engineering requirements
- Materials to be used
- Qualification requirements + Acceptance criteria
- Quality, traceability
- Pressure Equipment Directive – PED 2014/68/EU
- CERN safety rules

## CERN material specifications

- Materials acc. to standards in force ISO, EN, ASTM, and industry capabilities.
- Specific CERN requirements
- Technical quality ensured

Rev. No.	Date	Description of Changes (major changes only, minor changes in EDMS)
1.0	05/06/2014	First version
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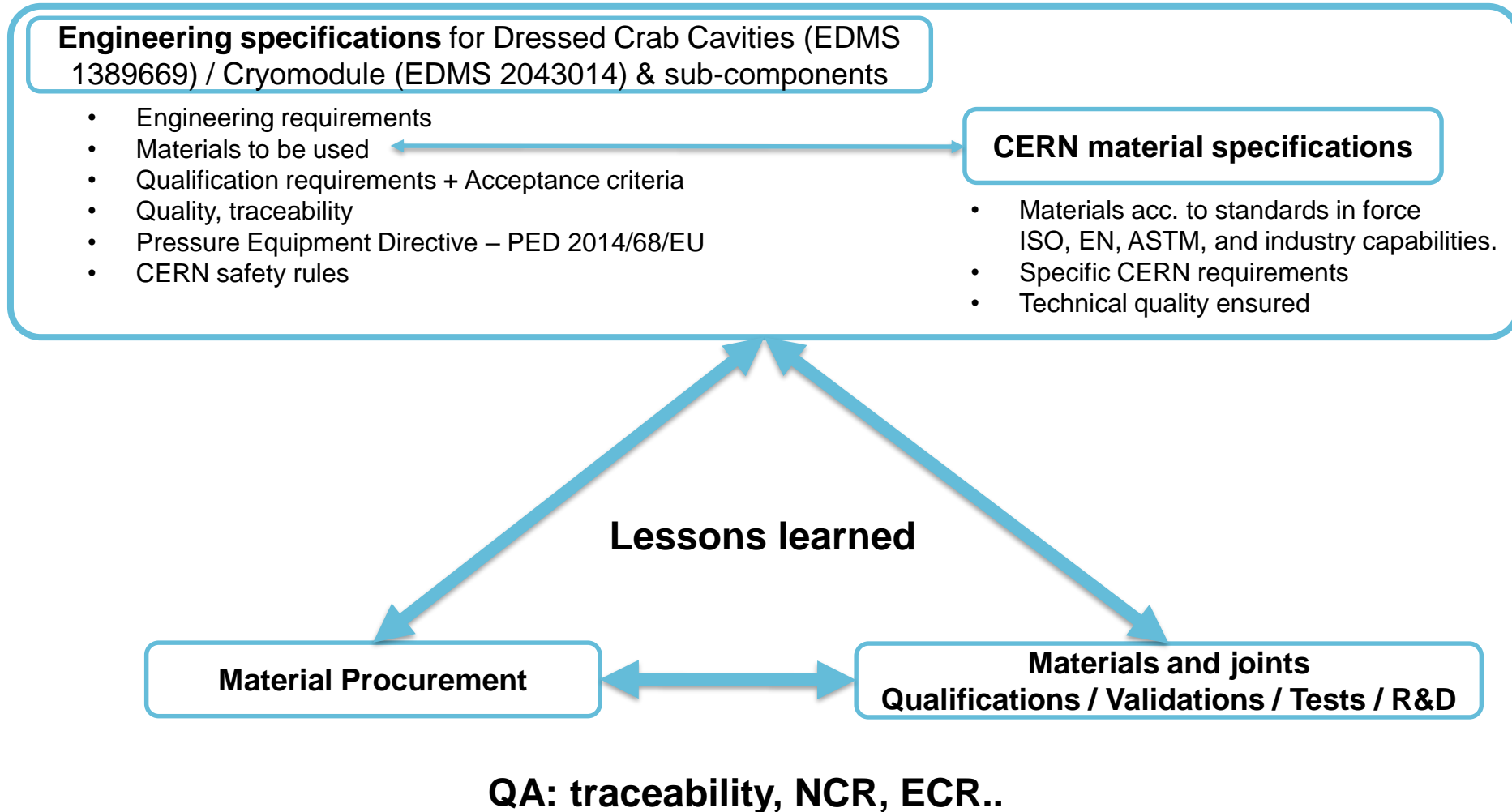
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# Our 'holy' documents



# Table of contents

1. **Niobium: procurement status** at CERN and challenges with **RFD pole forming**
2. **Cryomodule: stainless steel specification** challenges
3. **Advanced techniques** for characterizing parts and joints: update on several **case studies**  
(SS-Ti transition, Cu-Nb EBW antenna, tuner actuation bellows, AlN-Cu brazed ceramic)
4. Upcoming tests
5. Conclusions

# Niobium: procurement status at CERN

2018 - 2021  
2022

Items	Location	CERN Quality Check* Status
Nb and NbTi for DQW pre-series and series bare cavities	RI (working on series)	Done
Nb for DQW HOMS pre-series	CERN	Done
Nb <b>sheets</b> for DQW HOMS series (by Tokyo Denkai)	CERN	UT Done, the rest is pending
Nb <b>plates</b> for DQW HOMS series (by Ningxia)	Shipping ongoing	Pending
Nb <b>bars</b> for DQW HOMS series (by Ningxia)	Shipping Sept/Oct 2022	Pending

Summary report (ongoing) EDMS 2395238

\*Quality check including UT, tensile tests, RRR measurements, metallographic check





## Challenges with RFD pole forming



Min. thickness  
on corners  
~ 2.3 mm

RFD pole forming trials for pre-series cavities.

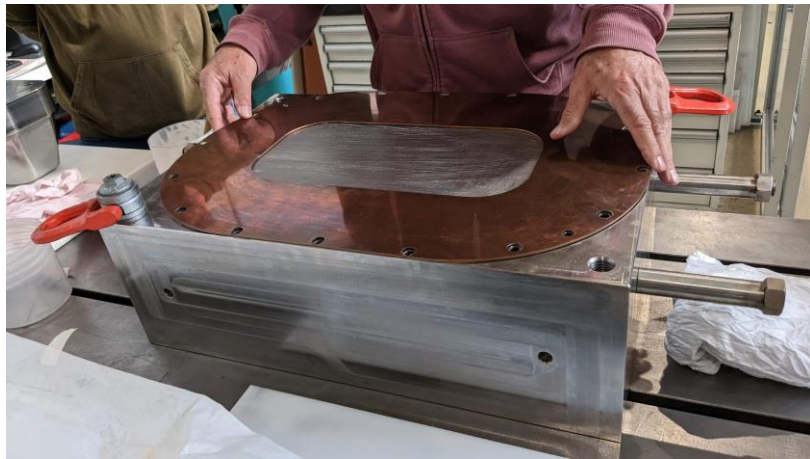
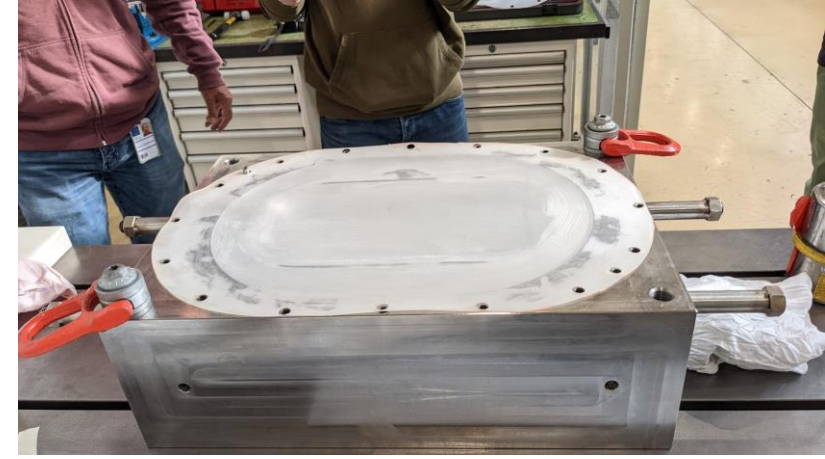
Poles formed with **material from a specific batch** showed **orange peel** appearance and **excessive thickness reduction** on certain regions (+ wrinkles) → **shape accuracy not guaranteed**



CERN-FNAL agreed to perform a forming trial at CERN, comparing two different material batches.

See talks of M. Narduzzi and A. Ratti.

# Challenges with RFD pole forming



Preparation of the RFD Pole forming trials held at CERN Main Workshop (May 2022).

# Challenges with RFD pole forming

Lot 1 – forming OK



Lot 2 – forming NOK



- Same material supplier
- **2 different material lots**
- Same tooling
- Same operators
- Same press machine



**Very different outcome!**

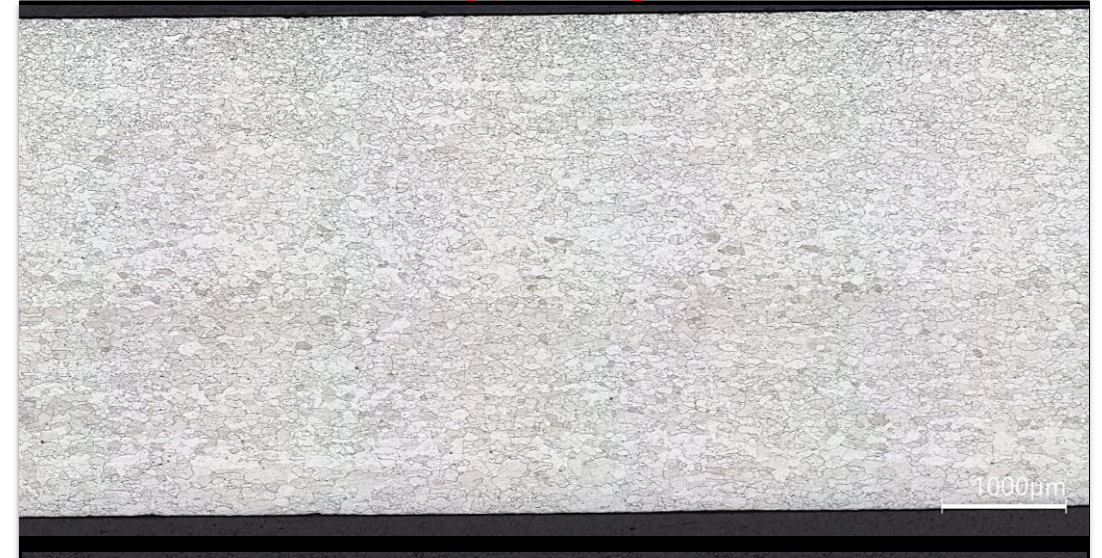
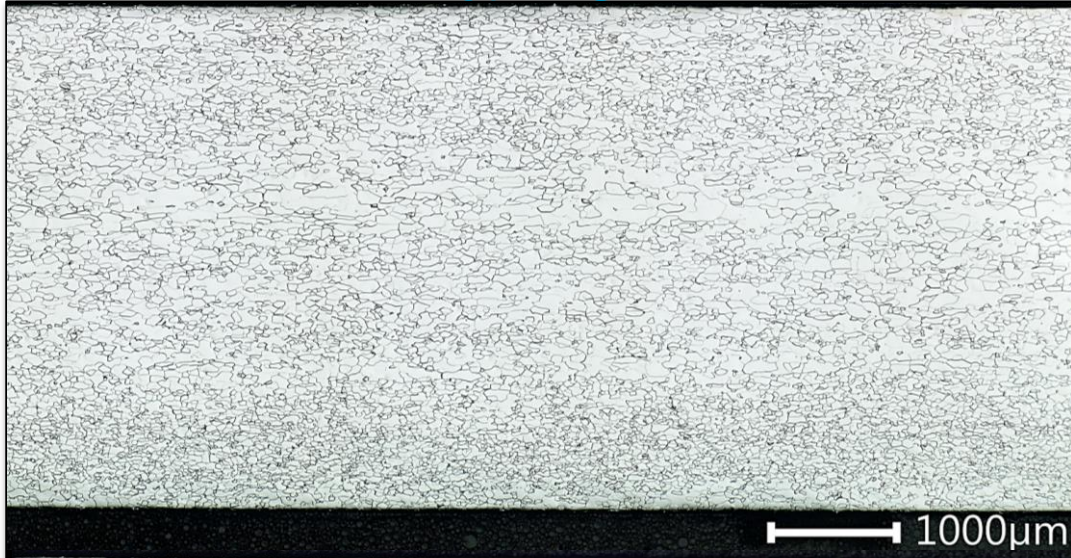
RFD Pole forming trials held at CERN Main Workshop (May 2022).

# Materials investigation – Microstructure check

Lot 1 - OK

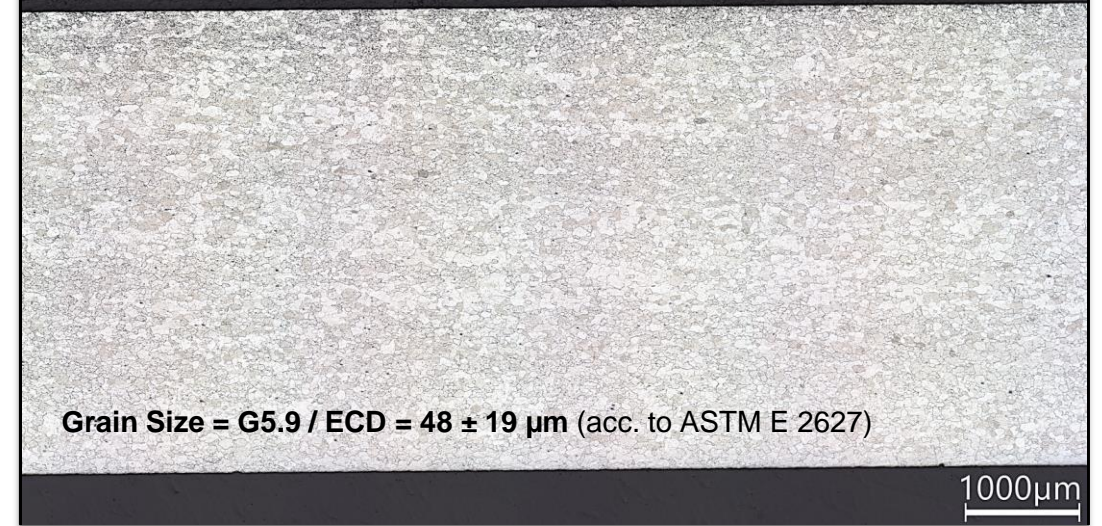
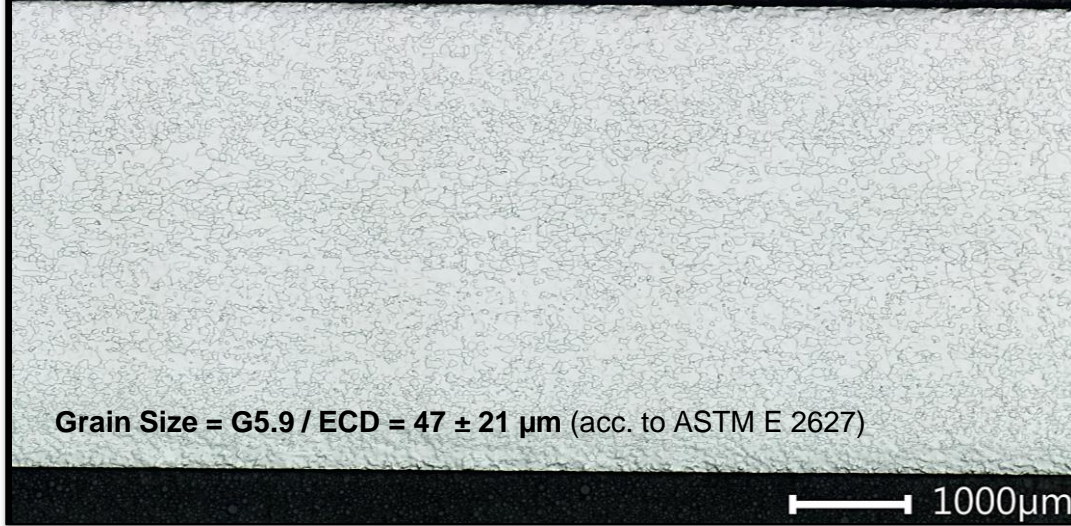
Lot 2 - NOK

ST  
RD



thickness 4mm

ST  
LT



Both sheets show a recrystallized, similar microstructure.

# Materials investigation – Mechanical tests

## Lot 1 - OK



Specimen designation	$R_{p0.2}$ MPa	$R_m$ MPa	$A_g$ %	$A_{24,45mm}$ %	$n_{0.02-0.20}$	$R_{p0.2}/R_m$
2082401_1L	53.0	161.6	30.9	52.6	0.38	0.33
2082401_2L	49.5	159.4	33.0	62.3	0.38	0.31
2082401_3S	51.4	167.7	31.3	59.2	0.37	0.31
2082401_4S	53.6	166.6	27.9	55.1	0.35	0.32

Material shows low  $R_{p0.2}$  (<65 MPa)

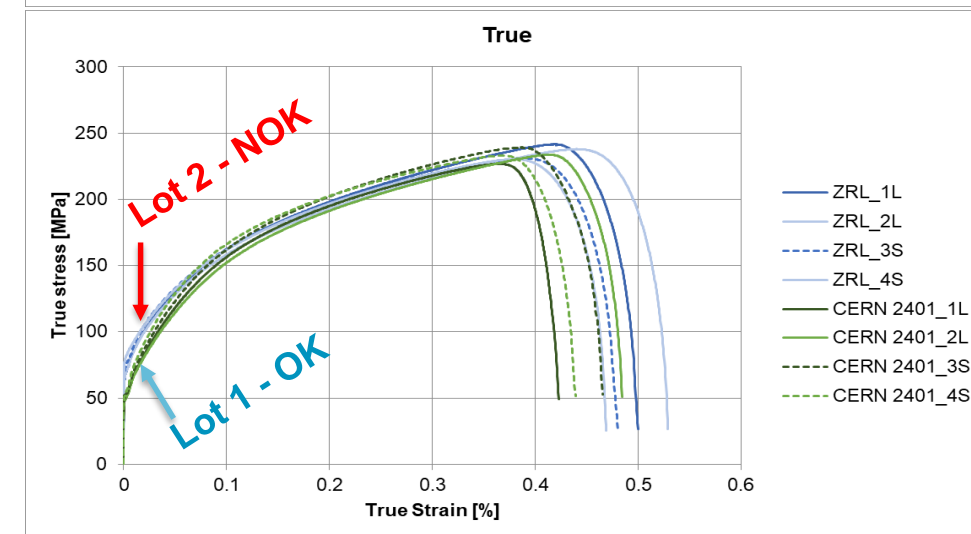
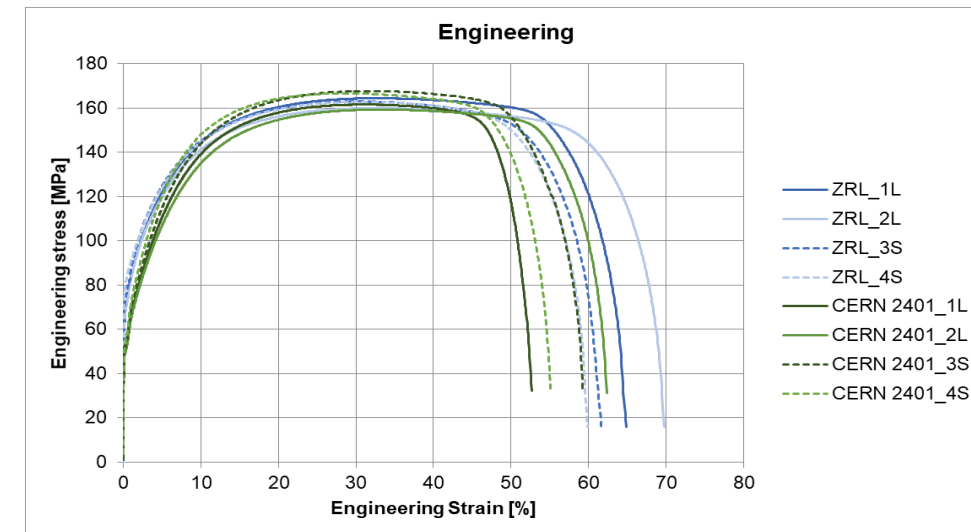
## Lot 2 - NOK



Specimen designation	$R_{p0.2}$ MPa	$R_m$ MPa	$A_g$ %	$A_{24,87mm}$ %	$n_{0.02-0.20}$	$R_{p0.2}/R_m$
ZRI_1L	68.7	164.5	32.4	64.9	0.30	0.42
ZRI_2L	69.1	160.0	32.3	69.7	0.29	0.43
ZRI_3S	74.0	163.0	29.5	61.6	0.28	0.45
ZRI_4S*	80.9	162.8	29.9	59.9	0.28	0.50

Material complies with CERN spec 3300 Ed.4!

'n' value seems to be significantly different, as well as the ratio  $R_{p0.2}/R_m$



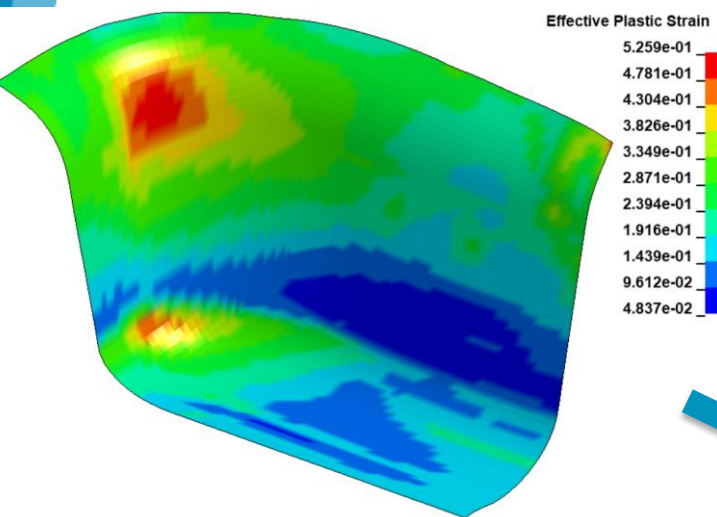
Note:  $A_g$  → elongation (engineering) at maximum force

$n_{0.02-0.20}$  → strain hardening index (interval from 0.02 to 0.2 true strain)

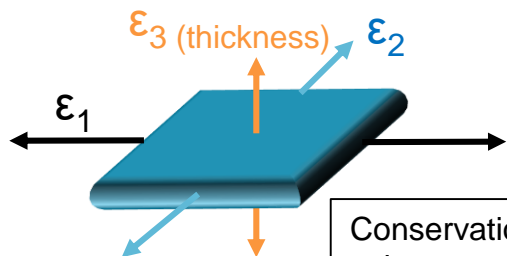
\*: for sample ZRI\_4S, the same test speed (0.05 1/min) was used during the whole test.

# Challenges with RFD pole forming – FE simulations

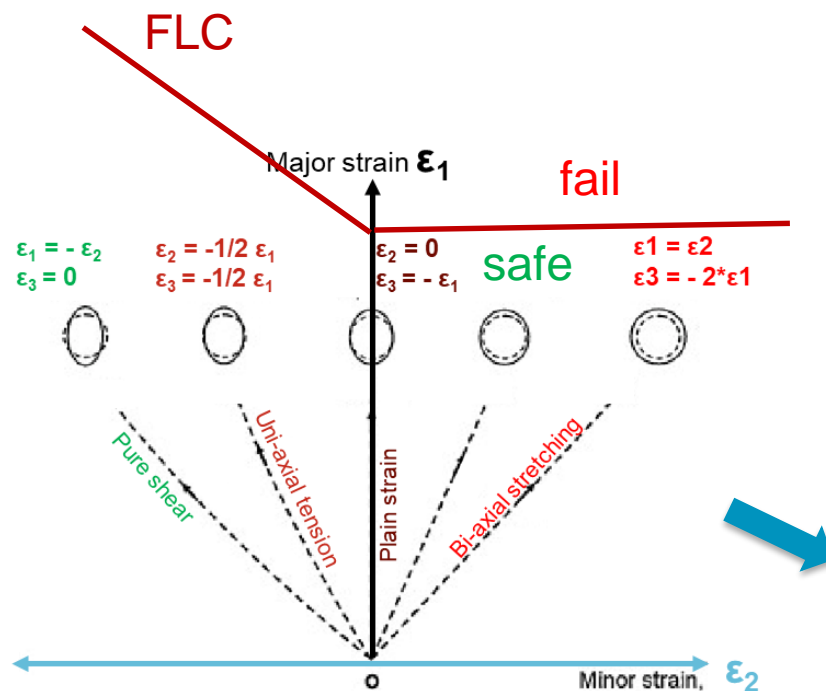
- FE simulations together with a failure criteria for membrane-like components (e.g. Forming Limit Diagram) can help understanding and optimizing the formability.



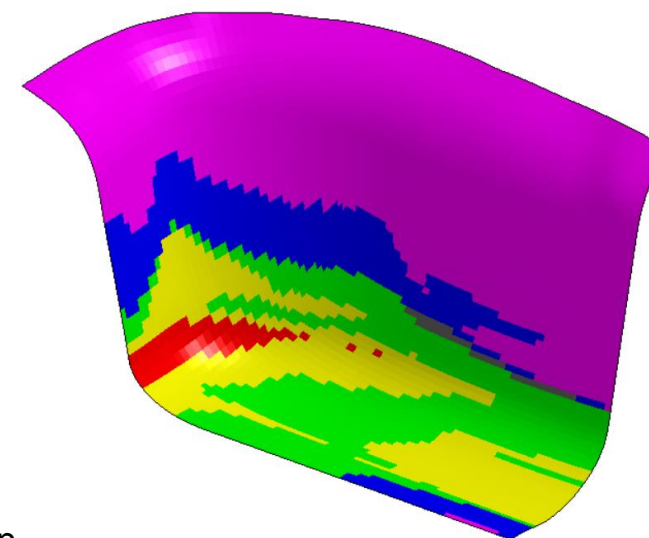
FE simulation with LS-DYNA, thanks to A. Amorim Carvalho, M. Garlasche



Conservation of volume :  
 $\epsilon_1 + \epsilon_2 + \epsilon_3 = 0$



Relationship between  $\epsilon_1$ ,  $\epsilon_2$  and  $\epsilon_3$  along the deformation process (i.e. **strain path**) matters:



FLD Standard Formability key	
Cracks	Red
Risk of cracks	Yellow
Severe thinning	Orange
Safe	Green
Inadequate stretch	Grey
Wrinkling tendency	Blue
Wrinkles	Purple

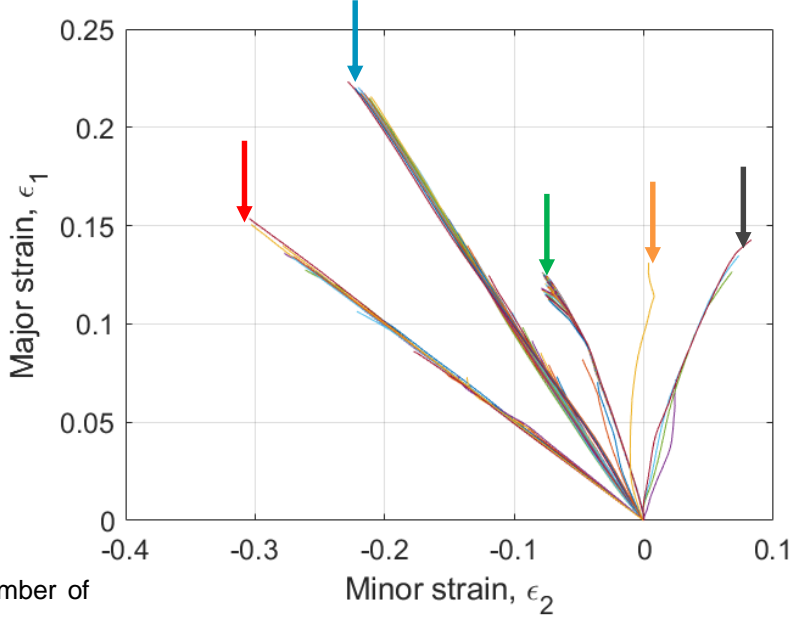
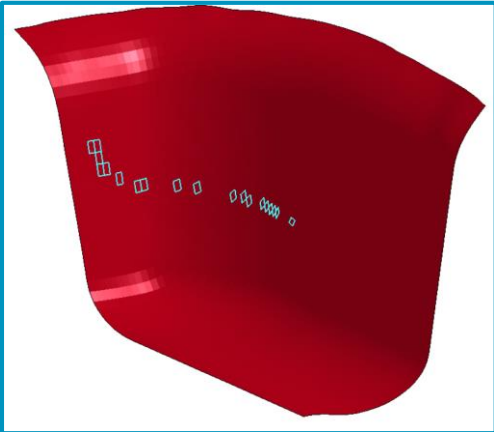
Thanks to J. Swieszek & E. Cano-Pleite

# Challenges with RFD pole forming – FE simulations

Uniaxial compression



Pure shear



\* Some elements were manually selected, the number of curves in the plot and the number of elements in the snapshots may not match.

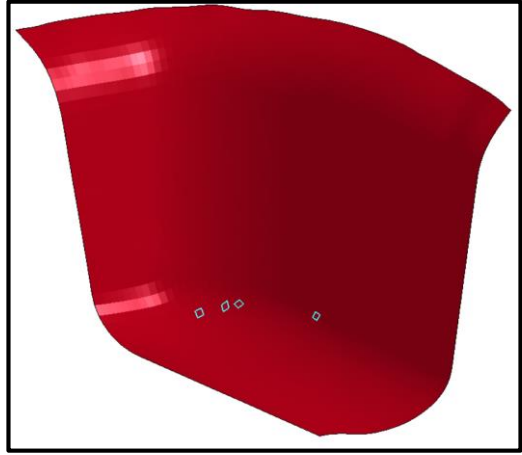
Uniaxial tension



Plane strain



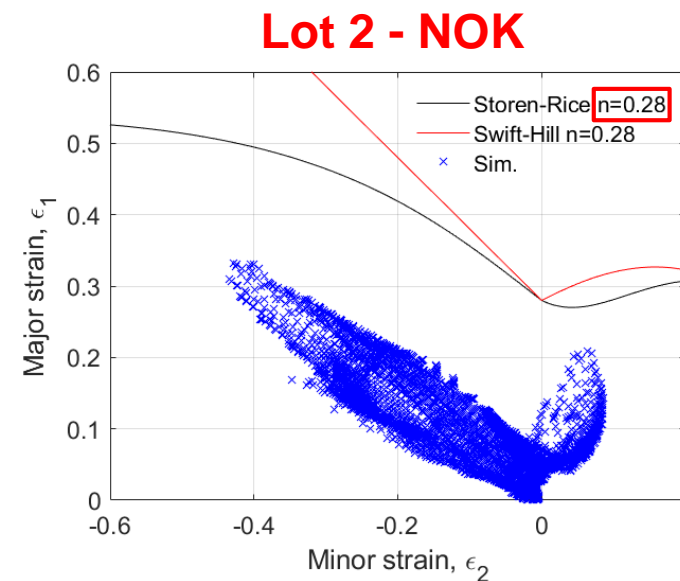
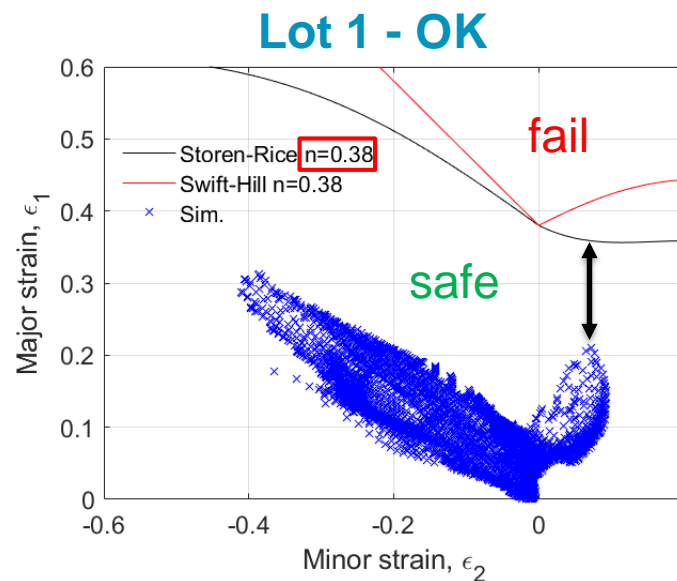
Biaxial tension



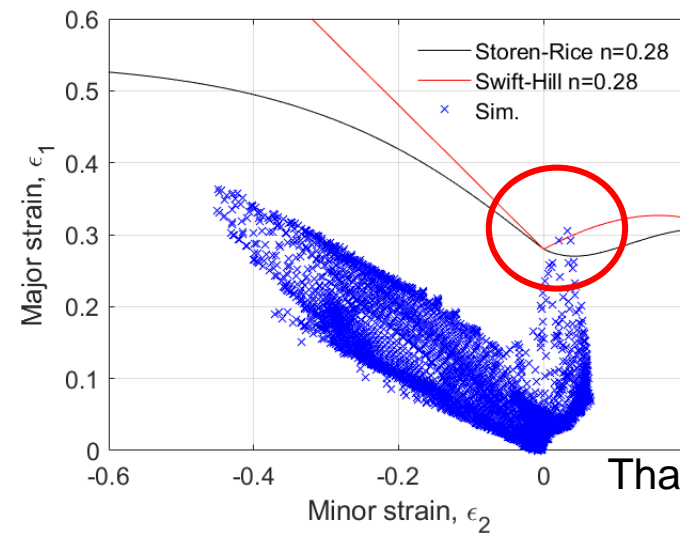
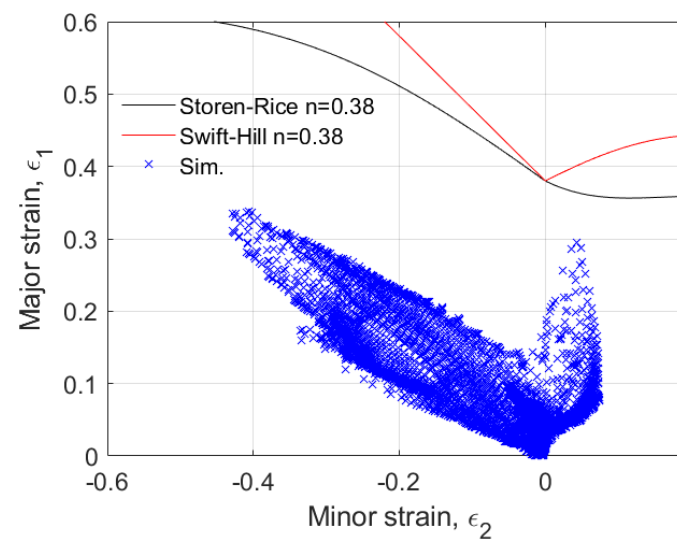
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# Challenges with RFD pole forming

Coefficient of Friction = 0.03



Coefficient of Friction = 0.18

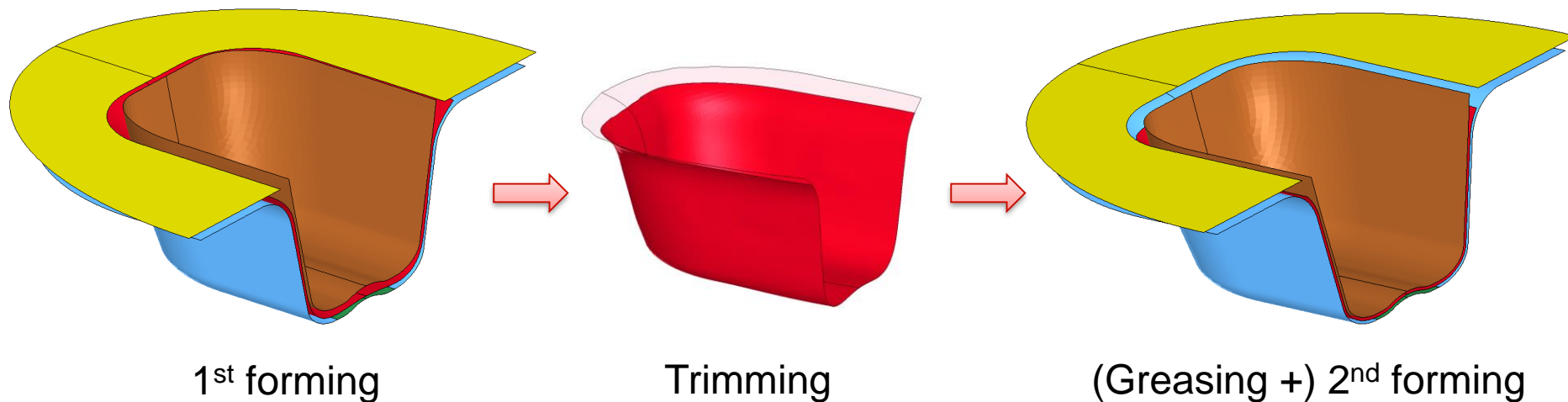


Thanks to J. Swieszek & E. Cano-Pleite



# Modified 2-step forming process

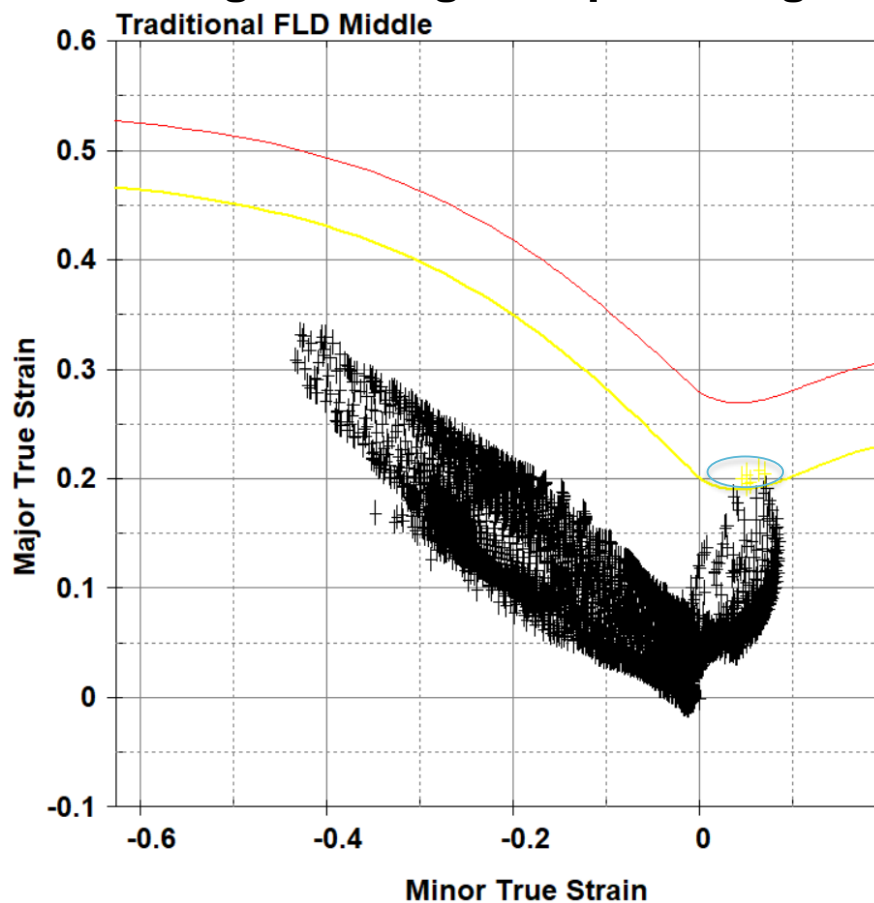
- Initial forming up to 5-15 mm from the end of the punch stroke.
- Trimming so that at least the nominal shape +25 mm of material is available.



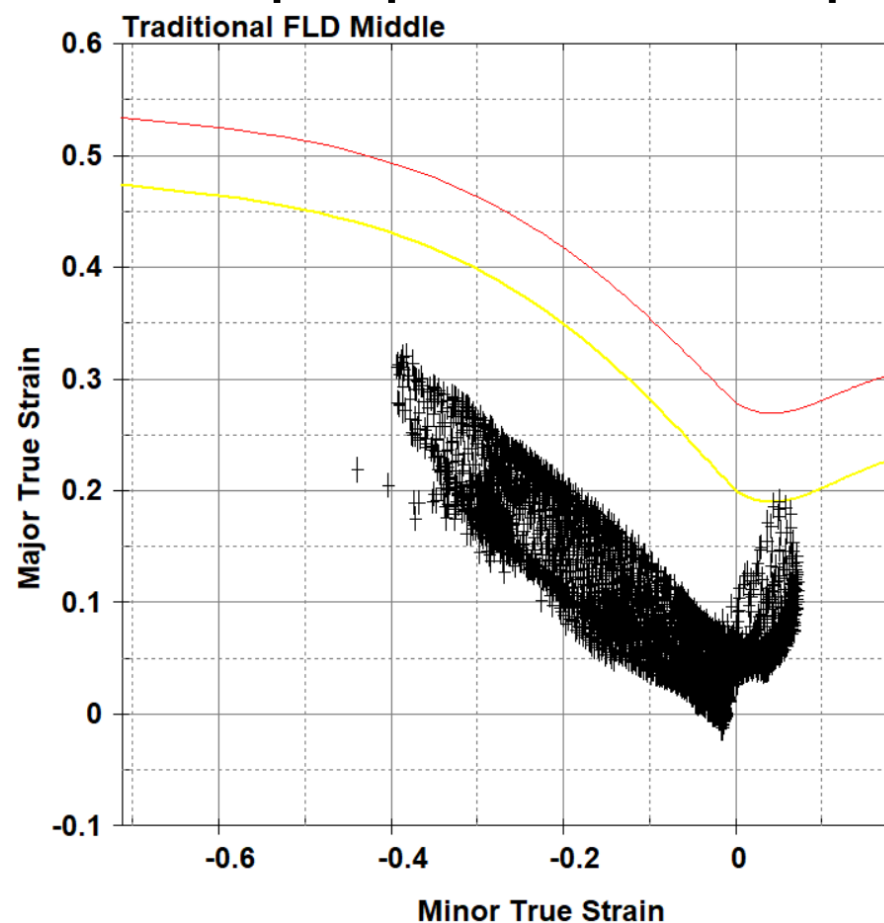
Thanks to J. Swieszek &  
E. Cano-Pleite

# Modified 2-step forming process

Original: single-step forming



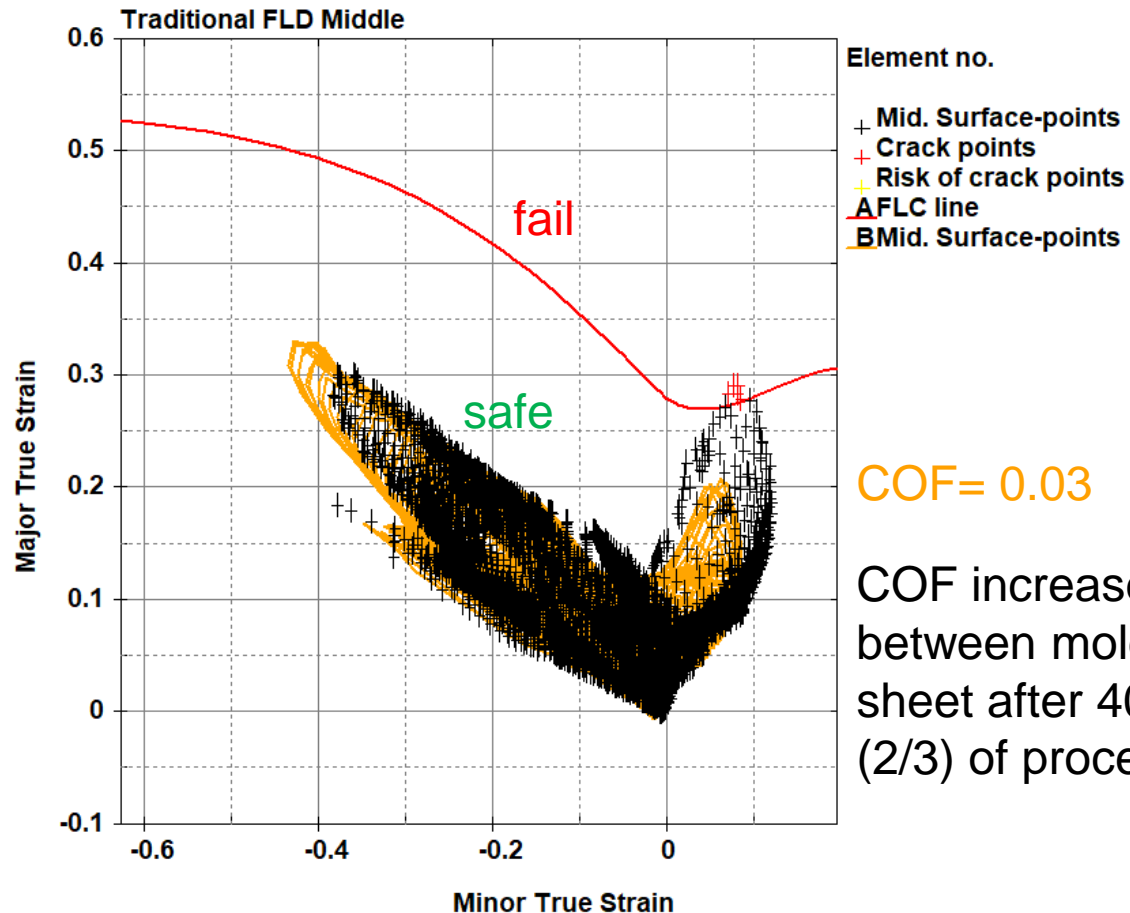
1<sup>st</sup> step stop at 15mm + 2<sup>nd</sup> step



The 2-step forming slightly reduces the value of strain in the conflictive region.

Thanks to J. Swieszek & E. Cano-Pleite

# Summary – Lot 2 NOK with increased CoF



Thanks to J. Swieszek &  
E. Cano-Pleite

The **2-step forming** process is **beneficial** mainly due to the **reduction in friction of coefficient** !  
The modified **forming procedure** by the **US-AUP** contractor has been validated by FE simulations.  
Some mechanical properties would play a significant role (**Rp0.2/Rm** & **strain hardening coefficient n**).

The CERN austenitic SS specifications (i.e. 1.4435, 1.4429) focus on several critical points:

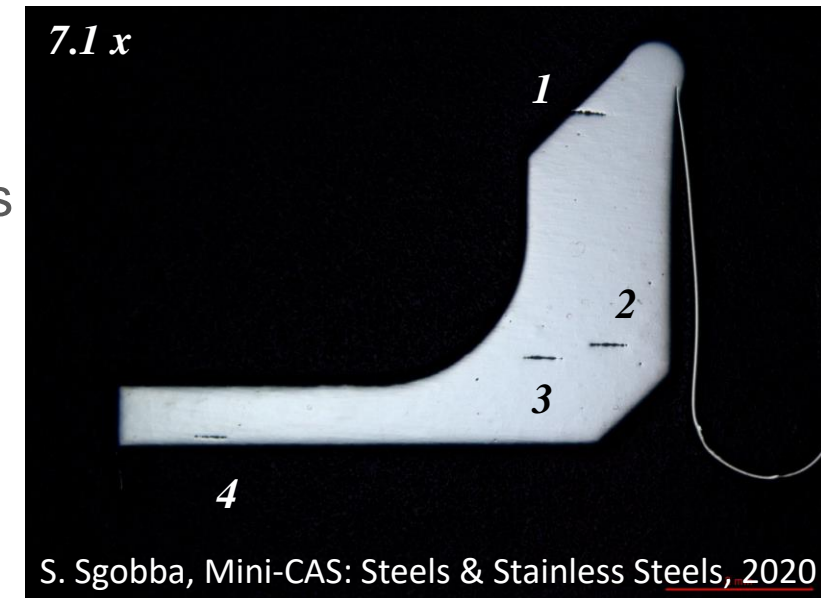
- **Chemical composition (grade) →**
  - **Avoid martensitic transformation**  $\gamma$  (austenite)  $\Rightarrow$   $\alpha'$  (martensite) (spontaneous when cooling or strain induced). General rule: the more alloying elements the better.
  - **Keep low magnetic permeability** (given by initial ferrite + 'transformed' austenite  $\rightarrow$  martensite).
  - Keep low P + S content  $\rightarrow$  improve **weldability** (avoid hot cracking)
  - Co content  $\rightarrow$  reduce **activation of cryomodule parts** (Derogation by HSE: up to 0.3 wt% allowed!)

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- **Keep low magnetic permeability** (given by initial ferrite + 'transformed' austenite  $\rightarrow$  martensite).
- Keep low P + S content  $\rightarrow$  improve **weldability** (avoid hot cracking)
- Co content  $\rightarrow$  reduce **activation of cryomodule parts** (Derogation by HSE: up to 0.3 wt% allowed!)

- **Grain size, inclusion content (& 3D forging) → UHV applications**
- **Mechanical properties and UT → structural soundness**
- **Certificate 3.1 (EN 10204) → ensure quality & traceability**



# Cryomodule : stainless steel specification challenges

via DeLong equation - ferrite content estimation:

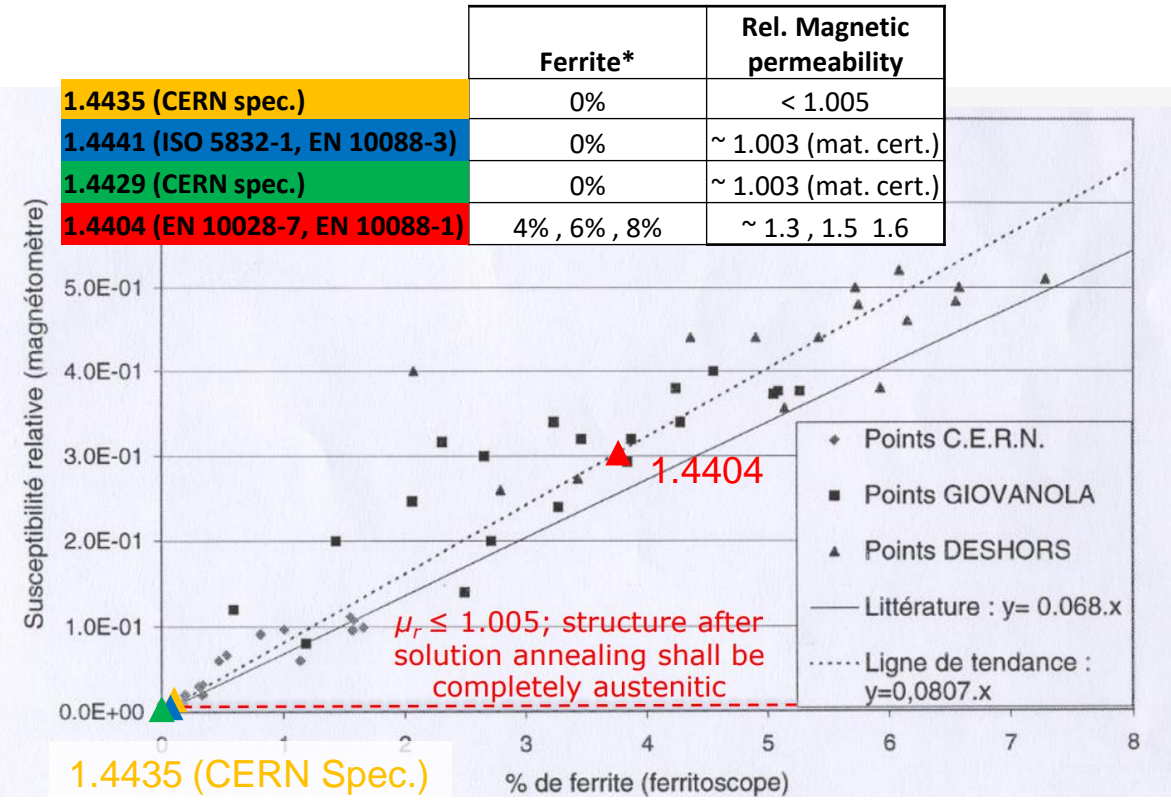
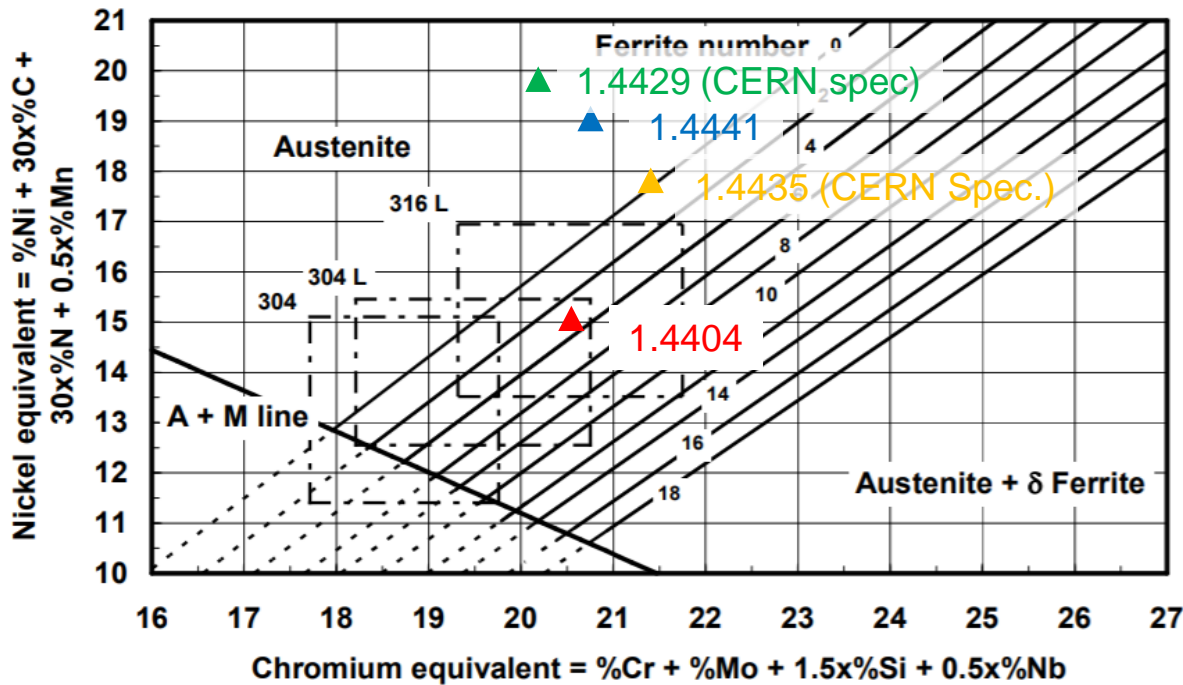


Fig. 5: Diagram for the determination of the ferrite content in austenitic stainless steel weld metal according to DeLong [41,42]. Diagrams more adapted to high N stainless steels are the ones of Hull [43] and Espy [44].

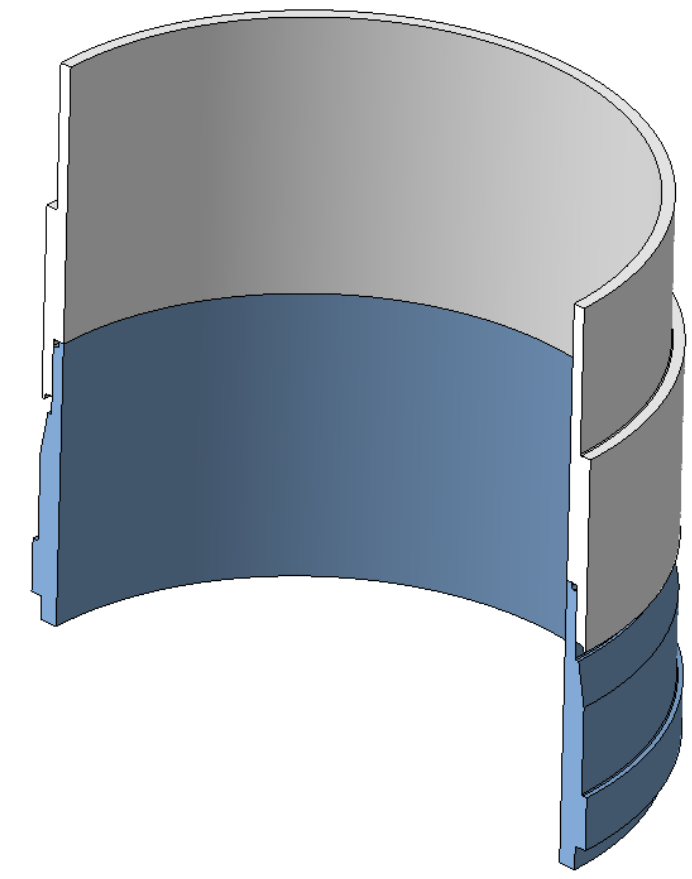
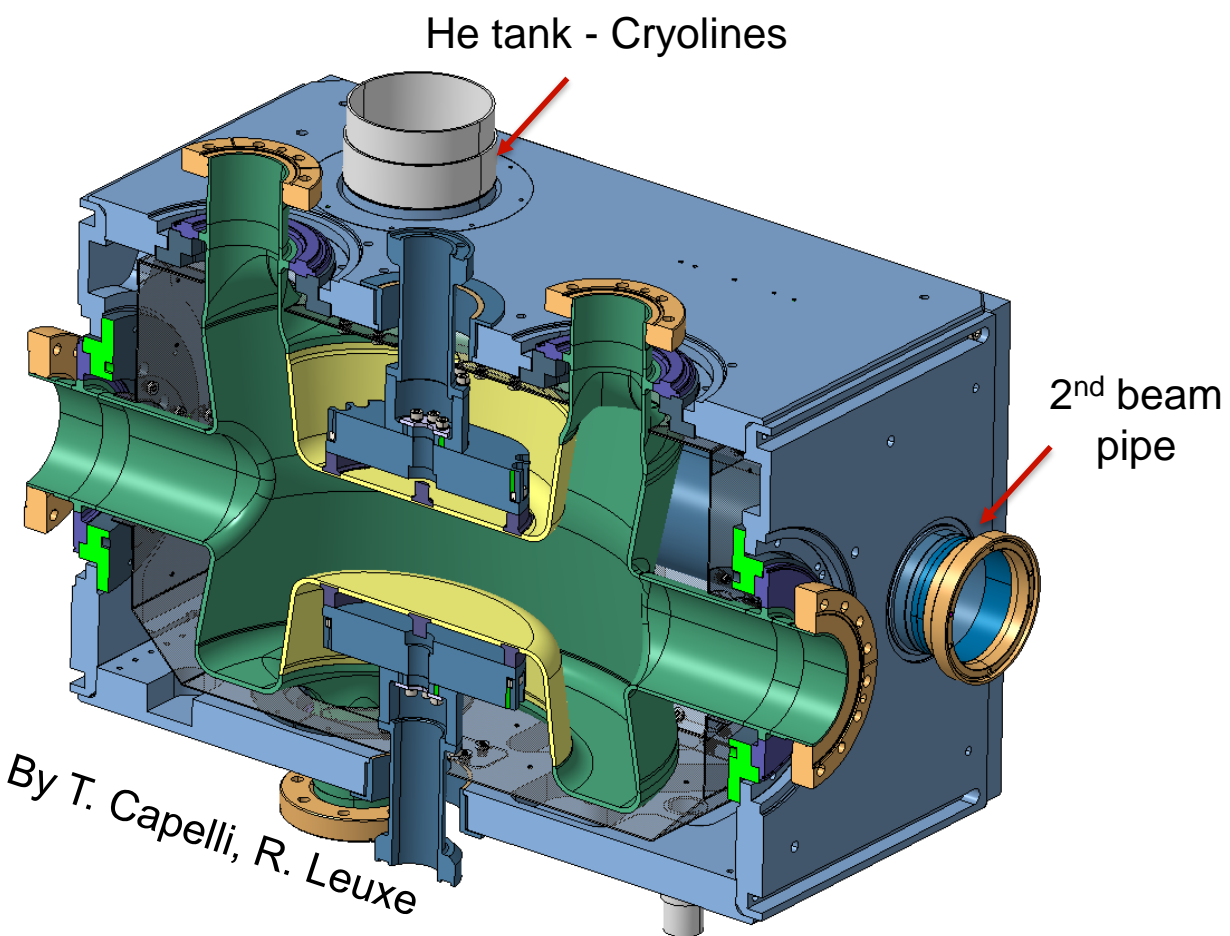
**Each ferrite % contributes ~0.08 to the relative magnetic permeability**

- Anticipate the purchase of specific grades (1.4429, 1.4435, 1.4441..).
- Check carefully Material Certificate vs. Eng.Spec & Application (UHV, cryogenics, position w.r.t. cavities..).
- In case of any doubts contact WP4 cryomodule responsables (M. Garlasche, T. Capelli)

# Advanced techniques for characterizing parts and joints: update on several case studies

# SS-Ti bimetallic transitions – new results

Brazing developed by F. Motschmann & MME-FW team

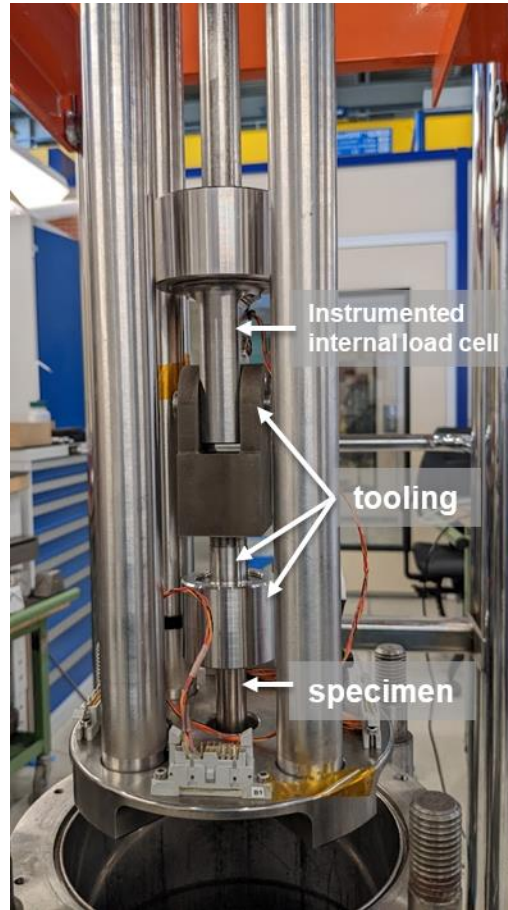
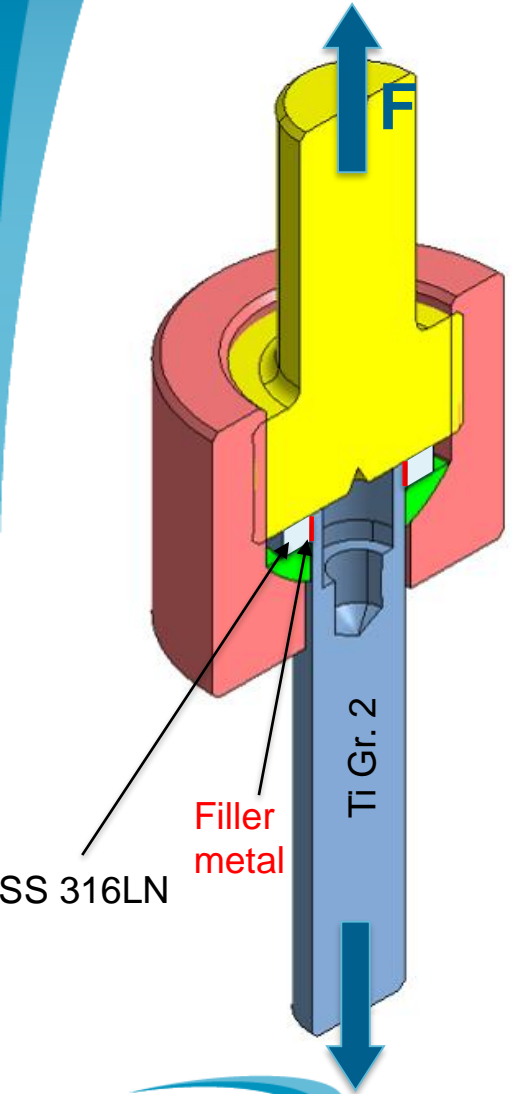


A dedicated qualification campaign was performed, see EDMS **2271509**.  
But mechanical tests set-up at 4K was not optimized (shear + non-desired bending)



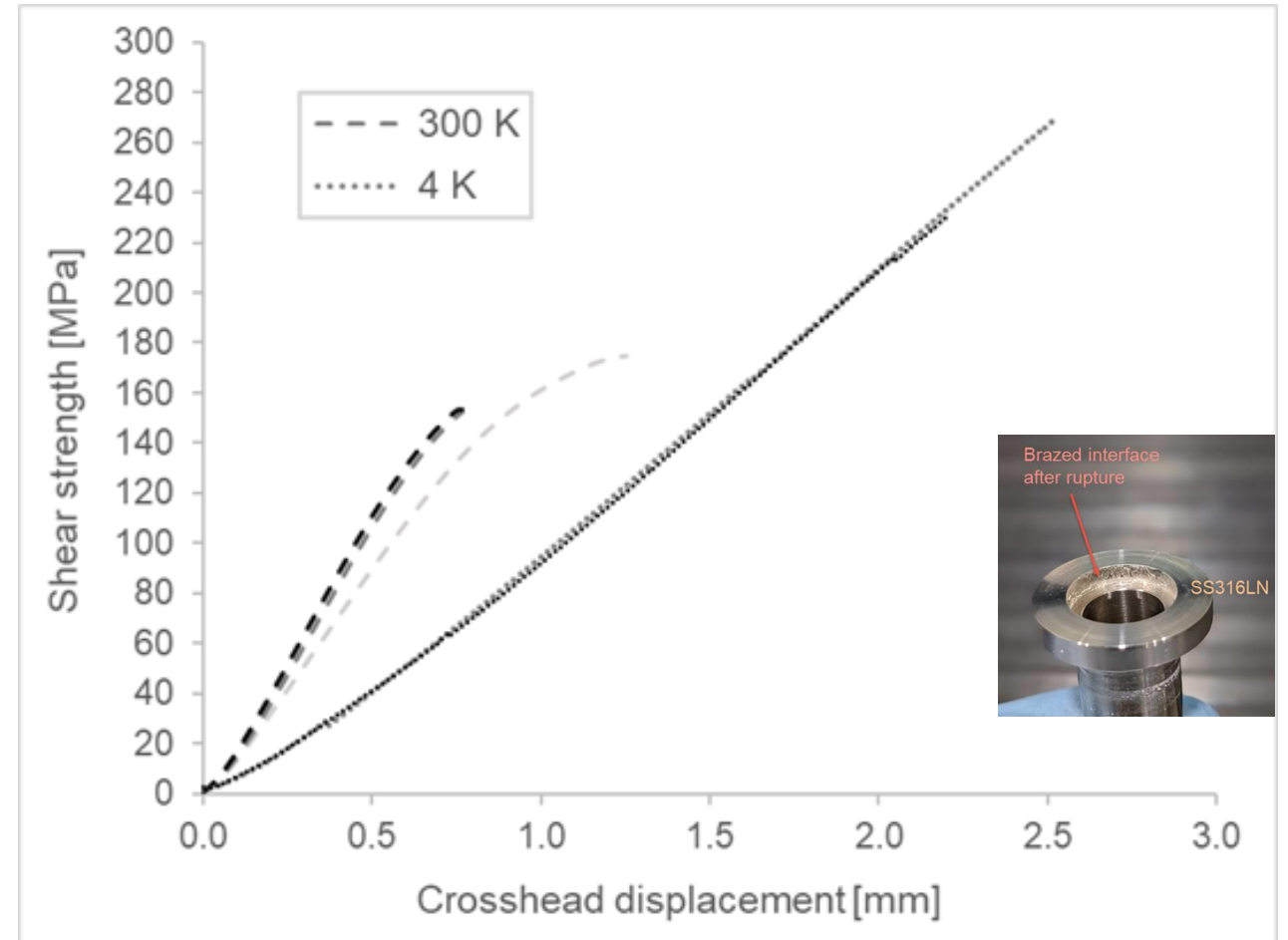
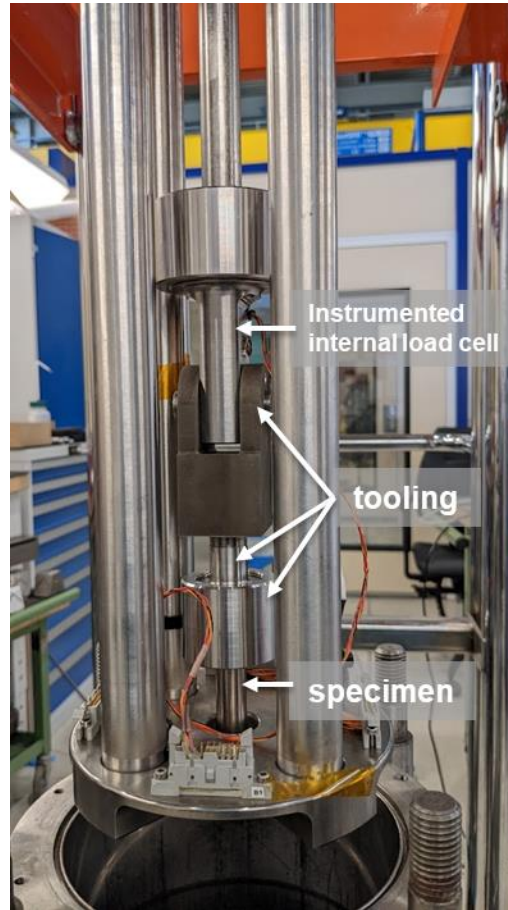
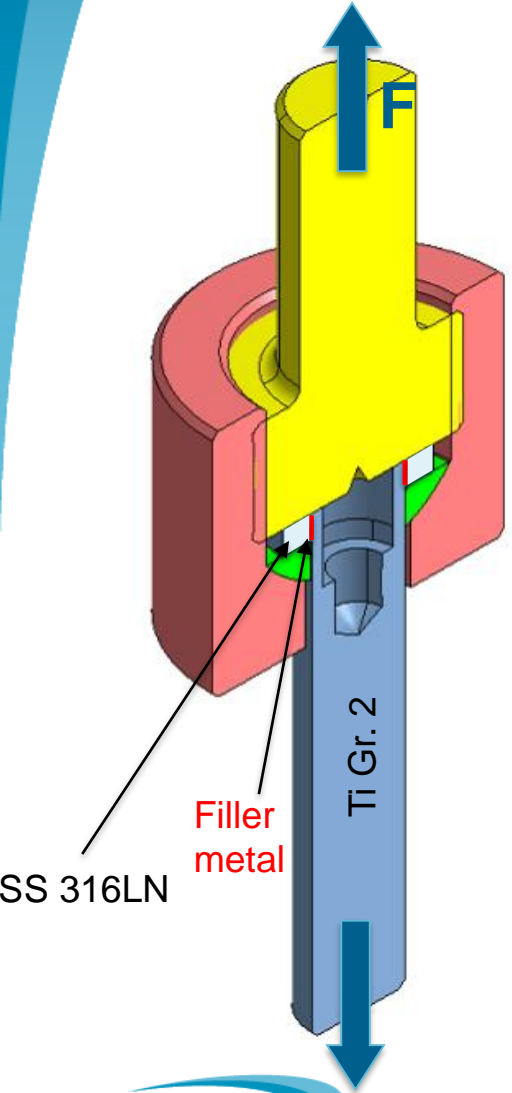
# SS-Ti bimetallic transitions – tests at 4K

New dedicated specimens and tooling adapted from ISO 5187



# SS-Ti bimetallic transitions – tests at 4K

New dedicated specimens and tooling adapted from ISO 5187



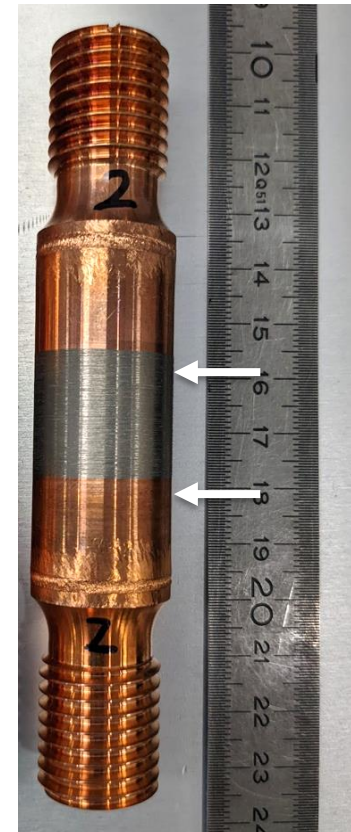
**Mechanical tests at 4K show significant shear strength improvement (min. 231 MPa) with respect to tests at RT (min. 153 MPa)**

“Vacuum Brazed Titanium-Stainless Steel Transitions for Cryogenic Applications”, Presented by F. Motschmann at the 13<sup>th</sup> International Conference on Brazing (LOT 2022), Aachen, Germany, 2022. Full results to be published.

# Cu-Nb Electron Beam Weld for the pick-up antenna



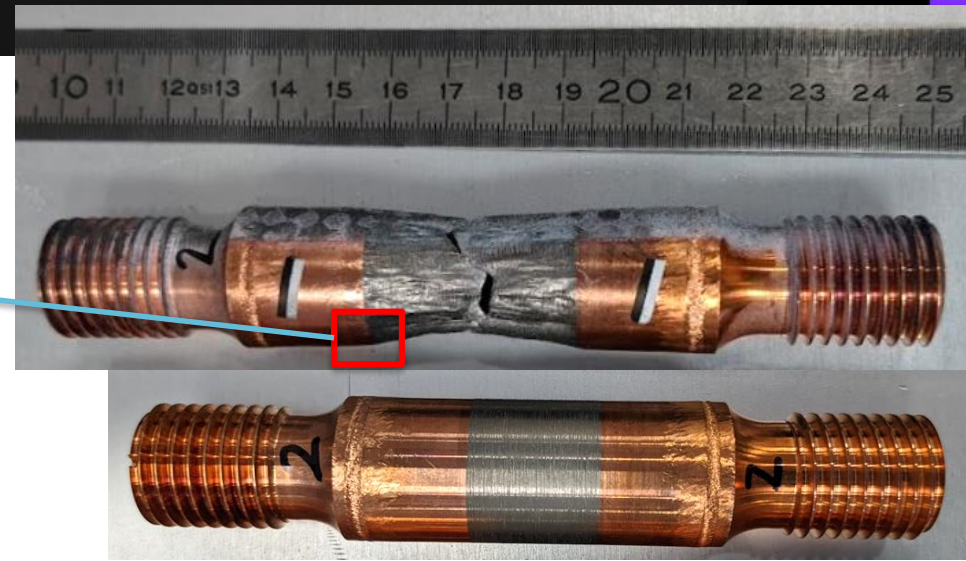
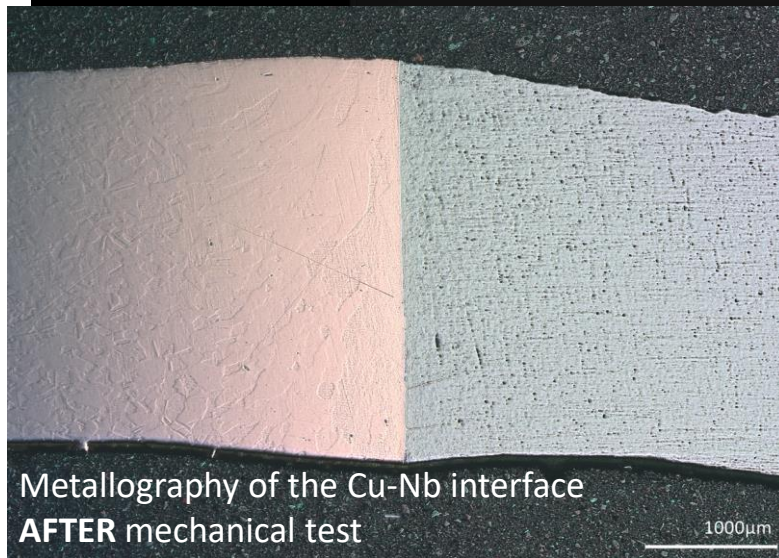
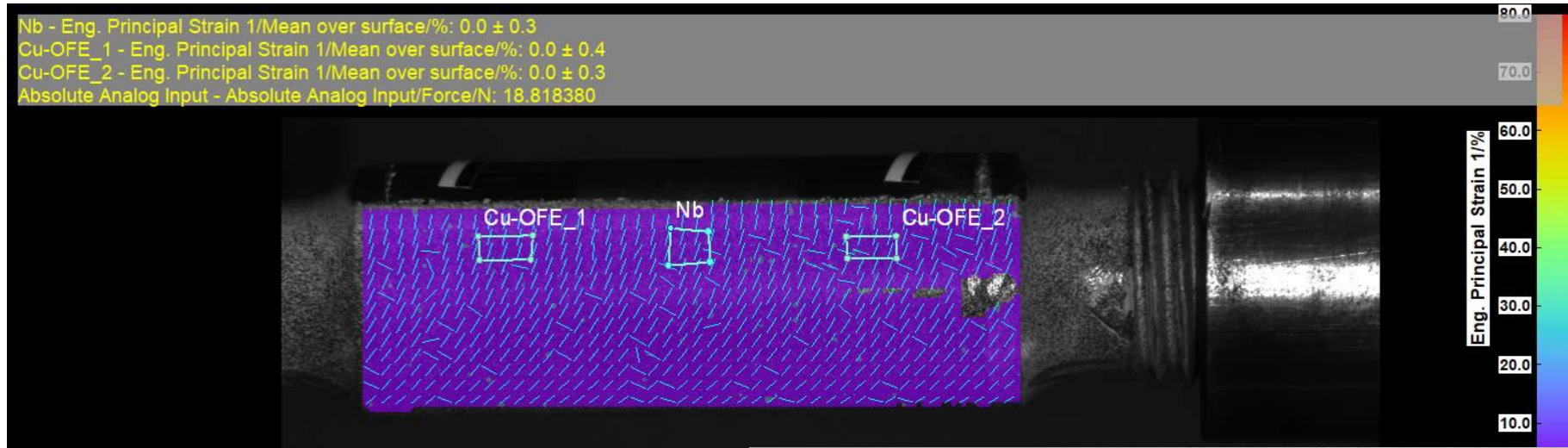
- Parts are leak tight but **mechanical robustness of interface was unknown.**
- New **dedicated specimens were conceived.**



Courtesy of S. Barriere, T. Demaziere.

Cu-Nb EB weld by T. Demaziere.

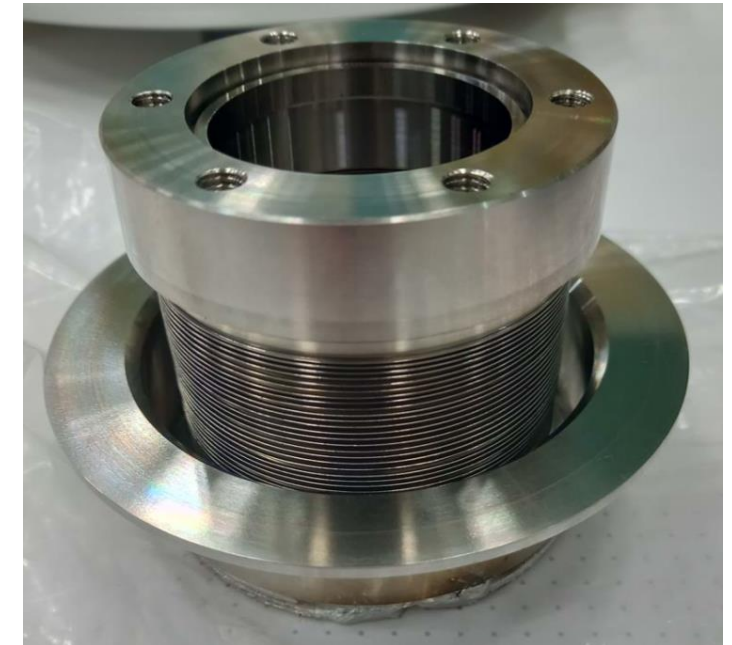
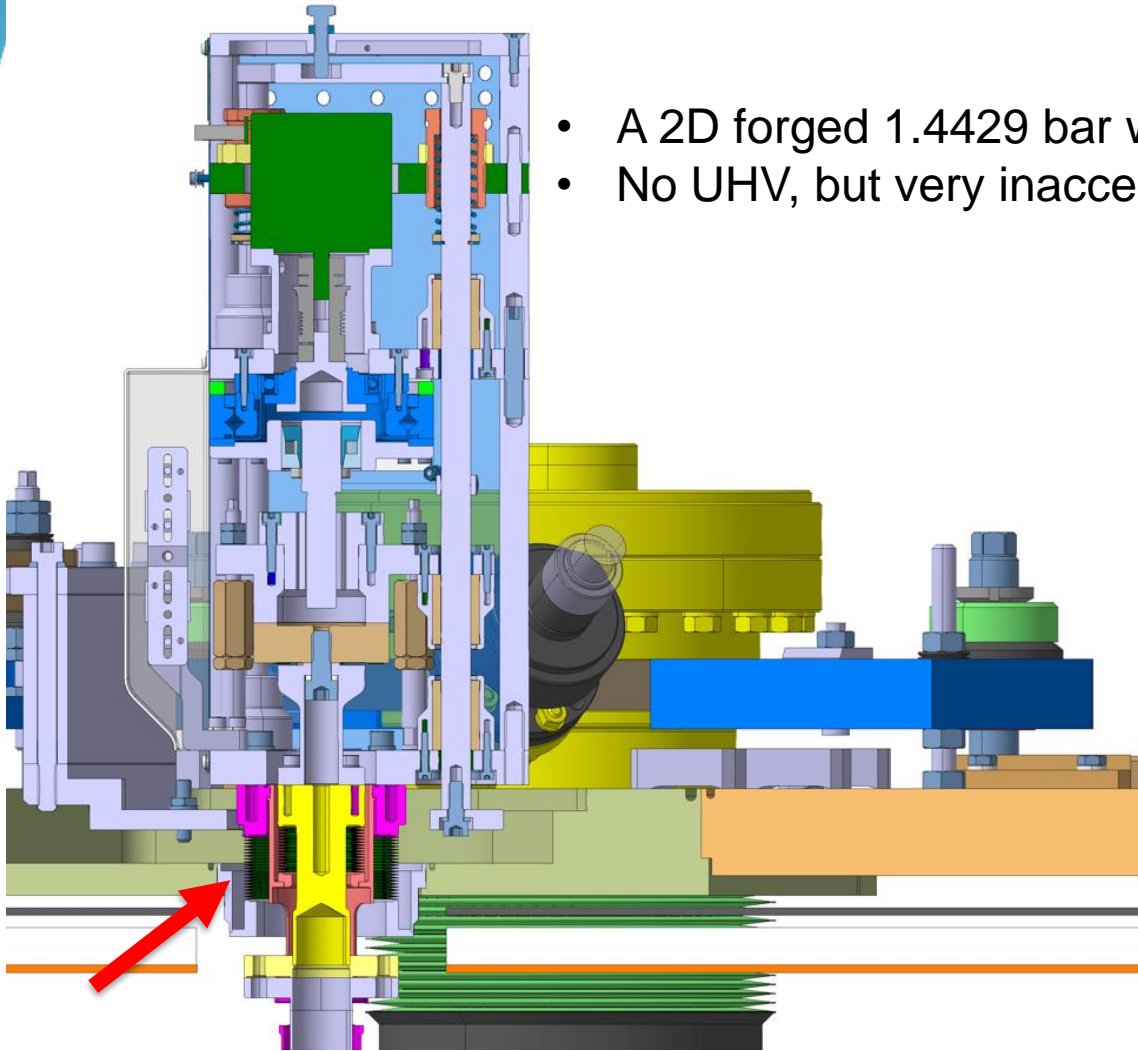
# Cu-Nb Electron Beam Weld for the pick-up antenna - DIC



The Nb-Cu EB weld is mechanically sound and even compliant with the Nb-Nb EBW qualification requirements! ( $R_m > 118$  MPa)

# Tuner actuation bellows

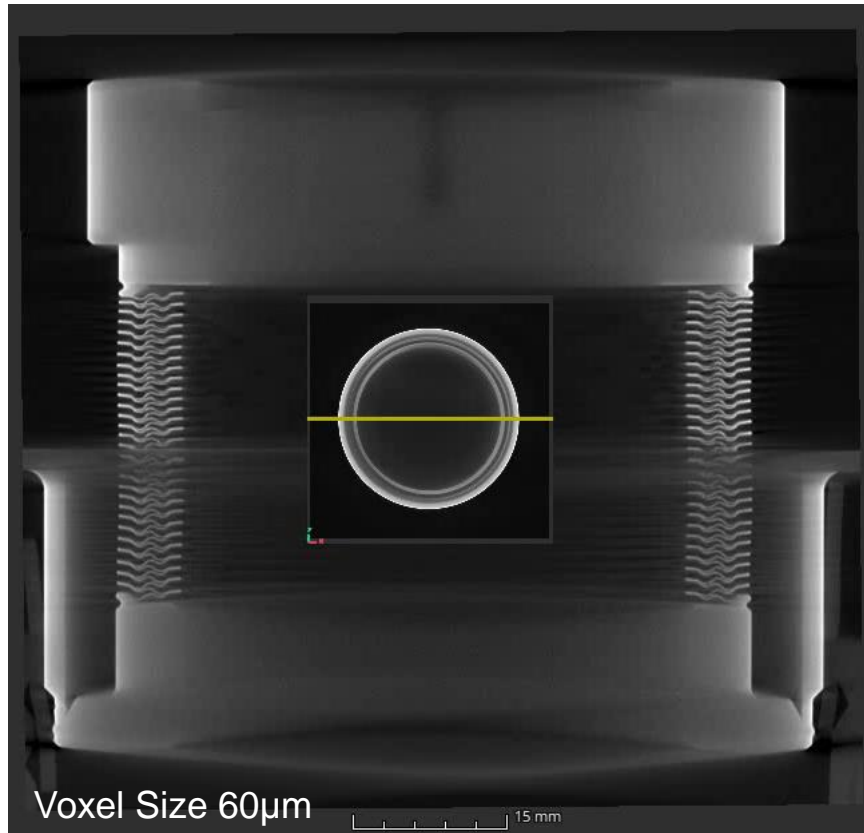
- A 2D forged 1.4429 bar was employed.
- No UHV, but very inaccessible part once is assembled.



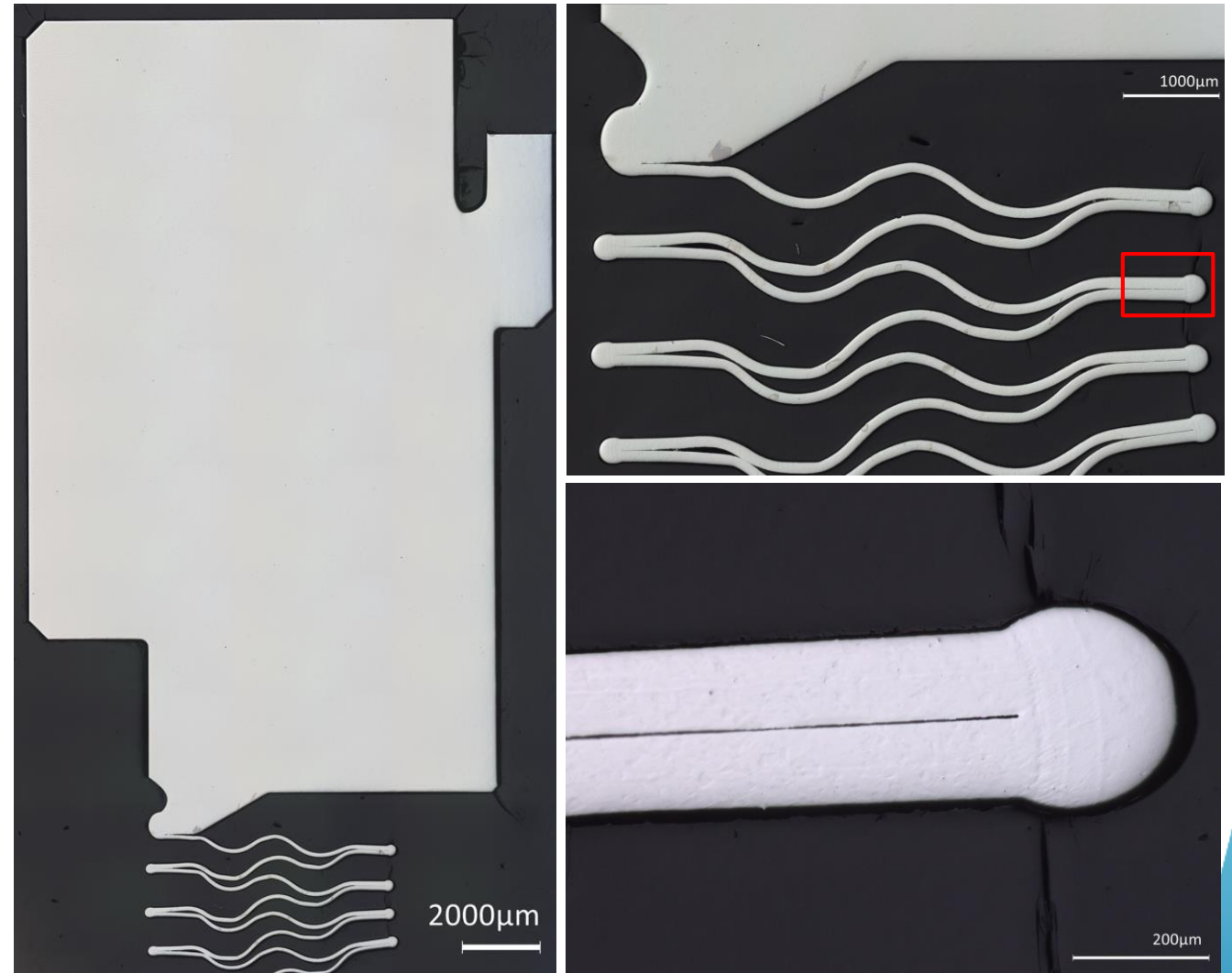
By K. Artoos, T. Capelli.

# Tuner actuation bellows - $\mu$ C Tomography and metallography

A 2D forged 1.4429 bar was employed.



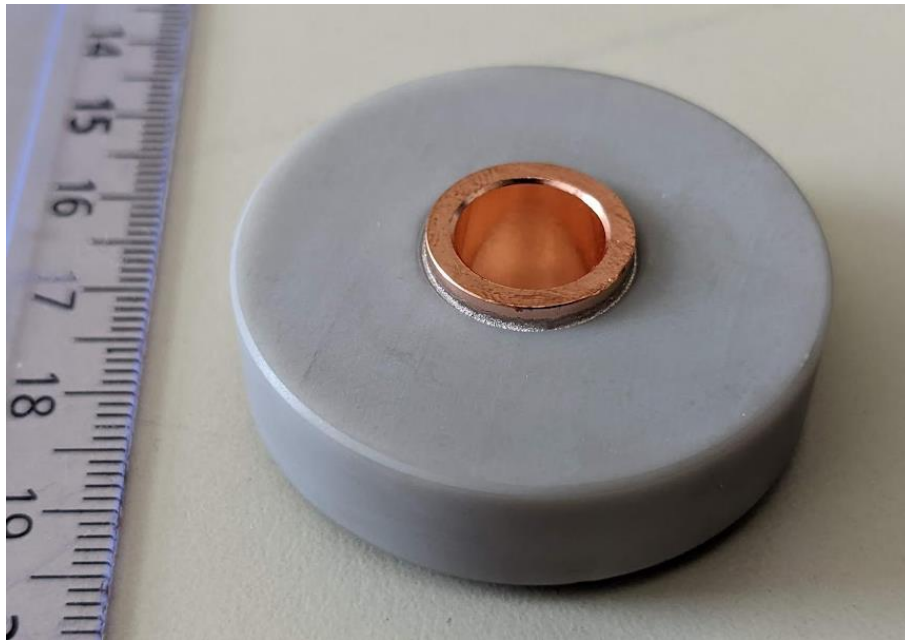
Thanks to M. Celuch



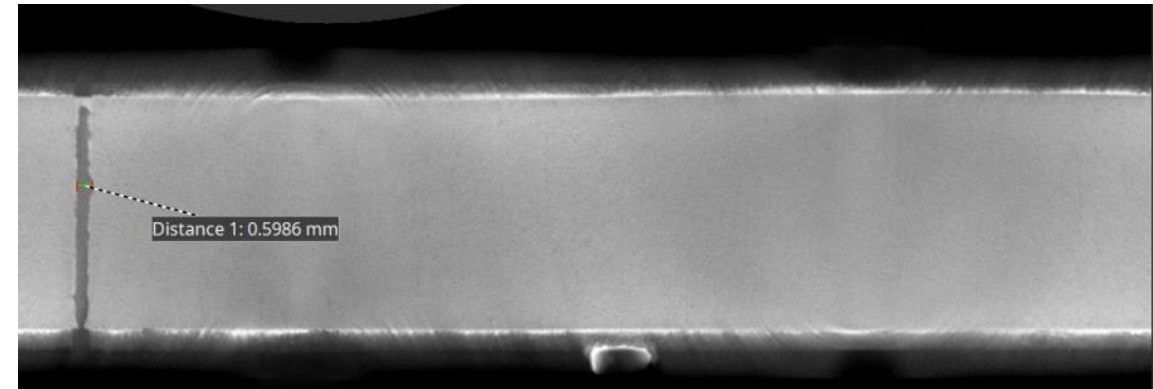
Very clean steel (316LN - 1.4429). Absence of aligned non-metallic inclusions despite being a 2D forged bar!

# AlN-Cu brazed joints for coaxial lines - $\mu$ C Tomography

Brazing by F. Motschmann

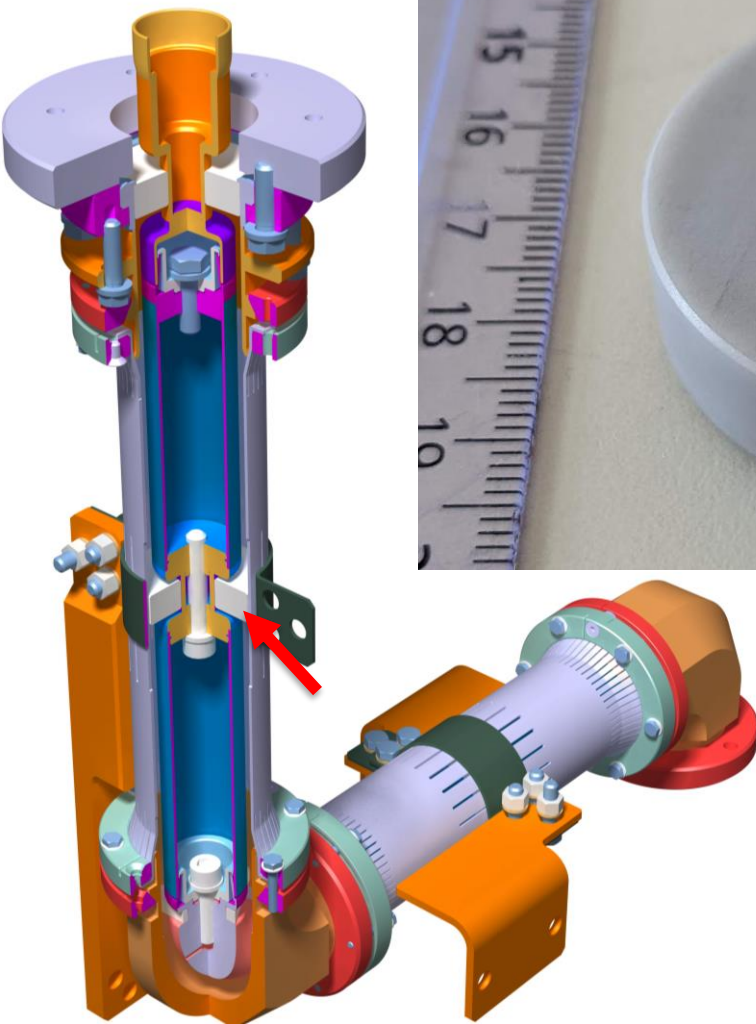
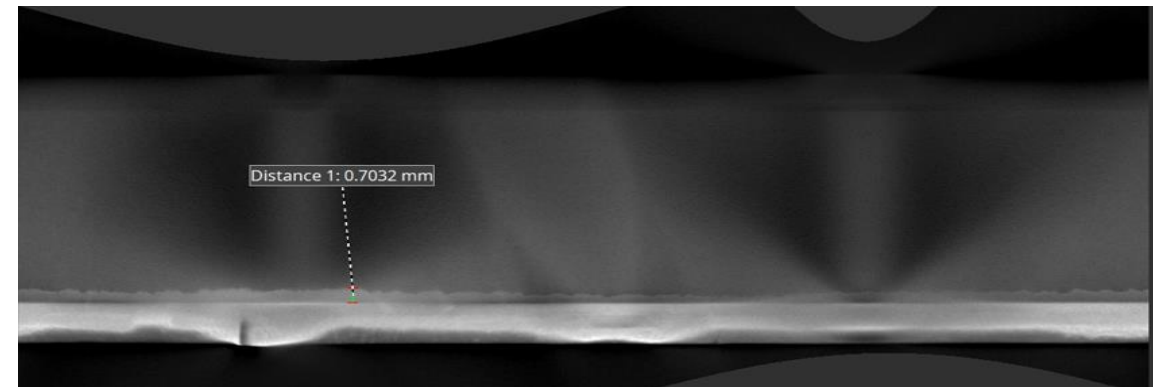


Foil 50  $\mu$ m



Vs.

Wire D0.75 mm

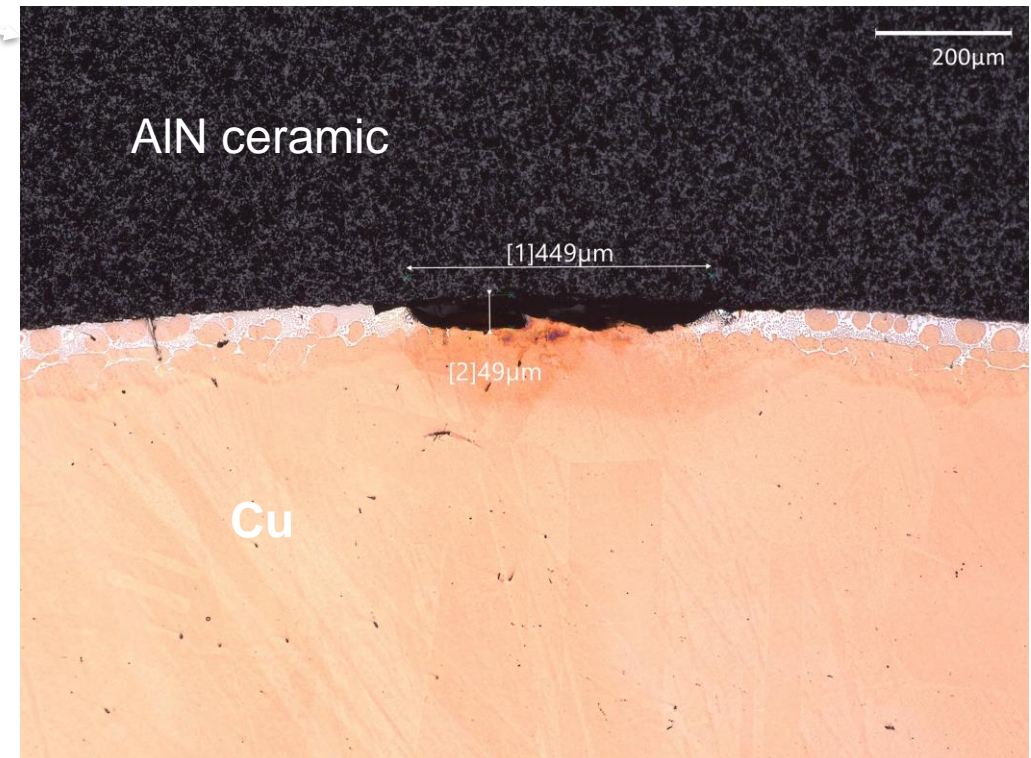
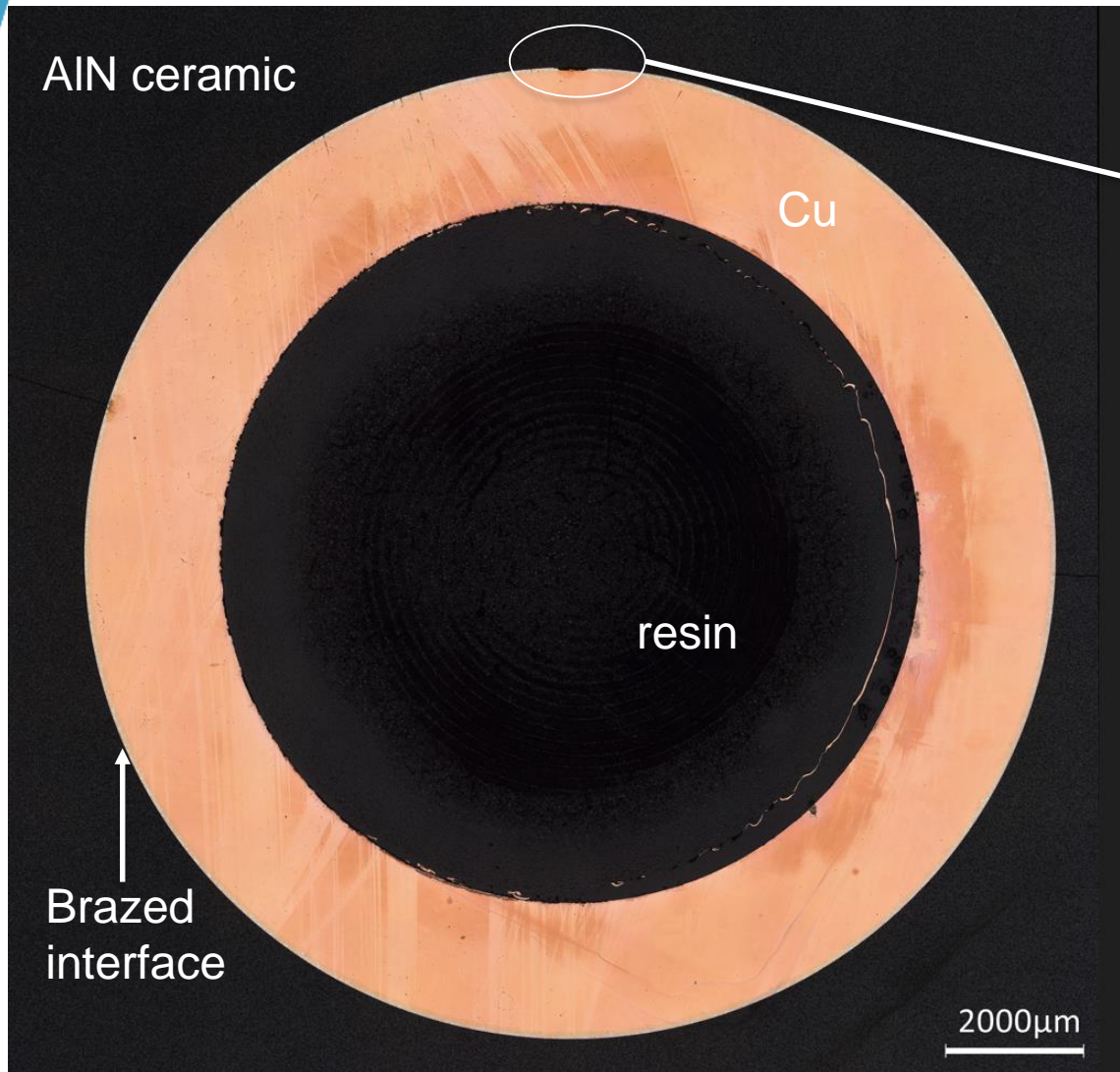


By T. Capelli

CT by M. Celuch.  
EDMS 2736630

# AlN-Cu brazed joints for coaxial lines

Brazing by F. Motschmann



uCTomography allowed to easily identify defects and optimize the brazing strategy.

Thanks to K. Buchanan

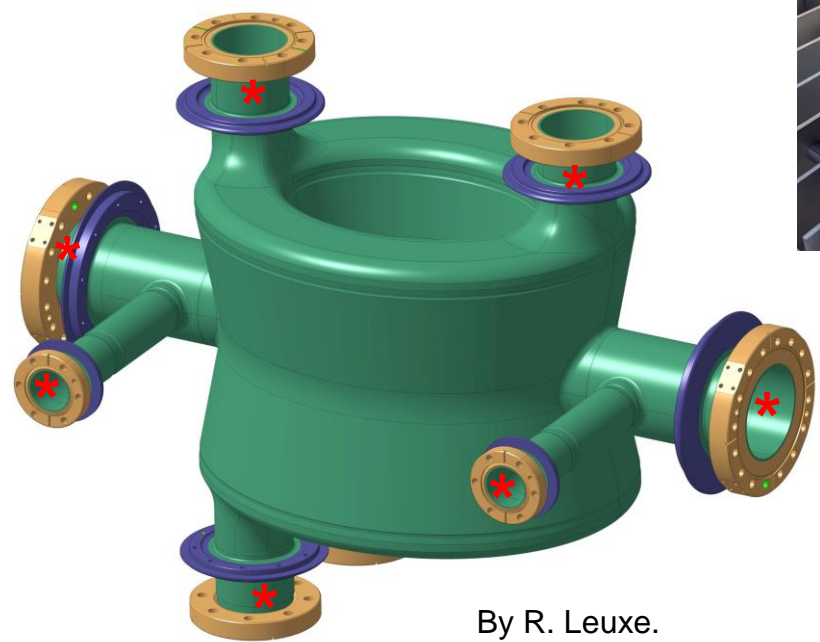


# Nb RRR drop during brazing heat treatment

CERN reference trials show a potential RRR drop up to 20%. (e.g. RRR 300 → RRR 240)  
 It is however possible to drop by a 70%-80% if the process and furnace cleanliness is not well mastered.  
 (e.g. RRR 300 → RRR 75)

Discontinuity in RRR → effect on RF performance?

Lost of ductility at cryogenic temperatures?



By R. Leuxe.



Brazed assemblies preparation, with Nb RRR witness samples. By F. Motschmann.

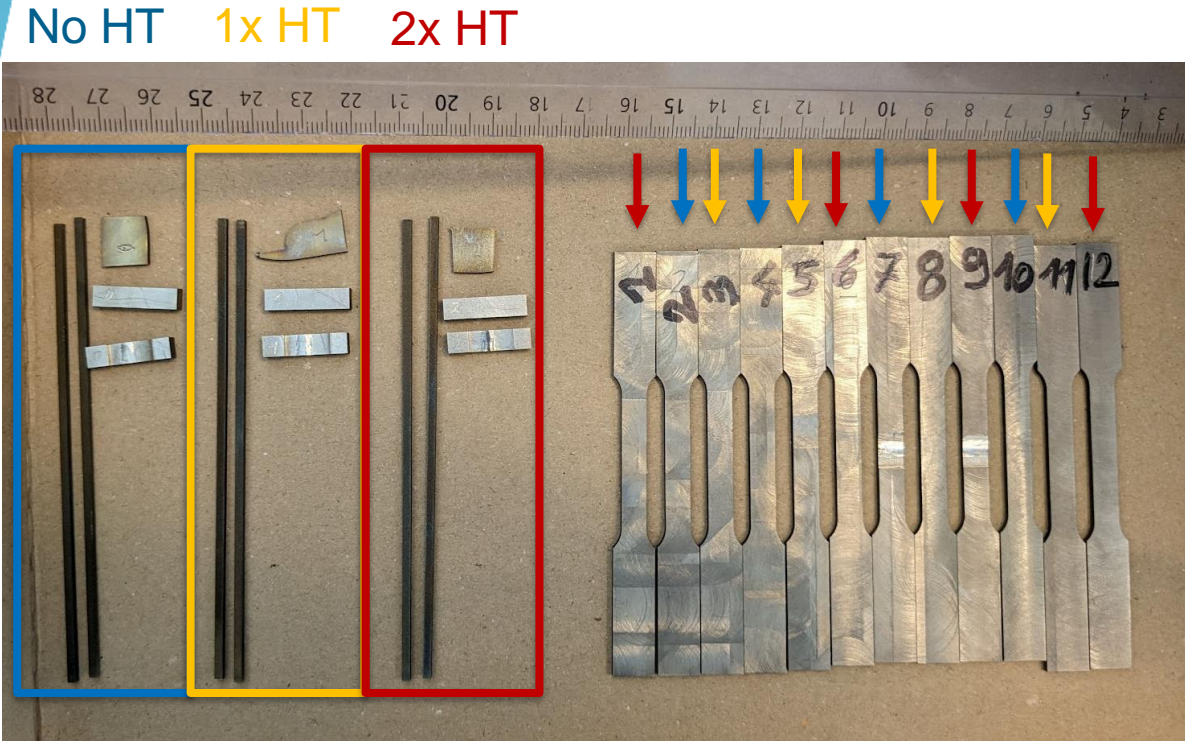
Table 2: Tensile test results.

Mat'l	Temp	Yield Stress	Tensile Strength	Red. in Area	Elong.-12.7 mm	Elong.-25 mm
	K	Mpa	Mpa	%	%	%
Nb	295	67	172	88.3		57.4
RRR	77	618	642	72.4		30.5
250	4	658	929	28.7		16.0
RRR	295	70	151	86.7		28.2
250	77	445	639	59.6		13.4
WELD	4	470	696	10.4	9.1	4.2
Nb	295	76	171	80.9		41.05
RRR	77	443	502	7.3		3.1
40	4		468	1.1		1.6
RRR	295	95.5	172	90.0		58.5
40	77		450	0.2		1.4
WELD	4		327	0.4		1.36



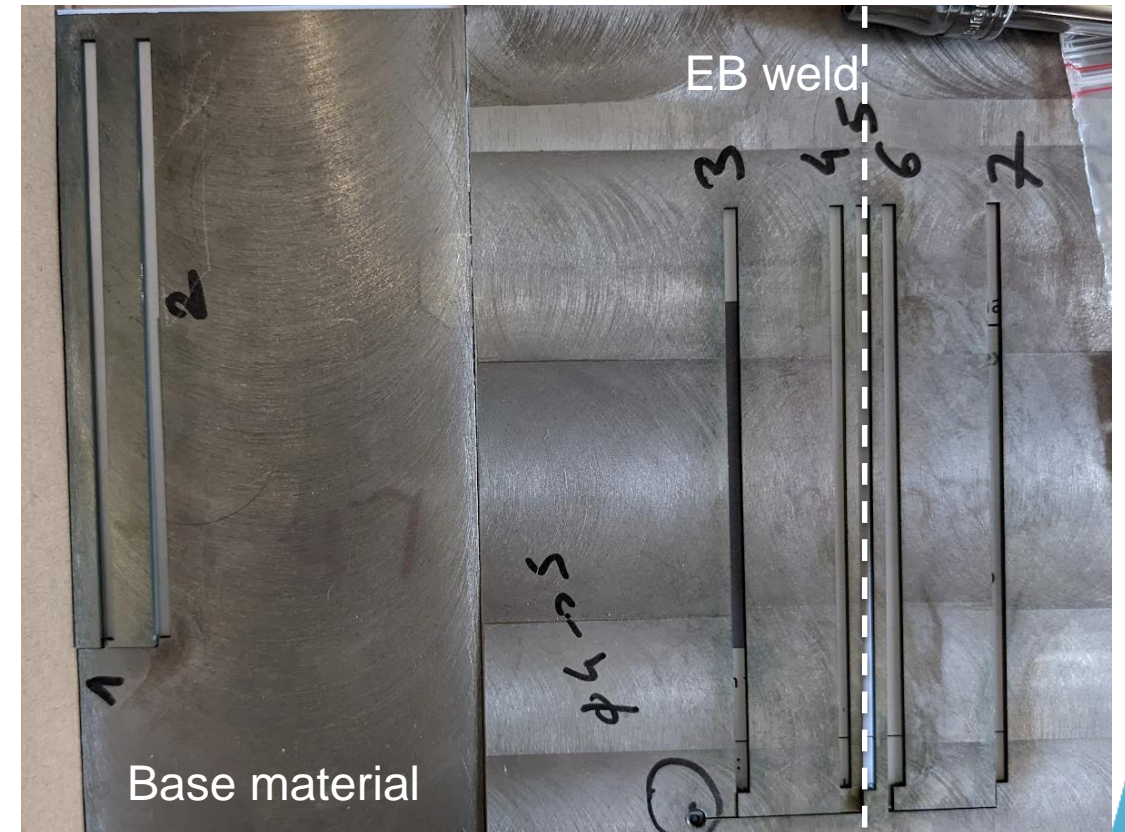
Walsh, R. P., et al. "Low temperature tensile and fracture toughness properties of SCRF cavity structural materials." IX Workshop on RF Superconductivity" Santa Fe. 1999.

# Effect of 2x H degassing heat treatments



Compare Nb mech. properties in as-received, welded and cold rolled condition.

# RRR after EBW US-AUP contractor



Assess RRR drop around the weld (0, 5, 20mm)

# Conclusions

- A **material** is not a name/number only, but it has to be **purchased according to a specification** (international standard, CERN specification, other). Product quality can dramatically change.
- **CERN material specifications are in the safe side** regarding product quality. **Derogations** are possible to meet industry standard practices but have to be **assessed case by case**. Contact WP4 and MME colleagues in case of doubts.
- **All NbRRR300 and Nb55Ti** for the DQW Crab Cavities (including HOMs) has been purchased. Internal quality checks pending for Nb to be used for DQW HOMs.
- **FE simulations + Forming Limit Diagram** were employed to deeply **understand** the RFD pole **forming** and the **2-step forming** strategy employed by US-AUP contractor was **validated**.
- **Critical components** (e.g. bimetallic SS-Ti transitions, RF feedthroughs) **validation tests are completed** (or well advanced) for the series.
- We are still climbing the learning curve, but thanks to the **lessons learned and the know-how** curated and maintained by WP4 and EN/MME, performance of the crab cavities is beyond expectations.



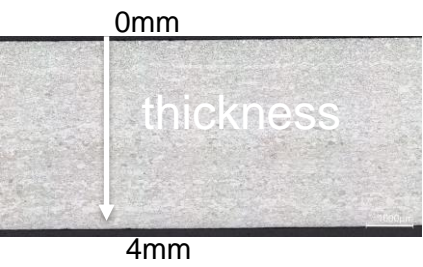
Thanks for your attention.

And thanks to all WP4 colleagues and MME-MM  
colleagues.

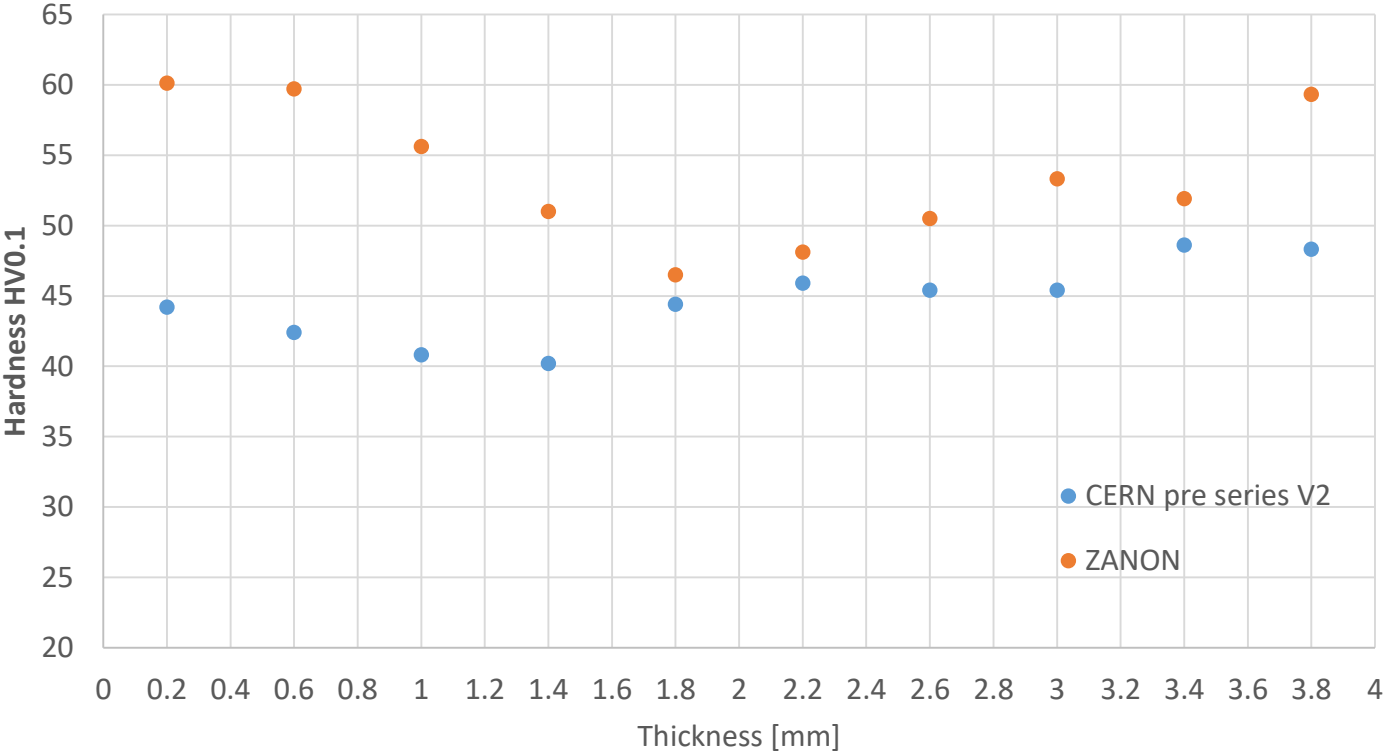
# Initial materials and tensile test conditions

- 'CERN' material:
    - CA7420650 (pre-series v2) – sheet ID 2282401
  - 'Zanon' material:
    - PO657756 (pre-series) – sheet ID 2411101
- 
- Zanon material → Cutting by Micro water jet cut
  - CERN material → Cutting by spark erosion
  - ASTM E8 sub-size specimens
  - 2x long (0 deg) and 2 x short (90 deg) specimens
  - Test conditions ASTM B393

# Hardness profiles



Hardness vs. thickness



Measurements done by J. Lequin-Souchon

- 'Zanon material' presents a higher HV0.1 hardness average ( $53.6 \pm 4.9$ ) compared to 'CERN material' ( $44.6 \pm 2.8$ ).
- 'Zanon material' hardness profile presents a more pronounced 'V-shape' presumably due to a levelling process, which would harden the surface.

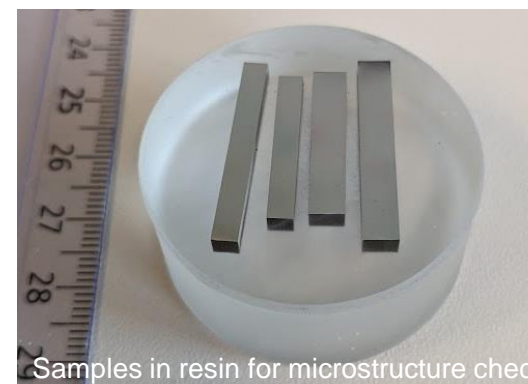
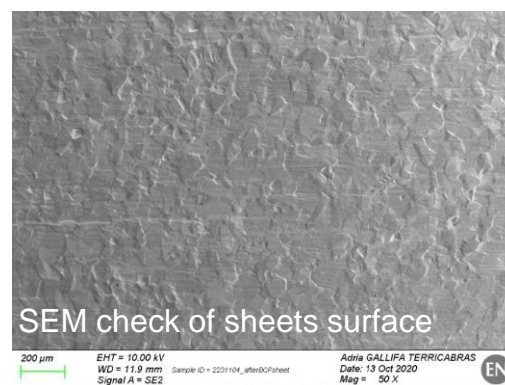
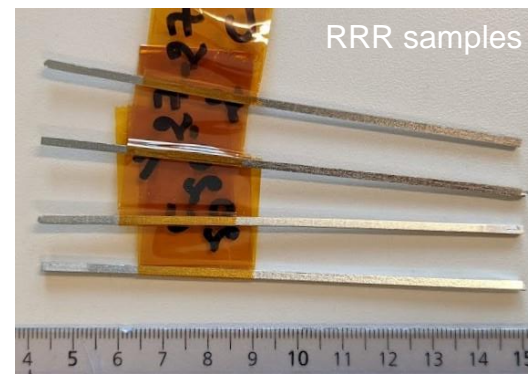
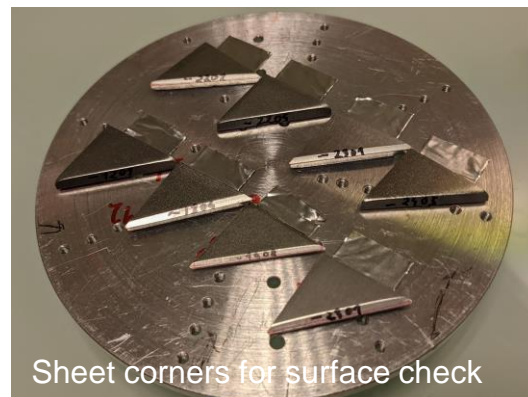
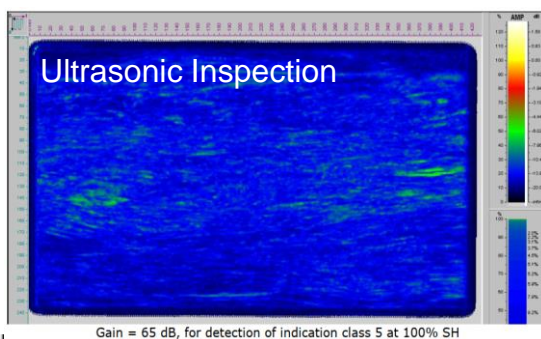


Figure 1. Detail of the exogenous substance on sheet ID 2221701.







# Specimens

- 0 HT: → please do not include them in the furnace
  - Tensile: x2 samples: #4, #2, #7, #10
  - RRR: x2 samples: #XX, #XX
  - Metallo: x 3 samples: base #0, weld #0, cold rolled #0

---

- 1 HT: → one hydrogen degassing heat treatment
  - Tensile: x2 samples: #5, #3, #8, #11
  - RRR: x2 samples: #XX, #XX
  - Metallo: x 3 samples: base #1, weld #1, cold rolled #1
- 2 HT: → two hydrogen degassing heat treatments
  - Tensile: x2 samples: #1, #6, #9, #12
  - RRR: x2 samples: #XX, #XX
  - Metallo: x 3 samples: base #2, weld #2, cold rolled #2