

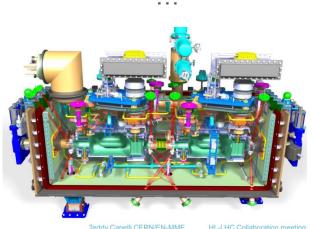
Material qualifications for Crab Cavities

Adrià Gallifa Terricabras – EN/MME/MM (CERN) on behalf of Materials, Metrology and NDT team and HL-LHC WP4.

11th HL-LHC Collaboration Meeting, CERN, 19-22 October 2021

Materials

- Nb RRR300
 - Ti alloys
 - Cu alloys
- Stainless steel
- Cryophy, mumetal
 - Ceramics (Al₂O₃)
- Filler metals: Ga-Pa-Ag



Components

- Bare cavities
- Brazed SS-Nb extremities
- Tuning system
- 2nd beam pipe
- He tank
- Ti-SS transitions
- Cryomodule
- Bellows
- HOMs and antenna couplers
- Cold & warm magnetic shield
- Coaxial lines
- RF feedthroughs
- Gaskets
- Fasteners

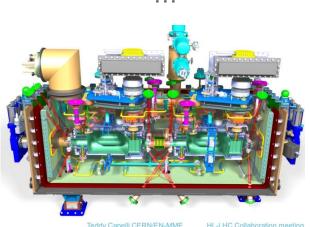
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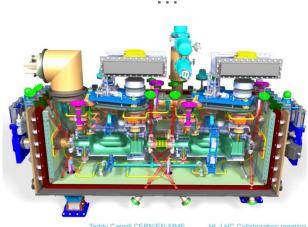




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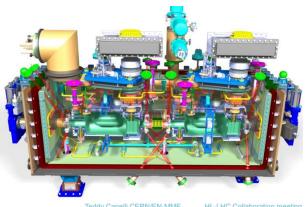
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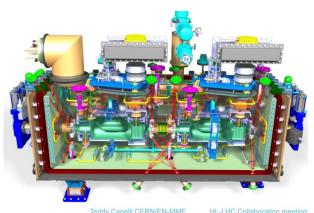




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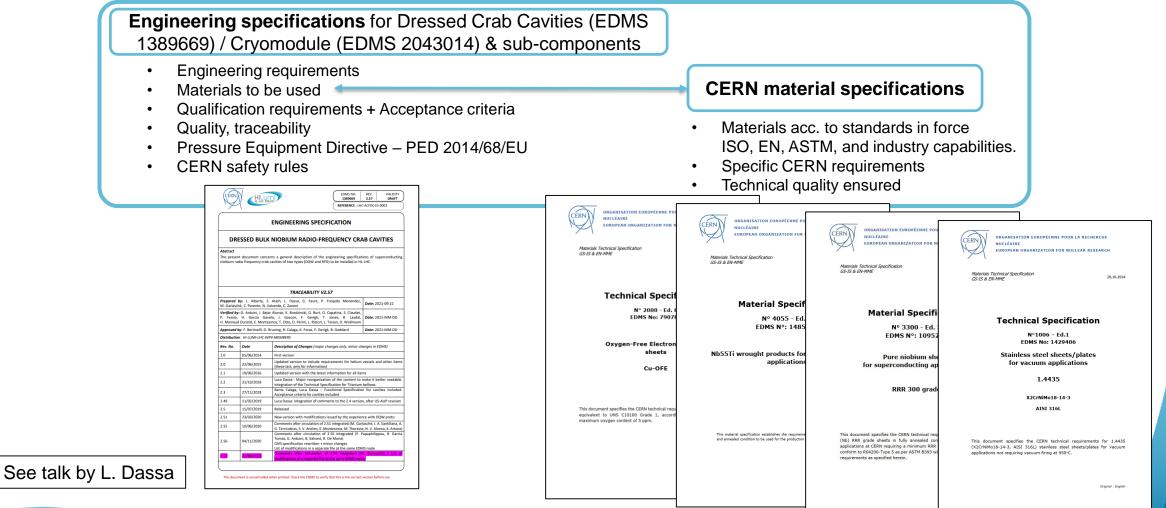
Manufacturing process

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



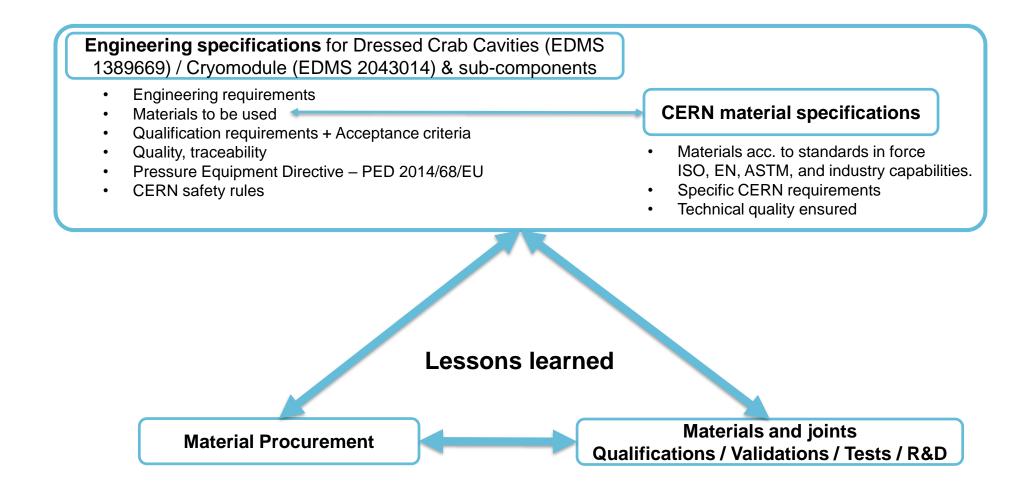
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Our 'holy' documents





Our 'holy' documents





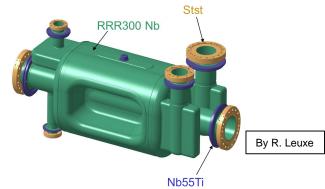


Nb RRR 300

 Back in 2018 - 2020: after DQW SPS proto, market survey and additional suppliers qualification: Ningxia (CN), Tokyo Denkai (JP), Ulvac (JP).

Procurement challenges

- Cost: > 700 CHF/kg
- Lead time: > 4 months
- Limited amount of suppliers: 3 are qualified (not all produce all products!)



Technical challenges (CERN spec. is stringent, but guarantees high quality material)

- Achieve a specific yield strength (65MPa < Rp0.2 < 100 MPa) → Low yield strength was measured in the past → important for strength assessment of the cavities according to PED.</p>
- Achieve a 'clean' surface → Abrasive particles embedded on the surface were detected → prevent field emission 'hot spots' during operation.
- For DQW series, systematic internal qualifications were performed consisting in:
 - **UT** (100%)
 - Surface check by SEM (+thickness measurements) (1-2/lot)
 - Tensile tests (~ 2 sheets/lot)
 - RRR measurements (1/each product type/shipment)
 - **Microstructure check** (1/each product type/shipment)

All NbRRR300 needed for DQW bare cavity series is already at CERN (sheets, plates).

See reception and internal qualification report in EDMS 2395238.

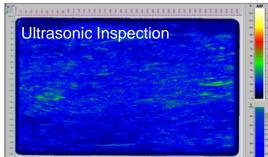


Nb reception and internal qualifications



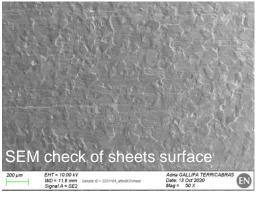


Figure 1. Detail of the exogenous substance on sheet ID 2221701.

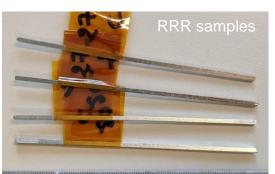


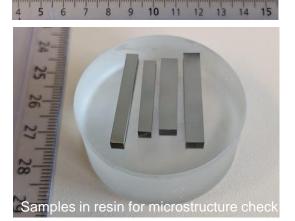
Gain = 65 dB, for detection of indication class 5 at 100% SH













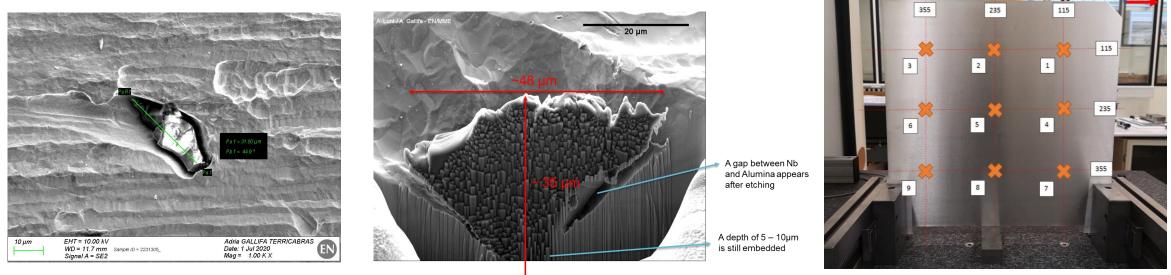




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Analyse Nb surface by SEM upon reception

 Issue: Some sheets of both Nb orders for DQW series had alumina particles embedded on the surface.



Embedded Al2O3 particle on as-received Nb sheet.

SEM-FIB tomography of the cross section of an embedded Alumina particle on Nb. By A. Lunt

Thickness measurement on 9 points of a Nb sheet.

 2x NCRs were created. Corrective actions: perform BCP20 um (per face) before forming operations (+ thickness measurements).



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Thanks to S. Forel, B. Bulat and M. Burkowski

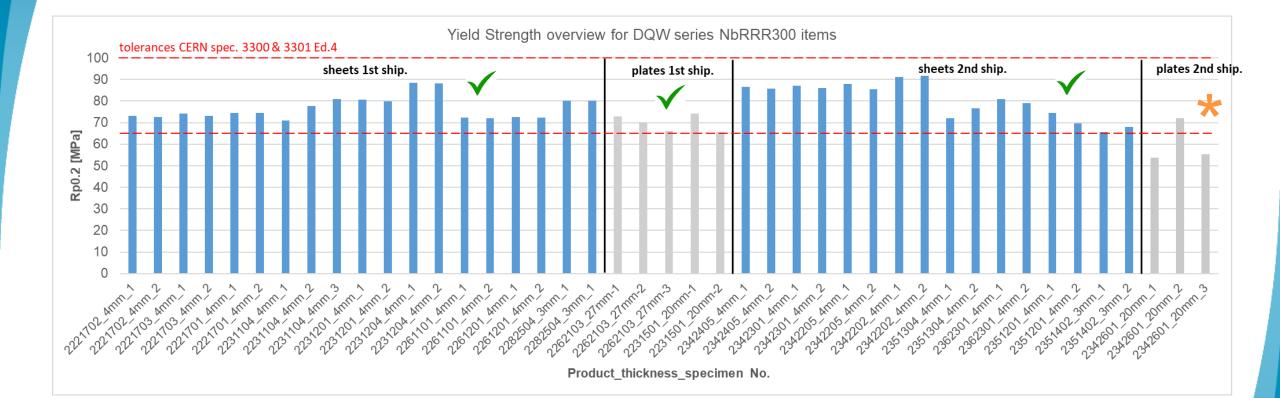
Microstructure check





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Tensile tests overview: Rp0.2

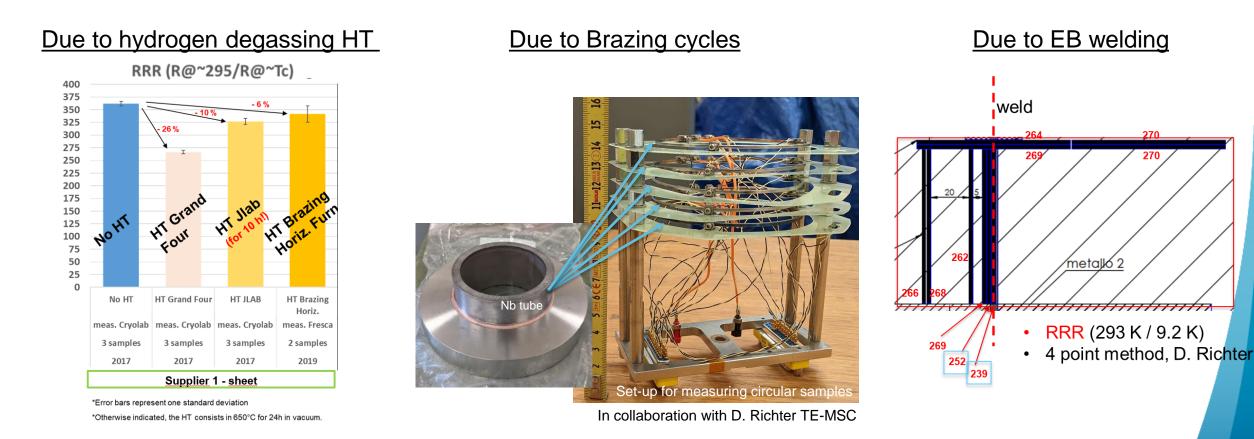


★ A NCR was created and after a dedicated FEA strength assessment considering Rp0.2=50 MPa the 20mm thickness plates were accepted for the series production.



RRR degradation after heat treatments

Thermal treatments can significantly degrade RRR:





RRR degradation after HT

- Thermal treatments can significantly degrade RRR
 - EB welding ·
 - Hydrogen degassing HT
 - Brazing cycles

RRR samples are requested in the Eng. Spec (4.2.7.6)!

Recommended to check RRR, useful to improve heat

treatment practices

Details as samples preparation, furnace manipulation and cleanliness, vacuum level and others can have a significant impact.

Some references:

Singer, W., Singer, X., Tiessen, J., Wen, H. M., & Schölz, F. (2003, July). RRR degradation and gas absorption in the electron beam welding area of high purity niobium. In *AIP Conference Proceedings* (Vol. 671, No. 1, pp. 162-175). American Institute of Physics.

Alonso, N. V., Atieh, S., Santillana, I. A., Calatroni, S., Capatina, O., Ferreira, L., ... & Vacca, A. (2014). Electron Beam Welding and Vacuum Brazing Characterization for SRF Cavities. In *Proc. 27th Linear Accelerator Conf. (LINAC'14)* (pp. 932-934).



Thanks to P. Freijedo, D. Richter. For further details contact A. Gallifa, F. Motschmann



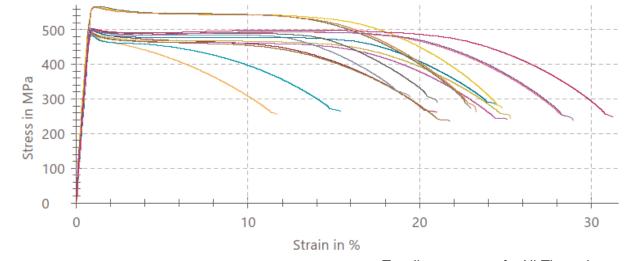


Nb55Ti reception and internal qualifications

- Additional pieces from the different lots were purchased for internal qualifications.
- Qualifications consisted in:
 - Ultrasonic Inspection (100%)
 - Mechanical Tests (1 item/lot and thickness)
 - Microstructural check (1 item/lot and thickness)



Additional pieces purchased for qualifications.



Tensile test curves for NbTi specimens.

All NbTi needed for DQW series is already at CERN (rings, rods, plates).



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See reception and internal qualification report in EDMS 2395238.



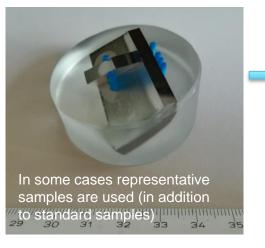


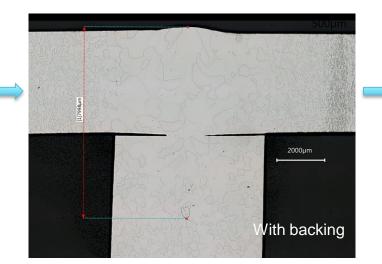
4000µm

EB welds Nb-Nb and Nb-Nb55Ti

With T. Demaziere

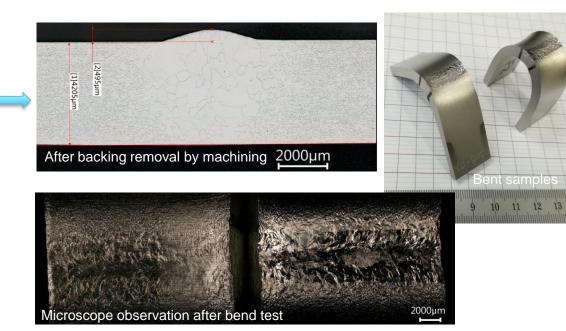
Welding Procedure Qualification Records (according to ISO 15614-11 and Engineering Spec EDMS 1389669)





Effect of <u>hydrogen degassing heat treatment</u> on

the mechanical properties of welds



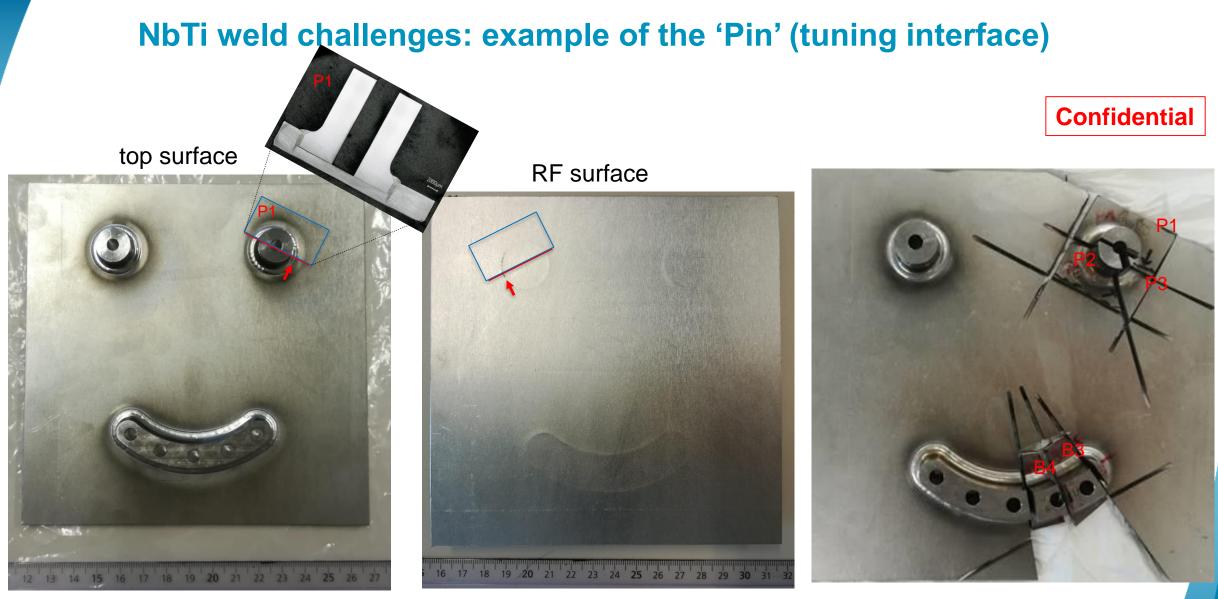
Percentage change of Rp0.2, Rp1.0 and Rm before vs. after HT.

Qualif. ID	Rp0.2	Rp1.0	Rm
Qualli. ID	change	change	change
WPQR-EBW-2019-01	-10%	-9%	-5%
WPQR-EBW-2019-03	-9%	-4%	-2%
WPQR-EBW-2019-04	-12%	-12%	-6%
WPQR-EBW-2019-05	-2%	-3%	-4%
WPQR-EBW-2019-07	-11%	-10%	3%
WPQR-EBW-2019-08	-18%	-17%	-6%
Average change	-10%	-9%	-3%



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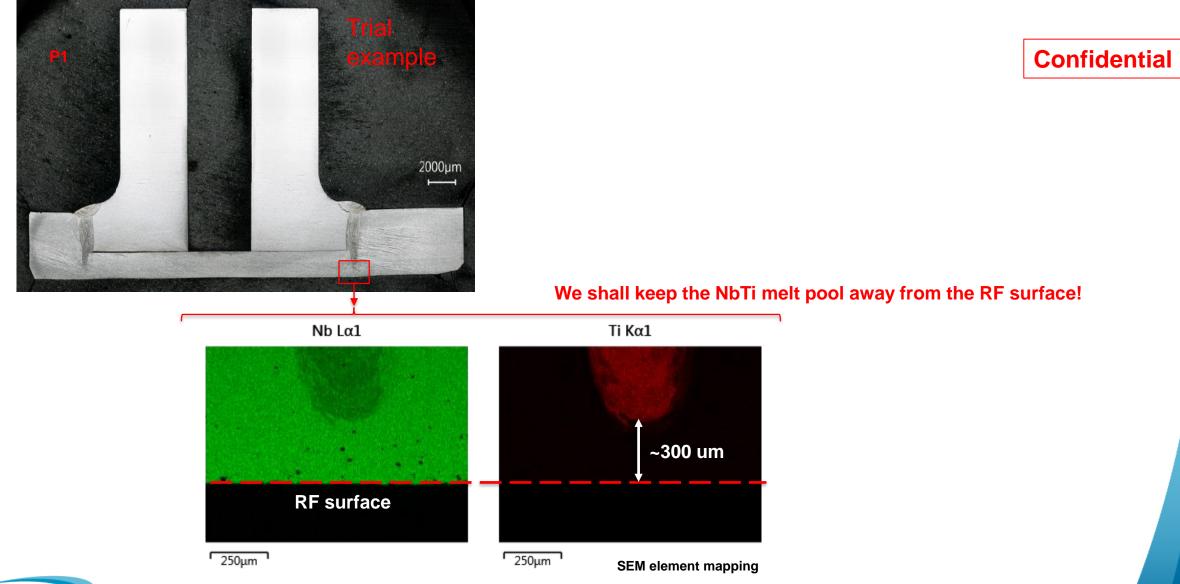
During manufacturing, NDT is not exploitable on the Pin and Banana.

 \rightarrow Several iterations needed to optimize penetration depth.



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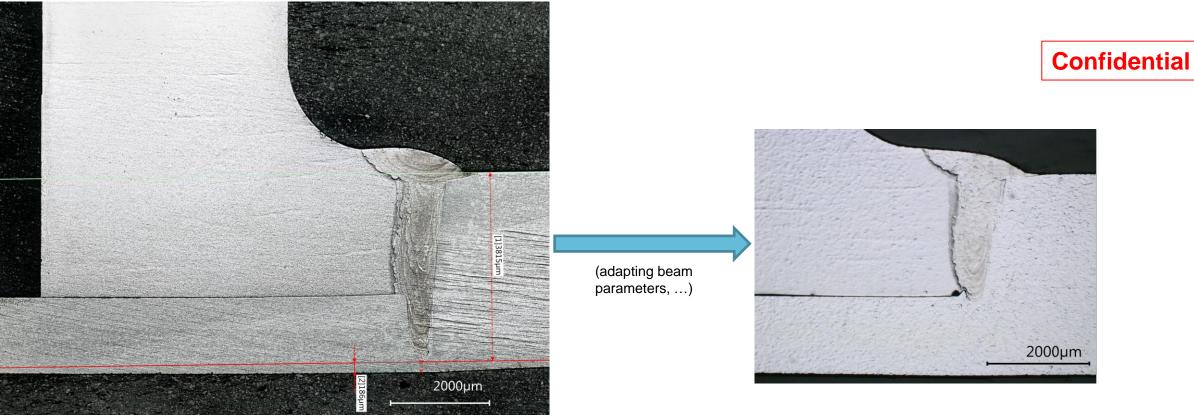
NbTi weld challenges: example of the 'Pin' (tuning interface)





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NbTi weld challenges: example of the 'Pin' (tuning interface)



First trial: Metalography P1, on the pin Penetration depth 3.815 mm After process improvement Penetration depth ~ 2.8 - 3 mm

Metallographic cuts are crucial for improving (and qualifying) welds.

The Nb-NbTi welds typically require several trial and error iterations.



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Image by M. Meyer

Ti Gr 2 procurement

- Crab Cavity project was relying on a Particular Material Appraisal (PMA) by DESY based on DIN specifications.
- A new PMA acc. to PED 2014/68/EU has been released together with CERN HSE team, in order to be able to use Ti Gr 2 acc. to ASTM/ASME standards.

2 MATERIALS UNDER THE SCOPE OF THE PRESENT PMA

This document presents the technical data needed to establish a Particular Material Appraisal (PMA) for the material **Titanium Grade 2¹/2H²** specified according to the following US standards:

- ASME SB-265 (version 13 to 20a) Specification for Titanium and Titanium Alloy Strip, Sheet, and Plate
- ASME SB-348 (version 13 to 19) Specification for Titanium and Titanium Alloy Bars and Billets;
- ASME SB-861 version 13 to 19) Specification for Titanium and Titanium Alloy Seamless Pipe;

and corresponding ASTM specifications with the additional requirements specified in §11.

Later versions of the aforementioned specifications are considered acceptable.

		RTICULAR MATERIAL APPRAISAL	1.1114	/	
		RADE 2/2H ACCORDING TO US	SPECIF	ICATIO	NS
This docum documental Titanium co equipment. In the frame	eent is written in tion to be drawn u mmercial grades ework of the HL-L	ng to US specifications: ASME BPVC.II.B, SB/B-20 the application of the European Directive 20 up by the manufacturer of pressure equipment are not covered by European harmonised stand HC WP4 'Crab Cavities & RF' project, the utilisa edicated PMA which is described in this docume	014/68/EU aiming co dards gran	J regarding onformity a nting their	g the technica ssessment. use in pressure
		ТКАСЕАВІШТУ			
Prepared by	y: L. Dassa, A. Gall			Date: 202	1-02-24
Verified by:		ifa Terricabras re, O. Capatina, M. Garlaschè, C. Gaignant, S. M	larsh,	Date: 202 Date: 202	
Verified by:	I. Aviles, S. Barrie N. Valverde, O. Wi	ifa Terricabras re, O. Capatina, M. Garlaschè, C. Gaignant, S. M	larsh,		1-05-03
Verified by: S. Sgobba, M Approved b	I. Aviles, S. Barrie N. Valverde, O. Wi yr. R. Calaga	ifa Terricabras re, O. Capatina, M. Garlaschè, C. Gaignant, S. M		Date: 202	1-05-03 1-05-20
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Verified by: S. Sgobba, M Approved b Distribution	I. Aviles, S. Barrie N. Valverde, O. Wi y: R. Calaga p: N. Surname (DE	ifa Terricabras re, O. Capatina, M. Garlaschè, C. Gaignant, S. M liiams P/GRP) (in alphabetical order) can also include r	reference s only, mil	Date: 202 Date: 202 to committe nor change ing circulat	1-05-03 1-05-20 tees s in EDMS)
Verified by: S. Sgobba, N Approved b Distribution Rev. No.	I. Aviles, S. Barrie N. Valverde, O. Wi yr. R. Calaga n: N. Surname (DE	ifa Terricabras re, O. Capatina, M. Garlaschè, C. Gaignant, S. M lilams P/GRP) (in alphabetical order) can also include r Description of Changes (major changes Revised version according to comments in E 0.5. Sections 4.2 and 4.3 added.	reference s only, mil	Date: 202 Date: 202 to committe nor change ing circulat	1-05-03 1-05-20 tees s in EDMS)
Verified by: S. Sgobba, N Approved b Distribution Rev. No. 0.6	I. Aviles, S. Barrie V. Valverde, O. Wi yr. R. Calaga Date 2021-02-24	ifa Terricabras re, O. Capatina, M. Garlaschè, C. Gaignant, S. M liams P/GRP) (in alphabetical order) can also include r Description of Changes (major changes Revised version according to comments in E 0.5. Sections 4.2 and 4.3 added. Range of validity extended: versions 2013 to	reference s only, mil	Date: 202 Date: 202 to committe nor change ing circulat	1-05-03 1-05-20 tees s in EDMS)

EDMS NO.

REV

VALIDITY



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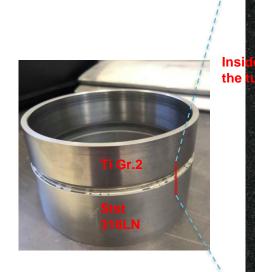
See talk by L. Dassa

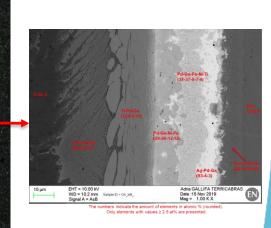
Brazed bimetallic transitions

SS-Ti bimetallic brazed transition validation

5'small' + 5'big' test samples were validated

- **1.** Leak tightness test
- 2. Ultrasonic test (UT) inspection
- 3. Thermal shock (TS) in liquid nitrogen (x5 times)
- 4. Leak tightness test after TS
- 5. Ultrasonic test (UT) inspection after TS
- 6. Visual test (VT) inspection
- 7. Metallograpy (OM and SEM)
- 8. Tensile tests
 - 1. 2x small tubes at RT
 - 2. 1x small tube at 77K
 - **3. 3x** specimens cut from one big tube, tested at RT
 - 4. 3x specimens cut from one big tube, tested at 4K





With F. Motschmann



HL-LHC Collaboration Meeting. 21 Oct 2021

SS316LN

Ti Gr.2

SS-Ti bimetallic brazed transition validation

Sample after rupture

at the brazed region



Tensile tests on real-size tube and on specimens extracted from a tube, at RT and cryogenic temperature.



Test conditions:

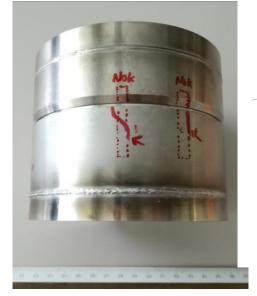
- Crossheads speed 0.4 mm/min (adapted from ISO 6892-1)
- 200 kN load cell
- Preload: 300 N

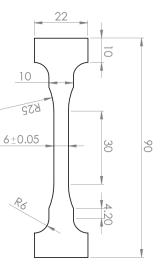
Extensive experience after internal tests:

Leak tightness: 10/10 parts OK (before and after thermal shocks) Ultrasonic test inspection: 9/10 parts OK

Mechanical tests

- Small tubes at RT: Fmax \ge 85 kN, Ultimate Shear Strength \ge 116 MPa
- Small tube at 77K: Fmax ≥ 143 kN Ultimate Shear Strength ≥ 190 MPa
- Specimens cut from big tube, at RT: Ultimate Shear Strength ≥ 72 MPa
- Specimens cut from big tube, at 4K: Ultimate Shear Strength ≥ 84 MPa





+ qualification with a notified body



Further information EDMS 2271509

For further details contact F. Motschmann, T. Demaziere.



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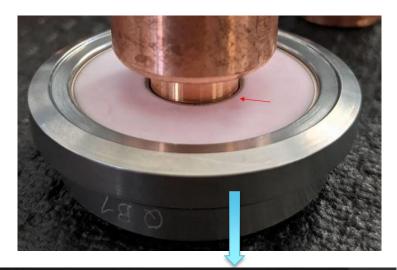
RF feedthroughs

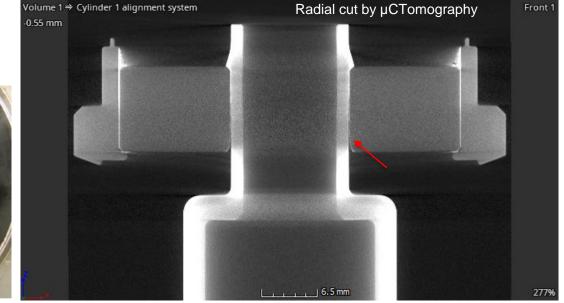
Validation Test Plan (after brazing)

- Leak Test
- Penetrant Testing
- Mounting on 'mock-up' chamber
- Thermal Shock in liq. N₂
- Leak Test
- Penetrant Test
- NDT
- Metallographic / microscope check

RF feedthroughs

See talk by E. Montesinos











With F. Motschmann and M. Crouvizier. Thanks to S. Bonnin, A. Porret.

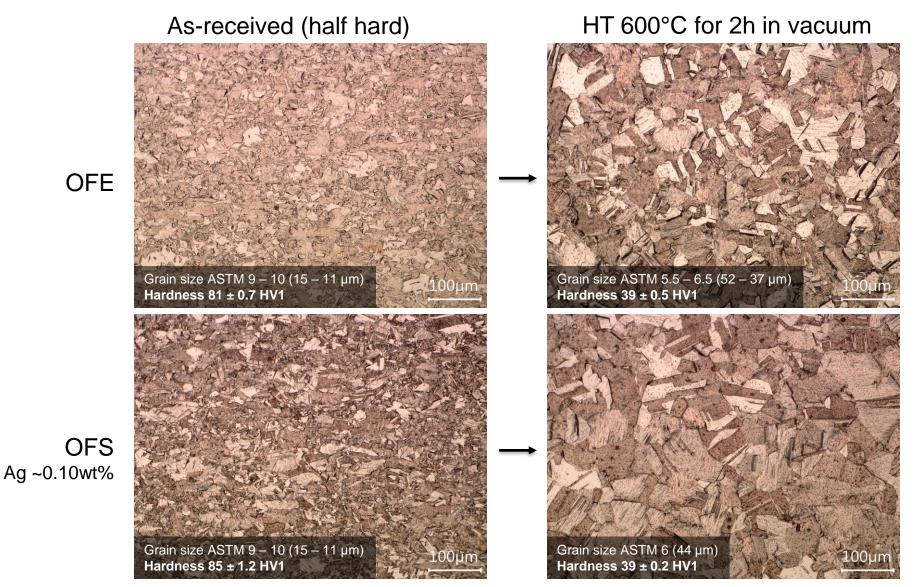
Acceptance criteria for series needs to be discussed.

Remark: As NDT, Ultrasonic Testing is preferable, but depending on the geometry µCTomography could be envisaged.

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Cu OFE vs. Cu OFS for RF gaskets

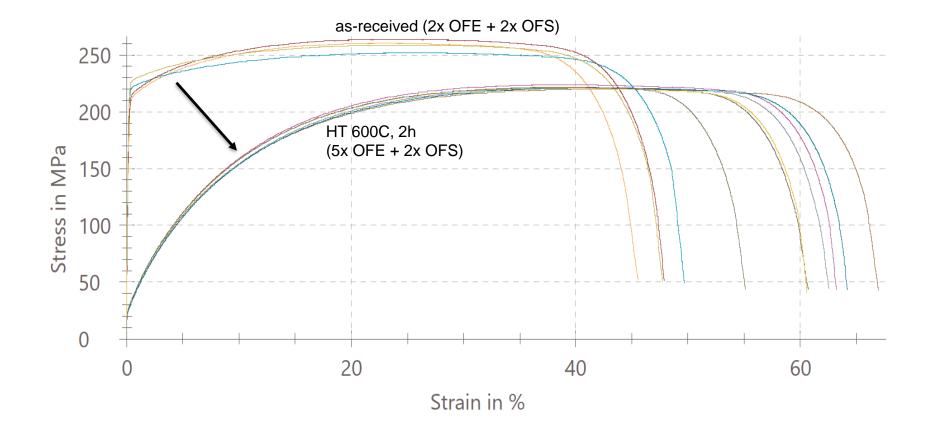




3 x hardness measurements in mid-thickness / Grain size values show the range measured within 5 fields / polishing up to 1 µm, etching with HNO3 50% vol.

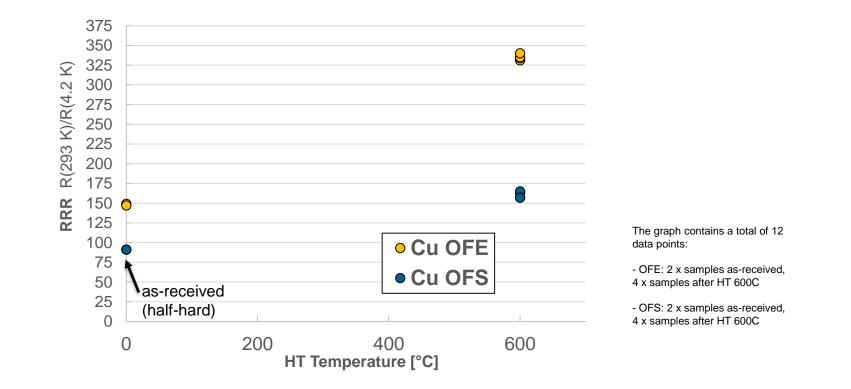
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Cu OFE vs. Cu OFS for RF gaskets – Tensile tests





Cu OFE vs. Cu OFS for RF gaskets - RRR

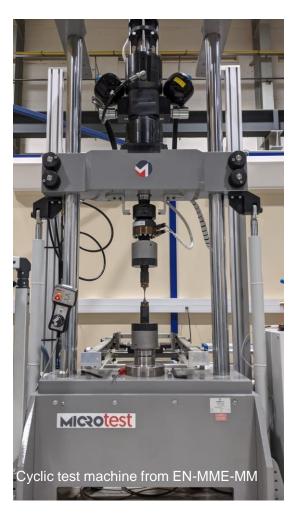


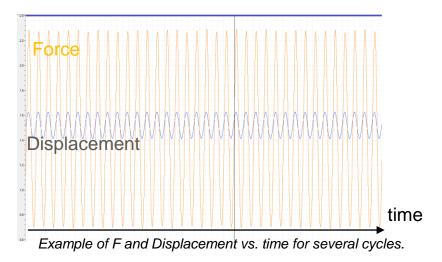
- In as-received condition, Cu OFE has a higher RRR than OFS by a factor 1.6
- After HT at 600°C for 2 hours, Cu OFE has a higher RRR than OFS by a factor 2
- The difference is **due to the resistance at 4.2 K** (at RT there is no significant difference)



Some future tests and R&D

- Ultrasonic parameters (attenuation..) vs. mechanical properties of NbRRR300
- Fatigue tests on NbRRR300







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.
- On critical materials for the project (e.g. NbRRR300) it is important to perform internal quality control tests and give feedback to suppliers.
- Raw materials for the bare DQW series (NbRRR300, Nb55Ti) are already at CERN. After internal tests, all items are validated for production.
- 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 to all MME and WP4 colleagues!

HILLHC PROJECT

And special thanks to I. Aviles, N. Valverde, L. Dassa, S. Barriere, M. Garlasche, T. Demaziere, F. Motschmann, K. Eiler, P. Freijedo, S. Forel, D. Richter, A. Eychenne, G. Arnau, S. Bonnin, M. Haavarstein, M. Pentella.



Adrià Gallifa – on behalf of CERN EN/MME and WP4



Questions?

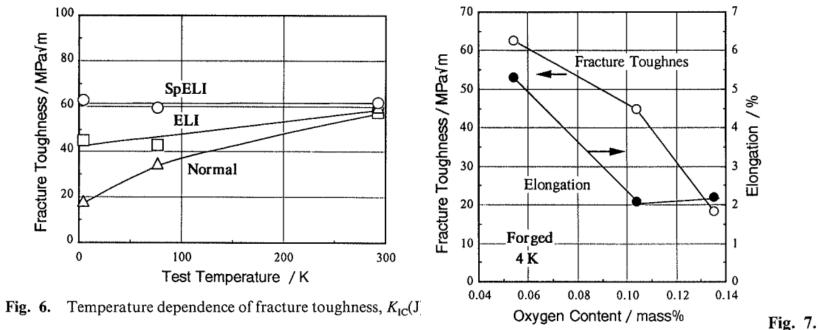
Adrià Gallifa – on behalf of CERN EN/MME and WP4

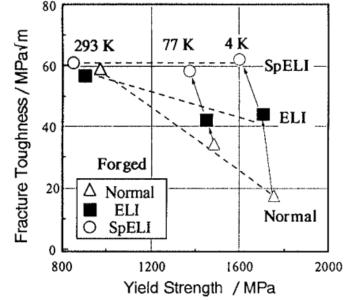


Backup slides

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Ti alloys: why to use Ti Grade 23?





7. Interrelationship between the fracture toughness of forged materials and other factors: (a) oxygen content and (b) yield strength. Elongation is also plotted in (a).

The fracture toughness and ductility for Extra-Low-Interstitial (ELI) grades, like Ti Grade 23, at cryogenic temperatures are significantly superior than for non-ELI grades, like Ti Grade 5.



Nagai, K., Yuri, T., Ogata, T., Umezawa, O., Ishikawa, K., Nishimura, T., ... & Ito, Y. (1991). Cryogenic mechanical properties of Ti-6AI-4V alloys with three levels of oxygen content. ISIJ International, 31(8), 882-889.

SS-Ti bimetallic qualifications (acc. to Eng. Spec. v2.57)

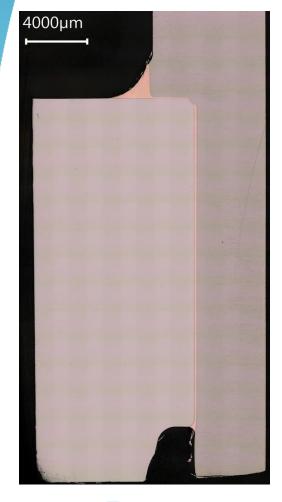
Table 27: Standards applicable to brazing qualifications

Description	Standards		
	American	European	
Brazing Qualification (BPQR&BPS)			
Brazing Personnel Qualifications (BPQ)	 brazing procedure variables (as detthe production of the cavity. No standard samples required by a The following tests are required for only 1 sample subjected to all the order: 100% Visual inspection Thermal shocks (at LN2 terr followed by warming to am Helium leak test (refer to Set 100% UT by immersion according to EN 12797 Shear test with test condition 	hts (see 8.1 of the EN 13134). roduced for every combination of efined in EN 13134), required for ASME are needed. or each qualification, considering following tests in the specified hperature) x 5 times, each of them abient temperature + final drying ection 4.3.5). ording to §4.2.6.4 n (on 2 sections of same sample) ons and documentation acc. to EN is possible to final product (to be	After the internal validation experience, the qualification requirements are integrated in the Engineering Specification. See talk by L. Das
Brazing	ASME BPVC Section IX Part-QB	EN ISO 13585	



Brazing qualification/validation of AUP brazed flanges (SS - Nb)

Acc. to Eng. Spec. for dressed cavitites EDMS 1389669 v2.56, table 11:





- 100% Visual inspection Done
- Thermal shocks (at LN2 temperature) x 5 times Done
- Helium leak test Done
- 100% UT by immersion Done
- Metallographic examination (on 2 sections of same sample) Done

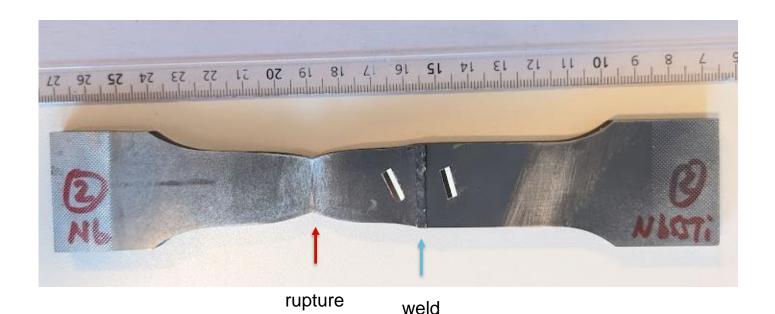




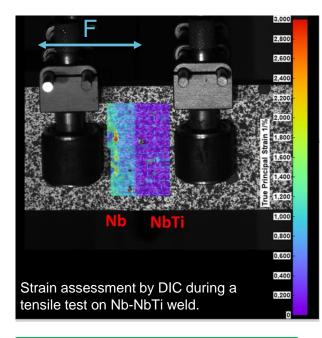
Reports stored in EDMS 2566930



Nb-NbTi EB welds



Specimen no. Fm Rm bo S₀ L0 ST Lc Rp0.2 Ag A16,45mm a_0 ΜE mm² % Legend No. GPa MPa % kΝ MPa mm mm mm mm 24.95 202.10 16.45 32.79 8.1 80 61 85 3.91 4.6 162 3 2 8.09 24.97 202.01 16.45 80 47 95 4.88 6.2 33.94 168 4



The NbTi part is much more stiff than the Nb. The specimens break in the middle of the Nb part.





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EDMS 2610033

Nb55Ti reception and internal qualifications

- Indications are sometimes identified by Ultrasonic testing:
 - Nb-rich regions

30mm

Microstructure inhomogeneities

74mm

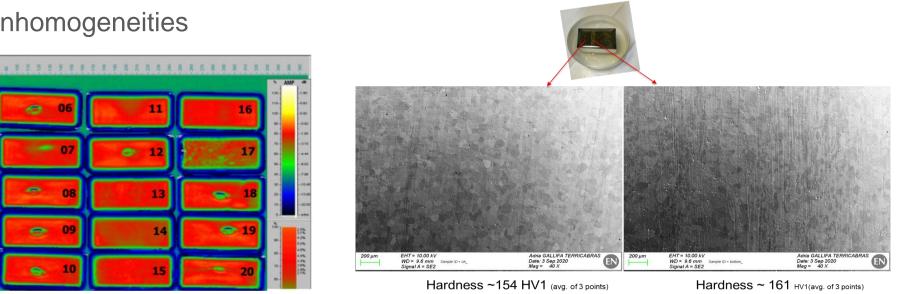


Figure 1. C-Scan Images – homogeneity of BWE of 18 mm thickness for NbTi pieces 1951201 to 1951240.

The defects encountered until now are considered non-critical for the Nb55Ti application, but they have to be studied case by case. Several NCR have been prepared to keep traceability.



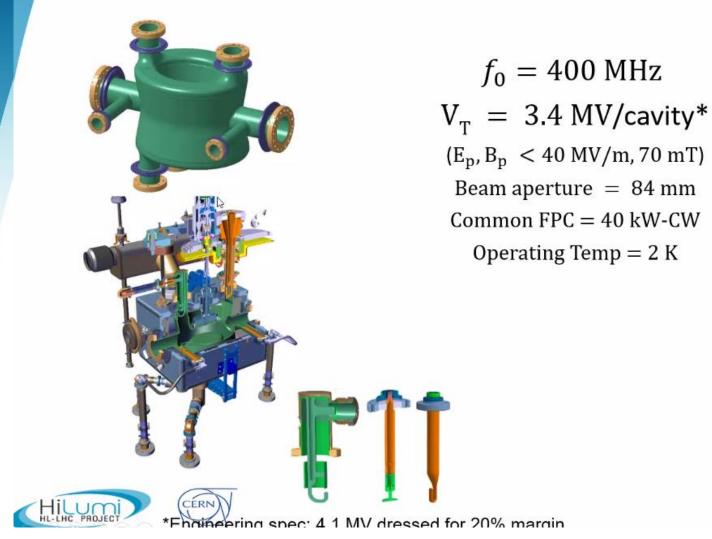


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Thanks to G. Arnau, S. Bonnin

Reminder: Dressed Cavity Geometries

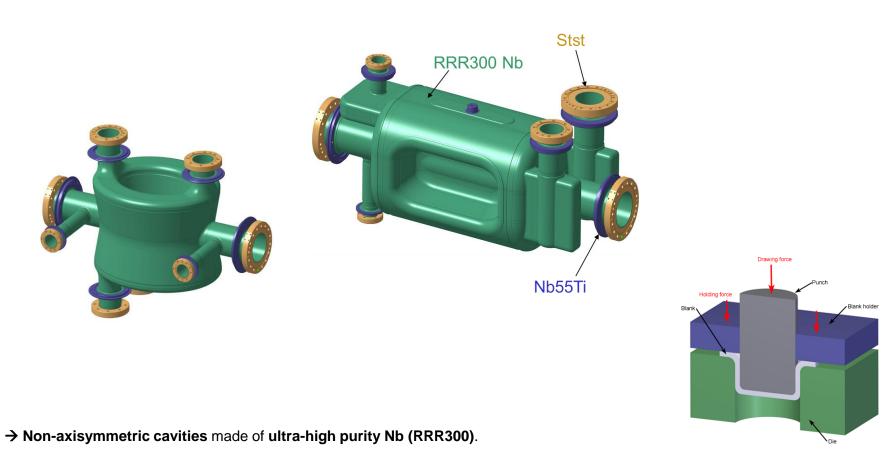
Double Quarter Wave



RF Dipole



HL-LHC WP4, Annual Meeting 2021



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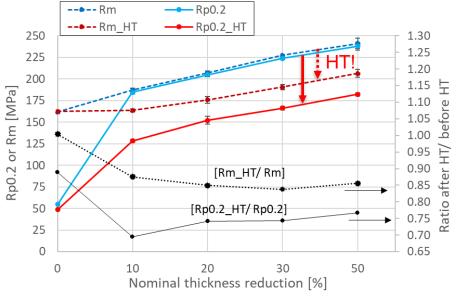
→ The main **components** of the CC are **plastically deformed** during manufacturing operations (i.e. **deep drawing**).

 \rightarrow The CC are submitted to a hydrogen outgassing <u>heat treatment</u> (HT) at 650° C during 24 hours.

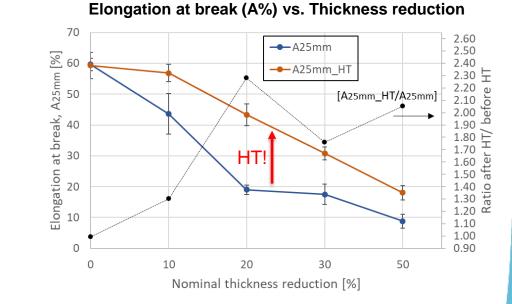


Adrià Gallifa – on behalf of CERN EN/MME and WP4

Mechanical properties of Nb after cold work and heat treatment



Yield strength (Rp0.2) and Ultimate strength (Rm) vs. Thickness reduction



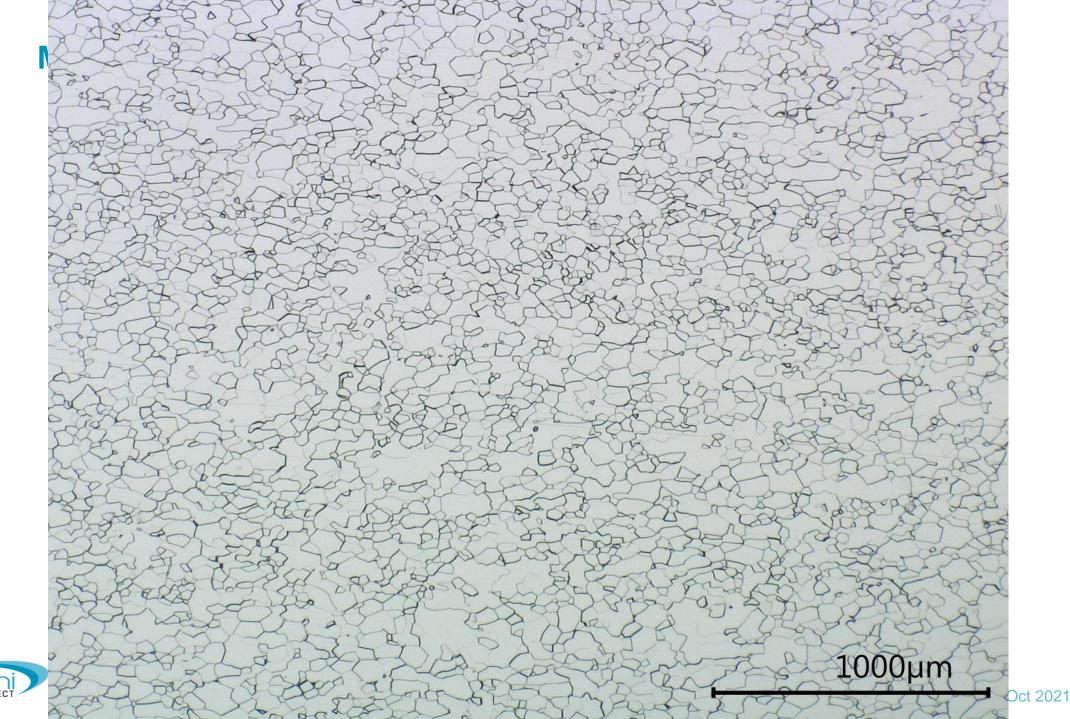
*Error bars represent one standard deviation

- No full recrystallization after heat treatment 650°C, 24h for none of the levels of plastic deformation studied.
- Some of the cavity components with lower Rp0.2 and Rm would be the ones non-plastically deformed during the manufacturing process!





EB welds for HOMs

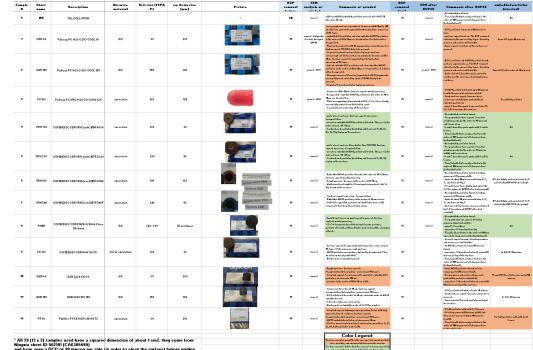




Studies of Nb surface cleanliness after use of several tooling & lubricants

Check surface after using several grinding tools (compare different brands, abrasive media, grit size...).
Determine minimum BCP thickness removal to eliminate embedded abrasives.

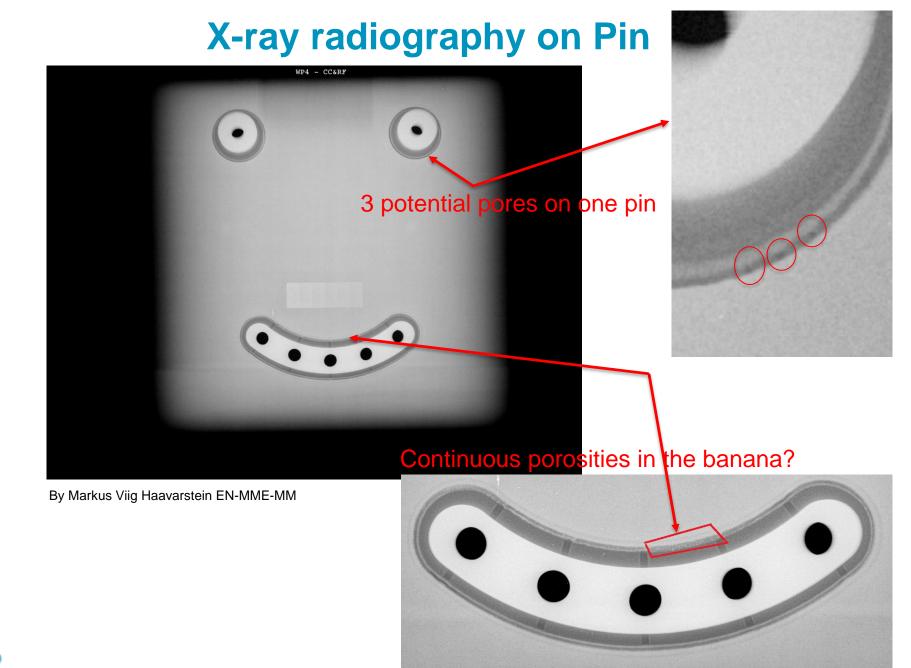




Summary table: EDMS 2258373

Thanks to M. Garlasche, L. Prever-Loiri, K. Eiler, A. Amorim Carvalho







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Ways to increase Rp0.2 in rolled products

Levelling

Skin pass

