TTC meeting, CERN, 4-7 February 2020

Status of infusion studies at IJCLab/IRFU

D. Longuevergne, IJCLab On behalf of all people involved :

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CNTS UNIVERSITÉ IJCLab





- Facilities (the furnace)
- Furnace qualification
 - Conventional H-degassing
 - Infusion-like degassing
- Infusion test
 - First on sample
 - Second on cavity
- Conclusion and upgrades

IJCLab

Furnace presentation

- Built by TAV Vacuum Furnaces Spa and commissioned in 2016
- Routinely used for hydrogen degassing (650°C, 10h) of SPOKE resonators with Ti jacket
- Dimensions : Max diameter : 700 mm. Max Length : 1600 mm
- Temperature up to $1300^{\circ}C$ ($1^{\circ}C 10^{\circ}C$ per minutes). Homogeneity : +/- $5^{\circ}C$
- Vacuum systems

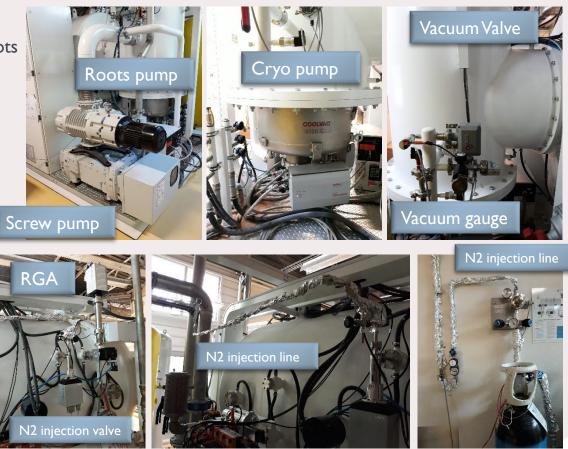
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FACIL

- Primary : Dry screw pump + roots
- Secondary : cryogenic pump
- Vacuum : 5^{E} -7 1^{E} -6 mbar





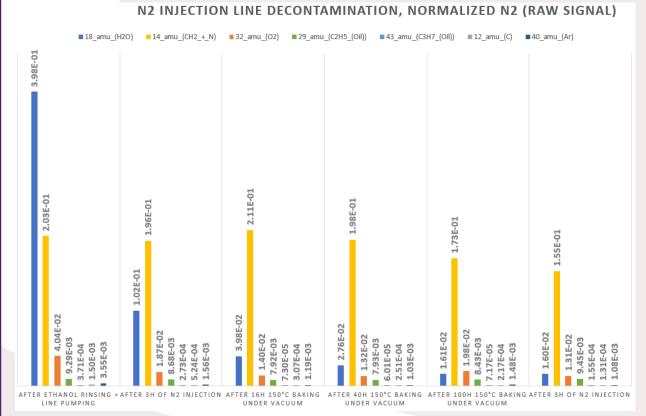
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N2 injection line

- Air liquide ALPHAGAZ 2 (>99.9999%)
- Stainless steel line

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(Ethanol rinsed + 150°C baking for > 100h)
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 Contamination measurement with RGA while injecting at 10⁻⁴ mbar in furnace

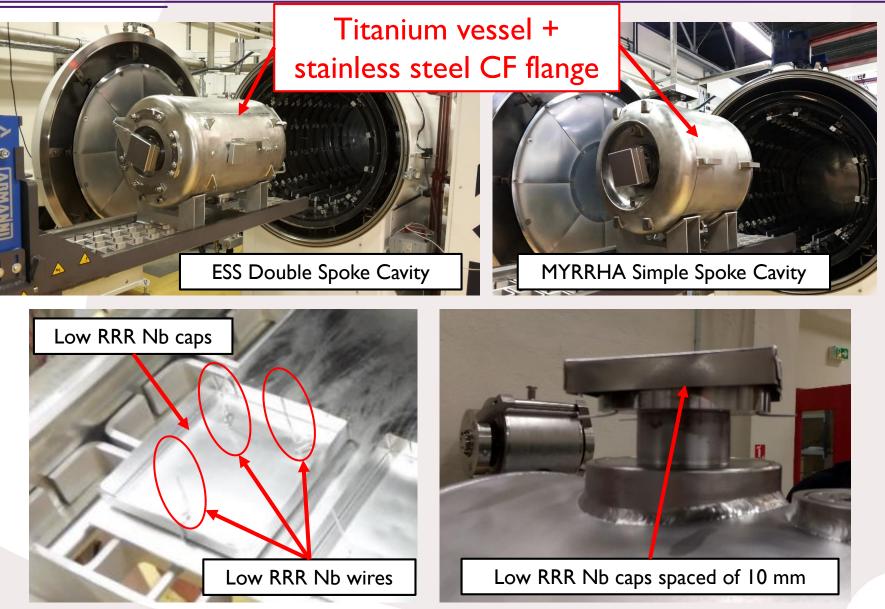


ALPHAGAZ 2 AZOTE

Pureté globale (% mol) N ₂	Impuretés (ppm-mol)		
≥ 99,9999	H ₂ O	≤ 0,5	
	O ₂	≤ 0,1	
	C_H_	≤ 0,1	
	CO	≤ 0,1	
	CO2	≤ 0,1	
	H_2	≤ 0,1	

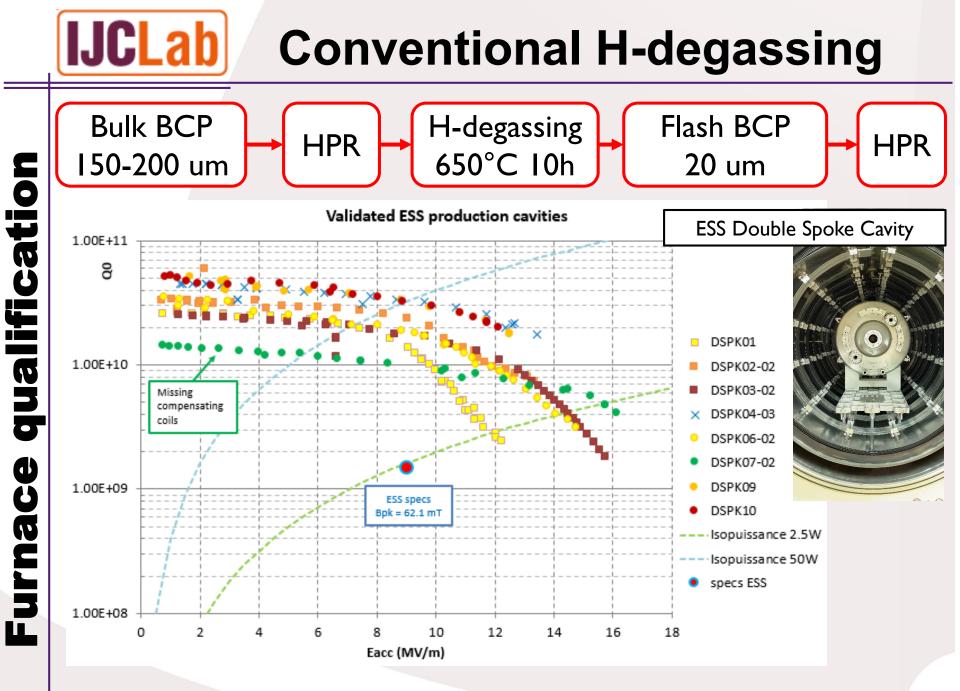


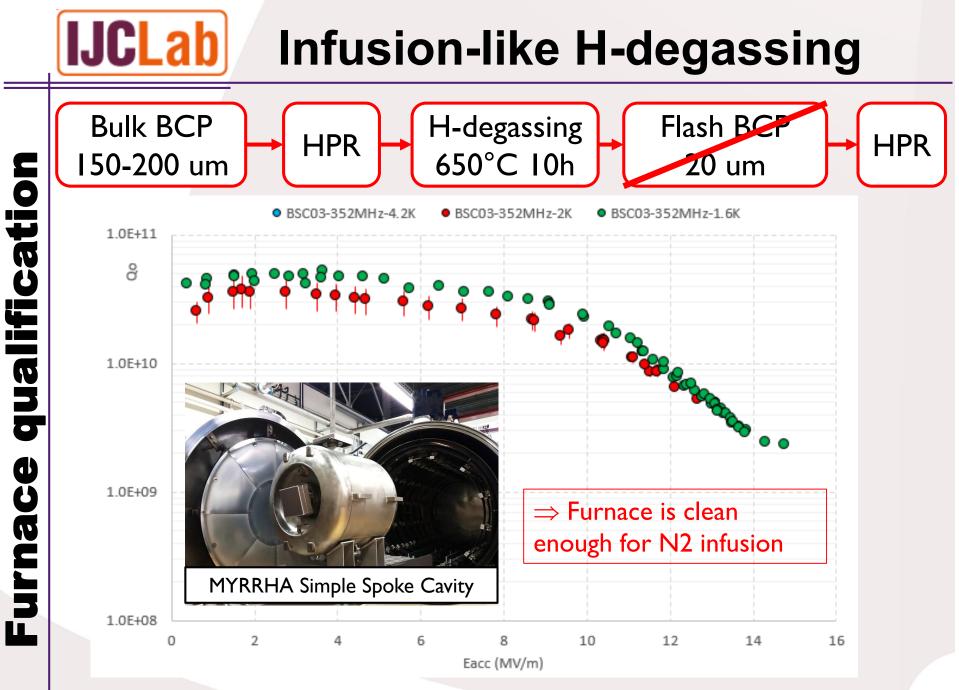
Cavities and caps



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IJCLab On samples

- First infusion test done on Niobium samples (50 um BCP) exposed directly in furnace (not in Niobium box)
- Cycle :

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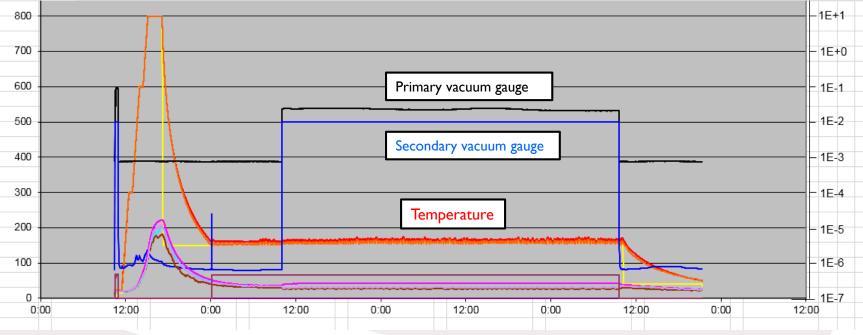
run

test

nfusion

- 800°C during 2h under vacuum
- Cooling down at 160°C under vacuum
- ▶ 160°C during 48h with 2.5^e-2 mbar N2
- Cooling down to 40°C under vacuum







#

run

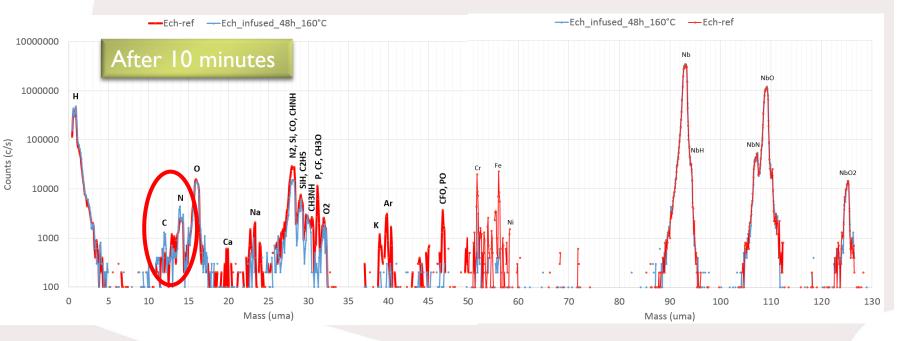
test

Infusion

Surface analysis : SIMS

- Compact SIMS from Hidden Analytical available at IJCLab
 - Argon 5 keV at 300 nA
 - Oxygen 5 keV at 300 nA
- Sample analysis with Oxygen :
 - Red : reference sample (just BCP)
 - Blue : sample infused with N2





IJCLab On cavity and samples

- Second infusion test done on I.3 GHz elliptical cavity (EP) with Nb caps (BCP) + Nb samples (BCP)
- Cycle :
 - 800°C during 2h under vacuum
 - Cooling down at 150°C under vacuum
 - ▶ 150°C during 48h with 2.5^e-2 mbar N2
 - Cooling down to 40°C under vacuum



10 00um

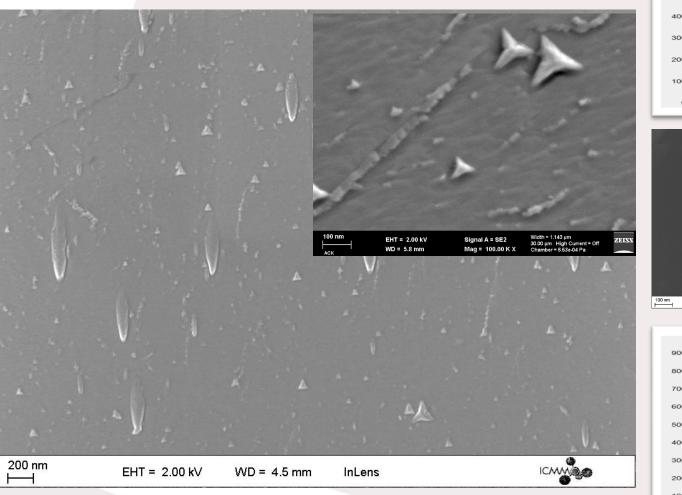
10.00um



Surface analysis: SEM + EDS

800-

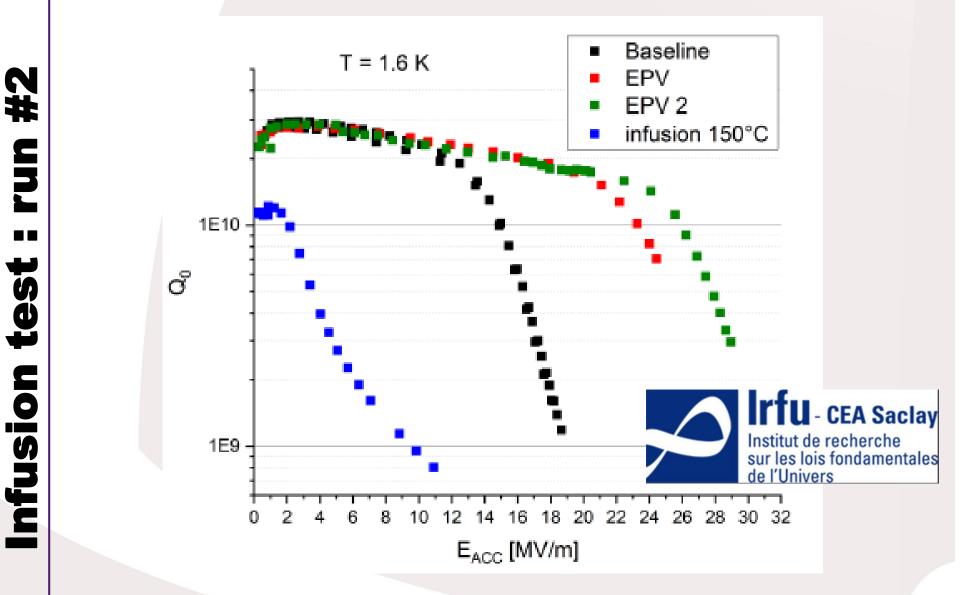
- SEM analysis done at ICMMO (F. Brisset)
 - ZEISS Sigma HD



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700-600-500-400-300-200-100-EHT = 5.00 kV WD = 9.3 mm ICMAN 900-800-700-600-500-400-300-200-100-

IJCLab Vertical cryostat



IJCLab

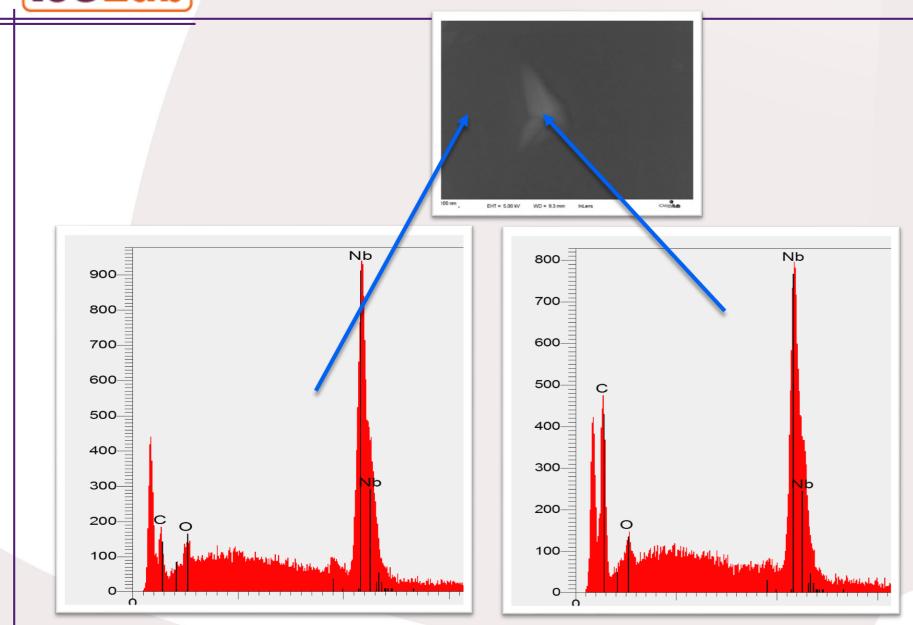
Conclusion

- Furnace cleanliness has been qualified several times
 - Infusion-like H-degassing gives very good cavity performances
- Niobium carbides grow on the surface when nitrogen is injected
 - Nitrogen purity is not good enough
 - Injection line has been cleaned but not thoroughly
 - Chemical filter has been received to filter nitrogen just before injection

SUBTRONIC® line	CATALYTIC REACTORS						
Туре	125	500	1 500	2 500	5 000		
Purified gas	Rare gases, Argon, Nitrogen						
Capacity - Nm ²	125	500	1 500	2 500	5 000		
Removed impurity	H2O, CO, CO2, O2, H2, CH4						
Impurity outlet level – ppb	<1						









100

0

5

10

15

Counts (c/s)

Ech_infused_48h_160°C Ech-ref 1000000 1000000 Н N2, Si, CO, CHNH C2H5 P, CF, CH3O 100000 ο CH3NH CFO, PO 10000 Ν Ar 02 Na К 1000 Ca

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25

Mass (uma)

30

35

40

45

20

50