650MHz Processing Plans at RRCAT

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PIP-II Technical Workshop

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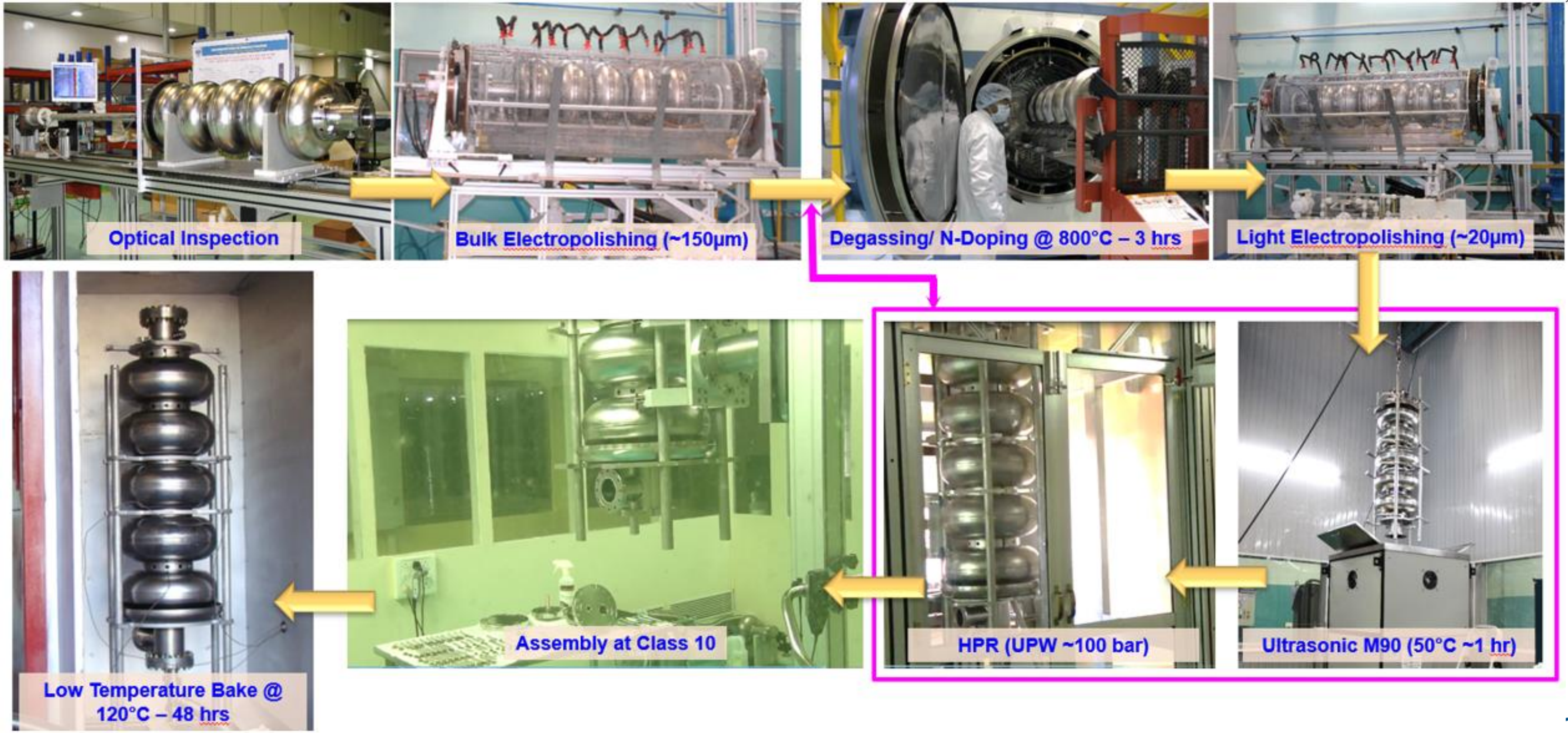
A Partnership of:
- US/DOE
- India/DAE
- Italy/INFN
- UK/UKRI-STFC
- France/CEA, CNRS/IN2P3
- Poland/WUST

Outline

- 650 MHz Cavity Processing and experiences at RRCAT
- Bulk & Light Electropolishing at RRCAT
  - Process and Parameters
  - Facility at RRCAT and its improvements
  - QA & QC
  - Areas of Discussion
- Thermal processing at RRCAT
  - Process and Parameters
  - Facility at RRCAT and its improvements
  - QA & QC
  - Areas of Discussion

650 MHz Cavity Processing at RRCAT



650 MHz Cavity Processing experiences at RRCAT

- Two no.s of Single cell HB 650 MHz cavity (HB102 & HB104)
- Five no.s of 5-cell HB 650 MHz cavity (HB501 – HB505) (Mid 2018 – Till Nov 2020)

HB501

HB502

HB503

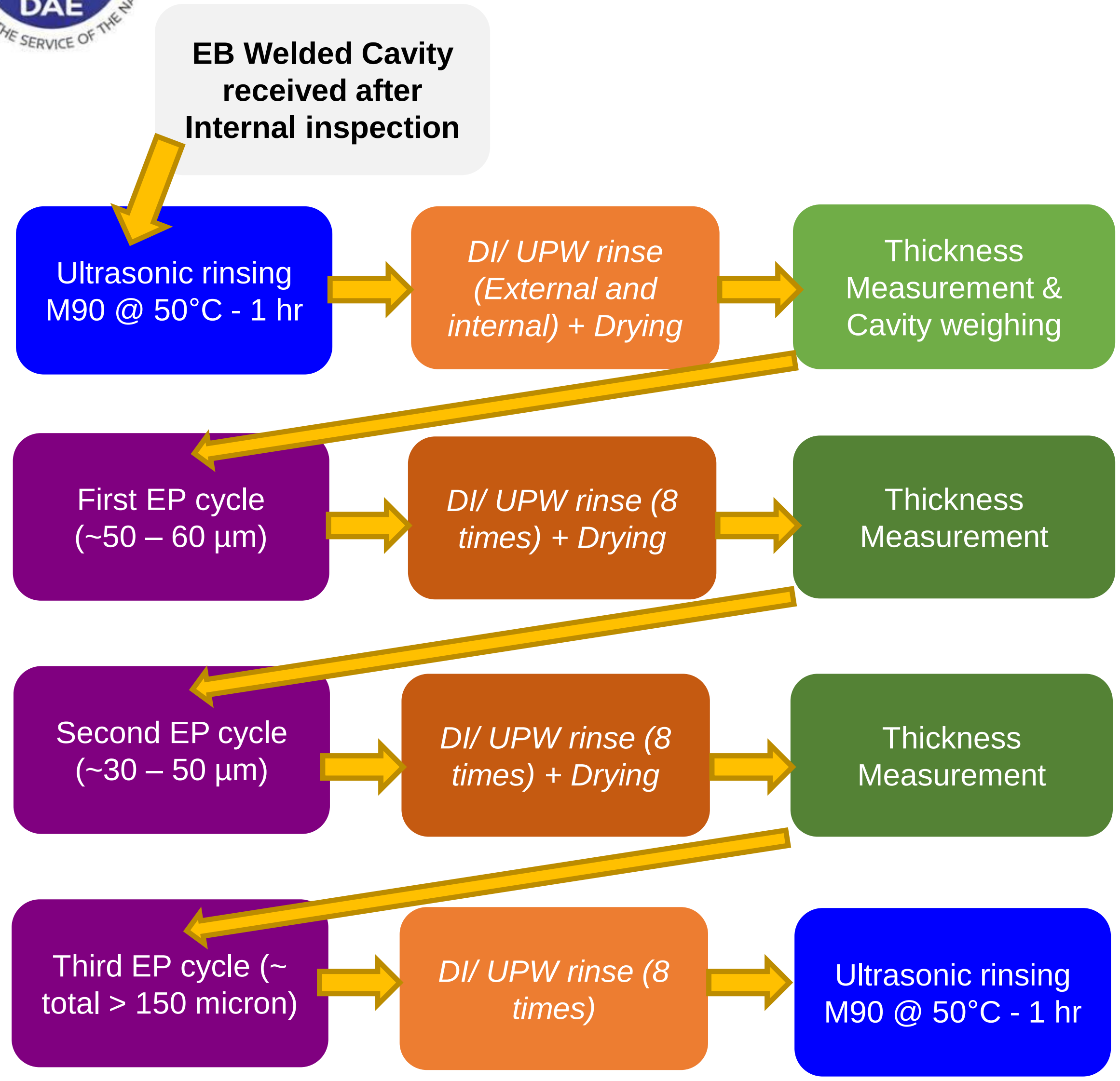
HB504

HB505

Electro-polishing - Brief

- **Purpose**
  - Bulk EP
    - Removal of damaged layer ~120 – 200 µm; Improve surface smoothness; Elimination of sharp discontinuities
  - Light EP
    - Removal of furnace contamination ~ 20µm post Hydrogen degassing process
    - Removal of nitride precipitate layer ~ 5µm post N – doping process

Bulk electro-polishing at RRCAT – Process and Parameters



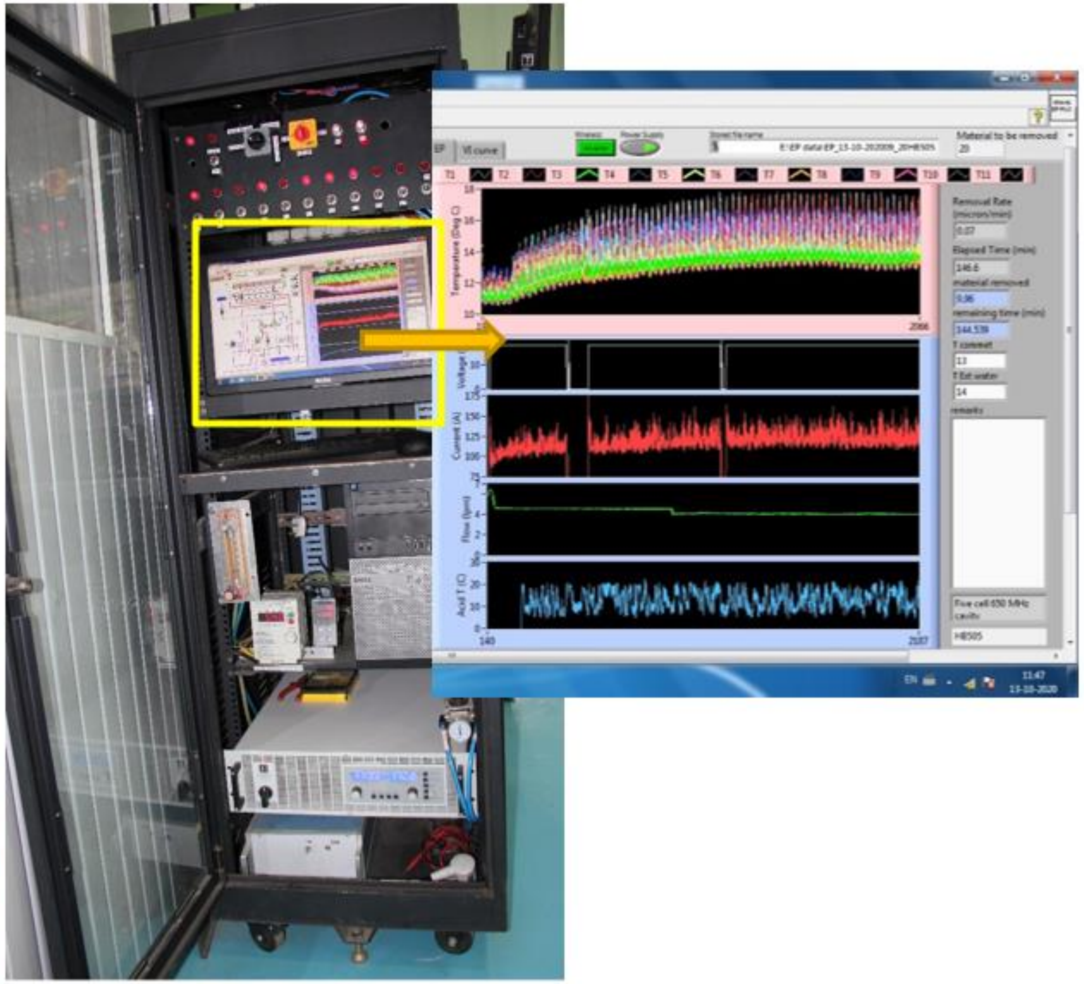
Parameters	Range
Voltage	15 – 18V
Current	175 – 225 A
Current oscillation range	~20 - 35A (peak to peak)
Acid temperature	18 – 23°C
Cavity temperature	17 – 25°C
Removal rate	0.1 – 0.12 µm/ min
Cavity rotation	0.5 – 0.75 rpm
Acid flow rate	4 – 5 lpm
Electrolyte quality (1st and 2nd cycle)	0.5 – 5 gm/ l of Nb (starting) – 10 g/ l of Nb (End)
Electrolyte quality (Final cycle)	2 gm/ l of Nb (starting) to 8gm / l (End)

Light electro-polishing at RRCAT – Process and Parameters

Hydrogen degassed/ N- doped Cavity tuned to > 98% field flatness → Thickness Measurement & Cavity weighing → Light EP cycle (~ 20 / 5 µm) → *DI/ UPW rinse (8 times)* → Ultrasonic rinsing M90 @ 50°C - 1 hr → *DI/ UPW rinse (external and internal)*

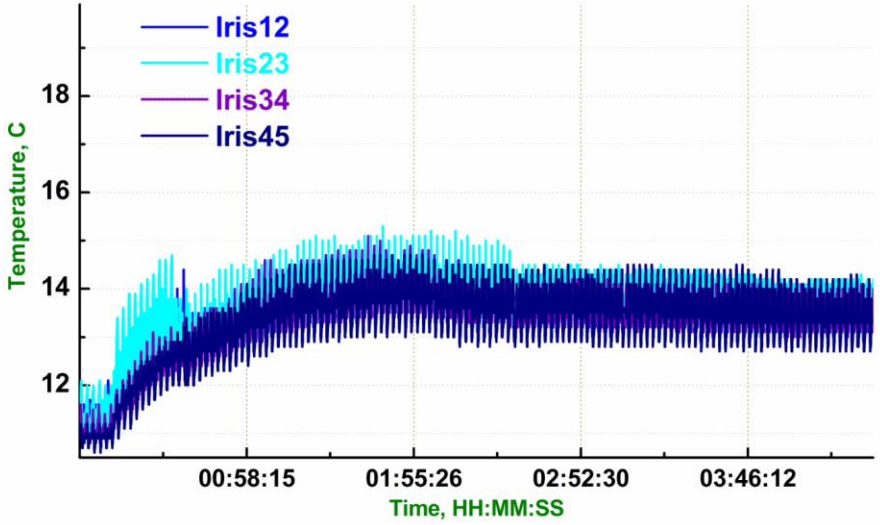
Parameters	Range
Voltage	18V
Current	110 – 135 A
Current oscillation range	~35 A (peak to peak)
Acid temperature	12 - 14°C
Cavity temperature	12 - 17°C
Removal rate	0.06 – 0.07 µm/ min
Cavity rotation	0.5 rpm
Acid flow rate	4 lpm
Electrolyte quality	Fresh electrolyte

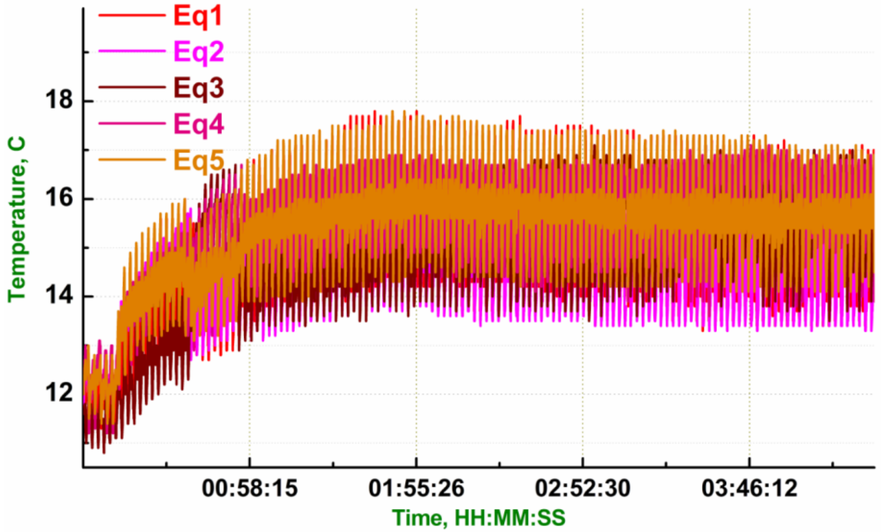
Electropolishing Facility at RRCAT



Team: S. K. Chouhan, A. Singh, S. V. Kokil, D. S. Rajpoot, K. Prasad, L. Soren, D. Vaskel

Improvements – Electro-polishing Facility at RRCAT





- Temperature monitoring of each cell with 2 thermocouples.
- Uniformity in external cooling of each cell with dedicated flow at each cavity wall. Each pipe maintains a flow rate of ~2 lpm.

QA & QC – Electro-polishing at RRCAT

Quality Assurance

- Cleaning of flow circuit prior to light EP cycle.
- Acquisition of minimum of three I-V curves at different intervals.
- Continuous monitoring of EP parameters at definite intervals.
- External surface rinsing with ultra pure water prior to ultrasonic rinsing.

Quality control

- Visual inspection of flange faces, internal surfaces of beam tubes, coupler and pick up ports.
- Optical inspection of internal surface post bulk EP and after VTS.
- Cavity wall thickness and weighing of cavity at different stages of EP process.

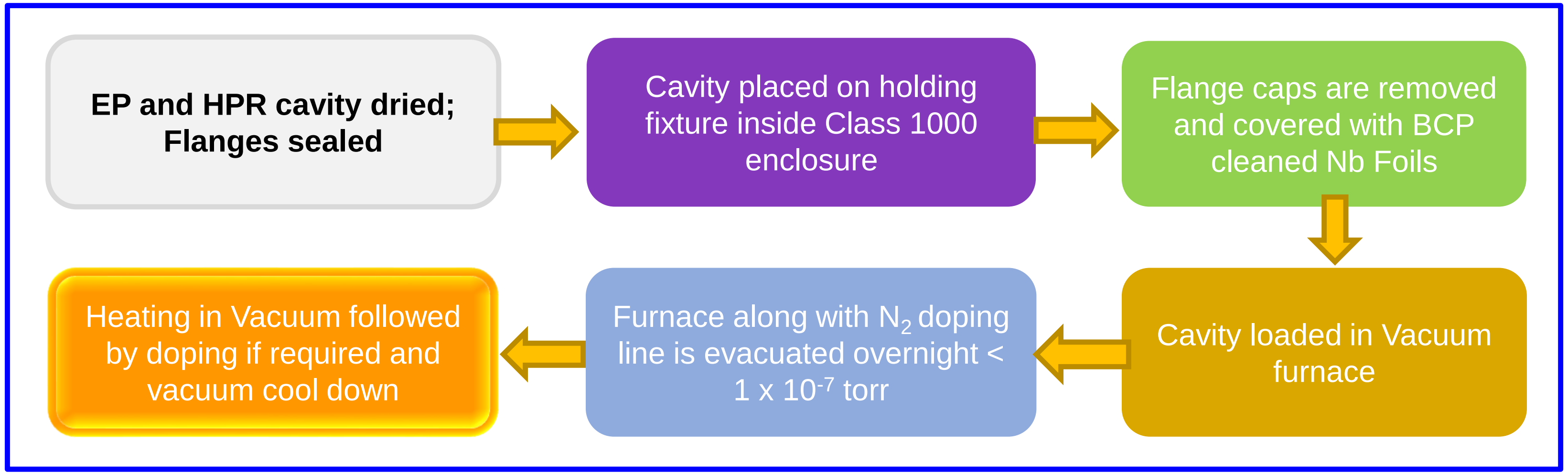
Areas of discussion – Electro-polishing

Areas of concern	Discussion
Cold EP – Current vs Temperature	➢ Cold EP specification - Current < 150A; Temperature < 12°C. ➢ RRCAT Cold EP - Current ~100 – 135A; Temperature oscillates 12 – 16A. ➢ **Since current and temperature are co-related, which is more important and why?**
Allowable Nb concentration in electrolyte for Cold EP	➢ Fresh electrolyte being used at RRCAT. ➢ **What is the maximum allowable Nb concentration for Cold EP?**
Importance of uniformity of Nb removal in N- Doped cavity.	➢ **What is the temperature difference being actually maintained to achieve uniformity?**
Qualification of EP process	➢ Optical inspection is being carried out at various stages of EP. ➢ **It would be beneficial if we can compare these images with other labs.**

Thermal processing at RRCAT – Process and Parameters

Purpose

- Reduce bulk hydrogen in Nb thereby inhibit hydride precipitation during cooldown.
- Improves quality factor (Q_o) of SRF cavity from 10^8 to $>10^{10}$.
- Introduction of N interstitials inhibits further H absorption during light EP doubling the quality factor (Q_o).



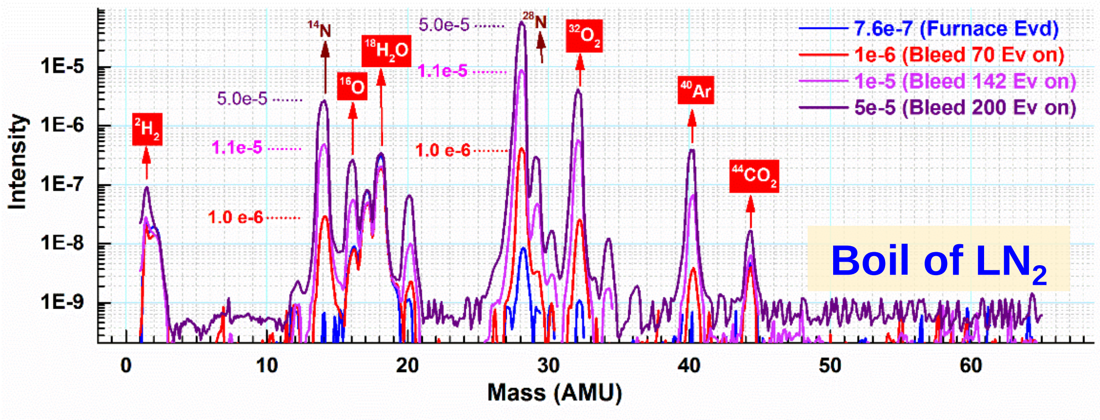
Hydrogen degassing	Nitrogen doping (2/6)
Vacuum before heating < 1 x 10^{-7} torr	Vacuum before heating < 1 x 10^{-7} torr
Heating @ 3°C/min to 800°C (in vacuum)	Heating @ 3°C/min to 800°C (in vacuum)
Soaking for 3 hrs (< 5 x 10^{-6} torr)	Soaking for 3 hrs (< 5 x 10^{-6} torr)
-----------	N_2 (5N) injection (25±2 mtorr – 2 min)
-----------	Evacuate to high vacuum + 6 min soak
Cooldown in vacuum to 50°C	Cooldown in vacuum to 50°C

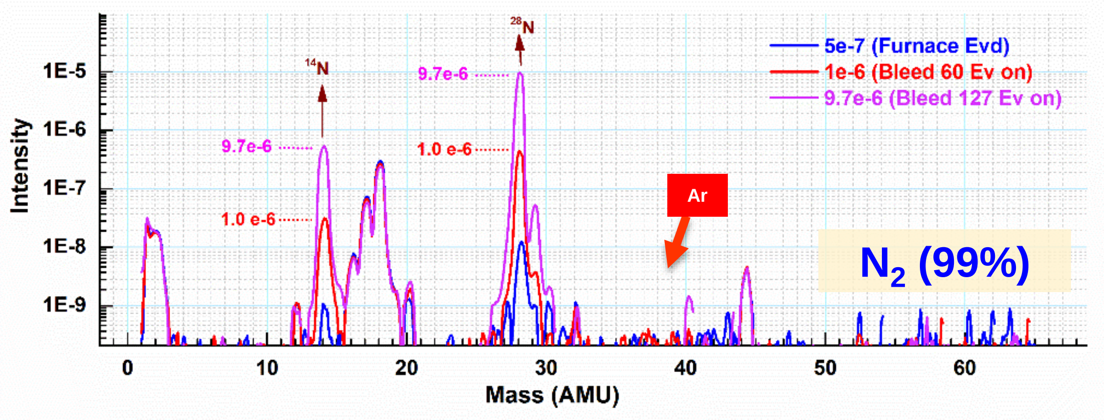
Thermal Processing Facility at RRCAT

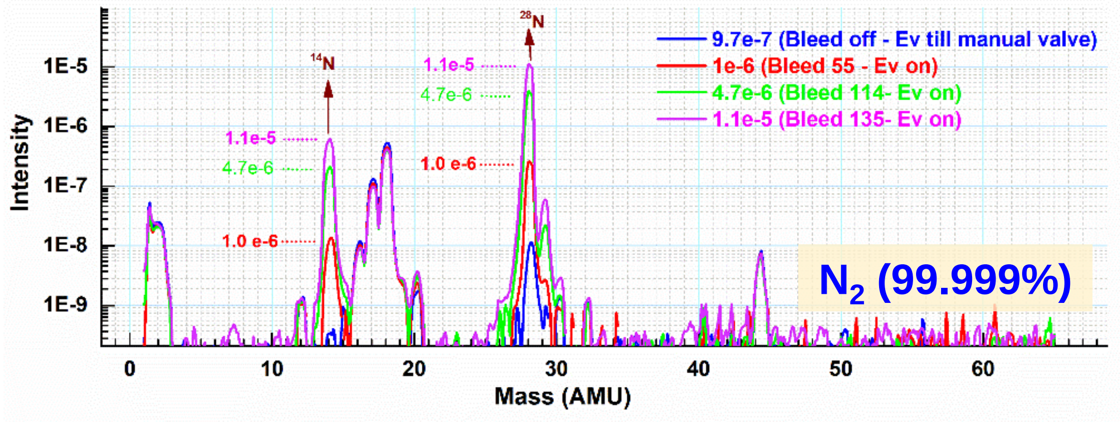
Team: S. V. Kokil, D. S. Rajpoot, L. Soren

Improvements – Thermal Processing at RRCAT

- Nitrogen line purity and leak check of furnace lines using RGA are meticulously followed.
- A comparison of nitrogen purity between boil off LN_2, commercial 2N N_2 cylinder and high purity 5N N_2 cylinder is presented using the same N2 bleeding line.







QA & QC – Thermal processing at RRCAT

Quality Assurance

- Class 1000 clean room protocol followed during assembly, loading and unloading of the cavity.
- Enclosure cleanliness monitored using particle counter.
- Cavity holding fixture and Nb supporting plates for holding cavity are stored in vacuum and cleaned using IPA.
- Nb foils are BCP cleaned, ultrasonically rinsed and dried using filtered air.
- Purity and leak tightness of the nitrogen line is checked at RT prior to doping cycle.
- Partial pressure of gases are monitored continuously during thermal cycles using RGA.

Quality control

- RGA data analysis.
- Impurities, specifically H and N are measured in samples after cavity degassing using TOF-SIM technique.
- The cavity is visually inspected to observe reduction in external surface contamination after the degassing cycle.

Areas of discussion – Thermal processing

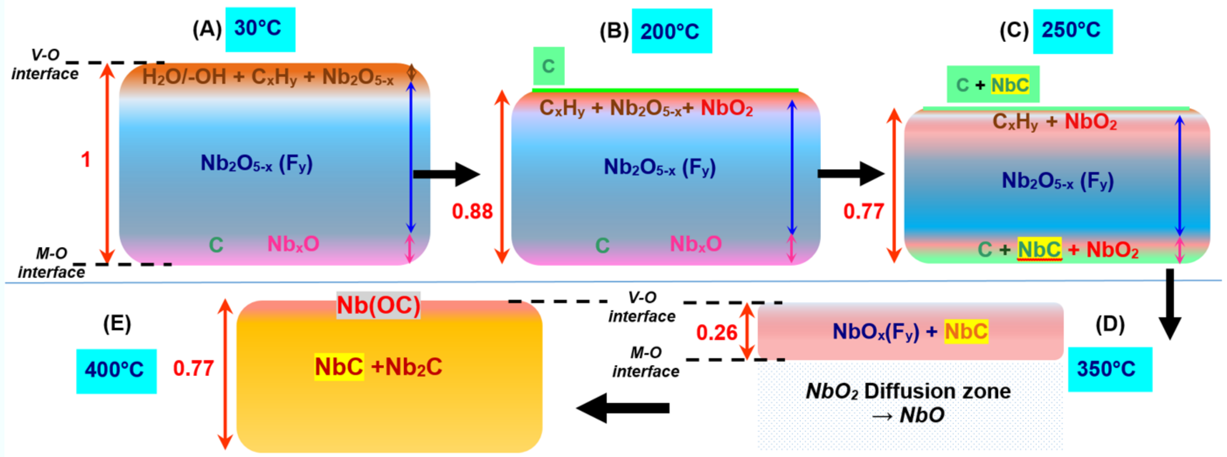
Areas of concern	Discussion
Upper vacuum limit during heating cycle	➢ RRCAT – As vacuum > 5 x 10^{-6} torr the furnace heating is "on hold" till vacuum improves < 2 x 10^{-6} torr. ➢ **It sometimes lead to long hold up time.**
Partial pressure of H_2 post thermal processing	➢ RRCAT - < 5 x 10^{-7} torr. ➢ **Is there a maximum allowable specification of the partial pressure of H_2 gas?**
Shielding process	➢ RRCAT – As shown in previous figure. ➢ **Any separate protocol that can be of advantage.**

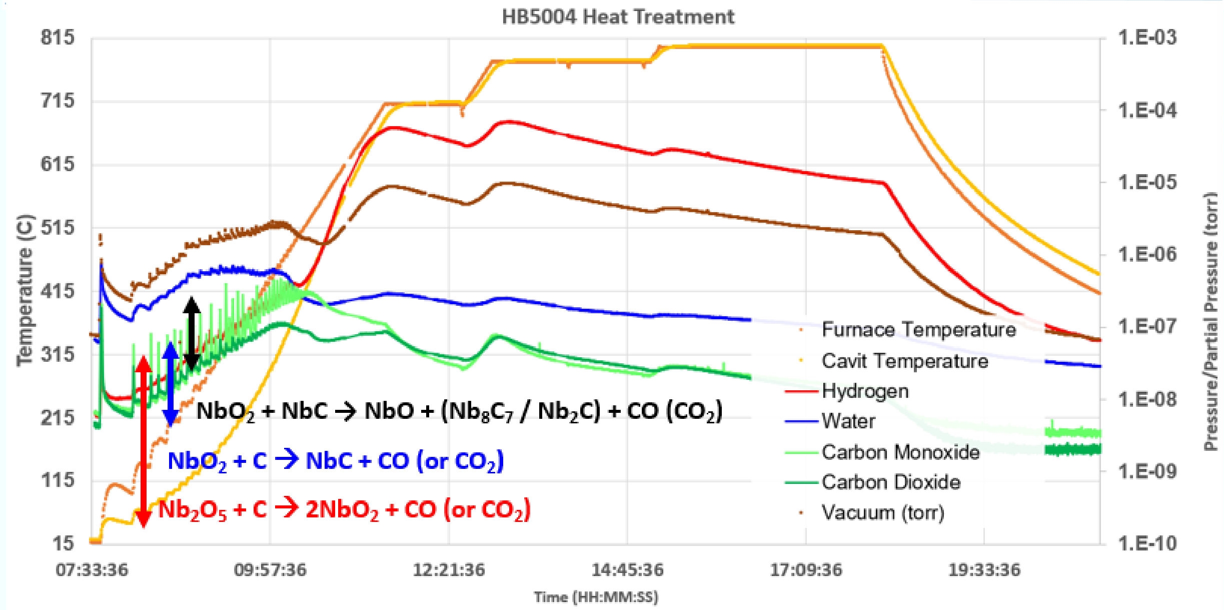
Thermal processing – Areas of concern and its resolution

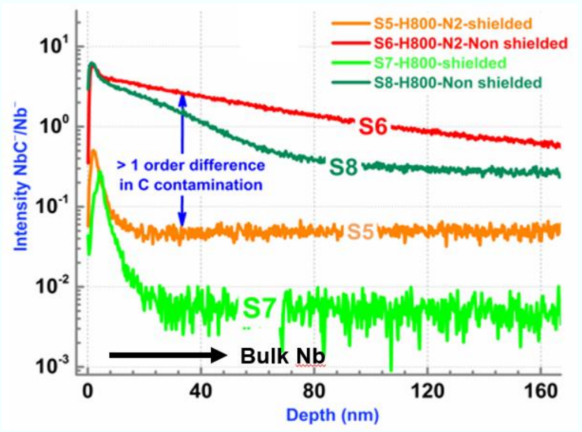
Highlights

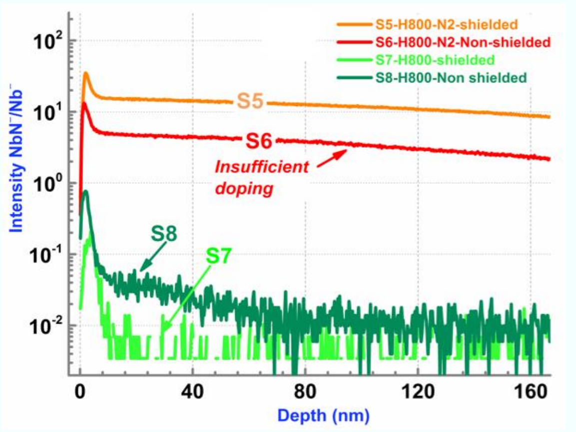
- ✓ Mechanism of impurity (H, C, O, N, S, F) evolution during degassing and N-doping
- ✓ Role of carbide layer in shielding and its beneficial effect
- ✓ Reason behind variation of impurities between various heating cycles.

A. Bose, et. al., Applied Surface Science 510:145464, Apr 2020









Thank You