



## WP13 Meeting – 19 April 2023

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on behalf of WP13 partners (CEA, CIEMAT, DESY, IFJ-PAN, INFN, STFC)



# Introduction:

## Group F: Platforms for characterization, treatments and test of materials

The platforms considered in this category serve for the characterization and treatment of materials/components used for accelerators and superconductive magnets. The platforms could be used for several purposes:

- Improvement of material performances
- Treatment Process optimization and qualification
- Materials treatment before tests
- Quality Control and material characterization
- Development of new materials

The platforms of group F are classified in 4 sub-categories:

- F1. Thermal treatment platforms
- F2. Chemical treatment platforms
- F3. Facilities for surface analysis
- F4. Electromagnetic, mechanical, thermal & associated material characterization



# F1. Thermal treatment platforms :

## @ CNRS: High Temperature Vacuum Furnace:

### Activities:

- **Hydrogen outgassing** of different type of SRF cavities :
  - 1.3 GHz single-cell elliptical resonators,
  - 352 MHz Double-Spoke(ESS) and single spoke (Myrrha & PIP-II) cavities
  - 702 MHz medium and high beta Elliptical cavities (ESS)
- **Advanced Heat Treatment R&D:** N-doping, N-Infusion, Medium temperature Annealing

### Technical characteristics:

- Thermal chamber volume: 4.5 m<sup>3</sup> → Maximum cavity diameter and length: 700 mm and 1600 mm,
- Molybdenum Heaters (3 heating zones, 5 Mo radiation shields) → Heating rate: 1-10 °C/min; Maximum temperature: 1400°C,
- Dry pumping system (Screw pump, Roots pump and Cryogenic pump) → Residual Pressure: 5.10<sup>-7</sup> mbar- 10<sup>-6</sup> mbar at 300K;
- Nitrogen doping/infusion circuit (AlphaGaz 2 grade N2) with micrometric valve and catalytic and adsorption filter (Gas flow @300K: 10<sup>-10</sup> et 500 mbar.l.s<sup>-1</sup>)



# F1. Thermal treatment platforms :

## @ DESY: High Temperature Vacuum Furnaces

### Activities:

- R&D program to **improve Q(E) behaviour** of 1.3 GHz cavities (N-infusion and mid-T bake under study) → The goal is CW gradients above 25 MV/m at  $Q_0$  larger than  $3 \cdot 10^{10}$  or + 30 MV/m in pulsed mode with  $Q_0$  above  $10^{10}$ .

### Technical characteristics:

- **Standard furnace:**
  - Can house **up to 4 cavities** of the 1.3 GHz 9-cell type (XFEL type)
  - Usually used for 800°C baking
  - Directly **attached to an ISO 4 cleanroom**
  - Two cryogenic pumps as vacuum systems (heater and niobium retort separated) → Starting pressure at room temperature of  $2 \cdot 10^{-8}$  mbar, and at 300° C about  $3 \cdot 10^{-7}$  mbar.
- A dedicated **sample furnace** as well as a **new UHV furnace** suited for **1.3 GHz single cell cavities** is operated by the Hamburg University, on DESY premises → offer more possibilities including the study of SIS layers



# F1. Thermal treatment platforms :

## @ INFN-LNF: High Temperature Vacuum Furnaces

### Activities:

Thermal treatments and vacuum brazing → R&D on X-Band Accelerating Structure development (EUPRAXIA@S)  
Open to companies and other RI's → vacuum firing treatments and brazing of other vacuum technology components

### Technical characteristics:

- Furnace operating volume: Cylinder of 400 mm (diameter) and 1300 mm (height)
- Can operate up to 1200°C with a base pressure out of thermal cycle is of  $10^{-6}$  mbar.

### Upgrading Plans:

Increasing the vacuum pumping to better treat larger mass components and reducing issues related to thermal cycling



## @ INFN-LNL: High Temperature Vacuum Furnaces

### Activities:

Production of copper modules by brazing of copper-to-copper or copper-to-steel with copper-silver and palladium-based alloys for several accelerator projects: ALPI, IFMIF/EVEDA, ESS and SPES.

### Technical characteristics:

- Homogeneous hot zone at temperatures **up to 1300°C** → 2m<sup>3</sup> (cylinder with diameter 1.3m and height 1.6m) with an homogeneity within +/-4°C. Possibility to **increase the height** of the furnace to 2.1 m by installing a volume extension.
- Dry vacuum system: primary screw pump & 2 cryogenic pumps during operation → Vacuum level in operation:  $10^{-6}$  mbar
- **Batch-type**, with a load capacity of up to 1000kg

### Upgrading Plans:

Development of a system for reactive or reducing atmospheres use is planned → In addition to a high vacuum level, atmospheres of high-purity gases such as hydrogen, nitrogen, and argon are expected to be available.





## F2. Chemical treatment platforms :

### @ CNRS:

#### Description:

A **BCP etching** can be performed on **large cavities** (max inner volume ~ 100L). 2 chillers allow to cool down the acid bath down to 8°C and the cavity (if equipped with helium tank) down to 10°C. **Rotational BCP has been recently implemented and validated.** A clean room (**ISO4**) is also in operation for the final assembly of cavities. It hosts a **High Pressure Rinsing (HPR)** unit for the final cleaning of cavities.

#### Activities:

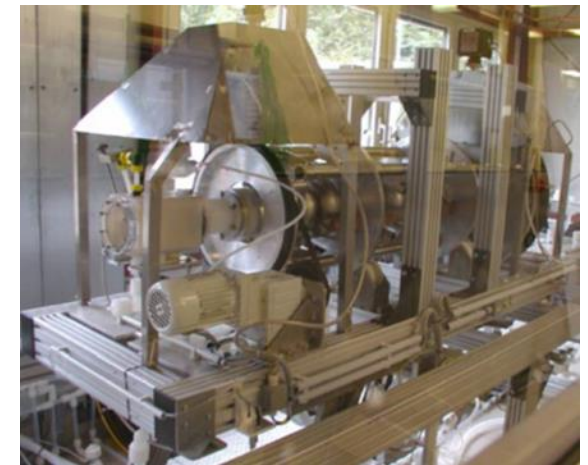
Several low-beta SRF cavities for different European project were treated in this platform: 14 QWR cavities at 88 MHz for SPIRAL 2, 26 Double-Spoke resonators at 352 MHz for ESS, and is today strongly involved in R&D and production phases for MYRRHA/MINERVA (Single Spoke at 352 MHz) and PIP2 (Single Spoke 325 MHz) projects.



### @ DESY:

#### Description:

A **BCP etching** as well as **Electropolishing** of single and 9-cell Niobium cavities at 1.3 GHz are possible. The platform was used as a mock-up for comparable platforms at cavity vendors. Today it is **only operated for DESY internal campaigns** (1.3 GHz Cavities and SRF Guns). **High pressure rinsing** (two stations) is available. An **ISO4 clean room** is right next to it, to allow assembly of cavities.



## F2. Chemical treatment platforms :

### @ CEA: Vertical electropolishing cabinet

#### Activities:

**Chemical and electrochemical treatment of elliptical Niobium cavities at different frequencies:**

- 5-Cell 704 MHz prototypes for SPL and ESS projects
- 1,3 GHz multi-cell and single cell cavities for several R&D programs (multilayers, thermal treatments, doping & infusion)

#### Technical characteristics:

- Maximum cavity height: 1.8 m and maximum mass: 200 kg
- Power supply: 20 V, 1200 A,
- Acid flow rate: 40 l/min, 300 l acid tank, Pipe and pumps made of PFA material, Teflon coating of the acid tank,
- Use of rotating cathode for uniform material removal (CEA-KEK-Marui collaboration)



### @ CEA: Chemical Treatment cabinet

#### Activities:

The platform has been used for several **prototypes** and **series cavities**: 1/2 wave and 1/4 wave resonators for Spiral2, DONES & IFMIF EVEDA, low beta cavities and high beta cavities 176MHz for SARAF accelerators, ESS 704MHz medium beta prototype cavities, 1,3GHz cavities for R&D activities (thermal treatments, ALD layer deposition).

#### Technical characteristics:

PNF bath: H<sub>3</sub>PO<sub>4</sub>48% – HNO<sub>3</sub>36% et HF40% in the proportions 2.4–1– 1, with an acid tank of 200 l, acid circulation from bottom upwards with a maximum flow rate of 30 l/min, gravity draining, PVDF piping, station under ventilated fume hood.



# F3. Facilities for surface analysis:

## @ CNRS: Vacuum and Surface platform

### Activities:

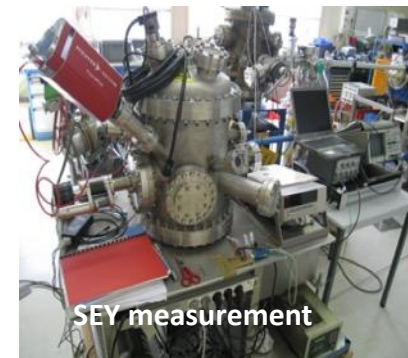
The platform is involved in all R&D activities and project of the accelerator division. It is today strongly involved in the **R&D for superconducting cavities** and the **study of dynamic vacuum in accelerators** and the **mitigation of e-cloud** (LHC, FCC).

### Technical characteristics:

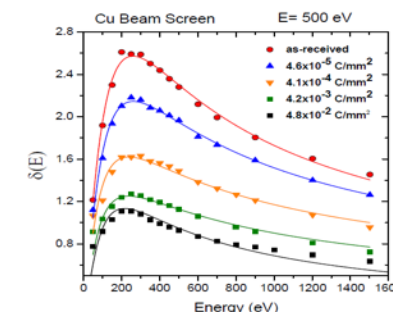
The platform is equipped with several commercial equipment: laser **confocal microscope**, **SIMS**, **SEM equipped with EDX and EBSD**, **GDRX** and as well **in-house developed equipment** for the measurement of **SEY** (Secondary Electron Yield) and **surface desorption yield** at room temperature. The platform is also equipped with **NEG deposition system**.

### Future upgrade:

The analysis capability of the platform will be greatly improved by the acquisition of a commercial XPS and a **multi-technique system** equipped with XPS and SEY **operational between 300K and down to 10K**.



SEY measurement



Outgassing rate



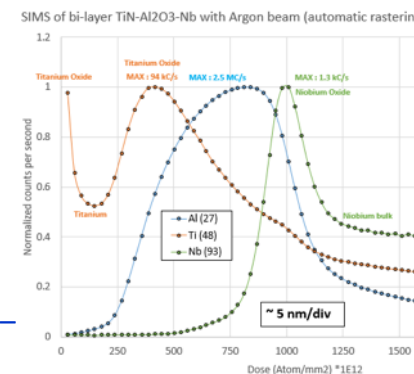
NEG Coating chamber



Scanning Electron Microscopy (SEM)



Compact SIMS



X-Ray Diffractometer





## F3. Facilities for surface analysis :

### @ CIEMAT: Microstructural Characterization Lab

#### Activities:

The facility has different rooms with **distinct electron microscopy and surface analysis devices** for microstructural studies of materials. It is a valuable tool in a variety of fields (materials science, metallurgy, nanotechnology...), crucial for advancing in the understanding of materials and developing new technologies with improved performance and reliability. This facility is composed by the following infrastructures: Scanning electron microscope with tungsten filament (**SEM**), Scanning electron microscopy (**FEGSTEM-EDX, BSE, EBDS**), **Scanning Auger microprobe**, **X-ray photoelectron spectroscopy (XPS/ESCA)** and **Transmission electron microscope (TEM)**.

Some of the utilities of the laboratory allow:

- Examination of the **crystal structure** and **materials composition** to understand their properties and behaviours.
- Analysis of the **morphology** and **distribution of particles, defects, and phases** in materials to assess their quality and performance.
- Observation of the **surface topography** and roughness of materials to evaluate their surface properties and behaviour.
- **Identification of any defects** or abnormalities in materials that may affect their functionality or reliability.
- **Development** and testing of new materials with **specific microstructural properties** tailored to meet specific applications and requirements.

## F3. Facilities for surface analysis :

### @ DESY: Quality Control of Niobium sheets

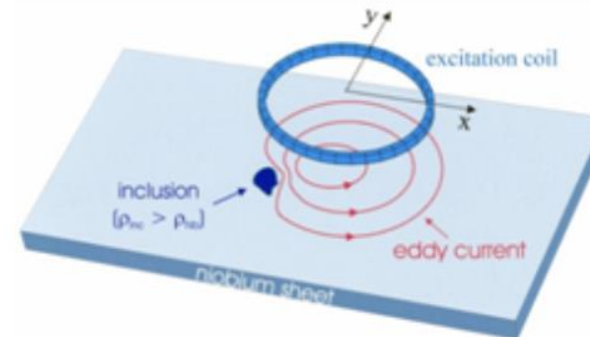
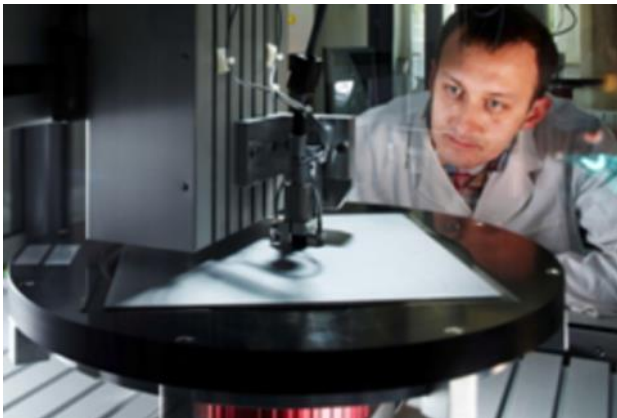
#### Activities:

**Scanning facility** to check **Niobium sheets** of different size. All sheets for the 800+ European XFEL cavities but also all sheets for LCLS-II and LCLS-II HE were scanned during the last years. Since the facility is unique, many more institutes in the SRF community asked for QC of sheets for a large number of research facilities (e.g. SNS, ESS, MYRRHA, SHINE, ...).

#### Technical characteristics:

Description : Scanning facility based on the **eddy current** in order to **determine the so-called RF side** of the Niobium sheet. In case of inclusions, additional detailed studies are possible to help identifying the cause.

**Future upgrade:** The platform is going to be upgraded according to the today known needs. It is used for many service contracts (research labs and industry)



Principle of eddy current measurement

## F3. Facilities for surface analysis :

### @ INFN: Outgassing characterization Facility - LATINO

#### Activites:

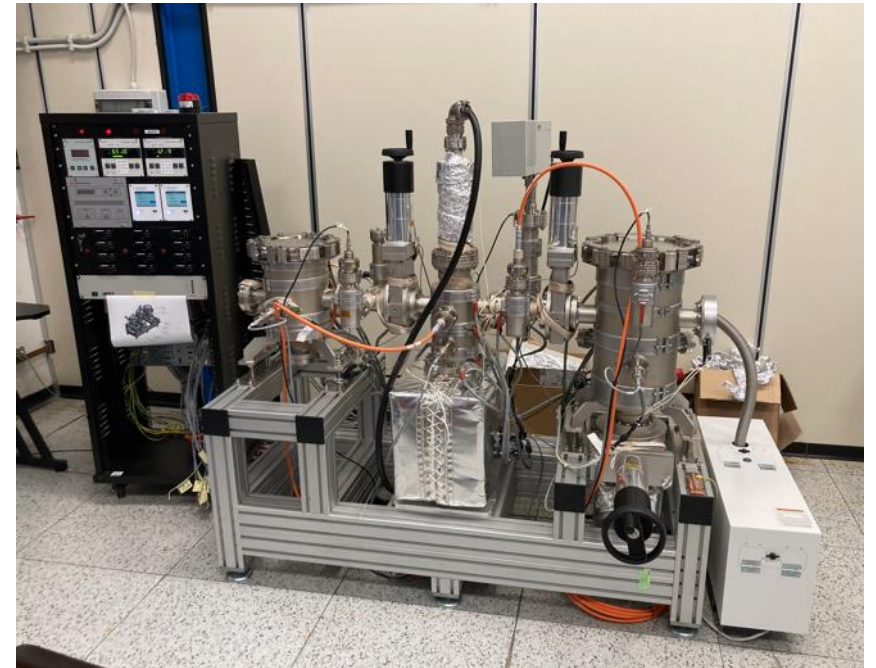
LATINO is High Vacuum Station for specific **outgassing investigation on materials** for vacuum applications. In these years the system was commissioned and used for the characterization of standard materials, such as **Stainless Steel and Aluminium** alloys. Presently, the facility is integrated in the **R&D activities** for the **characterization of materials** impinging on the vacuum system of the cryogenic tower of future gravitational wave detector, the Einstein Telescope.

#### Technical characteristics:

The installation is based on the Throughput Method and it is composed of a main chamber, the measurement one, and two other chambers dedicated to high outgassing materials and low outgassing materials. These two sample chambers are cylinders of 200mm(d)x300mm(h) and 250mm(d)x450mm(h) . The facility is also equipped with an RGA to complete the characterization with the spectrum of the gas emitted by the materials.

#### Upgrade plans:

In order to increase the sensitivity of the system, in the future plans the possibility of characterization with the accumulation method and/or the combination of accumulation and throughput one will be integrated.



## F3. Facilities for surface analysis :

### @ STFC: Vacuum Interface and Surface Technologies for Accelerators - VISTA Laboratory

#### Activities:

VISTA laboratory is the main hub for both **vacuum science** and **surface science R&D** within the UK accelerator community. Recent work programmes have been centred around activities such as **NEG** and **novel coating developments** for DLS-II and PETRA-IV; **secondary electron suppression** schemes for CERN; **superconducting thin film development** for iFAST; **CsTe photocathode** development for UK-XFEL. The facilities and expertise within the VISTA laboratory can be accessed by both **academic and industrial researchers**, subject to availability.

#### Technical characteristics:

The laboratory's suite of instruments cover specialisms such as **vacuum system analysis**, **vacuum metrology** and **gauge calibration**, and XHV processing for large vessels. As well as supporting **PVD**, **CVD** and **ALD deposition** development for complex freeform and long structures, the laboratory is equipped with full surface analysis capabilities including **RBS**, **NRA**, **MEIS**, **LEIS**, **AES**, **XPS**, **SIMS**, **SEM** and **TEM**.

#### Future plans:

Short-term development plans centred around the expansion of thin film coating capability to ever larger, longer and more complex substrates, and further developments in surface micro-structuring capabilities.





# F4. Electromagnetic, mechanical, thermal & associated material characterization :

## @ CNRS: Electrical resistivity & RRR measurements

### Activites:

These measurements are crucial for: 1) **Quality Control (QC)** of Niobium used for producing SRF cavities, 2) SRF **cavities processing qualification** and **thermal process** (H outgassing) **optimization**, 3) R&D on **Advanced Heat treatment** (N-Infusion, N-doping, Intermediate Temperature baking), 4) **Characterization of material** for High Power RF couplers (Stainless steel and copper films), 5) **Development of new materials** (e.g. superconducting thin films or produced by additive manufacturing or other methods)

### Technical characteristics:

**RRR measurements** are performed using the standard DC four probes method with reversing the sensing current to eliminate parasitic thermoelectric voltages. The electrodes are clamped to the flat test sample by means of copper-beryllium springs (contact pressure: ~12 Bars). The test sample (length: 80 mm), which is equipped with a calibrated Cernox thermometer, is immersed in a saturated liquid helium bath at  $T = 4.2\text{K}$ . The sensing current of 1 A, is delivered by a precise standard DC voltage supply, is on-line measured via the voltage drop across a precision resistor.

The **thermal conductivity  $k(T)$**  measurement device allows the test of four samples simultaneously during each run. The measurements are performed at low temperature (1.5 K- 30K) using the steady-state axial heat flow method with a careful control of heat leaks to the surrounding.

# F4. Electromagnetic, mechanical, thermal & associated material characterization :

## @ CEA: Characterisation laboratory at cryogenic temperature (LABCAF)

### Activites:

A **cryostat with a removable bottom insert**, currently used for first **critical field HC1 measurements** by local magnetometry. A second cryostat used for **RRR measurements**. The lab perform quality-control measurements on materials and is used on R&D programs.

### Technical characteristics:

The useful dimensions of the cryostat 1 are  $h = 1.33$  m and  $\Phi = 30$  cm,

The useful dimensions of the second cryostat are  $h = 1$  m and  $\Phi = 15$  cm.

Nitrogen and helium Dewars ( $T \geq 1.9$  K),

Precision voltmeters and amperemeters, temperature controllers

# F4. Electromagnetic, mechanical, thermal & associated material characterization :

## @ IFJ-PAN: Superconductor characterisation test stand

### Activities:

The platform can be used to characterise **critical current of superconducting wire strands** made of various materials. Additionally it will be possible to perform **resistance** and **RRR measurements**.

### Technical characteristics:

The platform is based on **16 T superconducting magnet** with a **Variable Temperature Insert (VTI)**. The temperature inside the VTI is controlled by a calibrated Cernox sensor and can be regulated between **1.8 K and 200 K**. The current of **up to 1000 A** can be delivered to the sample from external power converter using two current leads.

### Upgrading plans:

The capability of the test station could be **further improved** by the acquisition of the sample holder which enables **applying variable mechanical stress** to the sample

# Conclusions :

- Wide variety of complementary Technical Platforms (TPs) for accelerator developments in the several European labs.
- Implication of the TPs in the main accelerator projects realization in Europe and worldwide.
- The TPs are involved in the R&D effort made by the labs to optimise some pre-treatment recipe, Improve the performances, to develop new materials with new performances... and also in the prototyping and series production phases with single or batch treatment process, Quality Control and qualification of components.
- Some upgrading plan are foreseen for some TPs to meet required performances for some future needs.
- Most of the TPs are open to industrial collaboration.



# iFAST

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