

**Workshop "Advanced Mechanics in Accelerator Technology" (AMAT),  
19 April 2013, CERN**

# **Materials studies and tests at CERN**

- Mandate and expertise
- Equipment
- Examples of material studies and tests
- Perspectives

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EN-MME-MM

# EN-MME-



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*Aline Piquet*



*Ignacio Avilés*



*Barbora Bartova*



*Ana Teresa Pérez*



*Stephane Marcuzzi*



*Dominique Pugat*



*Ahmed Cherif*



*Stefano Sqobba*



*Gonzalo Arnau Izquierdo*



*Floriane Leaux*

*March 2013*



*Patrice Francon*



*Jean-Philippe Rigaud*



*Philippe Deweulf*



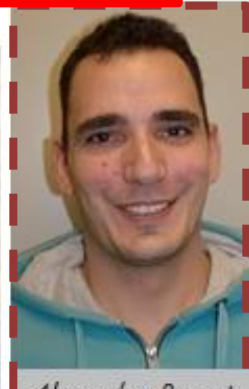
*Alexandre Gerardin*



*Melinda Hiltbrand*



*Dawid Marcinek*



*Alexandre Porret*



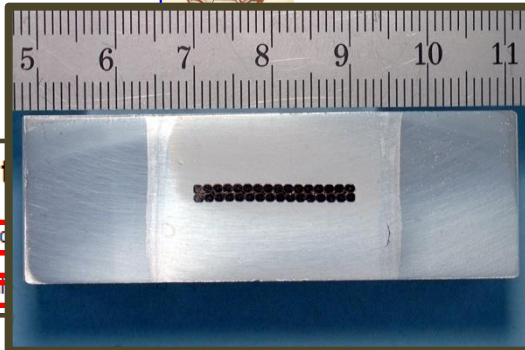
# EN Engineering Department



Home > Groups > MME > MM > - Mandate

## Metallurgy and Metrology Section

Undertakes the development, selection, specific control of materials and components including materials and components. Performs failure analysis

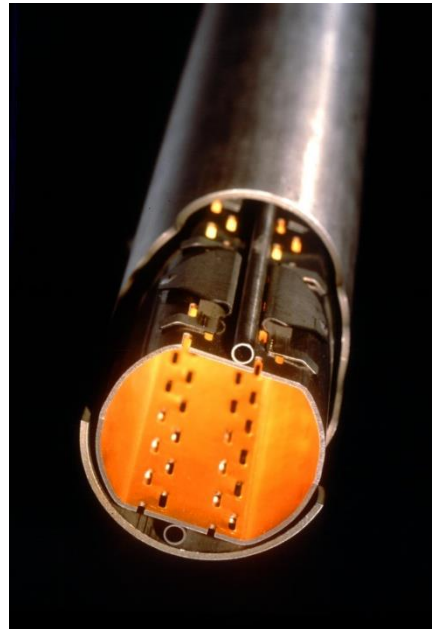


Mandate

Structure

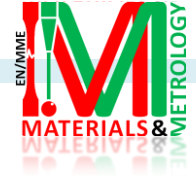
films. Provides support for the quality mechanical tests and measurements on

©CERN 2009





# Microstructural characterization. Specimen preparation



## Sectioning

Linear precision Saw. *Buehler IsoMet 4000*

Precision diamond wire saws. *Well 3242 and 3241*

## Mounting

Hot mounting presses. *Buehler Simplimet 1000 and 2000*

Cold mounting pressure chamber. *LamPlan M.M. 806*



## Grinding & polishing

Manual polishing machines. *LamPlan M.M. 8027 S. and Plamopol 2*

Automatic polishing machines. *Buehler Phoenix 4000 and LamPlan M.M. 8055*

Vibratory polishing machine. *Buehler vibromet 2*

Automatic electrolytic polishing-etching. *ATM Kristall 620 with electrolytic cell*



## n. Observation and analysis



### Optical microscopy

Various stereo microscopes.

Metallographic microscope. *Leica DMRM*

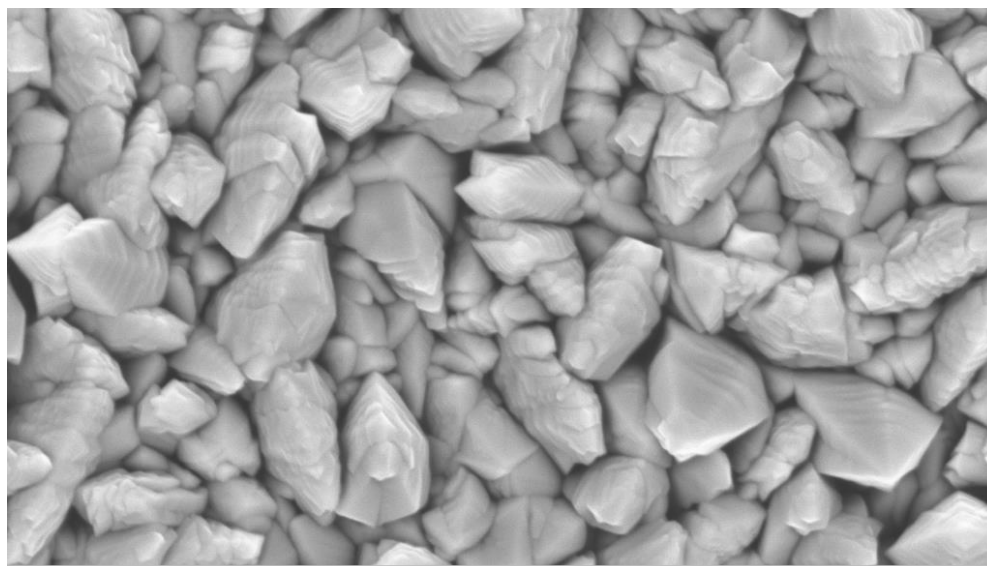
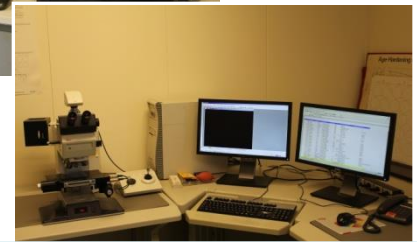
Objectives for magnifications from x16 to x1500

Image analysis system

Digital portable microscope. *Keyence VHX-1000*

Resolution max 54 MPixel

Objectives 0 - x50, x20 - x200 and x100 - x1000



Morphology of a Nb coating on Cu structure, original magnification 50000 x

200 nm 		EHT = 5.00 kV WVD = 2.9 mm Signal A = InLens	HIE-ISOLDE cavity Nb coating on Cu Test 43 - Be3 - pt 4	Mag = 50.00 K X Maud Scheubel Date :6 Jul 2011	
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SE and BSE imaging detectors. Max resolution 4 nm

Beam 0.5 to 30 kV, 1pA to 500 nA

EDS analyser *Oxford Isis*.

FE-SEM. Zeiss *Sigma*

Chamber  $\varnothing 365$  mm x 275 mm

SE, BSE, in-lens SE imaging detectors. Max resolution 1.5 nm

Beam 0.1 to 30 kV, 4 pA to 20 nA

EDS analysis *Oxford Inca* with 30 mm<sup>2</sup> SDD detector

EBSD analysis *HKL Chanel 5*



### Spectroscopy and X ray diffraction

Powder XR diffraction. *Siemens D5000*

Cu and Cr X-ray sources

Vertical and horizontal goniometers

Portable OES analyser. *Oxford Instruments PMI MasterPro*

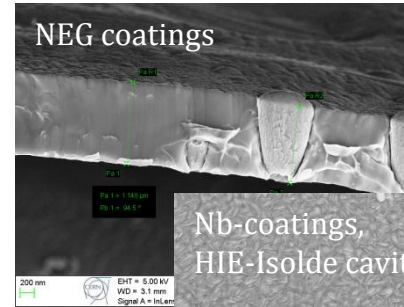
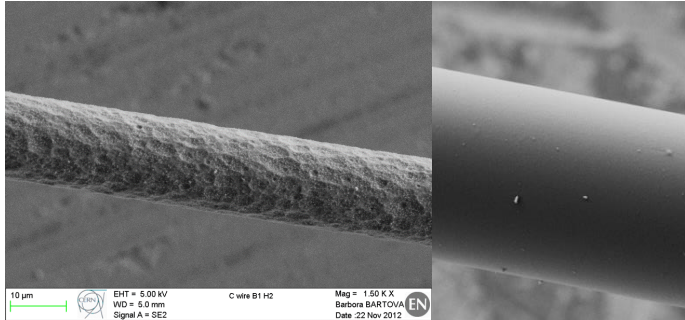
Calibrations for steels, Al, Ni, Cu and Ti alloys

UV probe for S and P

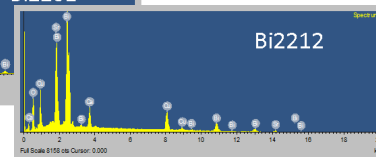
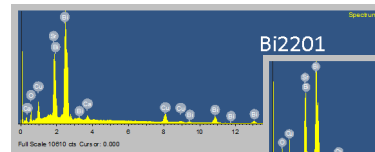
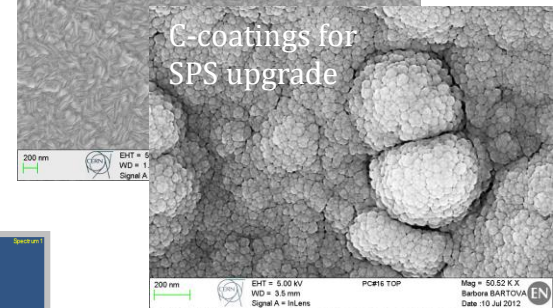
**New optical microscope  
being purchased in 2013**

**New SEM (replacement LEO) in 2015**

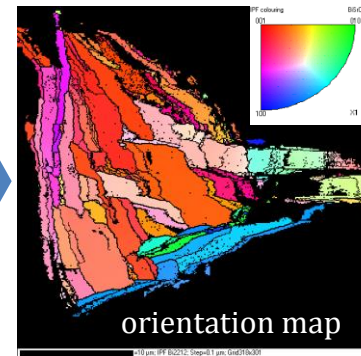
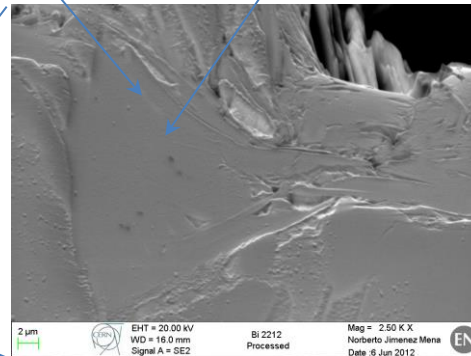
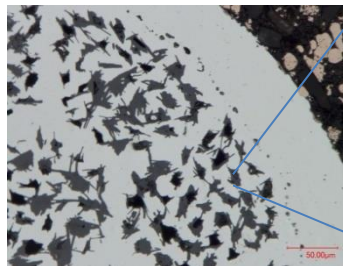
Carbon wires from SPS beam scan: region degraded by the beam (left) and unused reference (right, same scale)



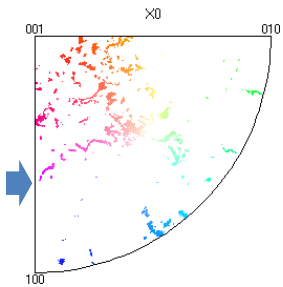
Thin films



Microstructural characterisation by EBSD of Bi2212 wire



orientation map

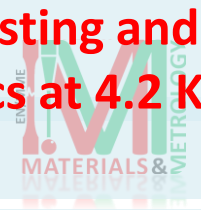


inverse pole figure



# Mechanical testing

**100 kN cryostat for tensile testing and eventually fracture mechanics at 4.2 K in construction (2013)**



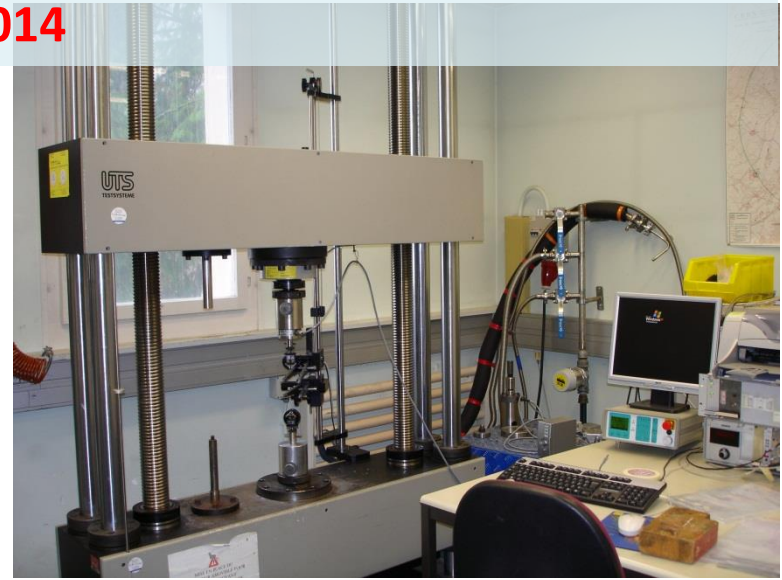
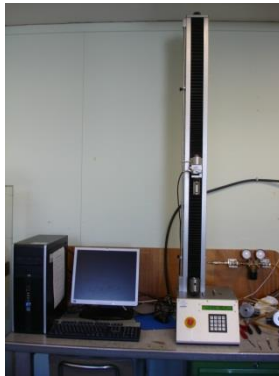
## Tensile testing machines

Two column electromechanical universal testing machine *UTS 200*  
Load cells 1 kN, 20 kN and 200 kN, stroke 800 mm  
Knives and clip-on extensometers

Tensile grips, compression plates, bending tools  
System for tests at 77 K and 4.2 K, 25 kN load cell

Single column press *ZPM 1000-500*. Load cell 1 kN, stroke 500 mm

**New dynamic mechanical testing unit in 2014**



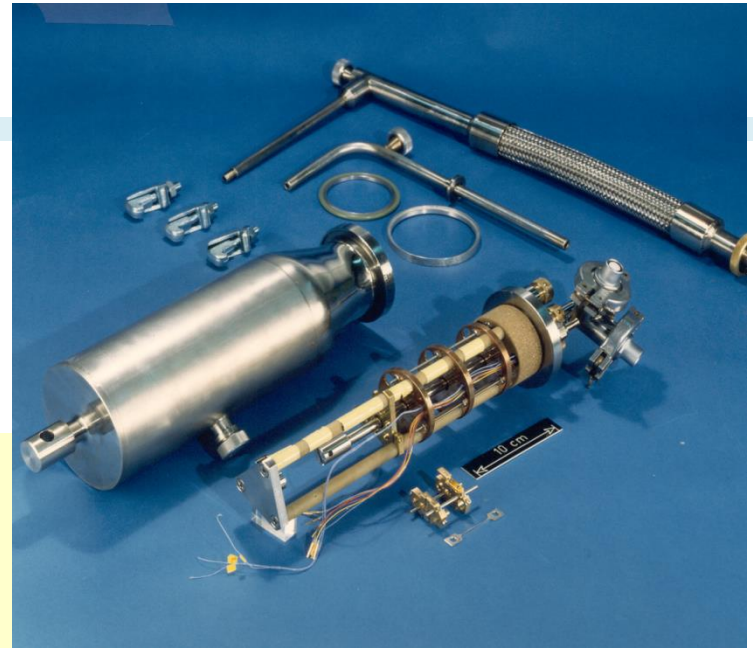
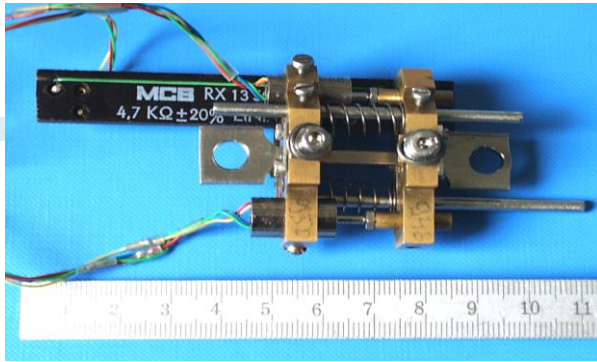
## Hardness

Hardness. *Wolfpert 2R*  
Load 1  
Brinell,  
Micro hardness autor  
Load 10

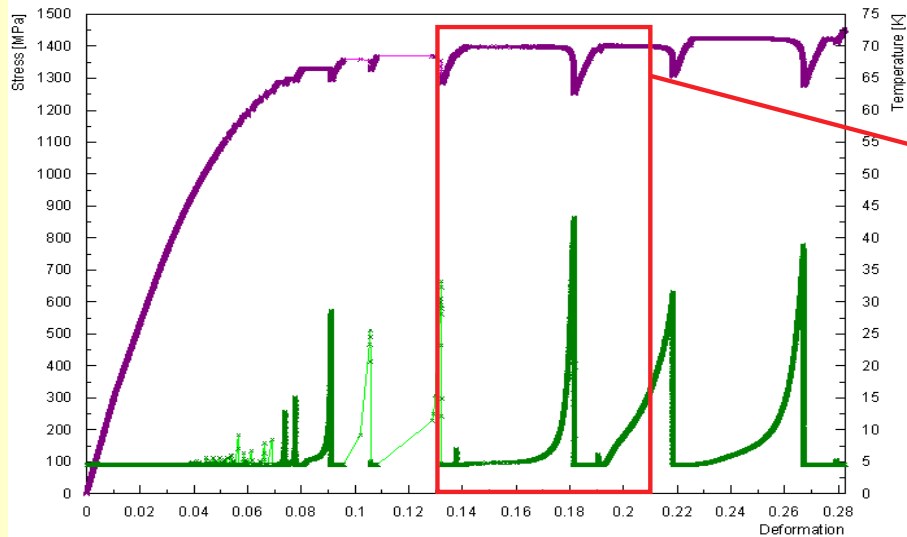




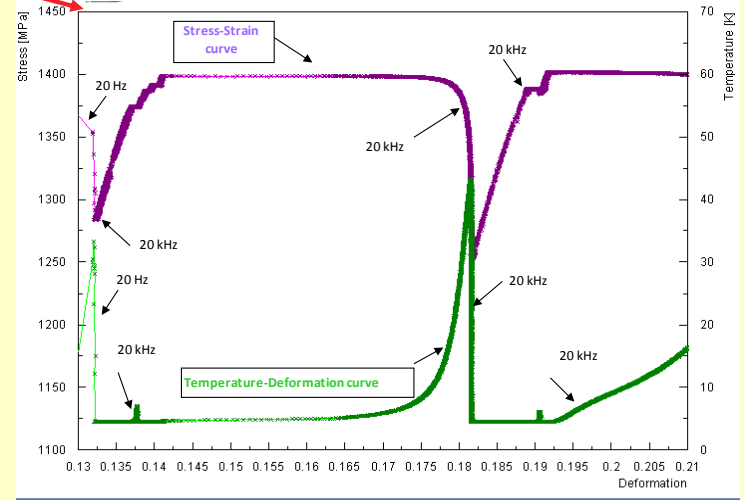
# Mechanical testing at 4.2 K, the past...



Sample 2 Ti5 Al2.5 Sn  
Detail of test 1 at 4.2K

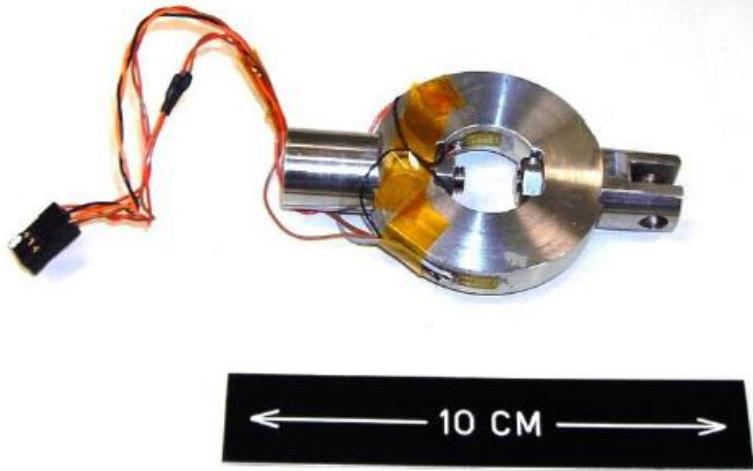


Detail of a Serrated Flow  
Sample 2 Ti5 Al2.5 Sn



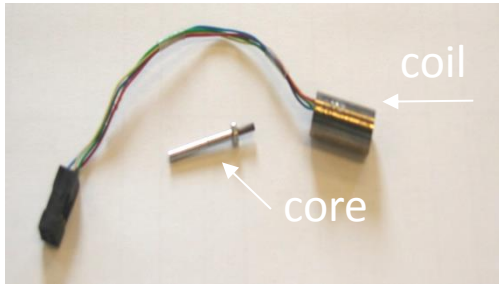


## Evolution of the equipment



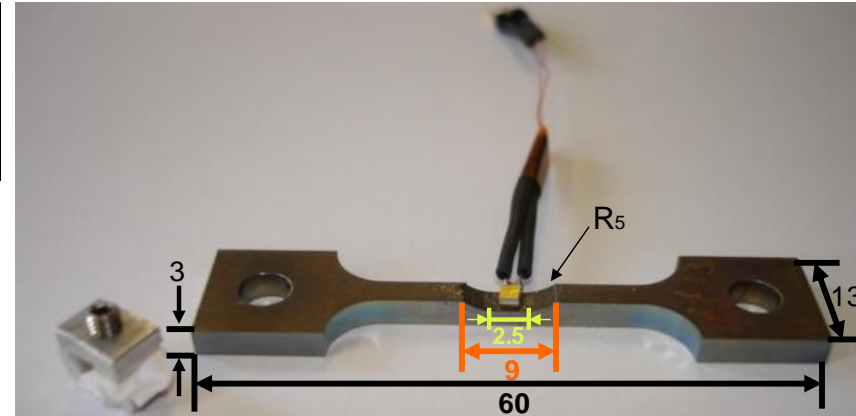
### Internal load cell:

immersed in liquid He, based on a deformable 316LN ring equipped by strain gauges  
*conditioner*: MVD2555 of HMB



### LVDT:

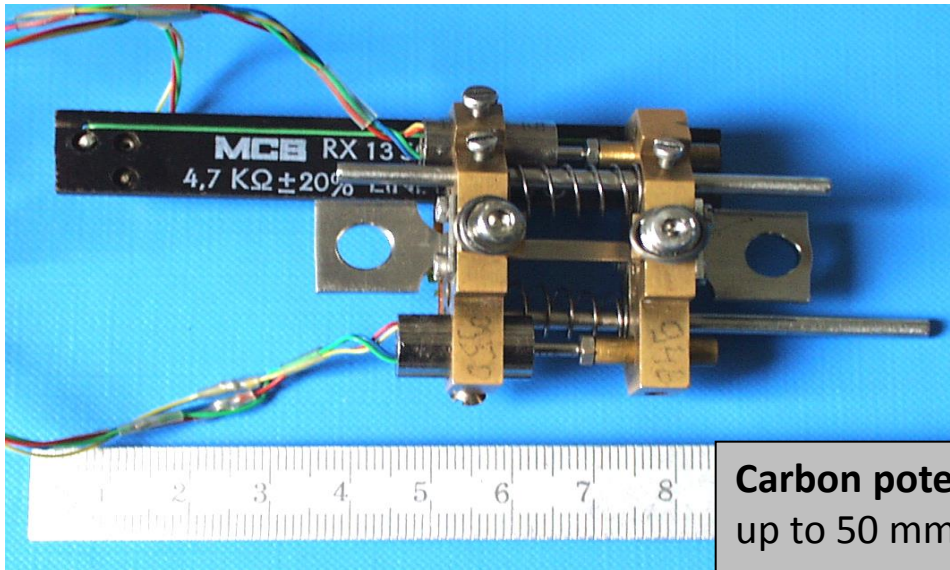
*range*: up to 3 mm  
*resolution*: 0.5  $\mu$ m



### Temperature probe, CERNOX CX-1050-SD-30

(Lakeshore Cryotronics):

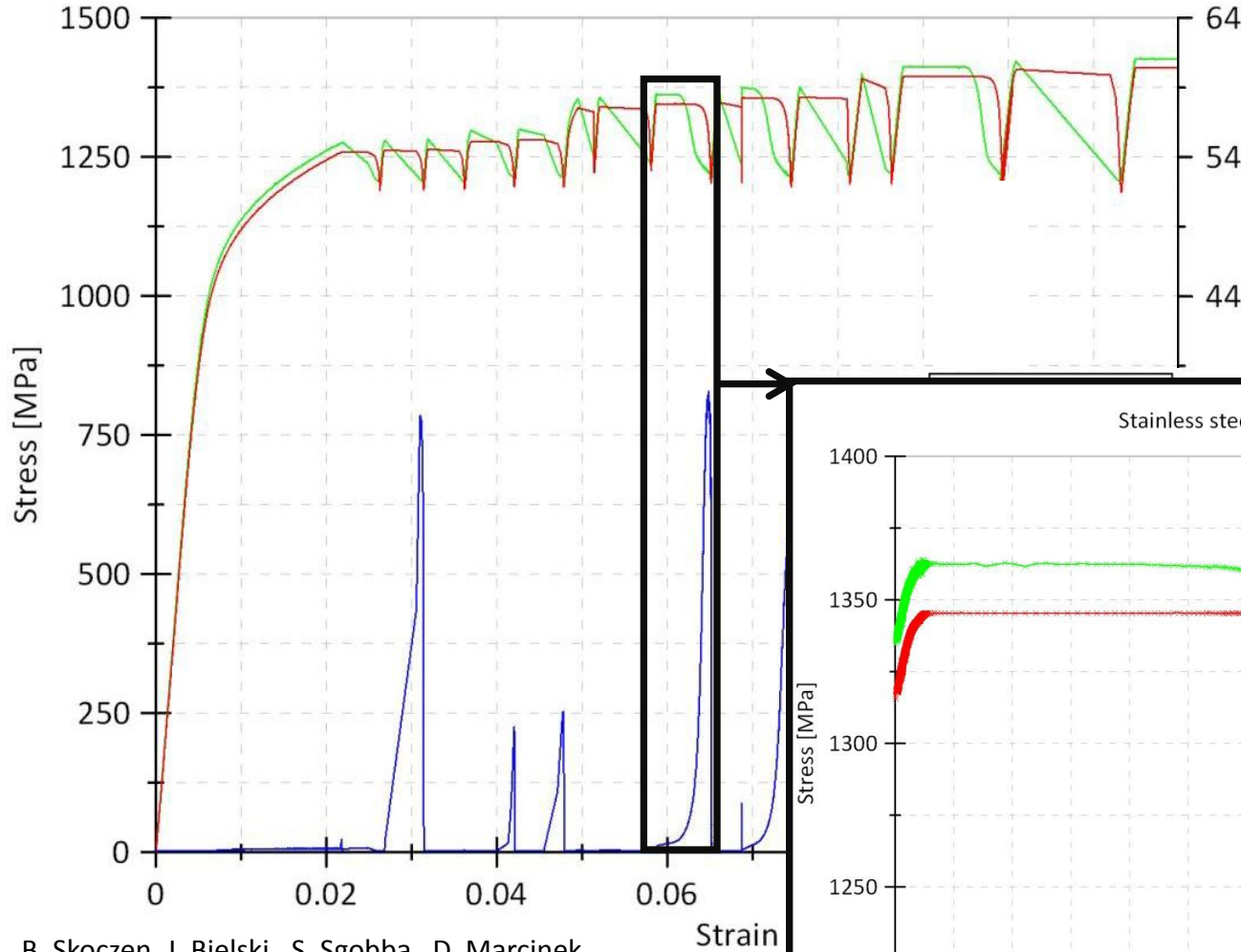
*thermal response time*: 15 ms at 4 K  
*resolution*:  $\pm 15$  mK



**Carbon potentiometer:** *range*:  
up to 50 mm *resolution*: 0.1 mm

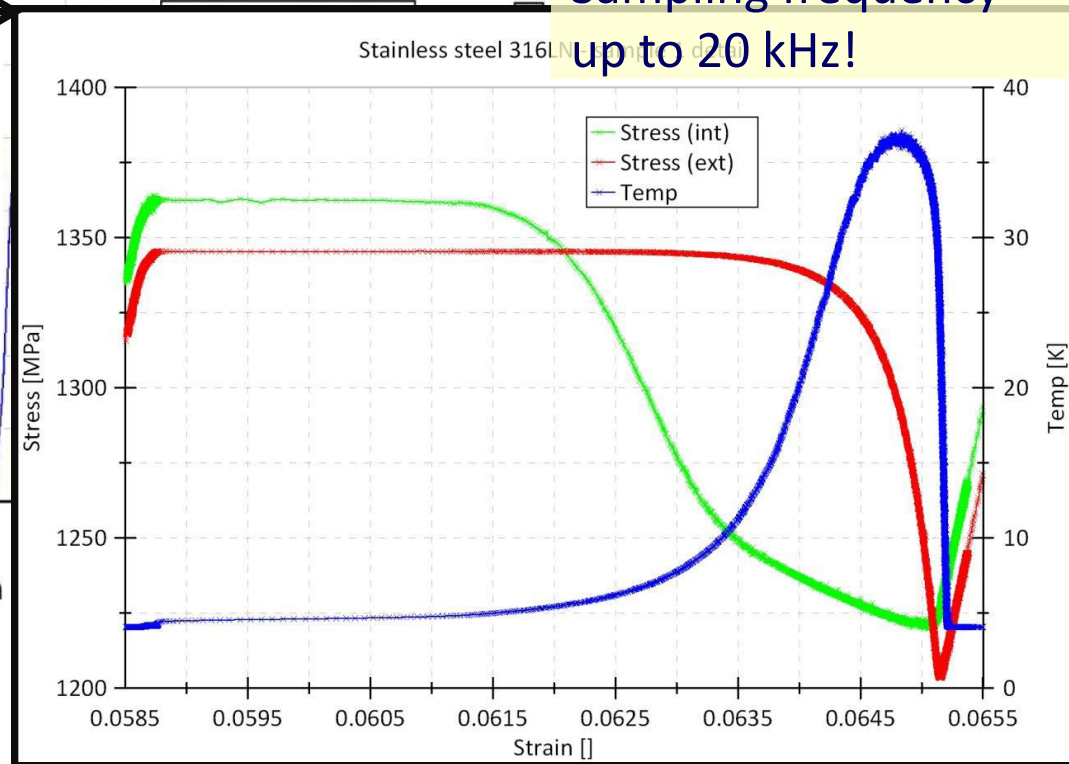
# Evolution of the equipment

Stainless Steel 316LN - sample 1



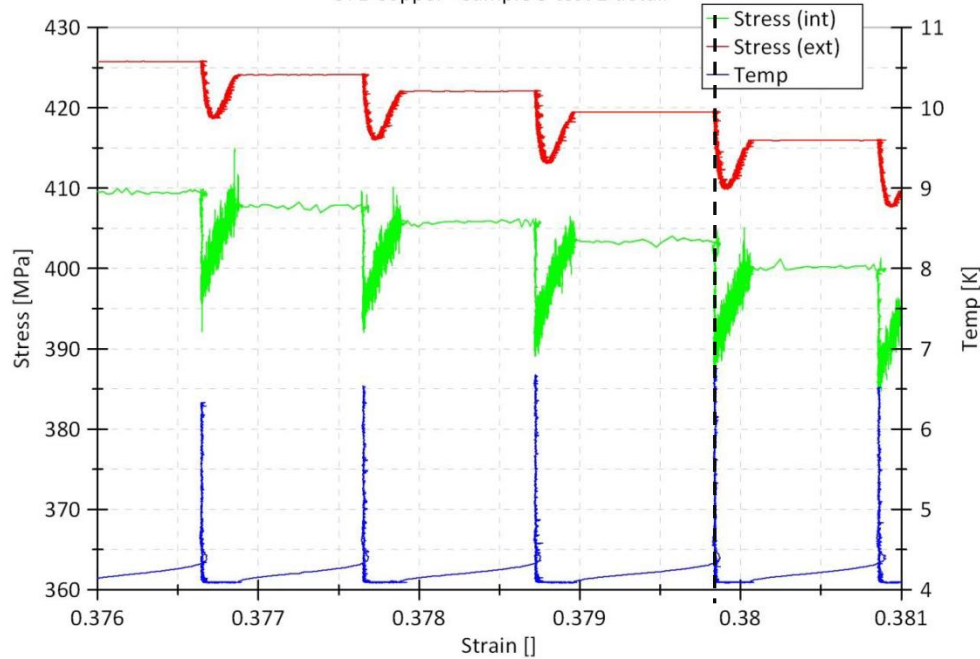
D. J. Marcinek, *Experimental Study of Discontinuous Plastic Flow, Phase Transformation and Micro-damage Evolution in Ductile Materials at Cryogenic Temperatures*, Master of Science Thesis, CERN and CUT, 2009

Sampling frequency up to 20 kHz!



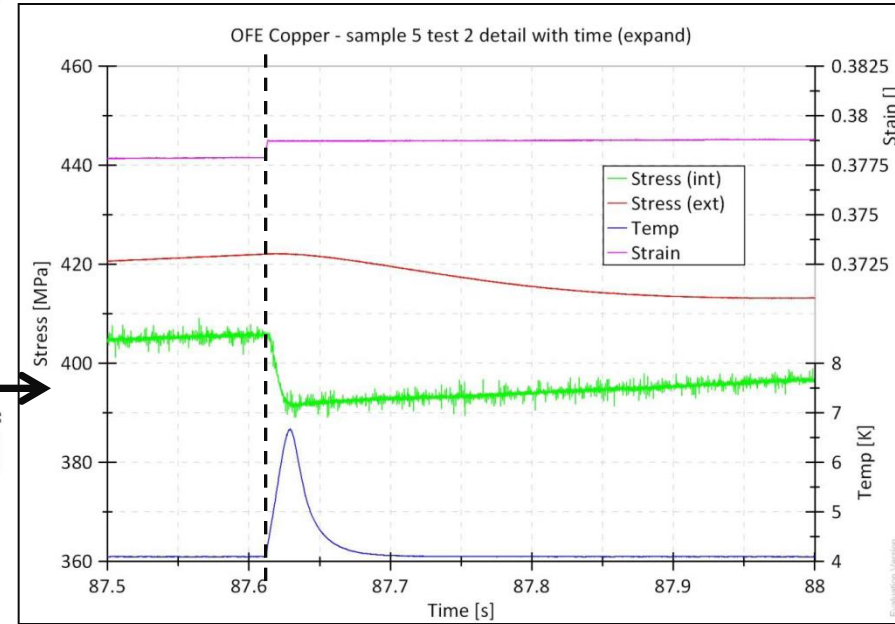
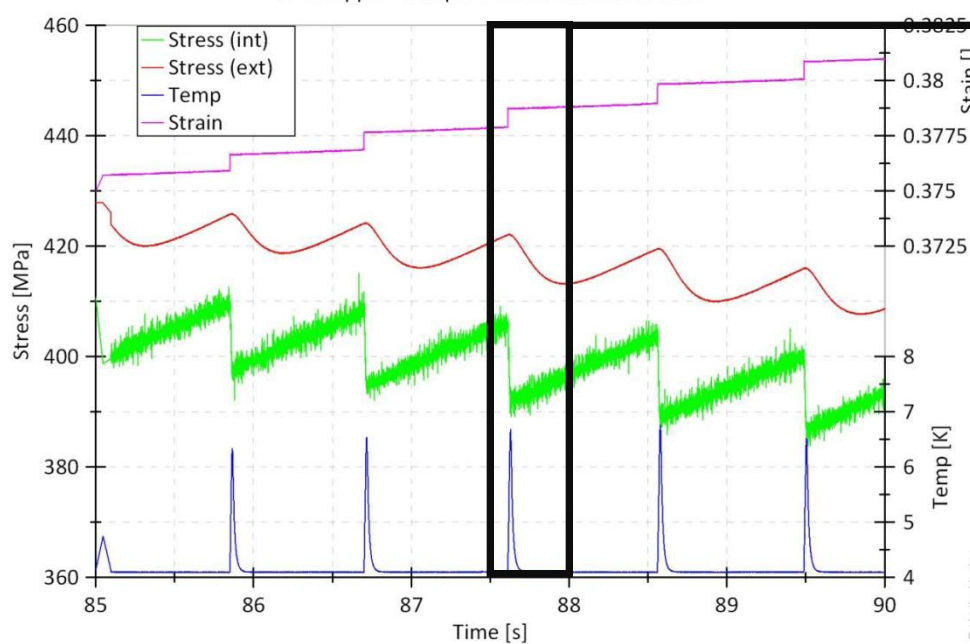
B. Skoczen, J. Bielski, S. Sgobba, D. Marcinek, *Constitutive model of discontinuous plastic flow at cryogenic temperatures*, International journal of plasticity, 26 (2010) 1659-1679

OFE Copper - sample 5 test 2 detail



OFE Cu, serrated yielding recorded during necking (engineering stress-engineering strain plot)

OFE Copper - sample 5 test 2 detail with time



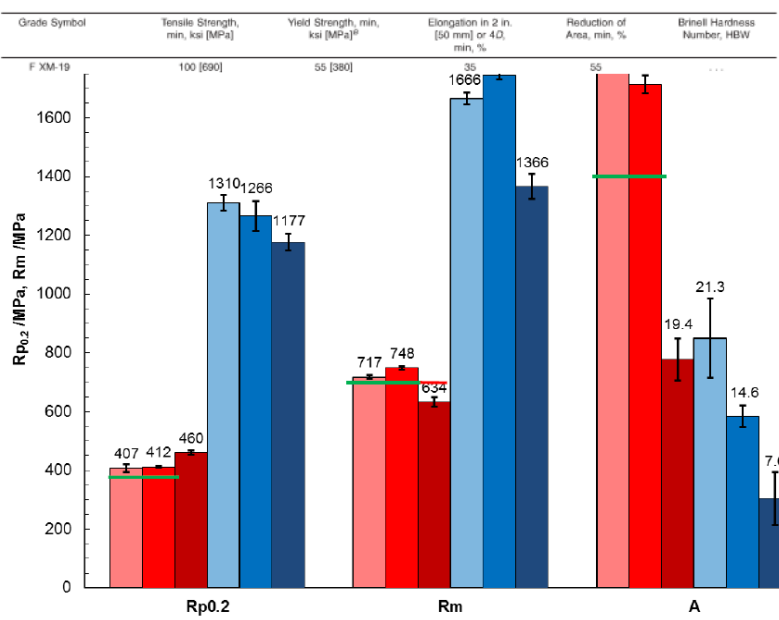
D. J. Marcinek, *Experimental Study of Discontinuous Plastic Flow, Phase Transformation and Micro-damage Evolution in Ductile Materials at Cryogenic Temperatures*, Master of Science Thesis, CERN and CUT, 2009

5.2 Mechanical Testing

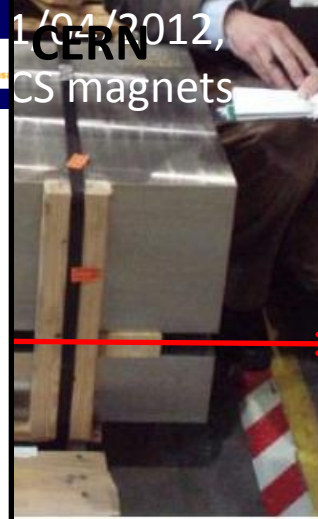
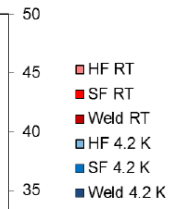
The mechanical properties of forgings supplied under this specification shall meet the properties required by ASTM A182, Grade FXM-19.

ASTM A182/A182M - 10a

TABLE 3 Continued



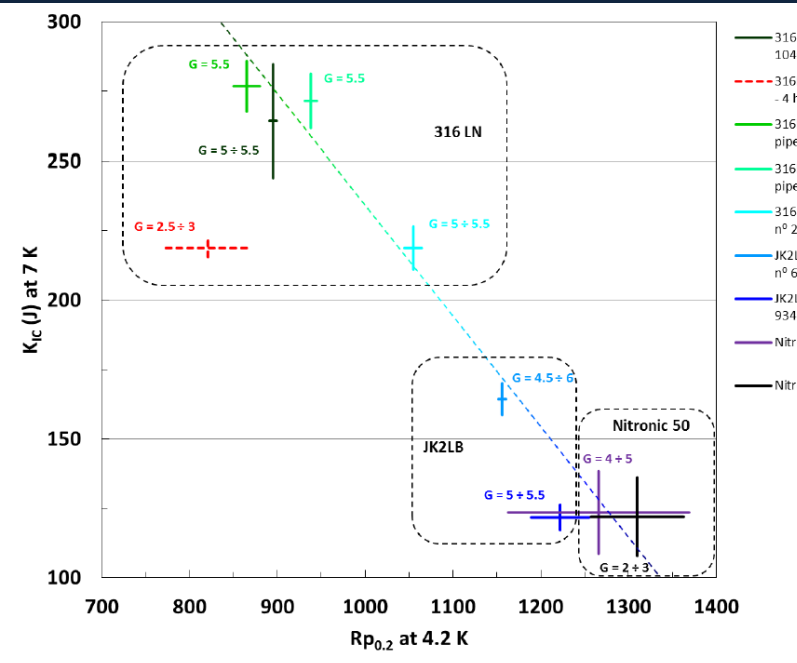
china eu india japan korea russia us



CERN + CEME



Mechanical testing at 4.2 K, the present and future

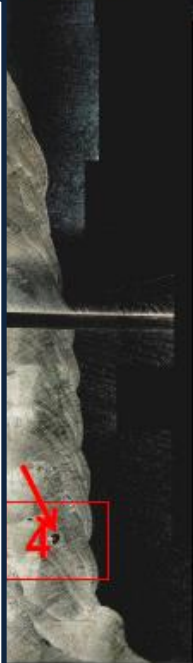
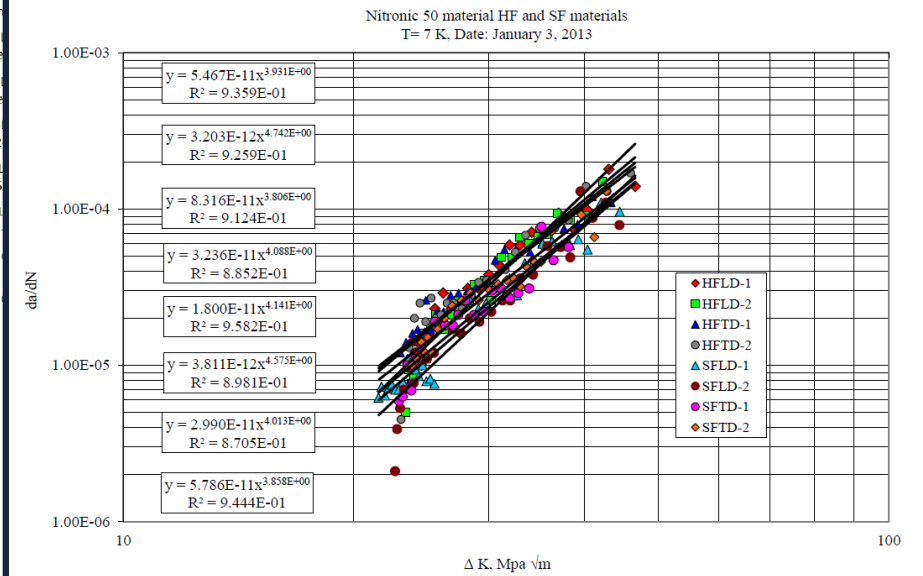


EN Engineering Department

FCGR results Base metal

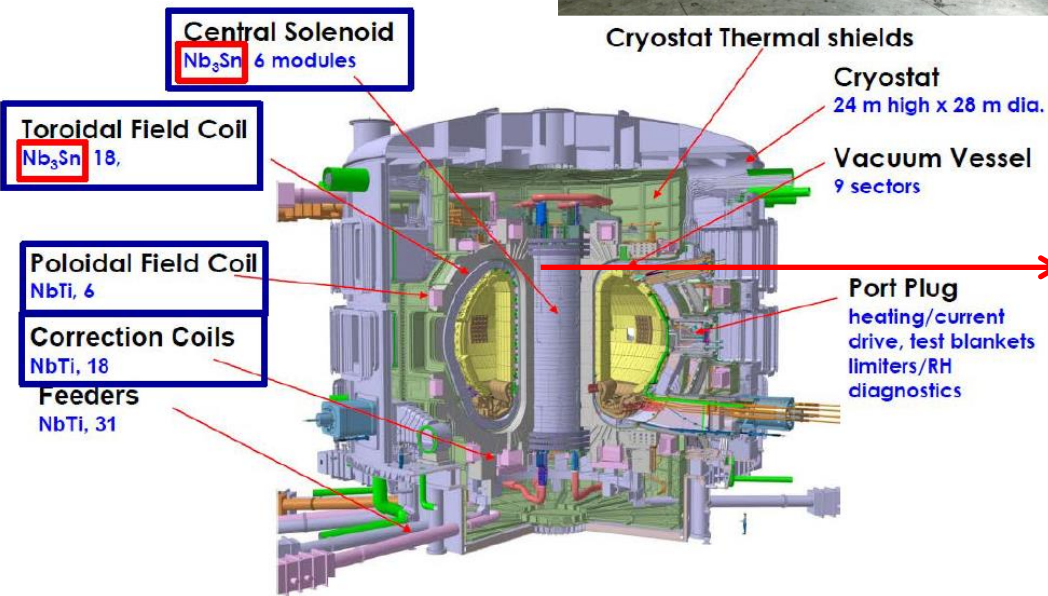
Document: 060741-14 © CEME: 2013

Fatigue crack growth rate and fracture toughness properties at 7 K of forged Nitronic 50 material and its welding





Courtesy of Kind





# Non destructive testing

Visual testing, VT, two level 2 certified

Penetrant testing, PT, two level 2 certified



## Ultrasonic testing, UT

Portable flaw detectors. *Krautkramer USN 60*

Gain 0 dB to 110 dB, probe from 0.25 MHz to 25 MHz,

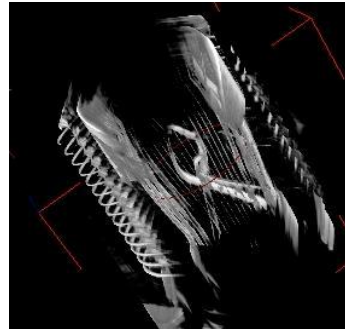
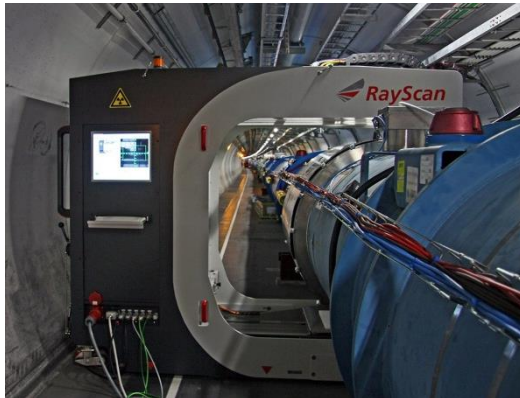
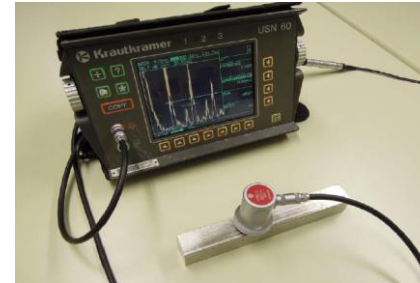
Portable flaw detector with Phased Array mode. *GE Phasor XS*

Gain 0 dB to 40 dB, probe from 0.5 MHz to 10 MHz,  
Various sectorial or linear transducers

Immersion tank scanner

Scan-surface: 500 mm x 500 mm, 0.1 MHz to 150 MHz

Level 3 and 2 certified operators



## Radiographic testing, RT

Mobile X-ray computed tomography

Mini-focus x-ray source

Flat panel detector

Software for 3D reconstruction

X-ray sources. *Philips 160 kV, 0.5 A*

Sieffert isovolt 160 kV

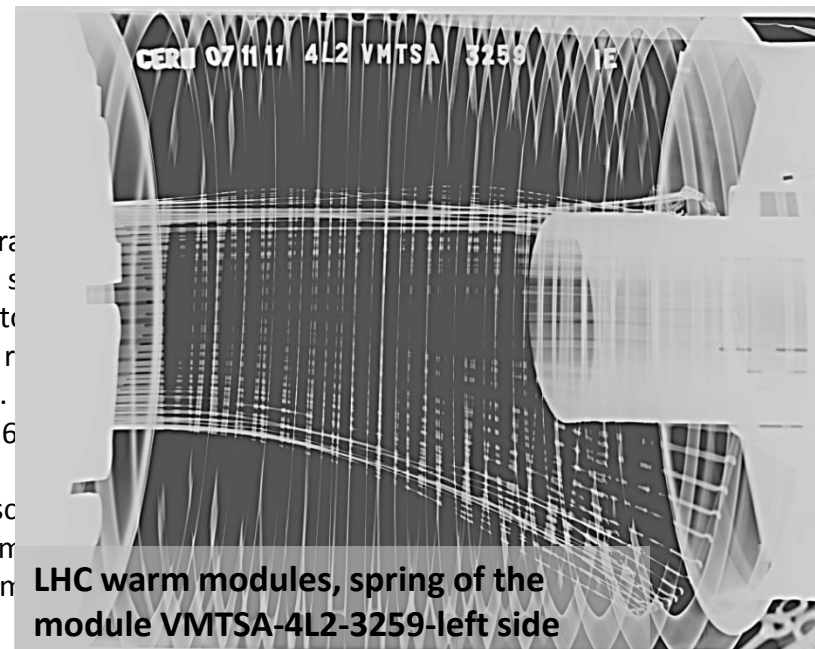
Computer radiography system

High definition software

High resolution monitor

Controlled area (bunker) of 30 m<sup>2</sup>

Level 2 certified operators



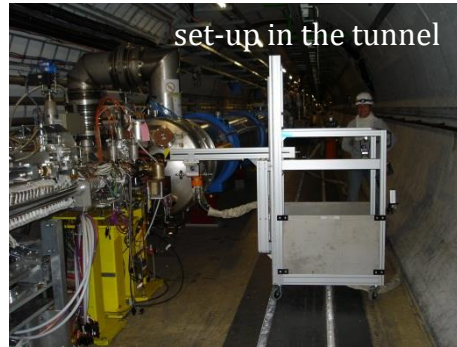
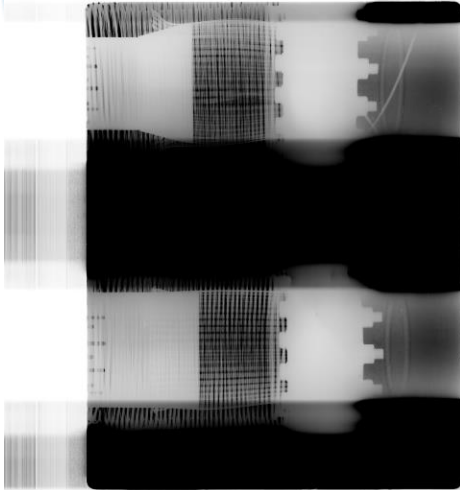
LHC warm modules, spring of the module VMTSA-4L2-3259-left side



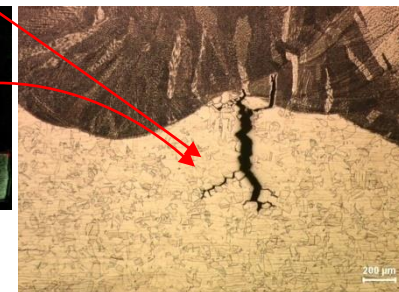
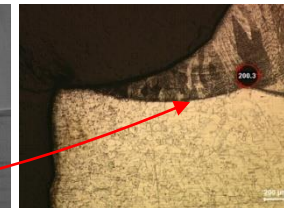
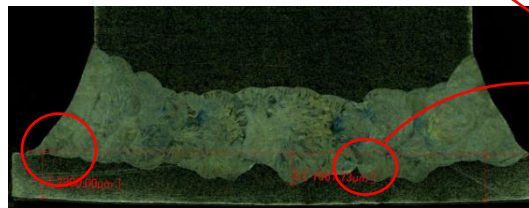
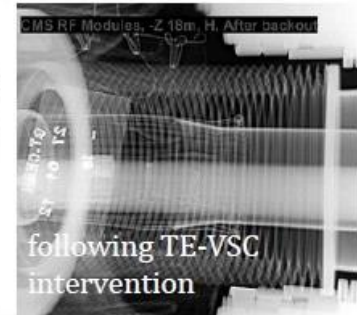
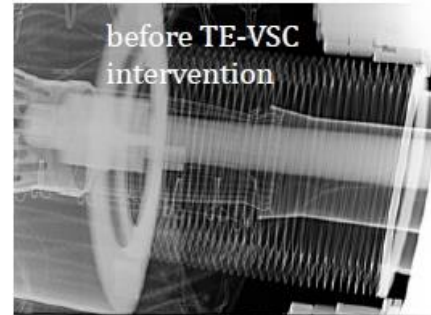
# Non destructive testing



⇐ Computed Radiography Testing (RT) of LSS warm modules. Completion of the radiography campaigns during LHC technical stops. 1767 modules examined, 107 non-conformities identified to be repaired during LS\_1, 46 nights.



⇓ Computed RT of CMS RF modules



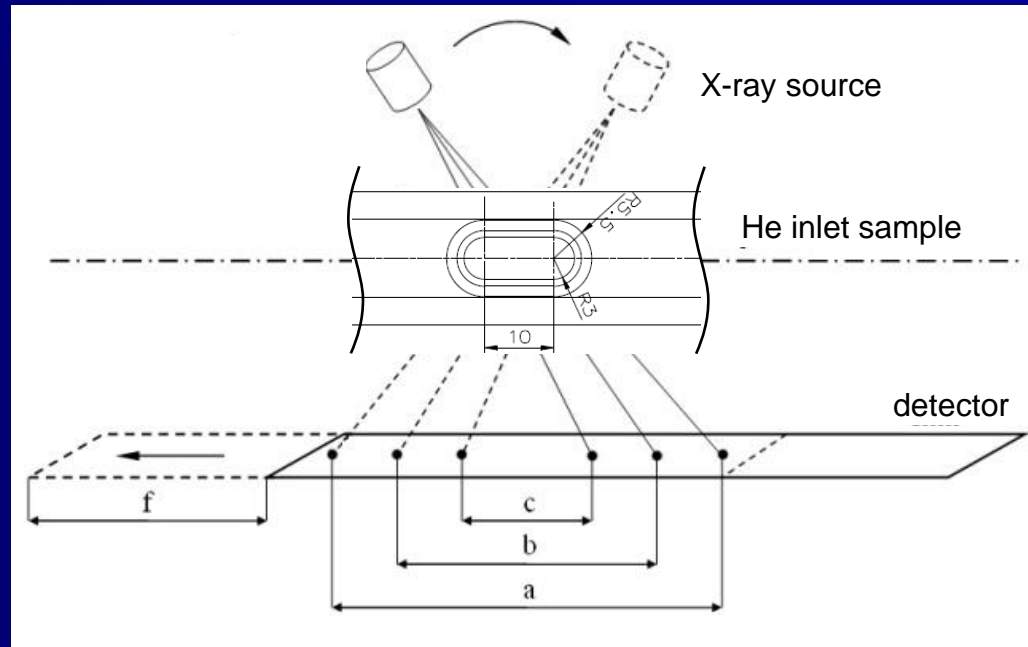
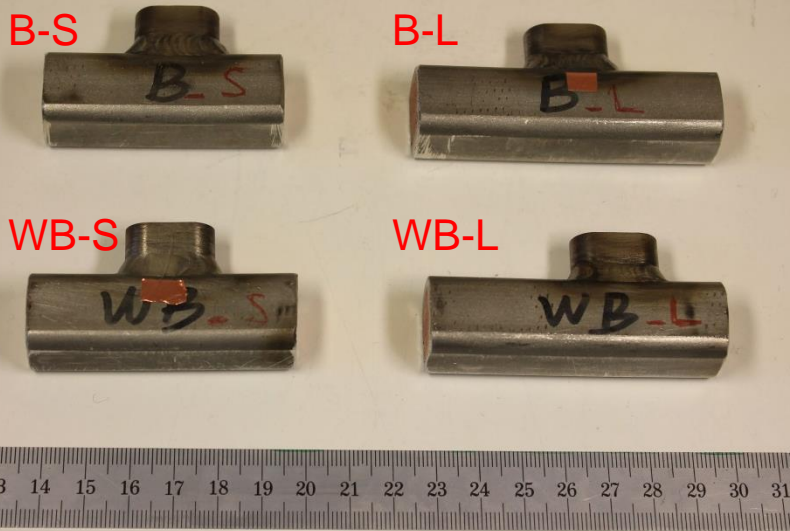
X-Ray tomography of ITER CC He-inlets: thin defects precisely identified and confirmed by metallography



⇐ Computed RT of ATLAS RF modules

# Delivery and test description

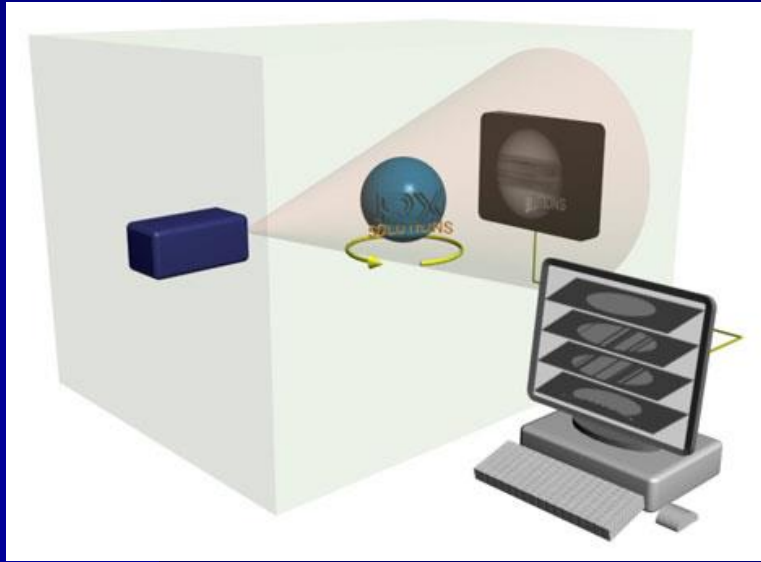
- ITER CC He Inlets samples delivered to CERN by ASIPP in March 2013
- Two welded with buttering (**B-L** and **B-S**) and two without buttering (**WB-L** and **WB-S**)
- Laminography (planar tomography) of all four samples performed by RX Solutions /FR, allowing to access defects in the whole weld volume with higher resolution than through computed RT
- Classification following ISO 6520-1 and judged with respect to EN ISO 5817 - level B







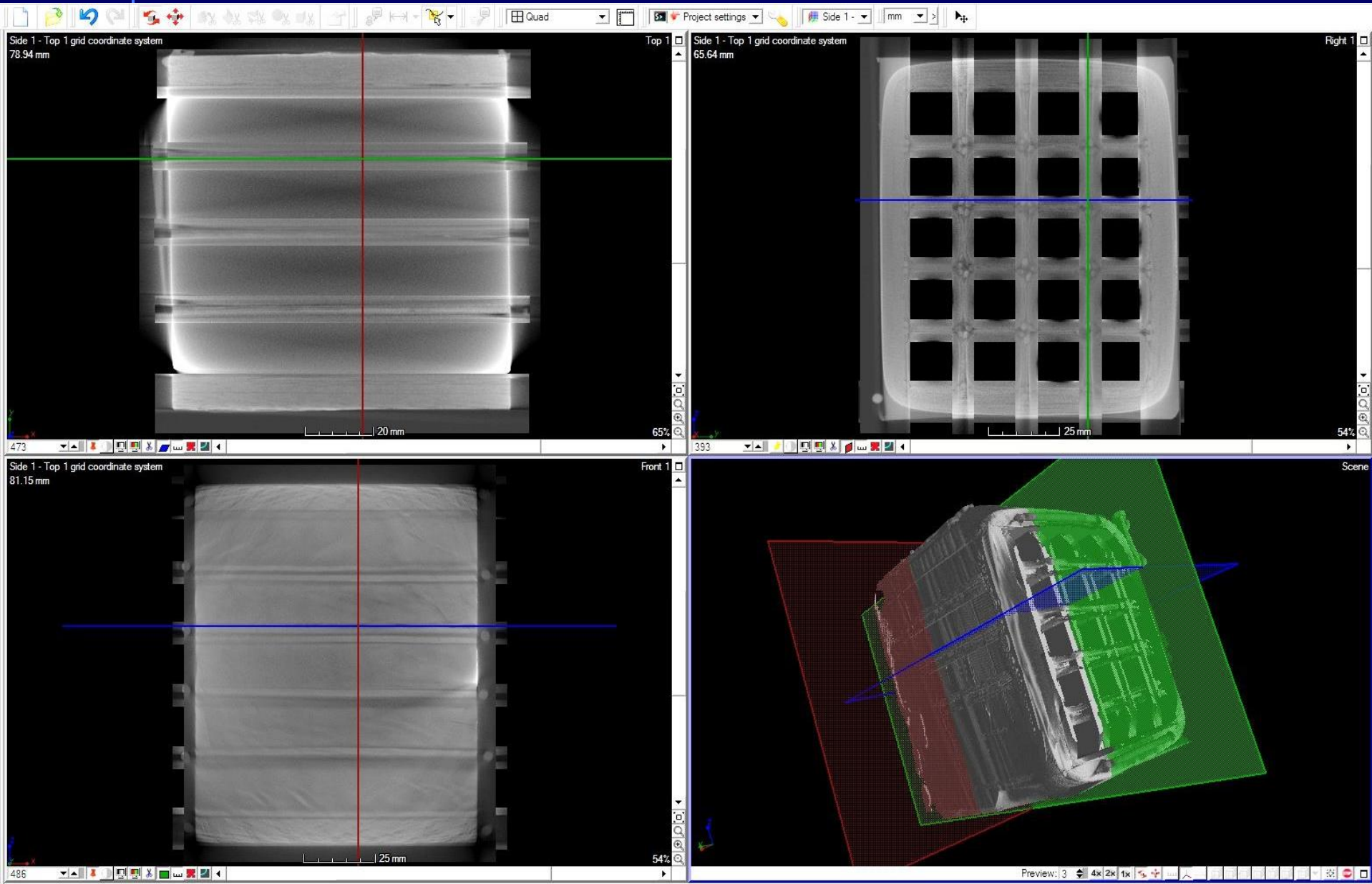
- Specimens placed on a mobile and rotatable stage
- Motorized zoom
- Full inspection of samples up to 240 mm length
- Sealed microfocus source, max. tension 150 kV
- HR area image sensor, 1920 pixels x 1536 pixels
- Detection surface: 200 mm x 250 mm
- 14 bits – 16000 gray levels
- 1-30 images /s



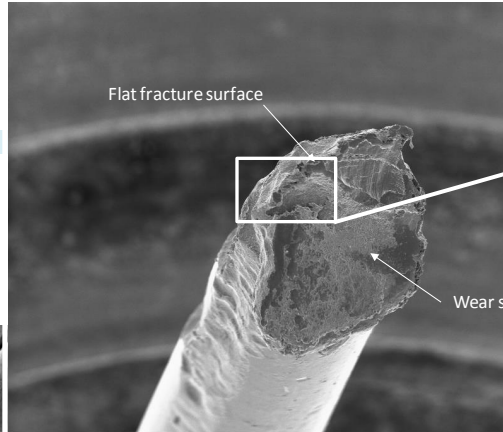
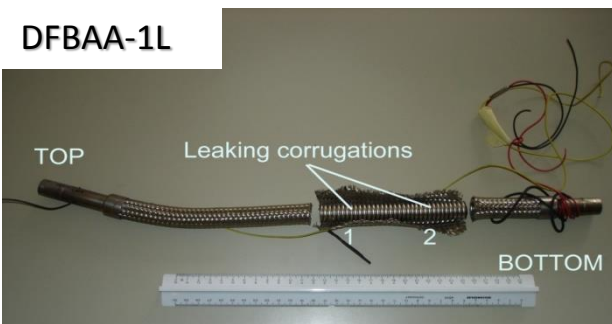
2nd sample of the VPI

11 separate 360° scans were combined to compile a final single 3D object reconstruction while avoiding shadows due to steel conduits





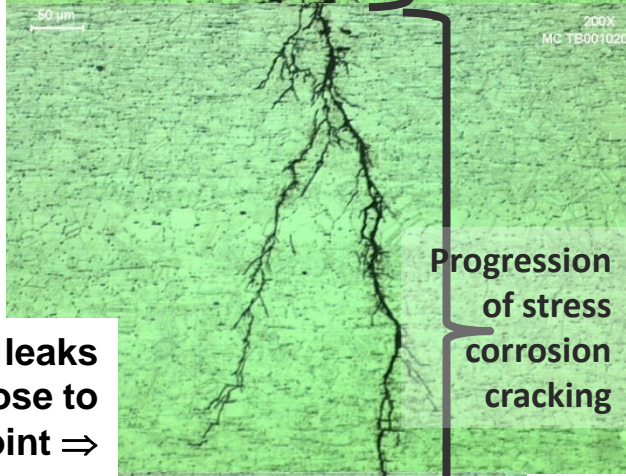
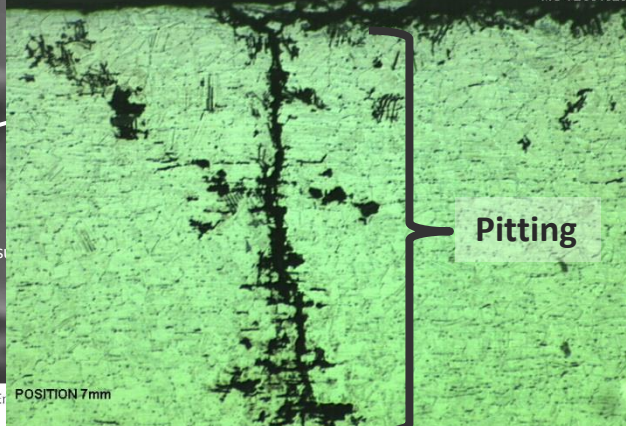
DFBAA-1L



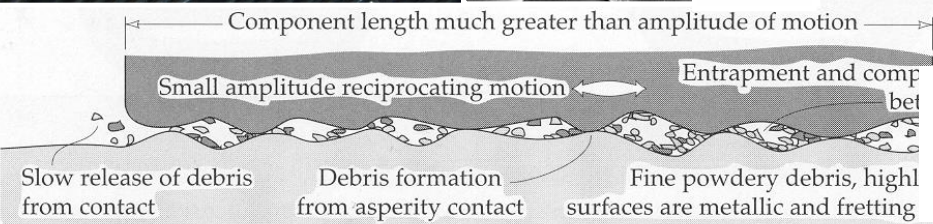
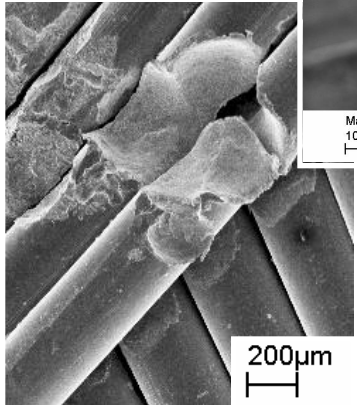
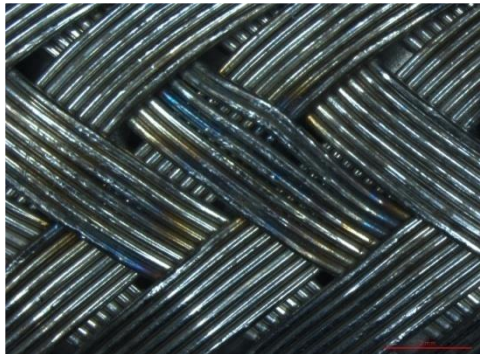
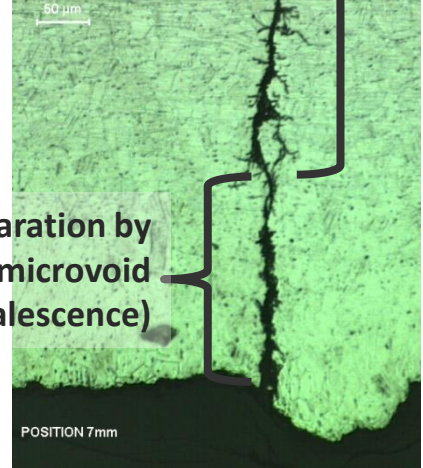
Mag = 100 X  
100μm  
Detector = SE1  
DFBAE-3L  
Date :7 Aug 2009

⇐ **Complex failure analysis of DFBA flexible metal hoses, TE-TM, 01/09/2009**

Divisional seminar, 01/02/2006  
MC TB001020

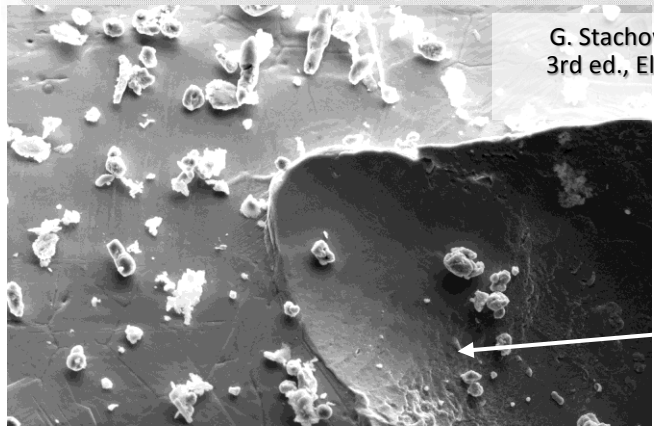


Final separation by overload (microvoid coalescence)

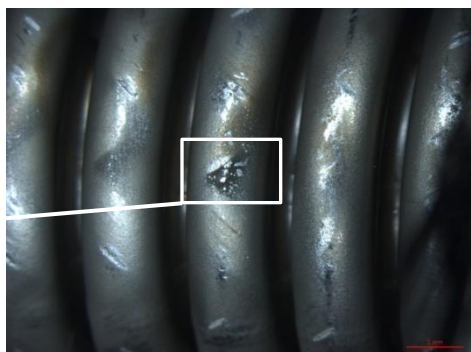


**QRL: corrosion leaks on the tube F close to the fix point ⇒**

G. Stachowiak, A.W. Batchelor, Engineering Tribology, 3rd ed., Elsevier Butterworth-Heinemann, Amsterdam (2005)



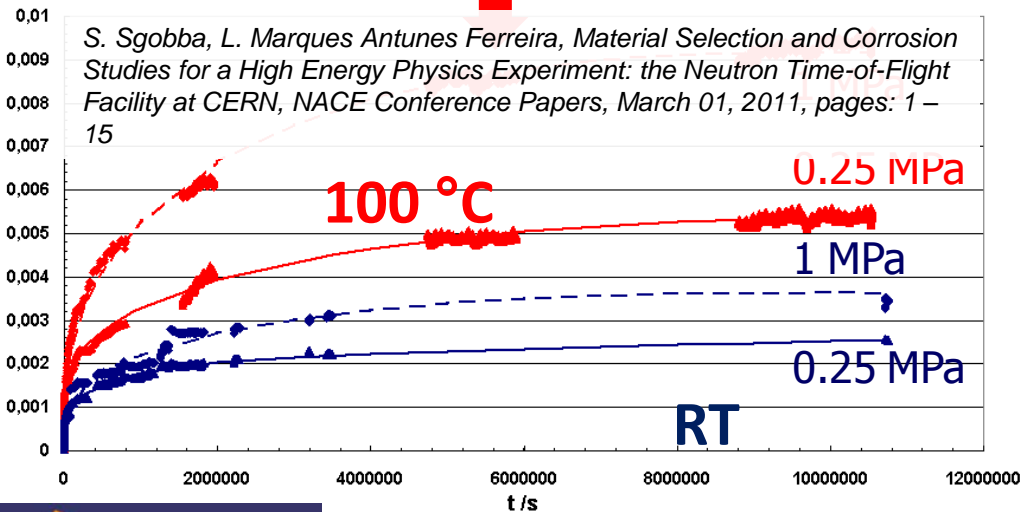
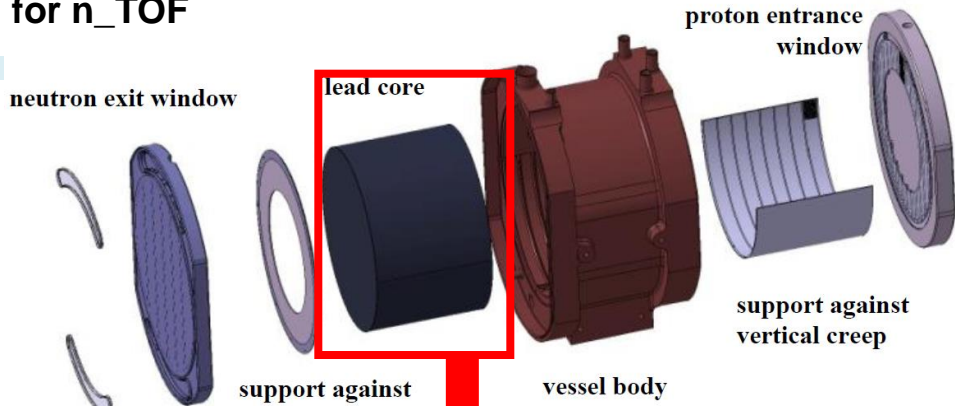
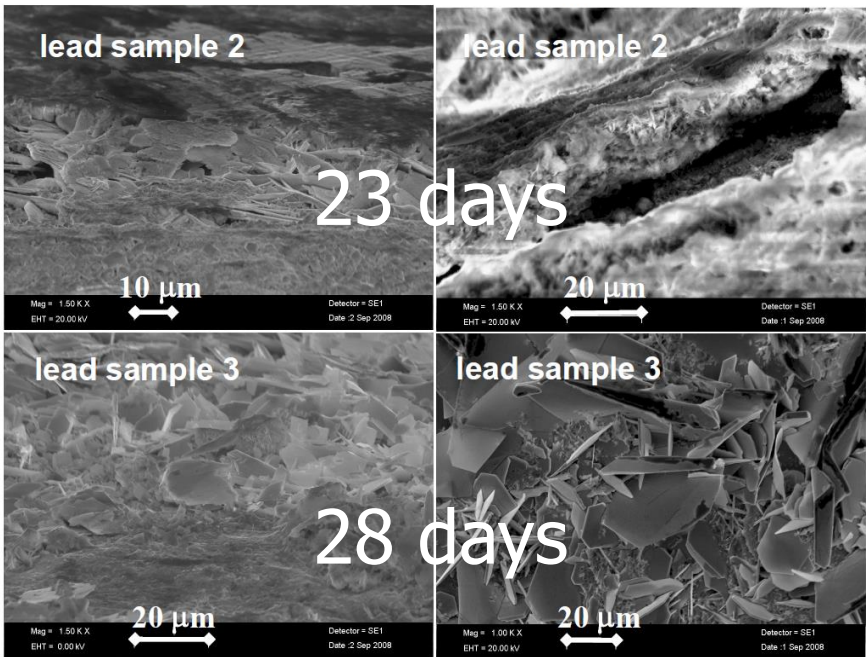
2 μm  
EHT = 10.00 kV  
WD = 9.7 mm  
Signal A = SE2  
Photo No. = 137  
Mag = 2.50 K X  
DFBAI 2009, fl  
Date :23 Jul 2009  
Time :19:15:41  
ZEISS



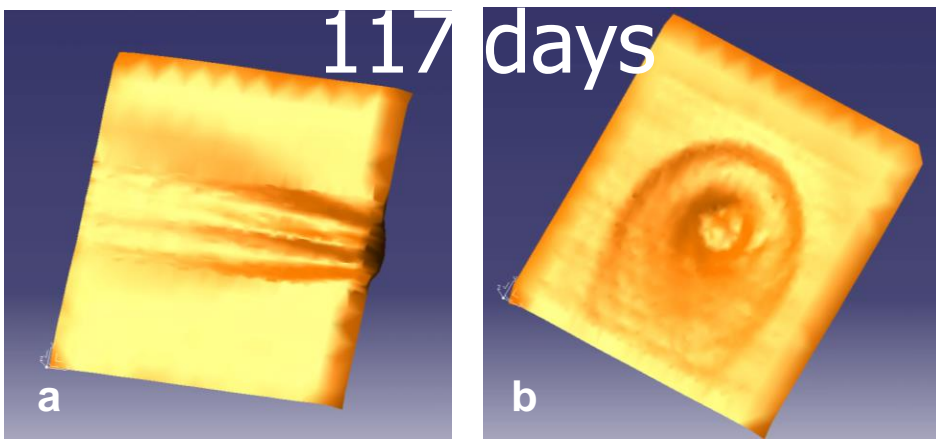
facing surface

opposite surface

### 2008-2009, material selection and corrosion studies for n\_TOF



erosion and impingement quantified by 3D metrology





## Conclusions



- CERN-wide (and beyond) activity
- Limited but selected and highly specialized equipment
- Covers a wide range of materials, welds and components
- Provides support to CERN stores (specifications, identification of suppliers, audit...)
- Several decades of experience, including audit and follow-up of industrial production
- In house expertise completed by an extensive network
- Prone to intervene in or pilot activities with a strong emphasis on materials or controls
- Section very exposed to international exchanges and contacts:
  - Industrial partners
  - National labs, academy
  - CERN-ITER cooperation agreement (IO, DAs via IO)
- Training of students and fellows, hosted a large (3 digit) number of trainees since the creation of the section in the early 90ies