JRC-CERN Collaboration Workshop
27 January 2014, CERN

Material and Surface Characterisation, Materials in Aggressive Environments

- Mandate and expertise of the Materials and Metrology section in the context of the EN-MME group
- Equipment
- Examples of advanced materials and surface characterisation activities and partnerships with industry and laboratories
- Aggressive environments in High Energy Physics (HEP) applications

Stefano Sgobba
EN-MME-MM

YEARS / ANS CERN

EN/MME MATERIALS & METROLOGY

CERN

EN Engineering Department

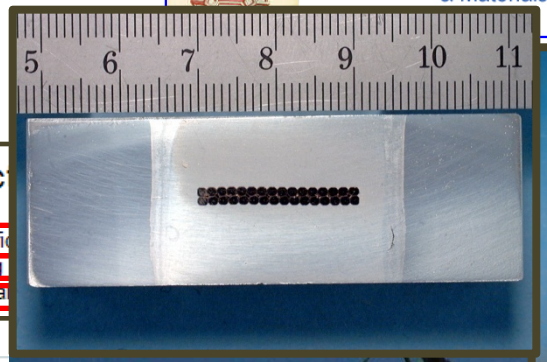
Mandate Structure

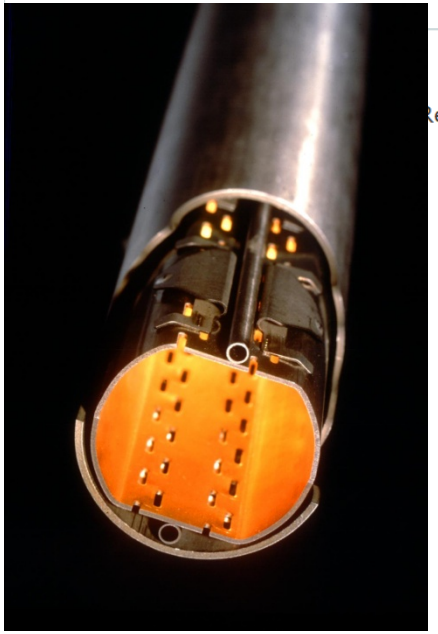
MME Mechanical & Materials Engineering

Home > Groups > MME > MM > - Mandate

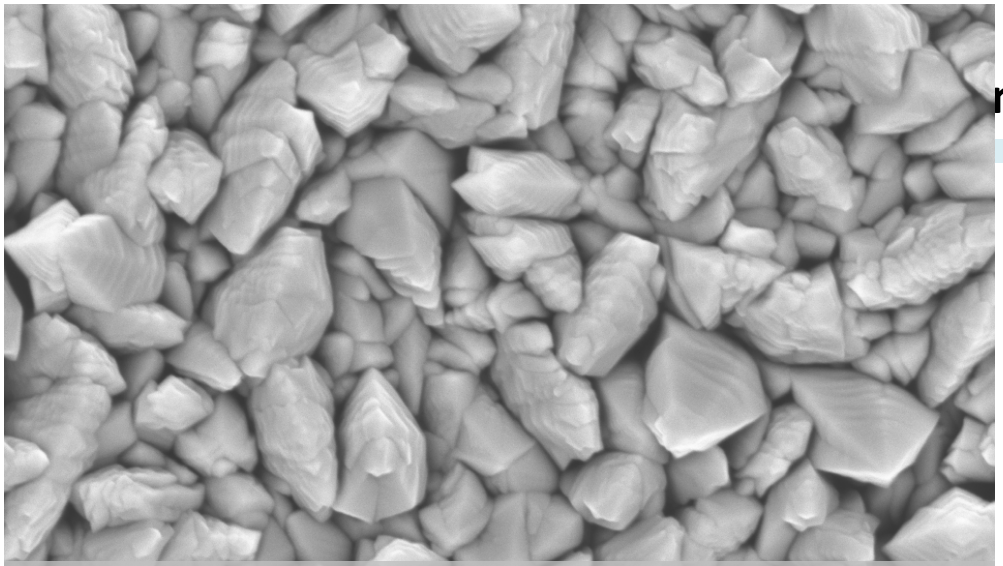
Metallurgy and Metrology Sec

Undertakes the development, selection, specifi films. Provides support for the quality control of materials and components including chanical tests and measurements on materials and components. Performs failure ana





n. Observation and analysis



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

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

Chamber 190 mm x 300 mm x 265 mm

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 Σigma

Chamber ø365 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^2 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*

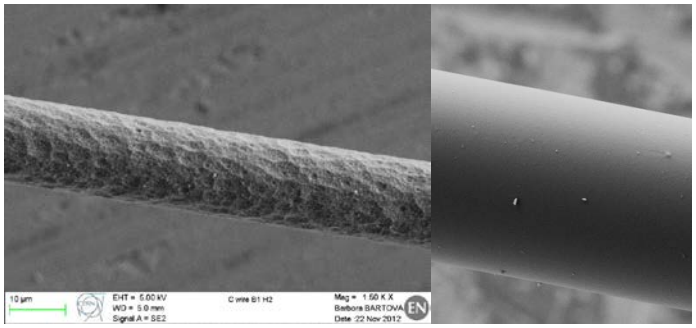
Calibrations for steels, Al, Ni, Cu and Ti alloys

UV probe for S and P

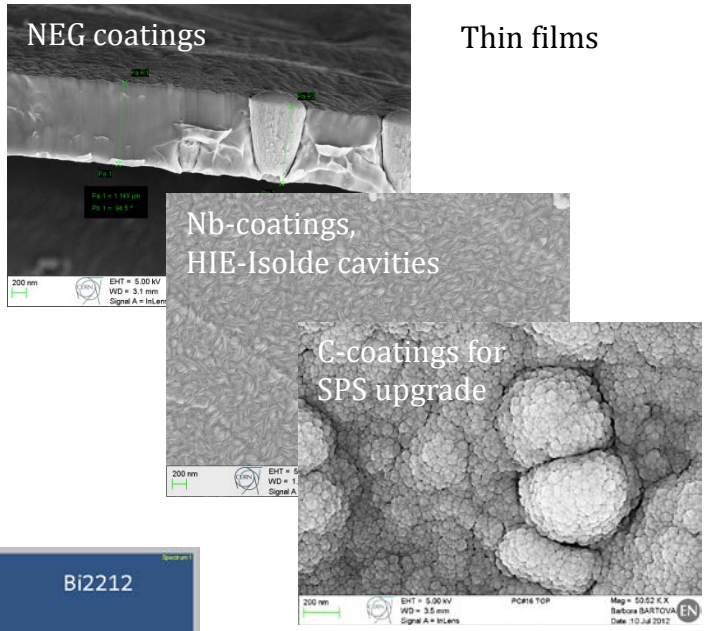
New optical microscope Zeiss Axio Imager purchased in 2013

Scanning Electron Microscopy and microanalysis support

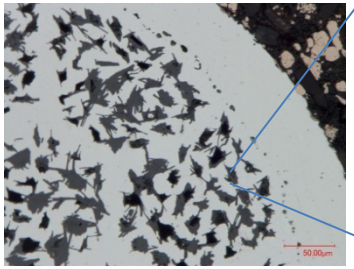
Carbon wires from SPS beam scan: region degraded by the beam (left) and unused reference (right, same scale)

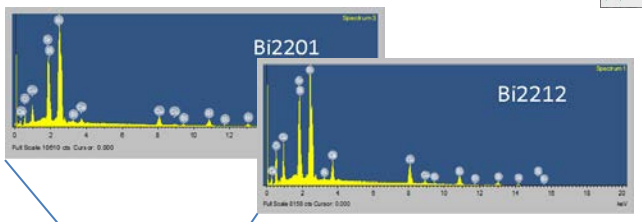


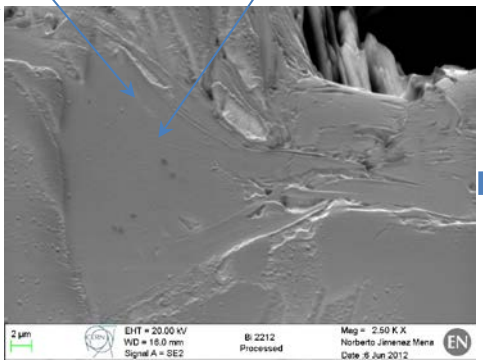
Thin films

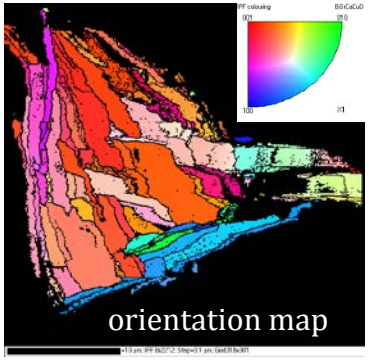


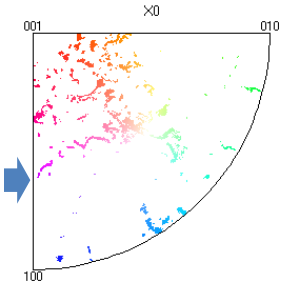
Microstructural characterisation by EBSD of Bi2212 wire











inverse pole figure

Mechanical testing

100 kN cryostat for tensile testing and eventually fracture mechanics at 4.2 K in testing and calibration phase

Tensile testing machines

Two column electromechanical universal testing machine *UTS 200*

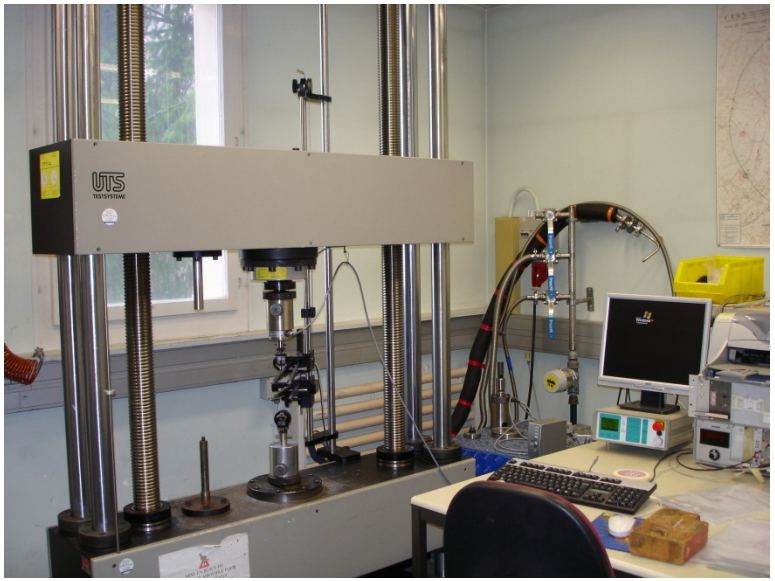
Load cells 1 kN, 20 kN and 200 kN, stroke 800 mm

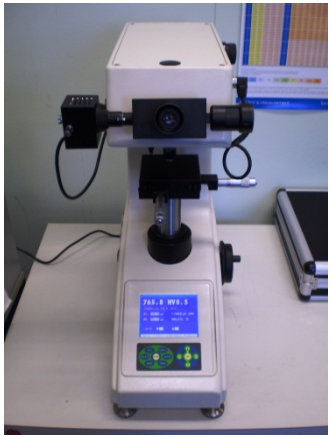
Knives and clip-on extensometers

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





Hardness

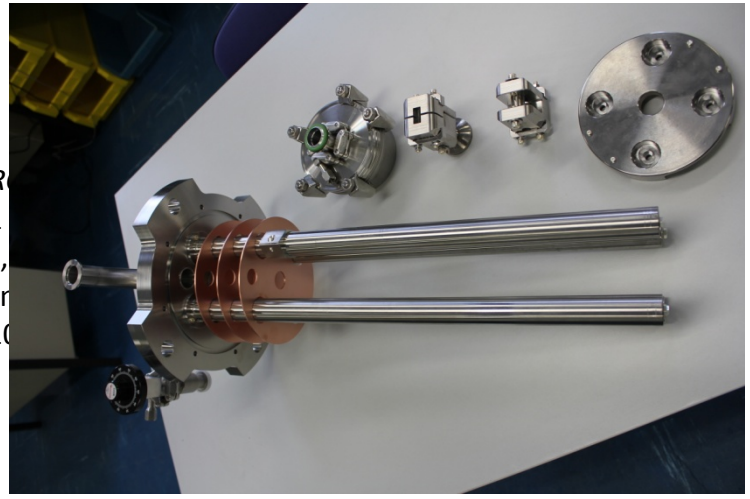
Hardness. *Wolpert 2R*

Load 1

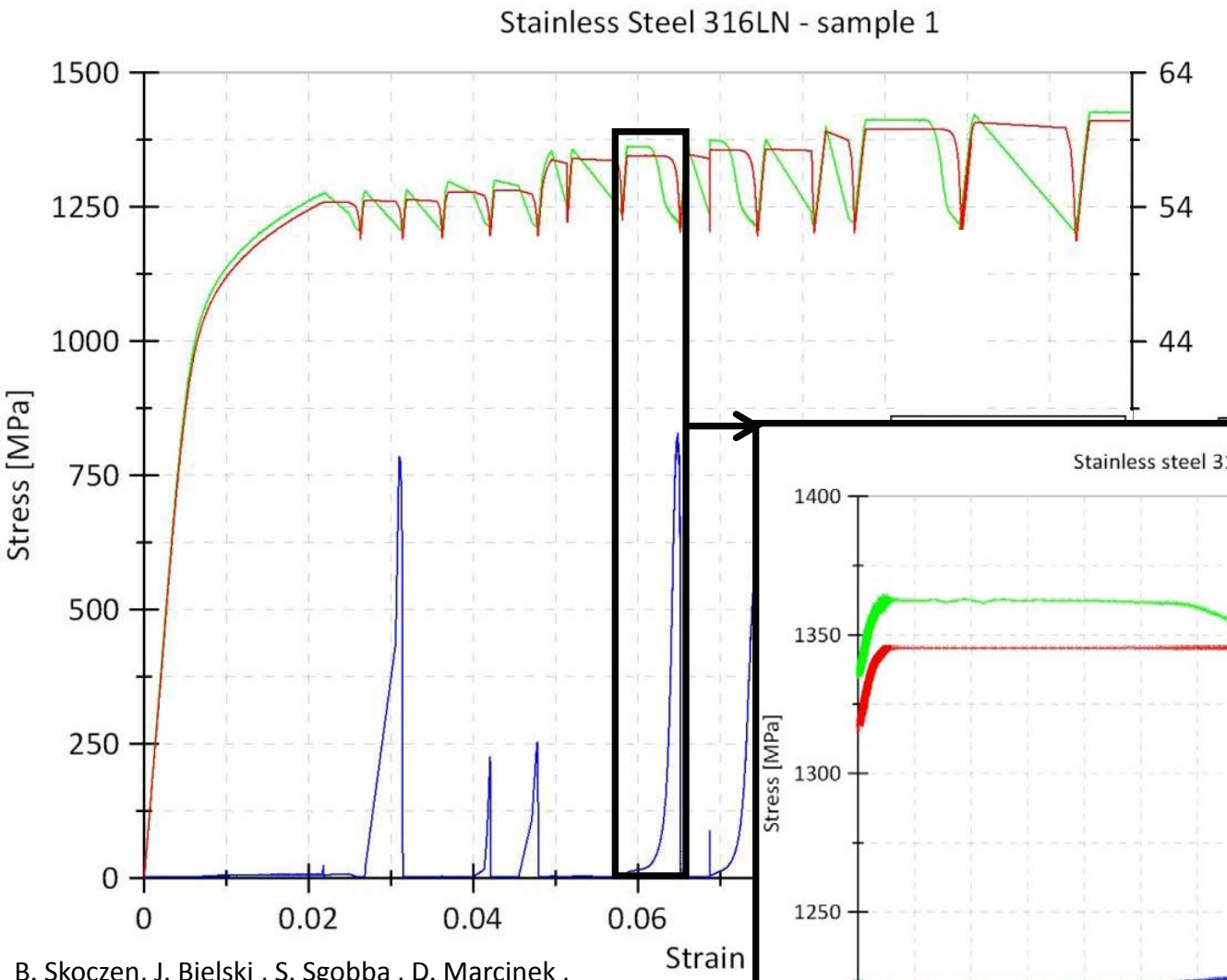
Brinell,

Micro hardness autor

Load 1

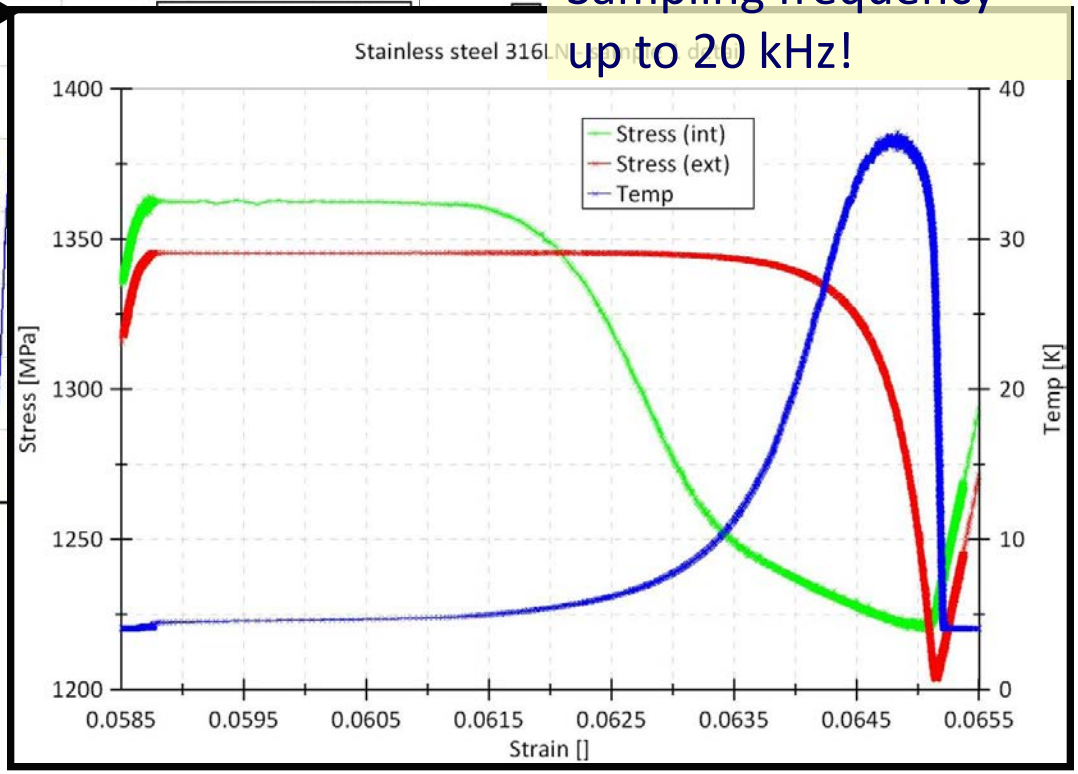


Capabilities of the equipment



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

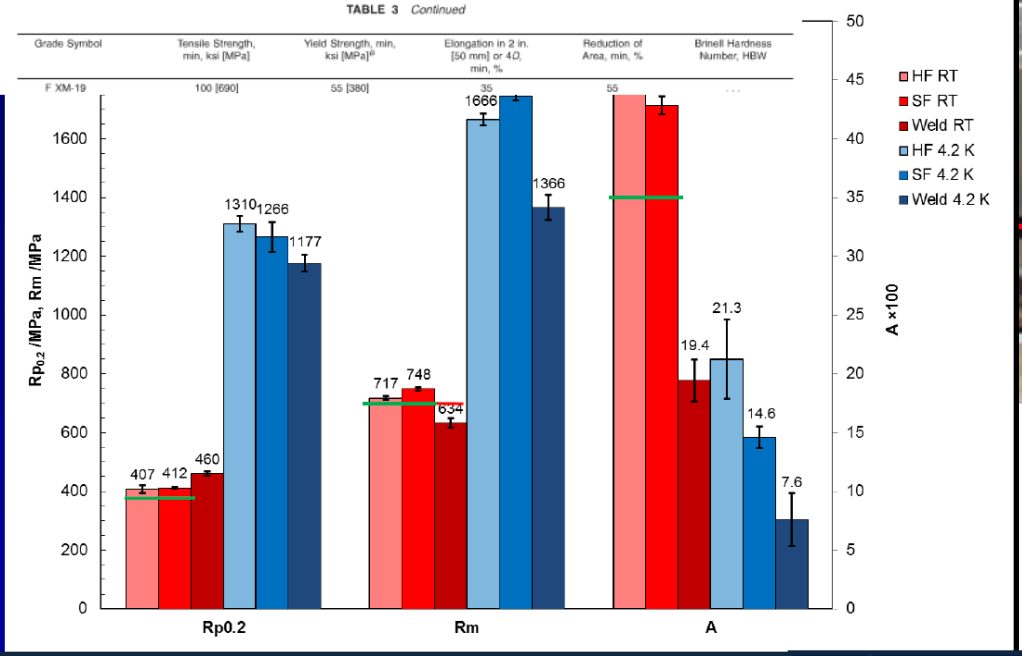
5.2 **Mechanical Testing**

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

A182/A182M – 10a

TABLE 3 *Continued*

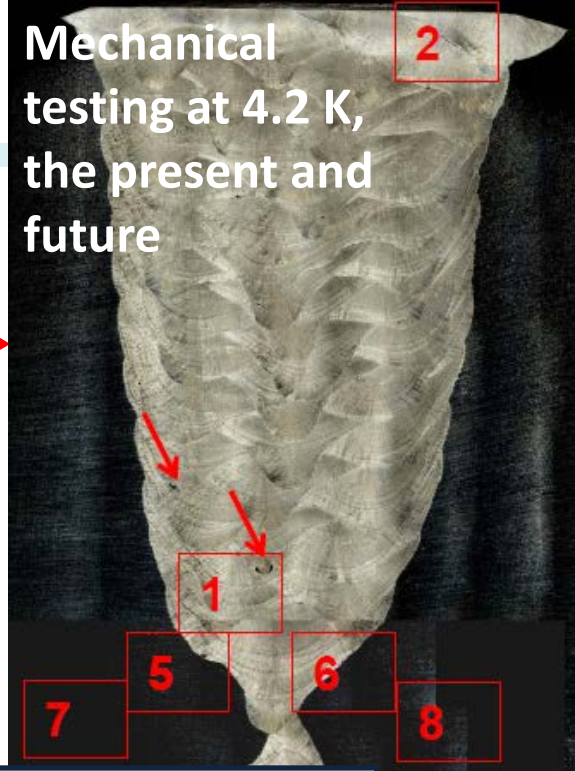
Grade Symbol	Tensile Strength, min, ksi [MPa]	Yield Strength, min, ksi [MPa][B]	Elongation in 2 in. [50 mm] or 4D, min, %	Reduction of Area, min, %	Brinell Hardness Number, HBW
F XM-19	100 [690]	55 [380]	35	55	...

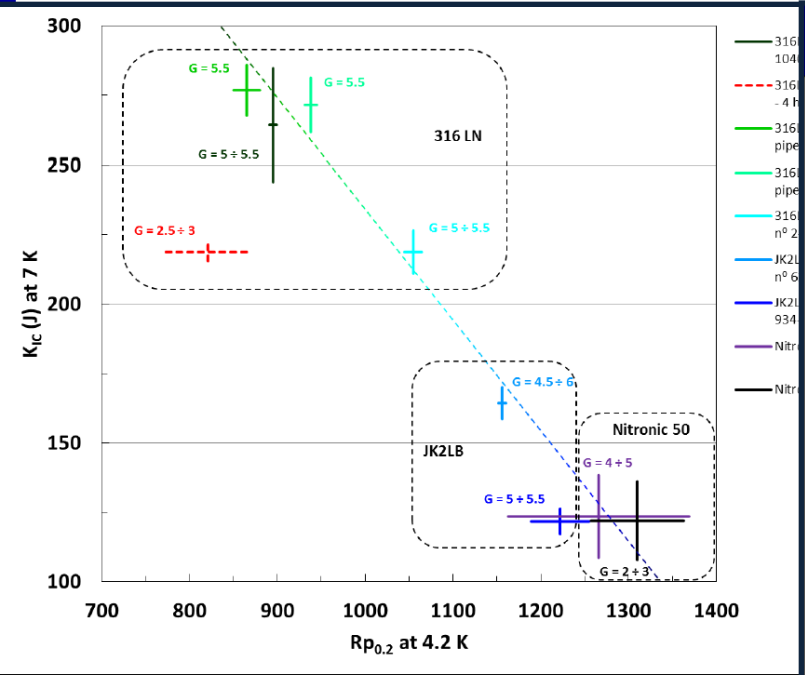


1/04/2012,
CERN
CS magnets

CERN + CEME

Mechanical testing at 4.2 K, the present and future



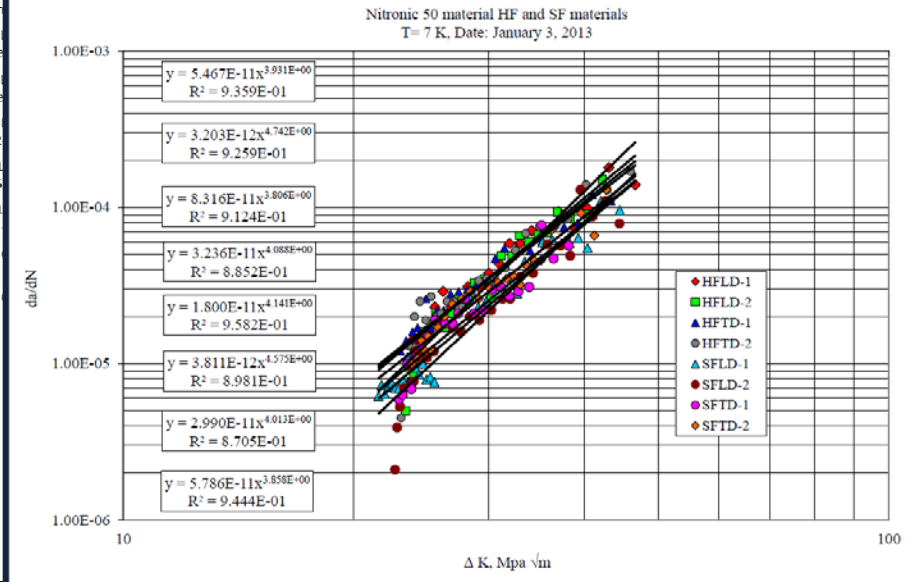


EN Engineering Department

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



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

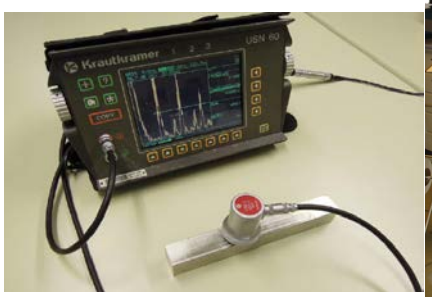
Gain 0 dB to 40 dB, probe from 0.5 MHz to 10 MHz,

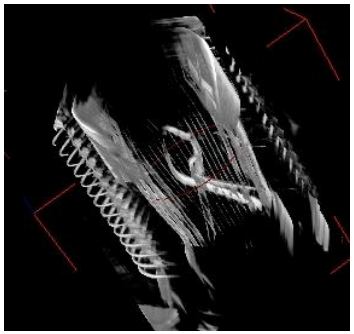
Various sectorial or linear transducers

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 tomogra

Mini-focus x-ray s

Flat panel detecto

Software for 3D r

X-ray sources. *Philips 160 kV*, 0.

Sieffert isovolt 16

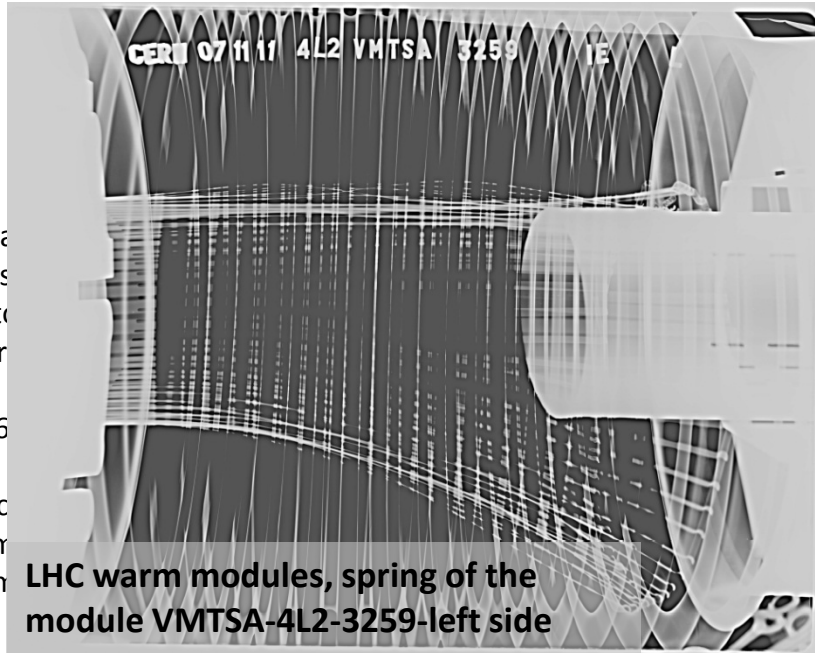
Computer radiography system

High definition sc

High resolution m

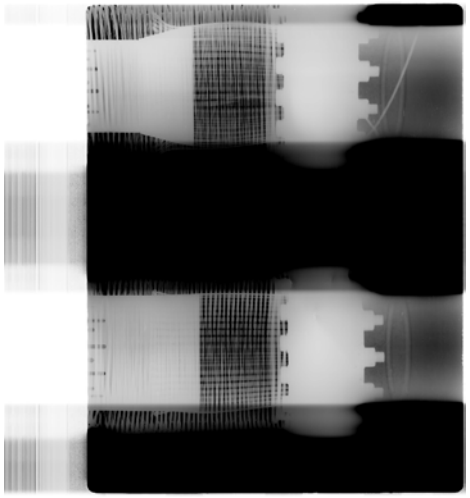
Controlled area (bunker) of 30 m

Level 2 certified operators

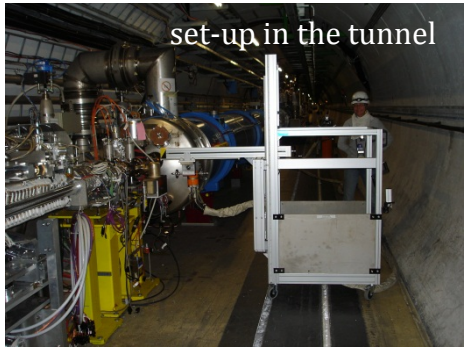


LHC warm modules, spring of the module VMTSA-4L2-3259-left side

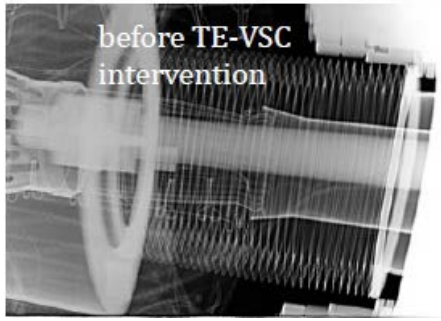
Non destructive testing

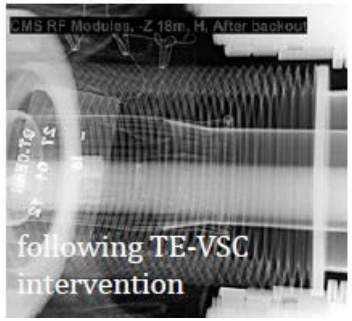


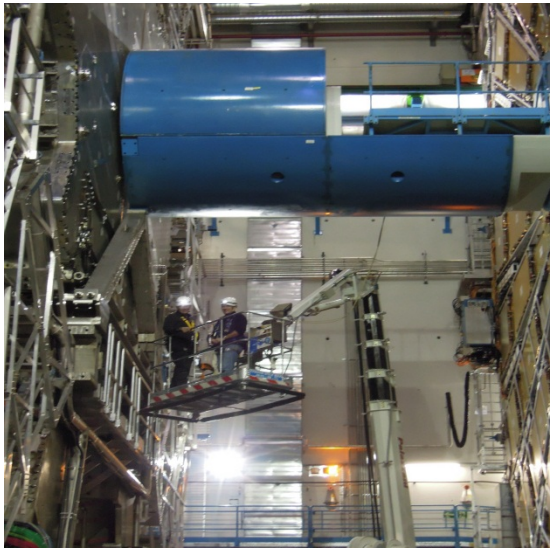
⇐ Computed Radiography Testing (RT) of LSS warm modules. Completion of the radiography campaigns during LHC technical stops. 1767 modules examined, 107 non-conformities identified to be repaired during LS_1, 46 nights.

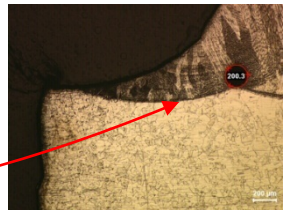


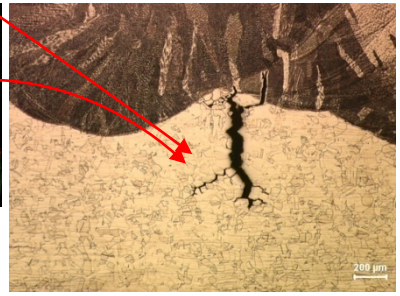
⇓ Computed RT of CMS RF modules











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

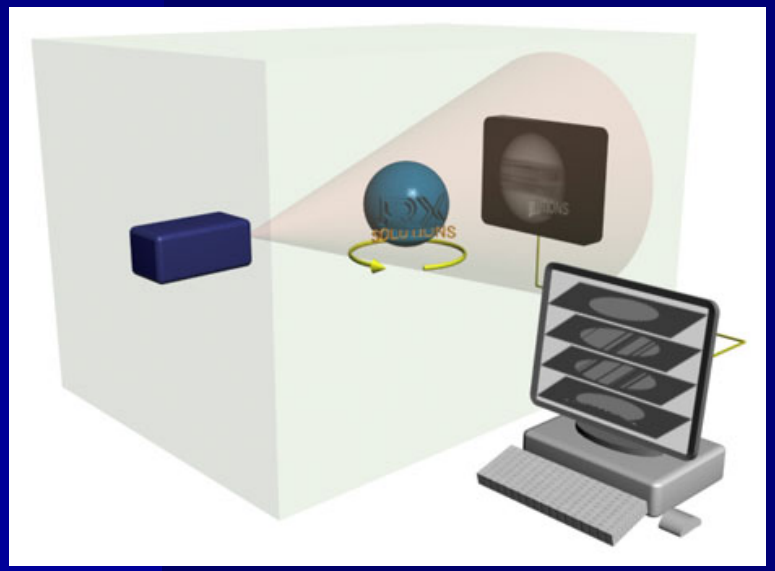
⇐ Computed RT of ATLAS RF modules

3D tomography
Technique description

Engineering Department

iter china eu india japan korea russia usa

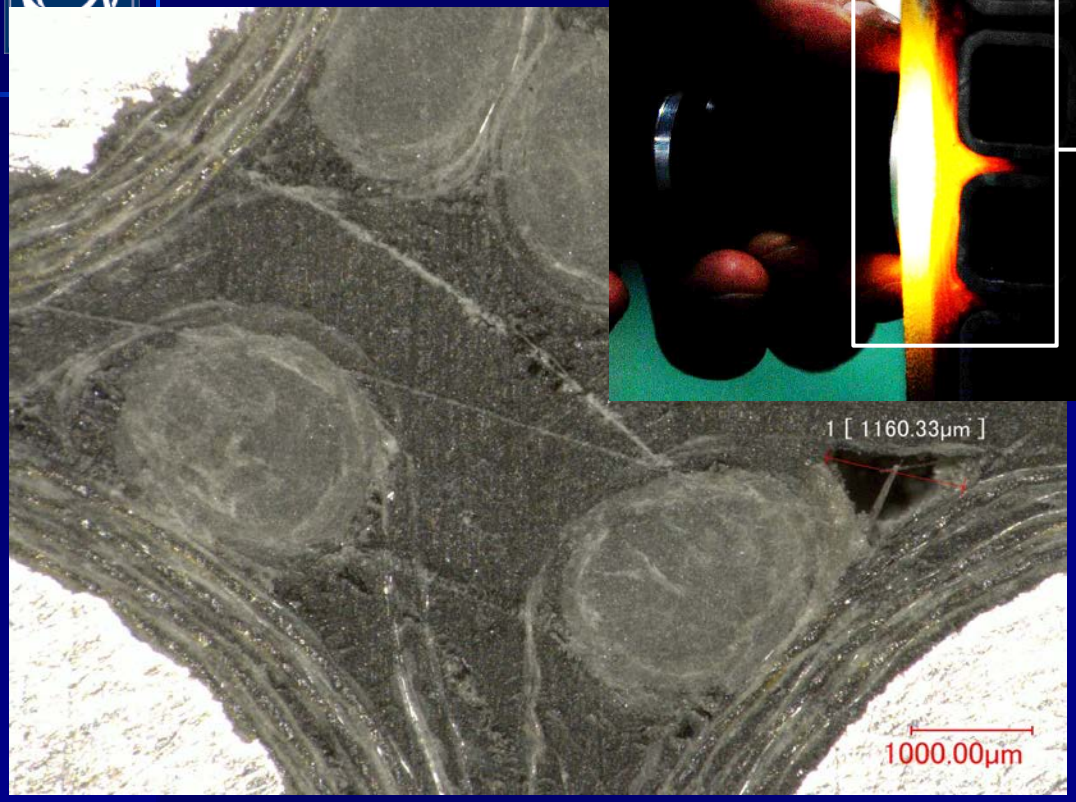
- Specimens placed on a mobile and rotatable stage
- Motorized zoom
- Full inspection of samples up to 240 mm length
- Sealed microfocus source, max. tension 150 kV
- HR area image sensor, 1920 pixels x 1536 pixels
- Detection surface: 200 mm x 250 mm
- 14 bits – 16000 gray levels
- 1-30 images /s

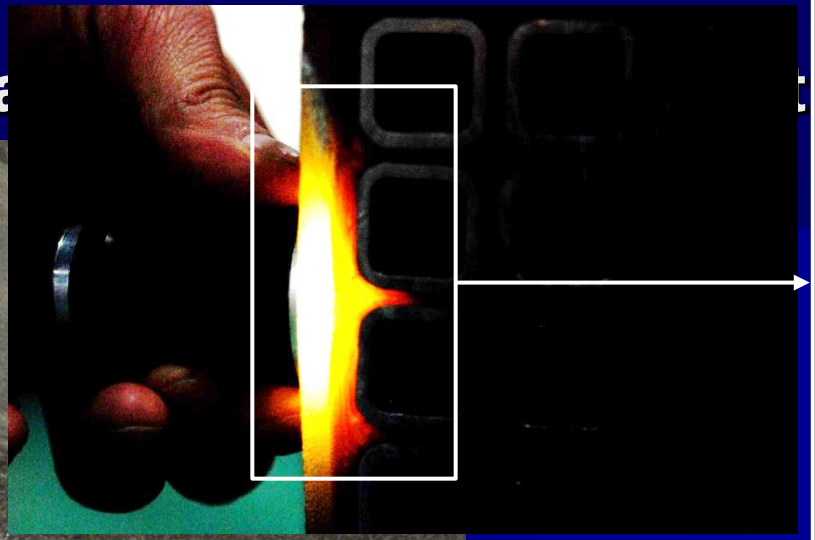


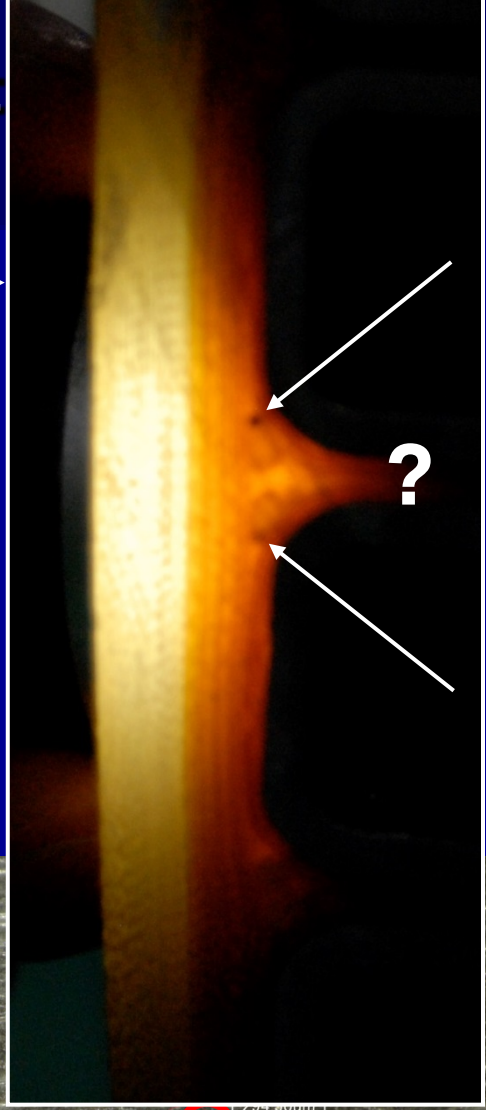
2nd sample of the VPI

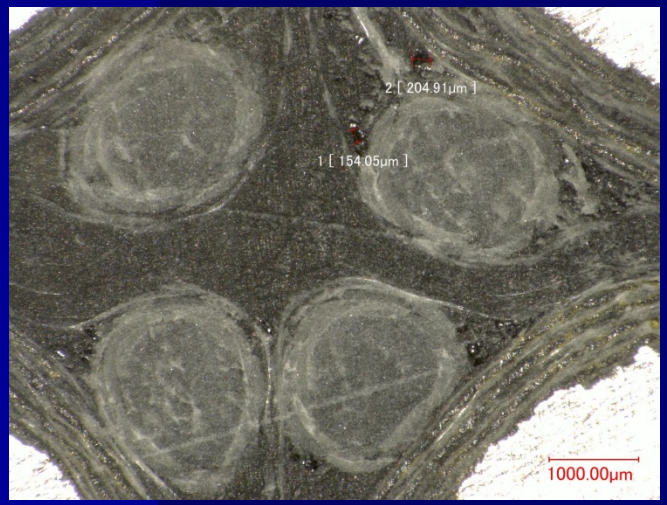
11 separate 360° scans were combined to compile a final single 3D object reconstruction while avoiding shadows due to steel conduits

Microstructura



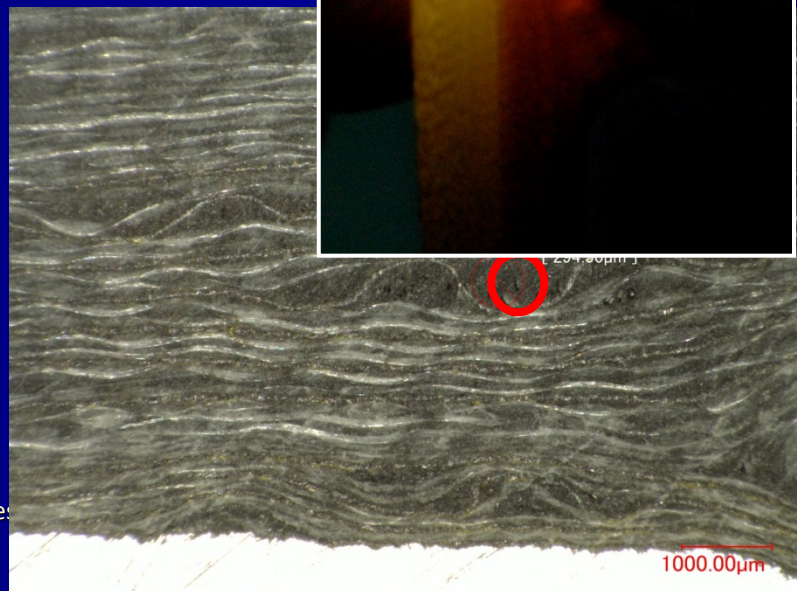




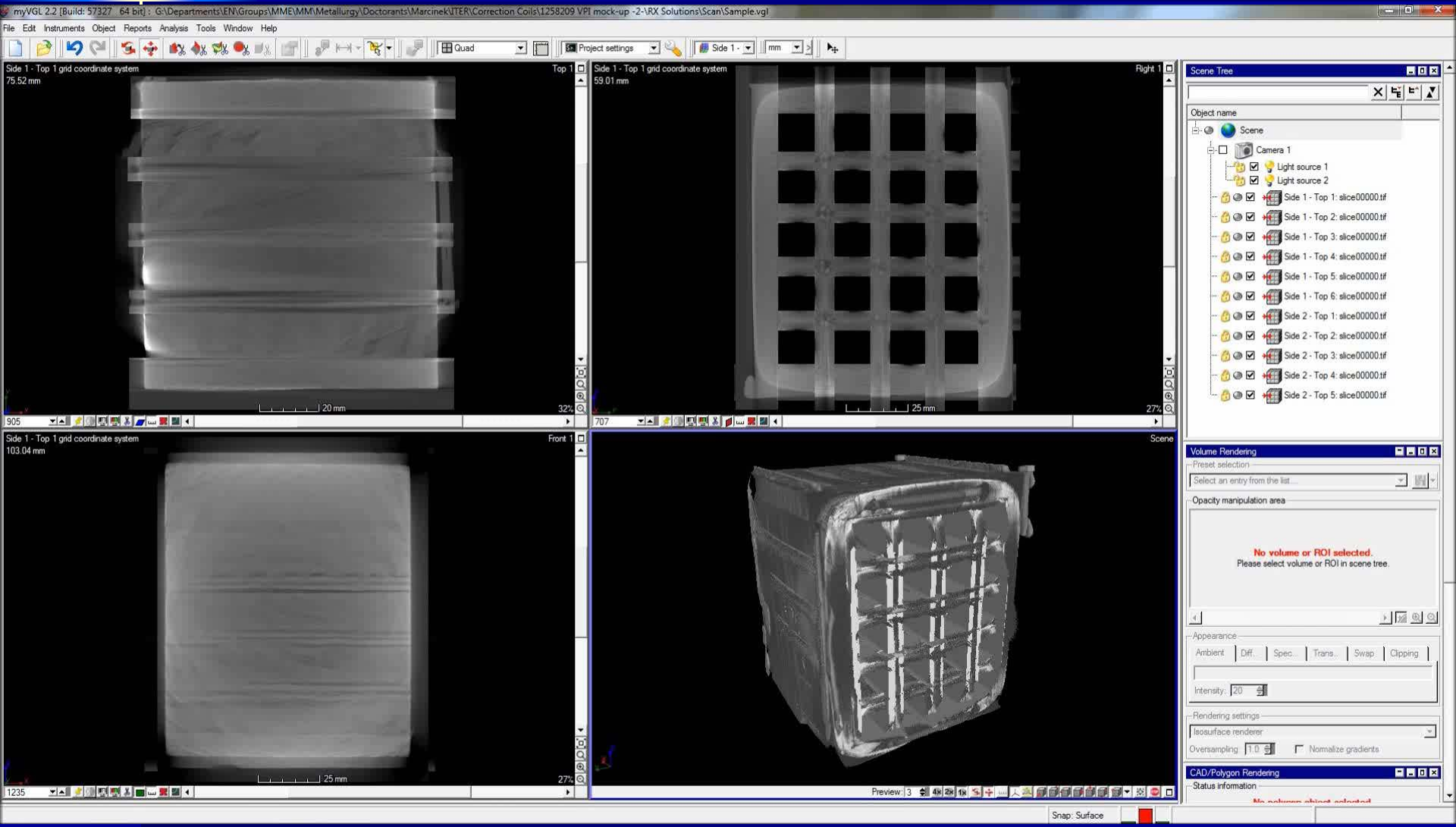


⇐ lack of resin in the cross-section

areas of pure resin ⇒

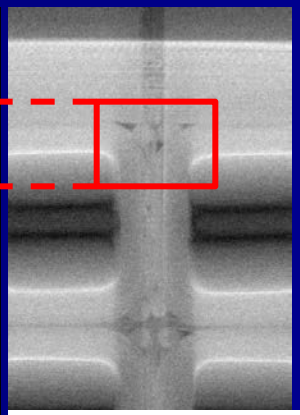


X-Ray Computed Tomography (CT)



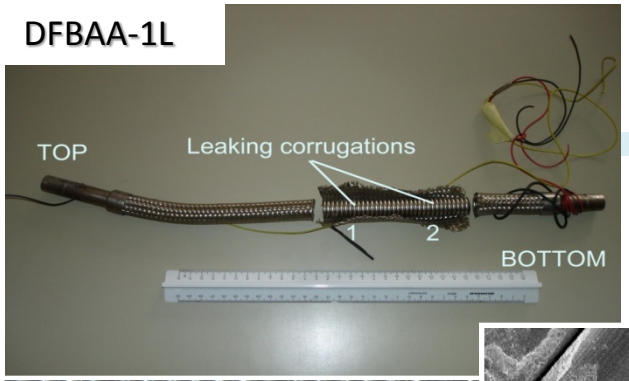
X-Ray
Computed Tomography (CT)

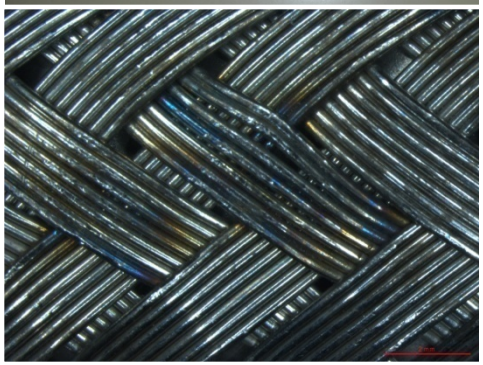
Slices extracted from the CT inspection confirming presence of voids along the fibers consistent with metallurgical cross sectional observations

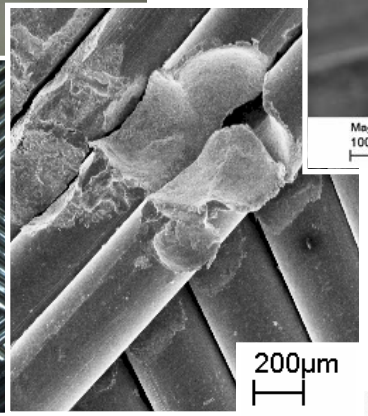


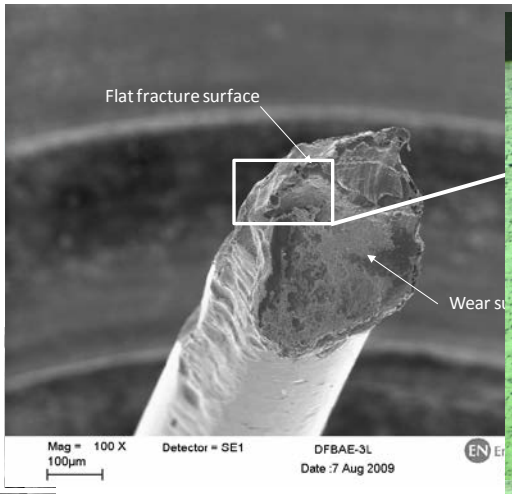
Elongated voids in resin along glass ropes between the steel conduits

DFBAA-1L

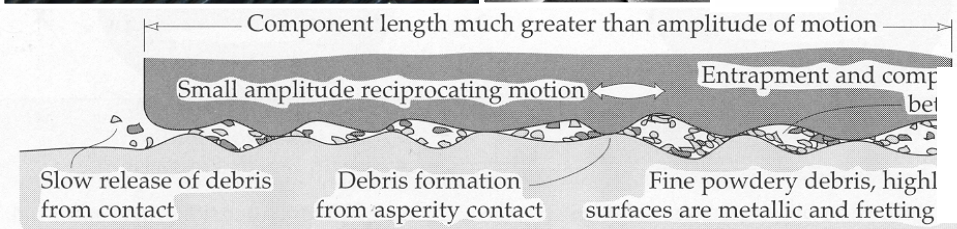






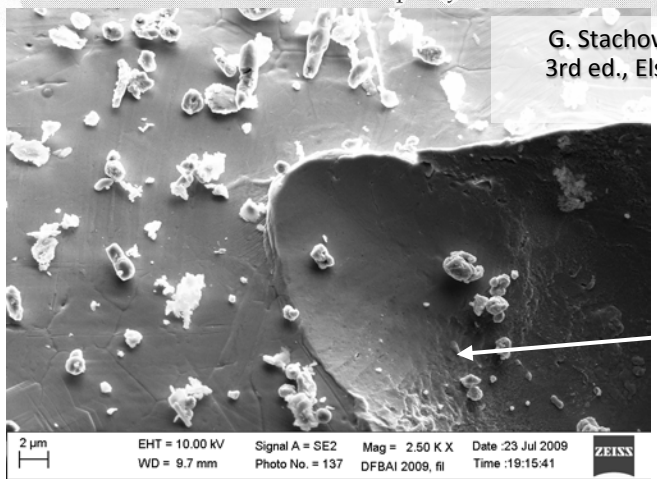


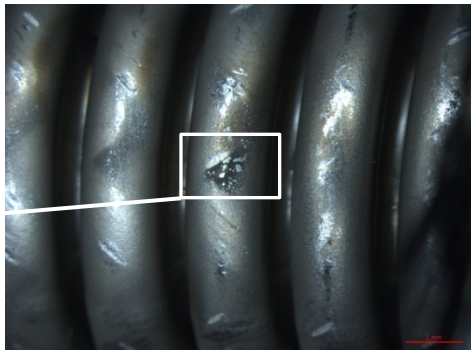
⇐ **Complex failure analysis of DFBA flexible metal hoses, TE-TM, 01/09/2009**

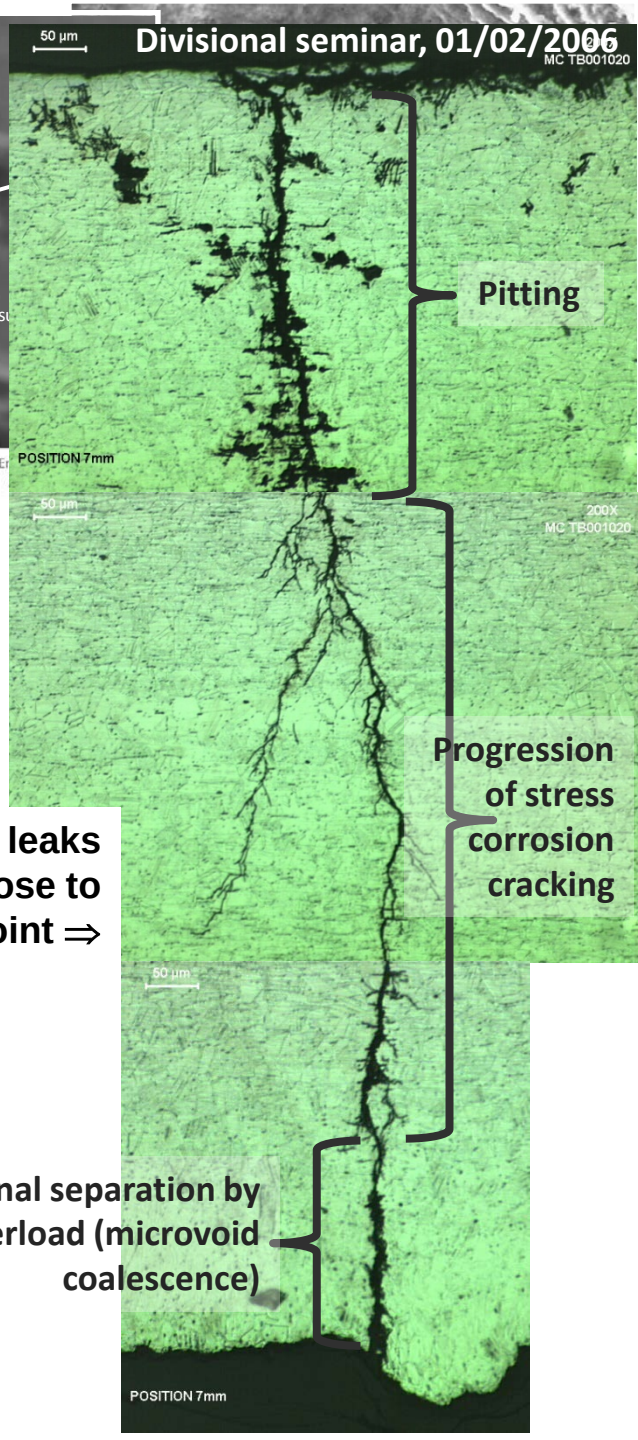


G. Stachowiak, A.W. Batchelor, Engineering Tribology, 3rd ed., Elsevier Butterworth-Heinemann, Amsterdam (2005)

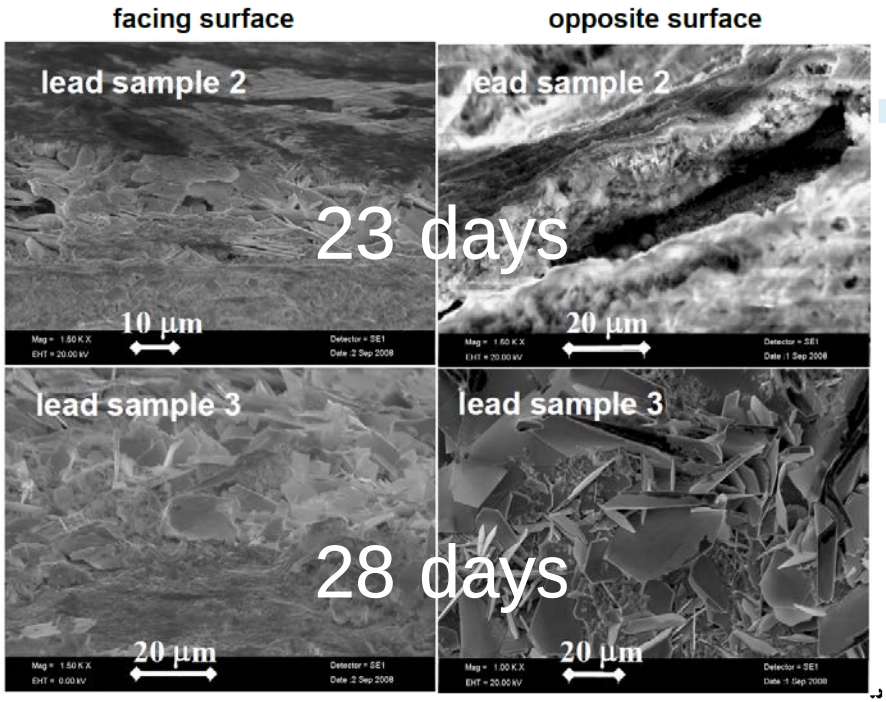
QRL: corrosion leaks on the tube F close to the fix point ⇒



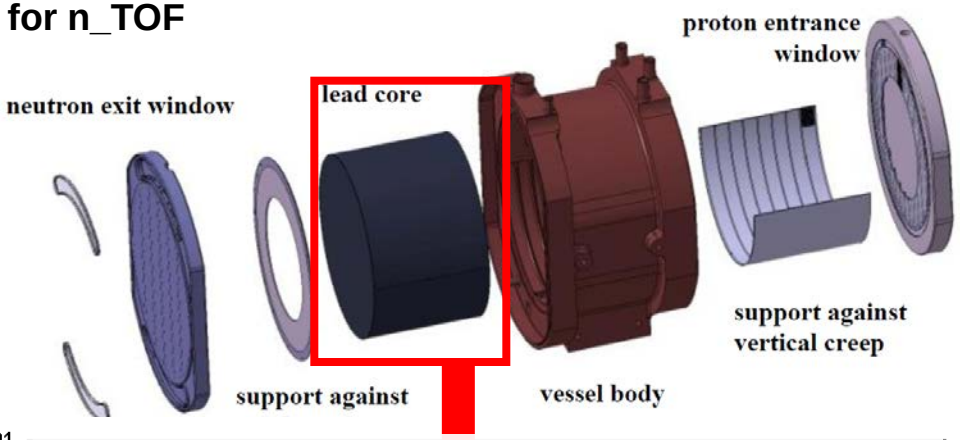




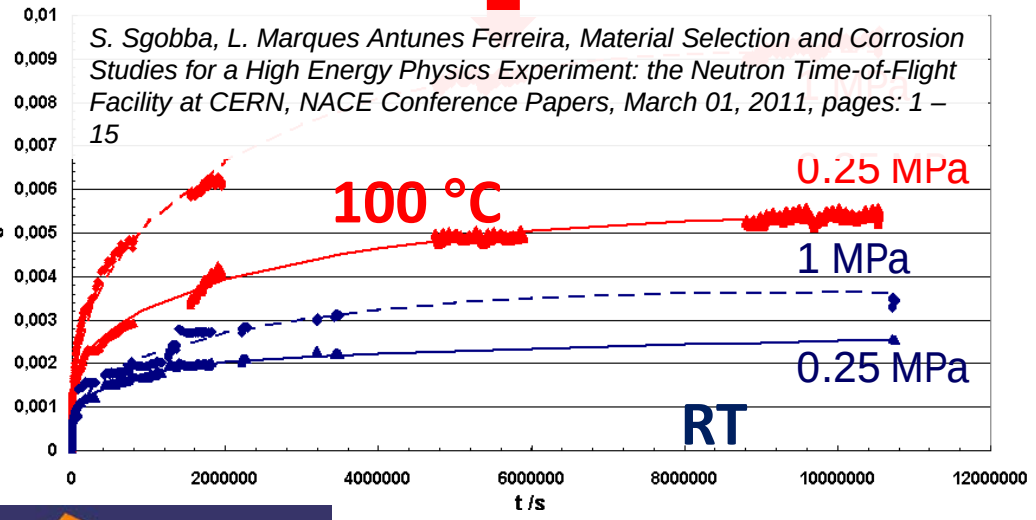
Final separation by overload (microvoid coalescence)



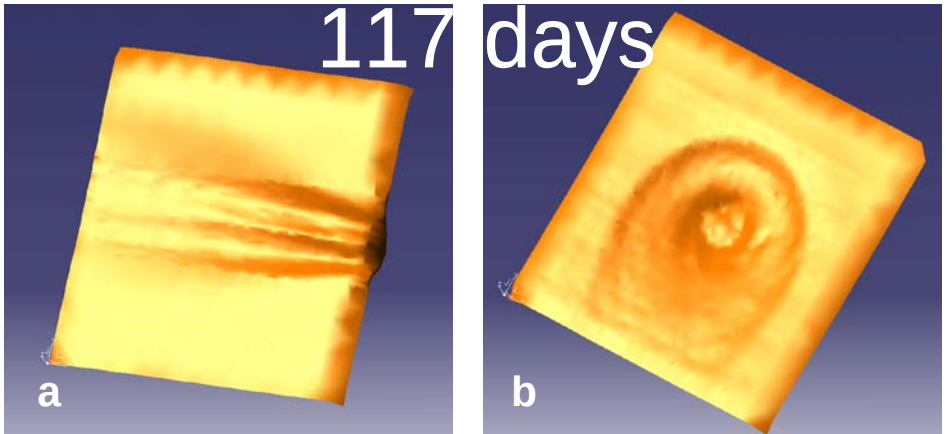
2008-2009, material selection and corrosion studies for n_TOF



S. Sgobba, L. Marques Antunes Ferreira, Material Selection and Corrosion Studies for a High Energy Physics Experiment: the Neutron Time-of-Flight Facility at CERN, NACE Conference Papers, March 01, 2011, pages: 1 – 15



erosion and impingement quantified by 3D metrology



Conclusions

- CERN-wide (and beyond) activity

- Limited but selected and highly specialized equipment

- **CERN expertise completed by an extensive network**

- Combined resources, facilities and expertise support advanced materials activities at CERN
    - Industrial partners
    - National labs, academy
    - CERN-ITER cooperation agreement (IO, DAs via IO)

- Materials can be submitted to aggressive environments in HEP applications
    - Corrosive environments (radiations, flux residues, erosion, impingement…)
    - Cryogenic temperatures (ductility and fracture toughness to be preserved, response to fatigue, fretting fatigue…)