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science transfer talent

Multiscale characterization of materials and processes: IMDEA's capabilities

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IMDEA Materials Institute

UNIT OF EXCELLENCE MARÍA DE MAEZTU

GOBIERNO DE ESPAÑA MINISTERIO DE CIENCIA E INNOVACIÓN

EUROPEAN UNION STRUCTURAL FUNDS

Comunidad de Madrid

The IMDEA Materials Institute, one of the seven Madrid Institutes for Advanced Studies (IMDEA), is a public research centre (**non-profit research organisation**) founded in 2007 by Madrid's regional government.

The **Mission** of the Institute is to do research of excellence at the forefront of Materials Science and Engineering, contributing to tackle the challenges of society and fostering the sustainable development.

science

excellence in materials **science** and engineering research

transfer

technology transfer to industry to increase competitiveness and maintain technological leadership

talent

attraction of talented researchers from all over the world to work in Madrid in an international and interdisciplinary environment

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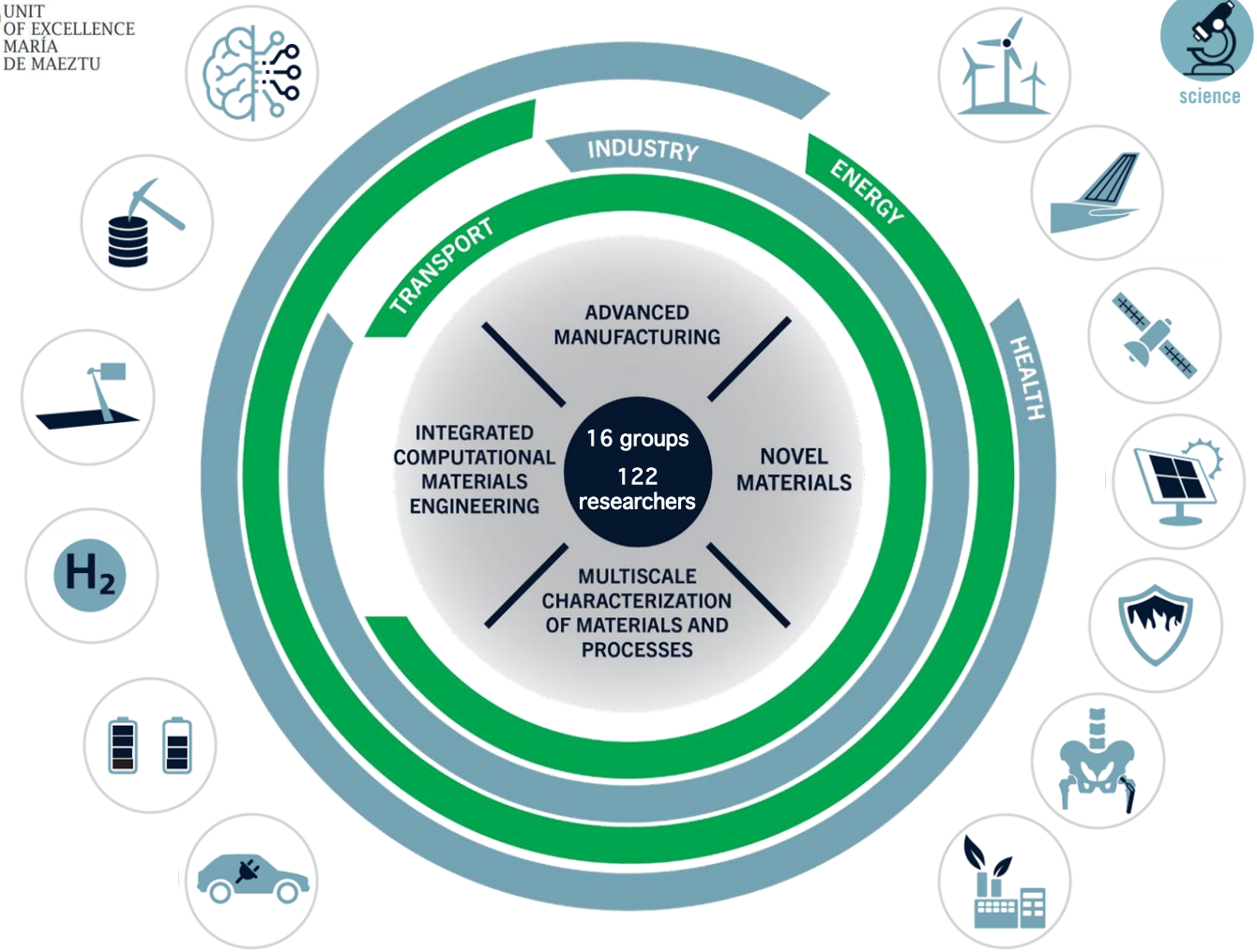
science transfer talent



Multiscale characterization of materials and processes: IMDEA's capabilities

OBJECTIVES

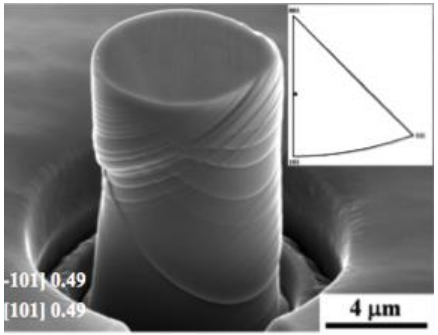
- **Understanding processing-microstructure-properties:**

- Microstructure and defect characterization, including 3D and 4D
- Mechanical properties from the bulk material to the individual phases and interfaces
- Microstructural development during processing

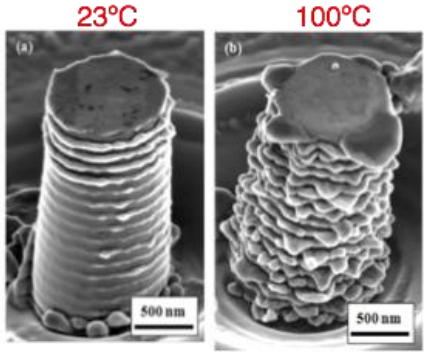
Fundamentals of plasticity and fracture	Bottom-up microstructural design
Develop new materials exploiting size effects	Inform microstructurally based models

- **Wide range of materials and applications**:

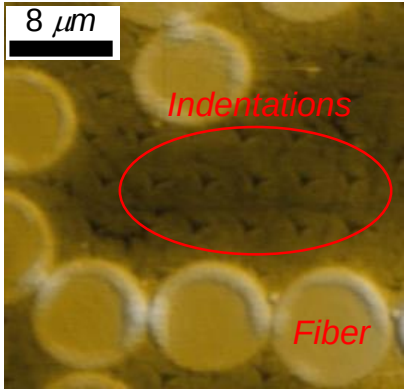
Metallic alloys: superalloys
Intermetallics ,steels, Mg



Coatings



PMCs



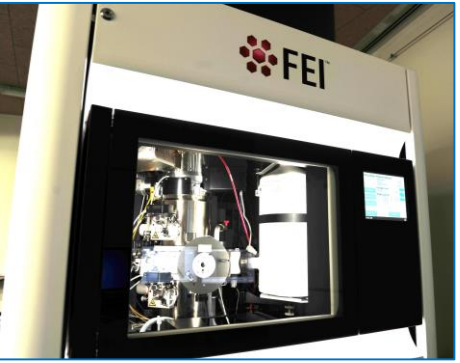
UNIQUE CAPABILITIES

- **3D characterization of materials,** across several length scales:

  - **FIB-FEGSEM**: including 3D-SEM, 3D-EDS and 3D-EBSD
  - **TEM**: including 3D-STEM and 3D-EDS.
  - **in-situ mechanical testing**

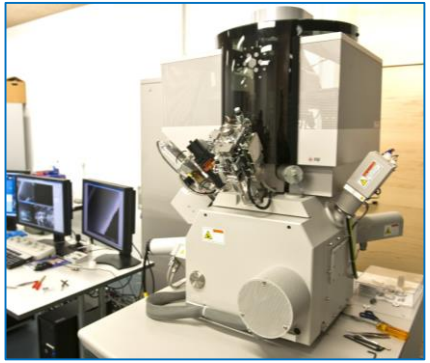
Compatible with in-situ mechanical devices for SEM

200 kV FEG(S)TEM (3D-STEM 3D-EDS)



FEI Talos F200X

FIB-FEGSEM (EDS and EBSD)



FEI Helios 600i

FEGSEM (EDS and EBSD)

Thermo Fisher Apreo 2S

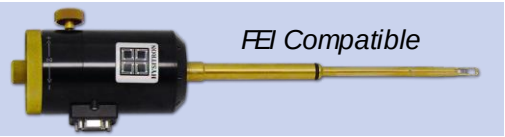
W filament (EDS)

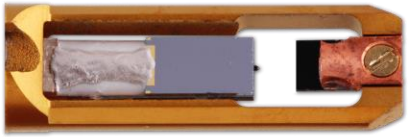
EVO MA15, Zeiss

UNIQUE CAPABILITIES: in-situ mechanical testing in TEM and SEM

- **Across several length scales and at different temperatures:**

Nano scale



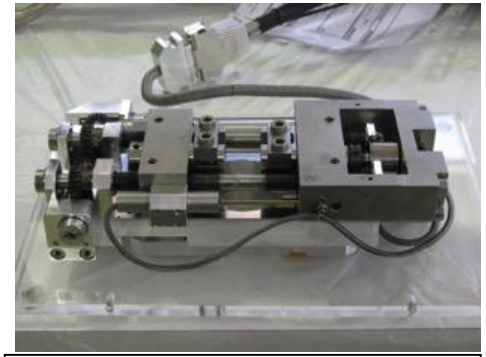


Hysitron PI95
10 mN

Micro scale

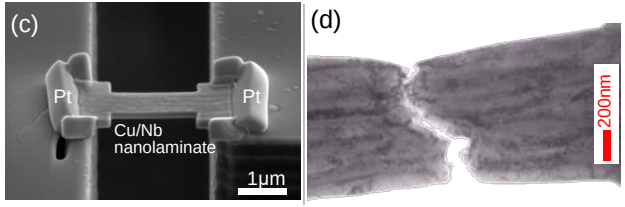
Hysitron PI87HT
150 mN, 800 ºC

Macro scale

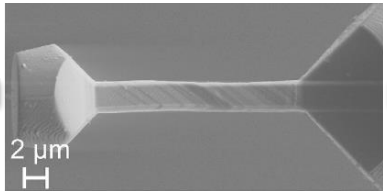


K&W Tension-Compression
10 kN, 650 ºC

From nm…



…to µm….



LOAD

…to mm

UNIQUE CAPABILITIES

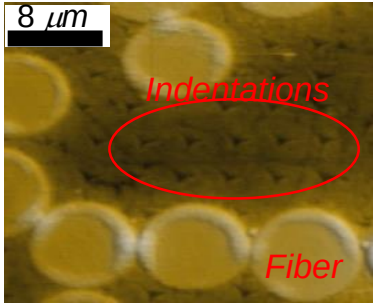
Hysitron TI950 Triboindenter

- 10 and 750 mN load heads
- Resolution: 1 nN, 0.1 nm
- Nanoscratch and nanowear
- Dynamic measurements
- SPM imaging
- Positioning with nm resolution

NANOMECHANICS

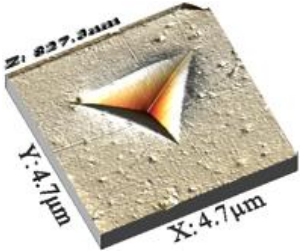
Micromaterials Nanotest

- 500 mN load head
- High temperature: 750 ºC
- Active tip heating
- Inert atmosphere: Ar



Park XE 150 AFM

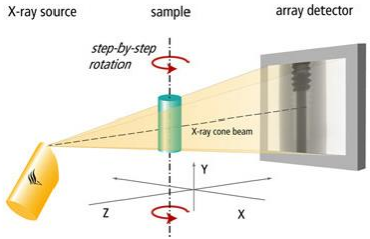
- Contact and non-contact
- Magnetic and thermal measurements
- High temperature: 250 ºC

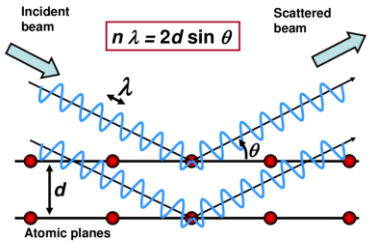


UNIQUE CAPABILITIES

- **3D characterization of materials,** using **X-rays**, NDT, correlative:
  - Microstructure and defect characterization, 3D and 4D
  - Microstructure and relationship with mechanical properties, **ex-situ** and **in-situ** testing
  - Microstructure development during processing, **in-situ** devices

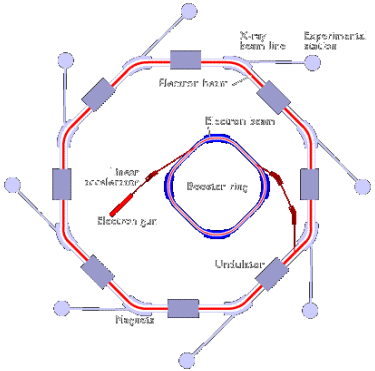
Aided by X-rays (lab & synchrotron) + 4D evaluation (in-situ devices)





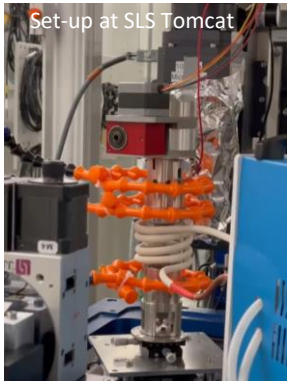
Nanotom 160NF – Lab tomograph

Empyrean Panalytical – Lab diffractometer



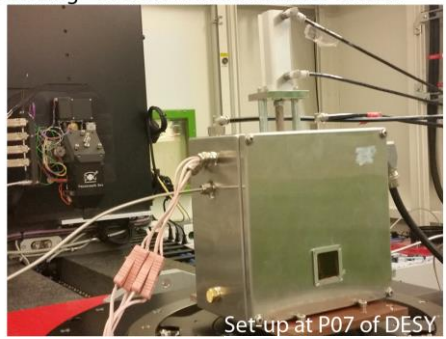
ESRF / SLS / PETRA III / BESSY / ALBA

Creep testing device for high temperature and in-situ XCT/XRD. Resistive heating.

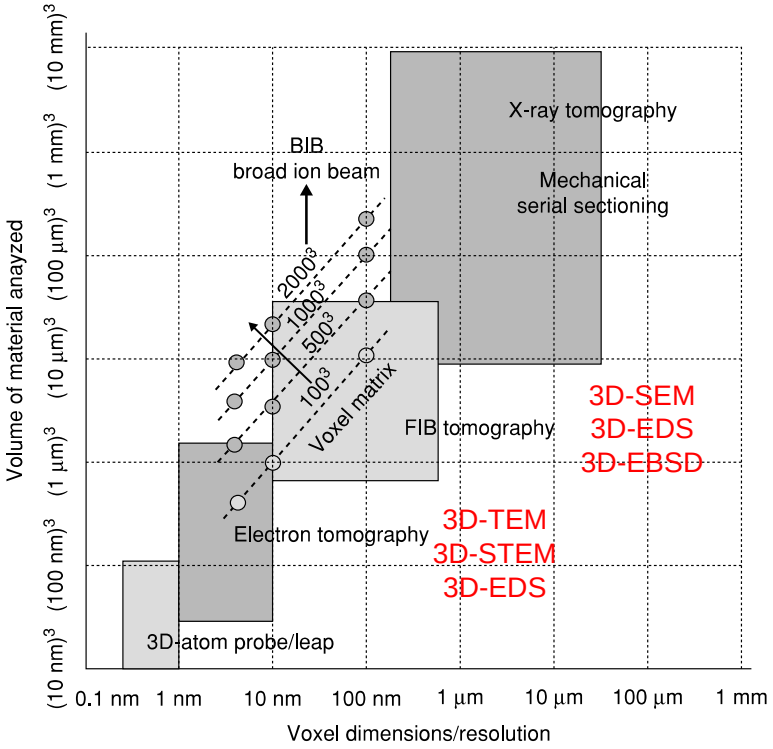


Mechanical testing device for RT & high temperature (600°C) and in-situ XCT & XRD. Induction heated

Processing - In-situ HT infiltration for metals



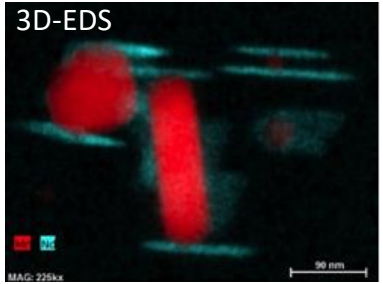
TOMOGRAPHY ACROSS LENGTH SCALES



After Holzer and Cantoni

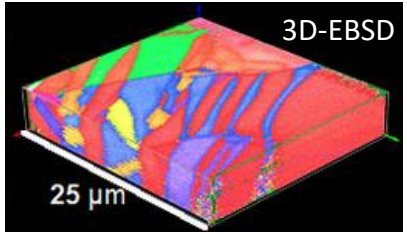
From nm…

TEM



Precipitates in Mg-Mn-Nd alloys

SEM



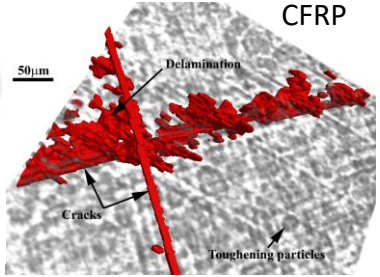
3D twin structure

…to µm…

XCT

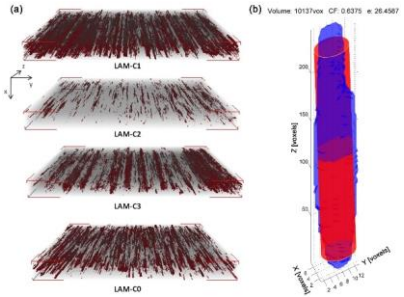
Porosity in die-cast Mg

Damage in CFRP



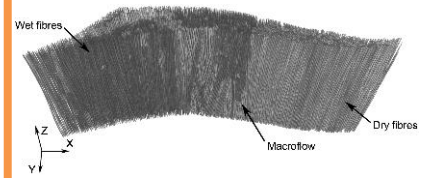
Research topics in Composites

Effect of curing in OoA composites

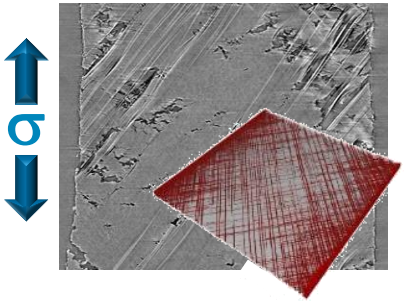


In-situ infiltration – microflow investigation

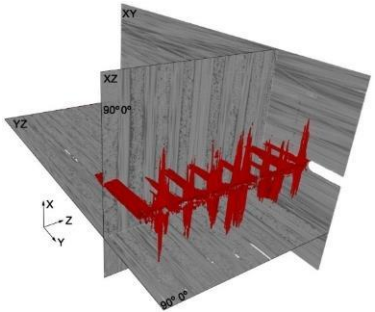
In-situ curing



Multiscale XCT for damage evaluation

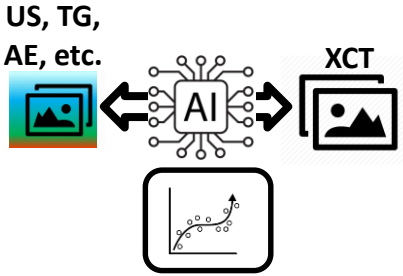


Sequential loading in 3PBT



Analysis of impact damage

Smart detection of defect



Crack formation in CFRP under thermal cycling

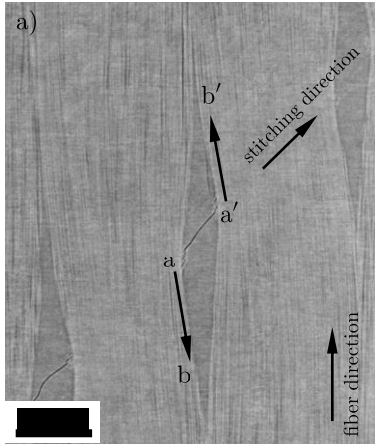
Objective

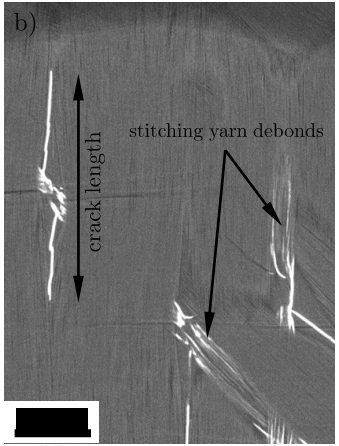
1) Non-crimp fabric composites subjected to thermal cycling => Evaluate crack generation and growth under thermal conditions (-55°C to 72°C)

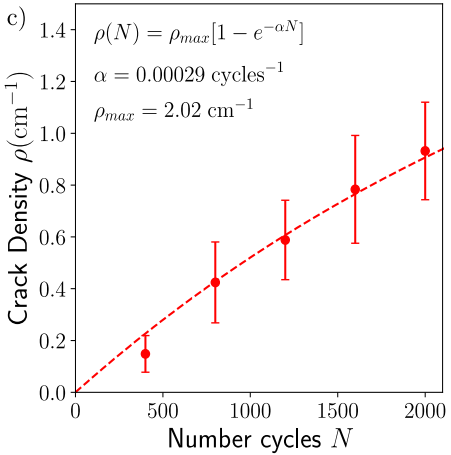
2) XCT allowed for a detailed characterization of the individual cracks, their shape and spatial distribution within the composite.

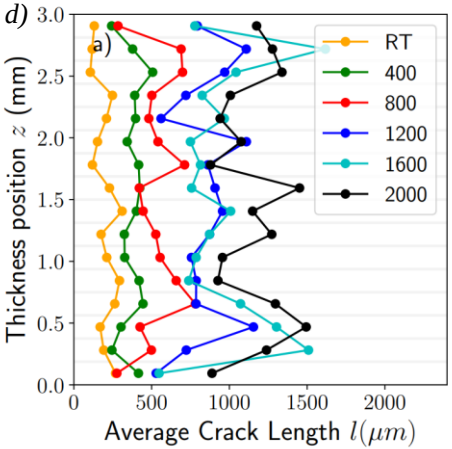
3) Automated ML-based segmentation to obtain robust and precise data

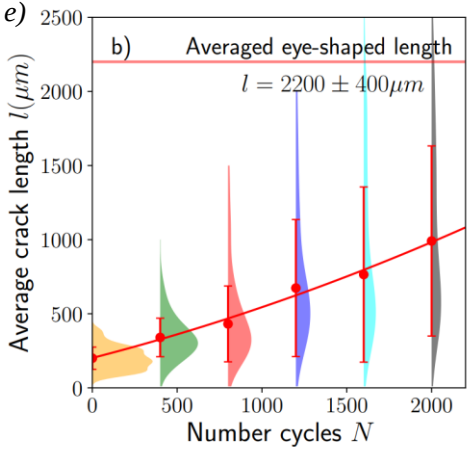
- TOP view: *a) XCT of crack within the resin rich region and b) XCT after dye penetrant (increased contrast of cracks)*
- *c) Crack density vs Number of thermal cycles*
- *d) Average crack length vs. thickness position*
- *e) Average crack length vs. number of thermal cycles and length distribution. Huge quantity of cracks were analyzed.*









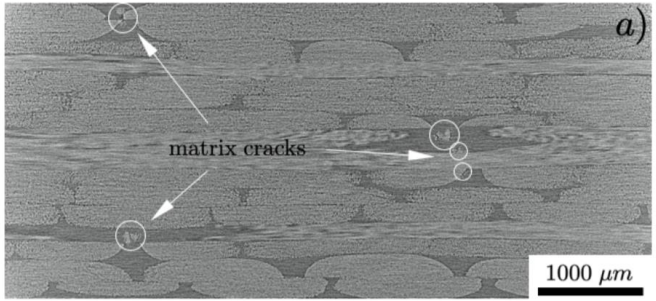


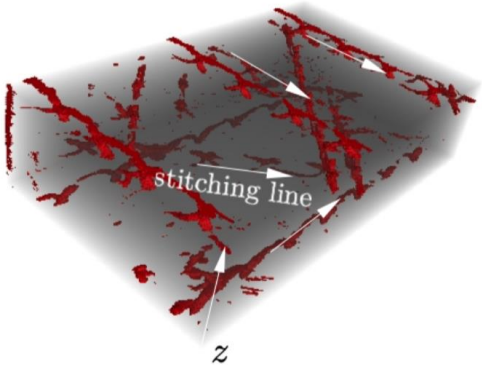
Lizarralde, I. https://doi.org/10.1016/j.compositesa.2021.106699

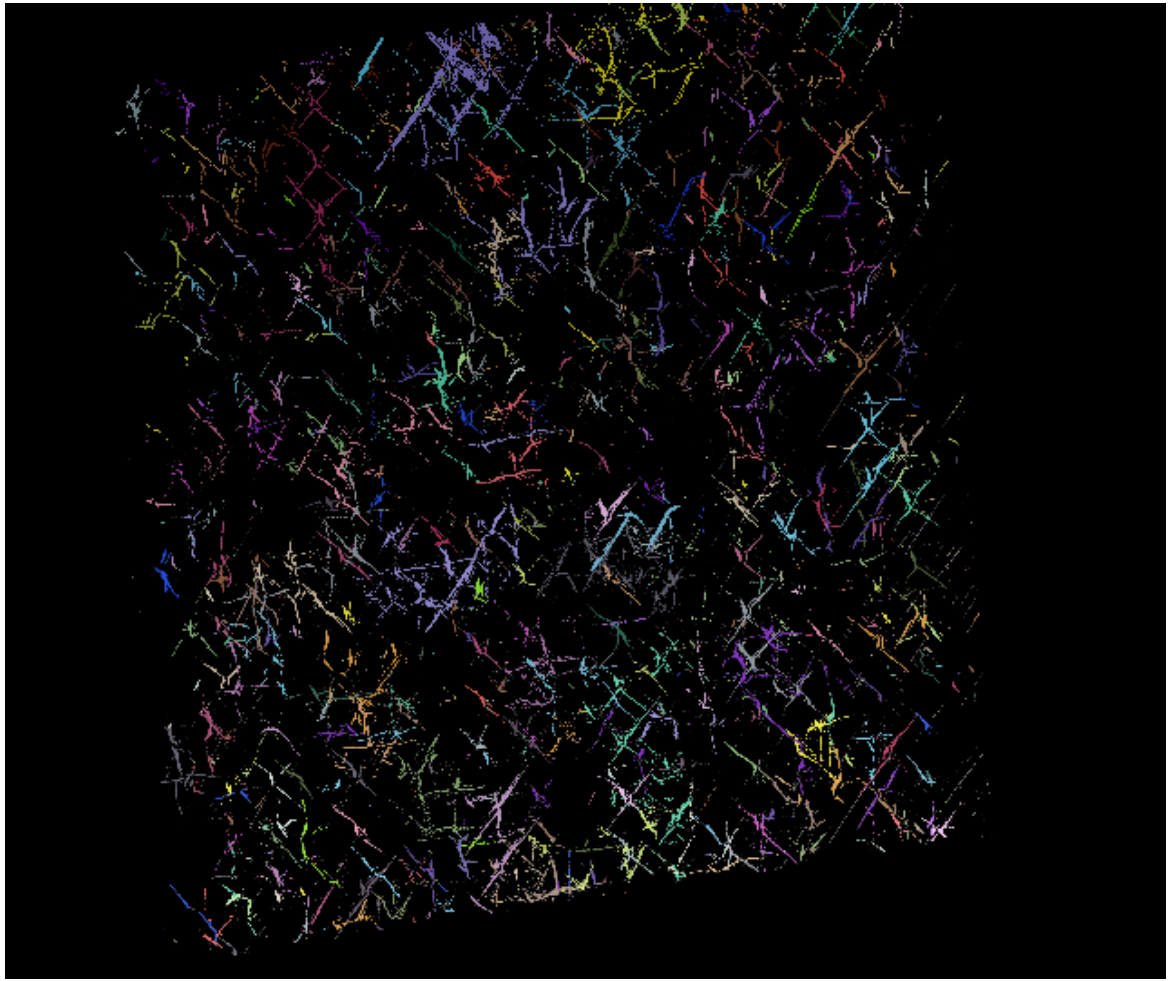
Crack formation in CFRP under thermal cycling

- *Crack clustering 3D:*
- *Algorithms: K-Means*
  DBSCAN
  OPTICS

- *Determination of crack percolation path possible in 3D*

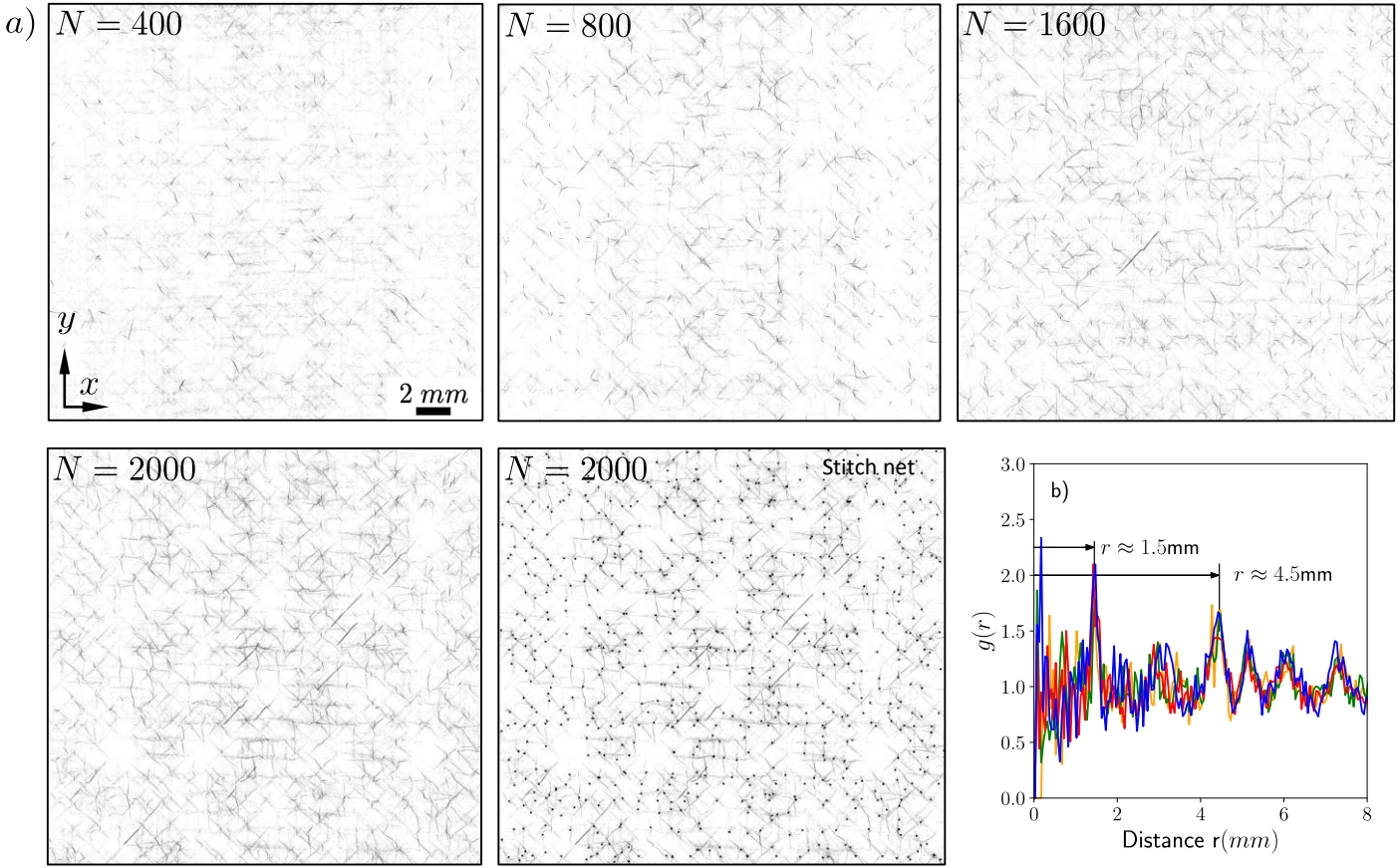






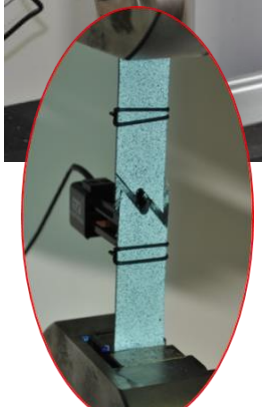
Crack formation in CFRP under thermal cycling

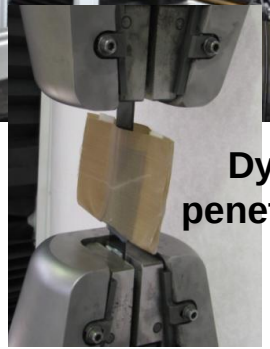
- *a) Projection in the through-the-thickness direction of detected cracks at several number of thermal cycles.*
- *N=2000, overlapping of stitching points with crack*
- Pair correlation function *g(r), to determine cluster regions. Cluster-to-cluster distance of about 3mm.*



Matrix cracking evolution: [90/+45/-45/90/0]s – Mechanical testing in OH specimen

Tensile tests + DIC + Cyclic loading

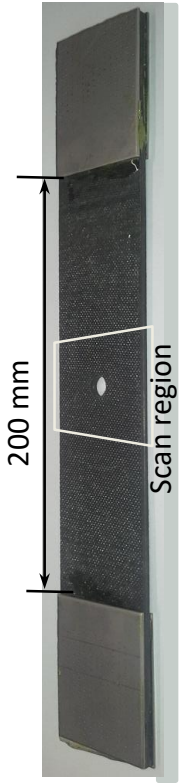




Dye penetrant

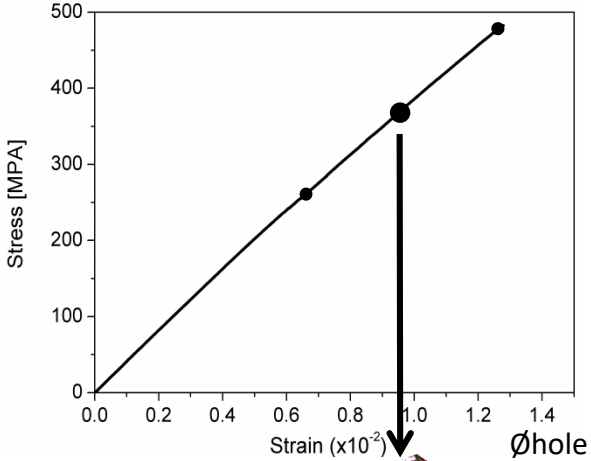
Material

UD300/M10.1



$[90/+45/-45/90/0]_s$

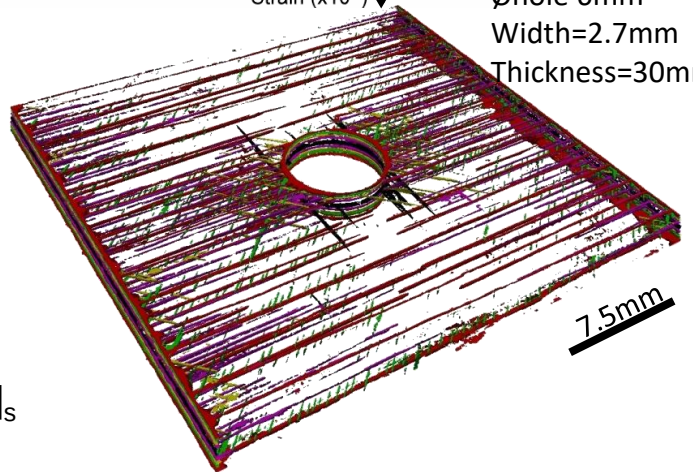
OH



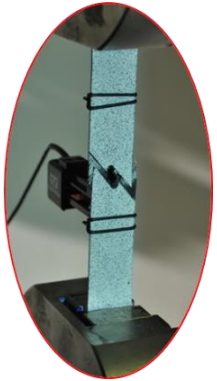
Øhole 6mm

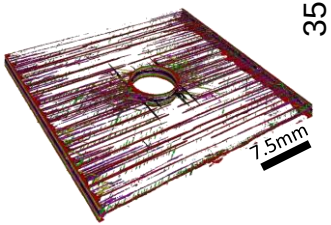
Width=2.7mm

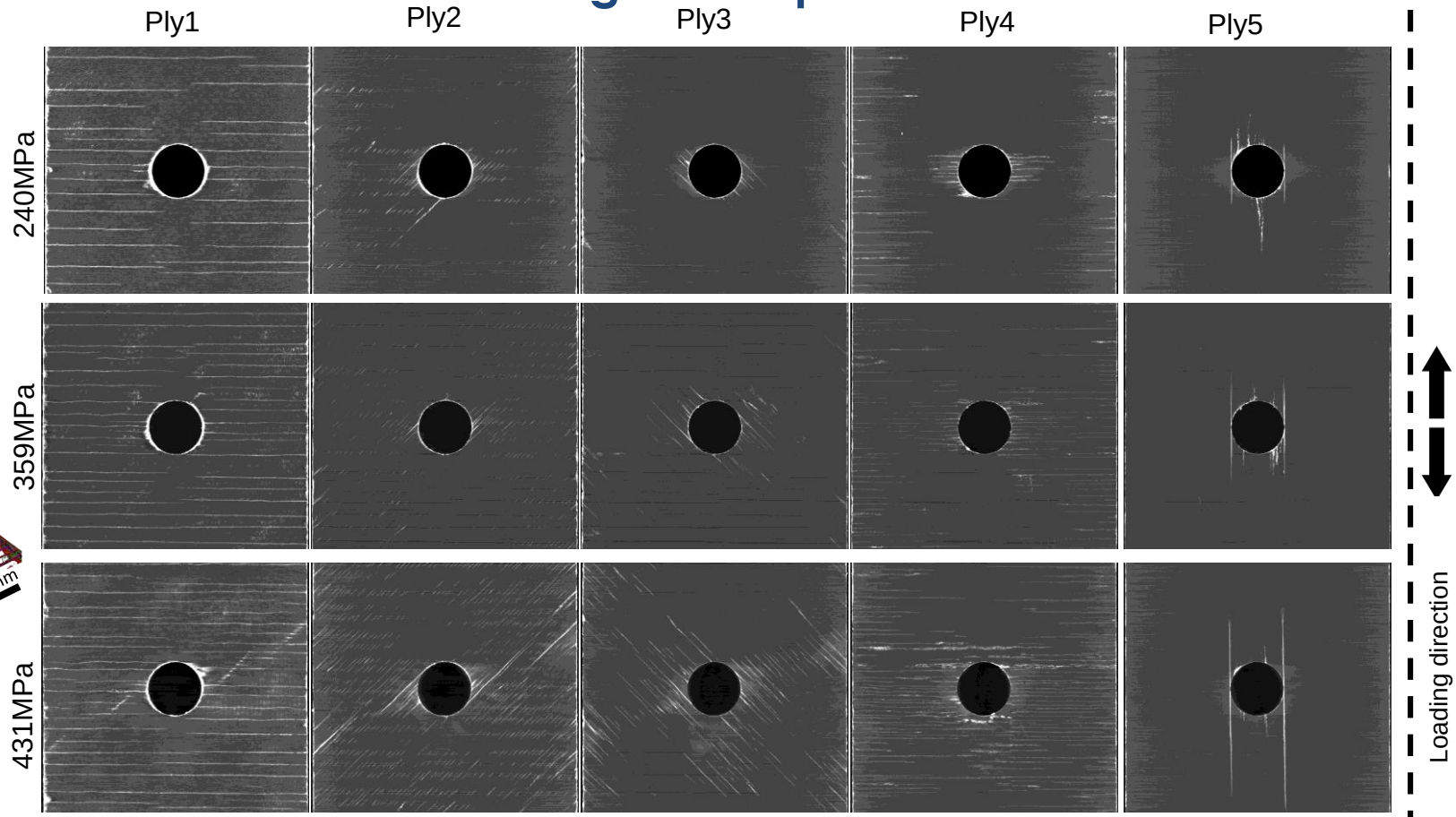
Thickness=30mm



Matrix cracking evolution: [90/+45/-45/90/0]s – Mechanical testing in OH specimen







Conclusions

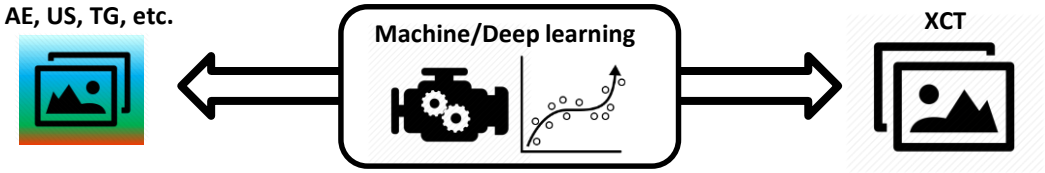
1) XCT resolution is high enough to detect cracks with thicknesses in the order of 5µm

2) Image analysis is a powerful tool that allows quantification of many cracks parameters in CFRP subjected to thermal cycling, such as: geometry, size, position, clustering, percolation or interconnectivity, evolution (4D), etc.

3) The methodology of image analysis can be extrapolated to study other types of solicitations (sequentially ex-situ or in-situ), such as mechanical or thermomechanical.

4) The 3D distribution of cracks and its evolution with thermal cycle can be correlated with the permeability evolution in the material.

5) Because of its relatively high resolution, XCT can be used as a validation technique (in 3D and 4D) for the correlation of other types of signals that provide complementary information: for example from Acoustic Emission (AE), Ultrasounds (US), thermography, etc. through the use of e.g. artificial intelligence.



We are working on this…

Multiscale characterization of materials and processes: IMDEA's capabilities

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