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Innovative polymer-based composite systems
for high-efficient energy scavenging and storage

Training on (X-Ray) Micro (μ)-CT for Non-destructive Inspection of Structural Failure of Flexible Supercapacitor

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Training Objectives

After this training, the learner shall understand

- General working principle, advantages and limitations of μ-CT.
- When μ-CT can be used?
- How to use μ-CT for researching flexible supercapacitor?
- Where μ-CT can be used?

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Table of Contents

1. X-Ray µ-CT (Basics)
2. X-Ray µ-CT (Equipment)
3. µ-CT for Supercapacitor Failure Research
4. X-Ray µ-CT (Imaging)
5. µ-CT Application Cases

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1. X-Ray µ-CT (Basics)

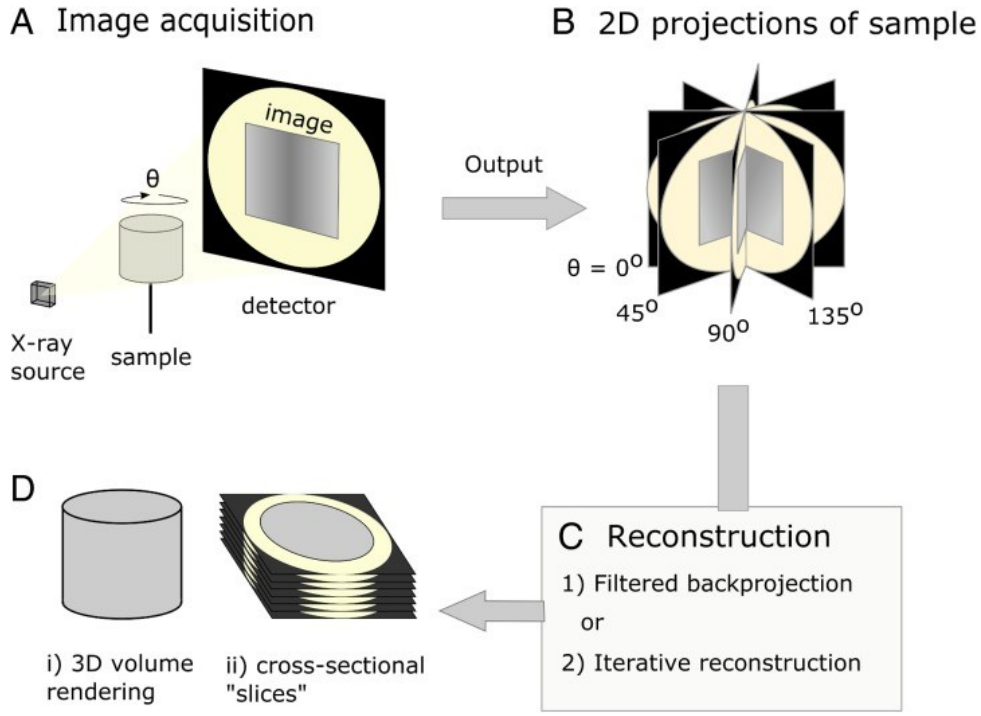
History

First in-vivo MicroCT system in the world in 2002.
Still new, mainly used in biomedical fields.
There is potential to use it more for wider fields.

Working Principle

It is an X-ray transmission image technique.
It works with the help of a micro-focus X-ray source that illuminates the sample. By rotating the sample, several views can be acquired from different angles. These multiple angular images are reconstructed to create a high-resolution 3D image of the structure.

It calculates its internal 3D structure based on the **density** distribution of the material.



Schematic illustration of X-ray CT acquisition and reconstruction processes.

J.D.B. Sullivan *et. al.*, *Parasitology* **2018**,145(7):848-854.
E. N. Landis and D. T. Keane, X-ray microtomography, *Materials Characterization* **2010**, 61(12): 1305-1316.
G.L.J. Paterson, *et. al.*, *Memoirs of Museum Victoria* **2014**, 71: 237-246.

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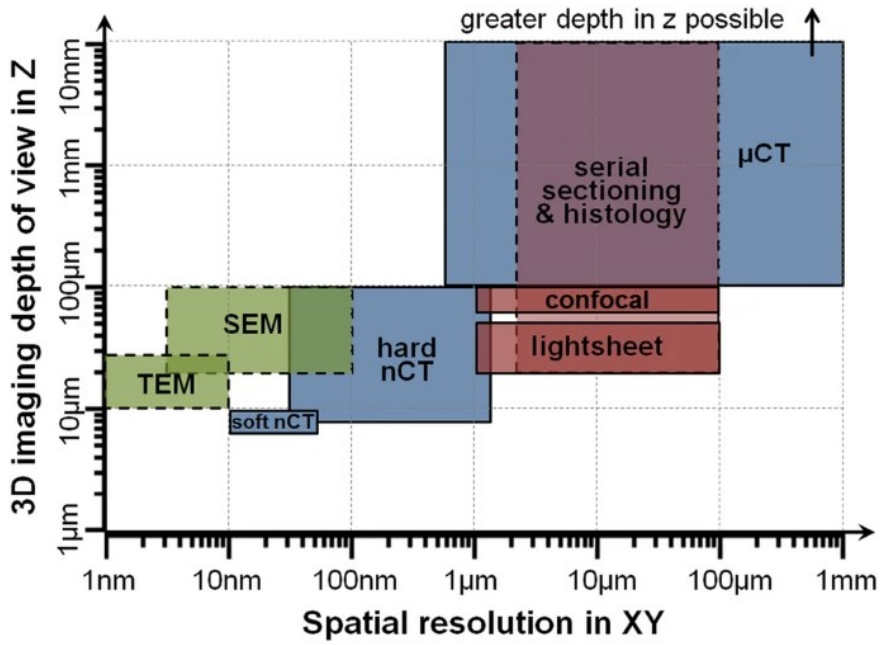
1. X-Ray µ-CT (Basics)

Advantages

- Non-destructive to the sample
- No need for supercapacitor sample preparation (sample preparation is needed for e.g. biological samples)
- High resolution imaging: 1–100 µm
- Can inspect anywhere, any layer of the sample
- High resolution images
- Rapid technique that gives results within 40 min to 12 h
- Less cost than SEM

Disadvantages

- Use of radiation which can be harmful to animals at high dosages
- Stains are unavailable for some type of tissues
- Storage and retrieval of large number of files
- Requires good IT infrastructure and data pipelines
- Not suitable for distinguishing similar types of tissues



S.D. Rawson, *et al.* X-ray computed tomography in life sciences. *BMC Biol* **18**, 21 (2020).

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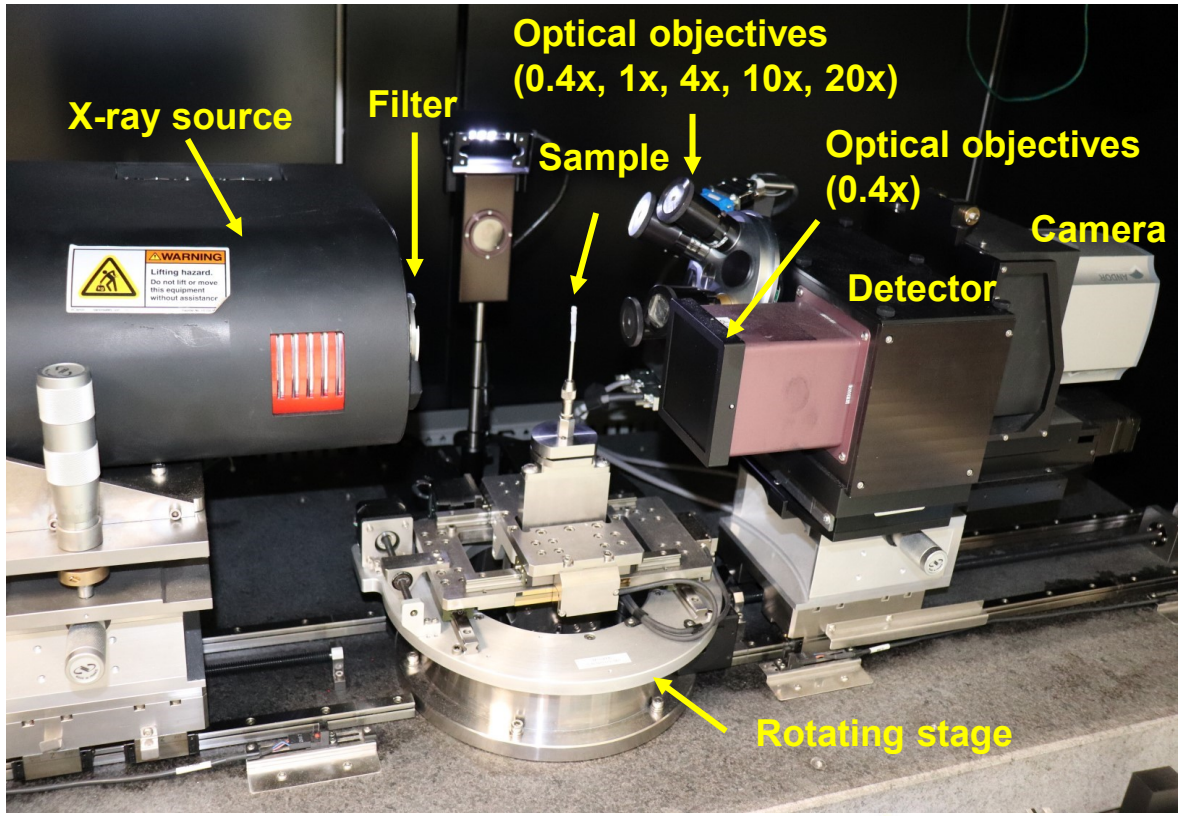
2. X-Ray µ-CT (Equipment)

- spatial resolution < 1 µm
- resolution varies depending on the field of view: 1-70 µm
- maximum sample size: 20 cm
- maximum sample weight: 15 kg
- field of view: 5 cm



Xradia MicroXCT-400 in Computational Biophysics and Imaging Group (CBIG), Tampere University (Kauppi campus)
For details about micro-CT use, please contact Markus Hannula (markus.Hannula@tuni.fi)

https://www.tuni.fi/en/research/x-ray-microtomography#switcher-trigger-services

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3. μ-CT for Supercapacitor Failure Research

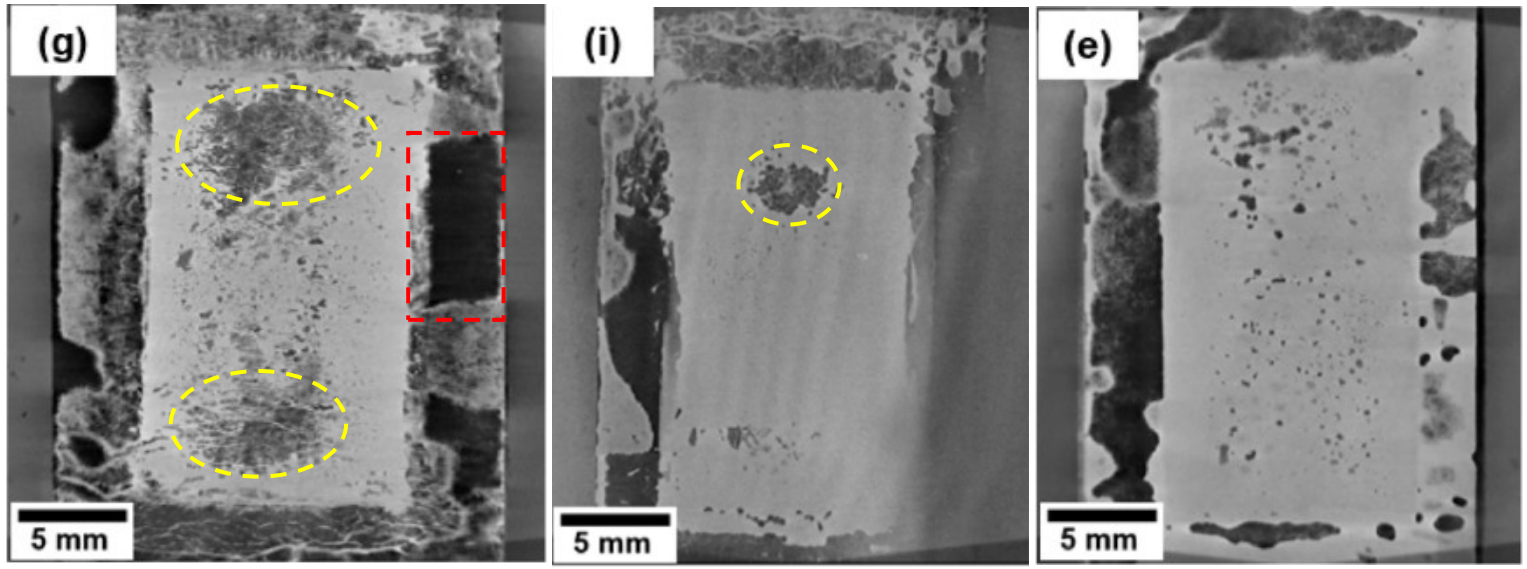
Principle: in X-ray μ-CT imaging: darker areas indicate lower density of the material.

Step 1: imaging the whole sample, showing the relatively darker areas (marked in Fig.g).
In comparison, SC in Fig.i has smaller area with lower density; reference sample does not show concentrated area with lower density

Super large dark areas are due to the lower thickness of stacked SC components, rather than failures.

By now, we can close to identifying **failure location**, but:

in which layer the failure is?



Tested SCs

Reference – not tested SC

Z. Fu *el al.*, *ACS Appl. Mater. Interfaces* 2022, 14, 35, 40145–40157

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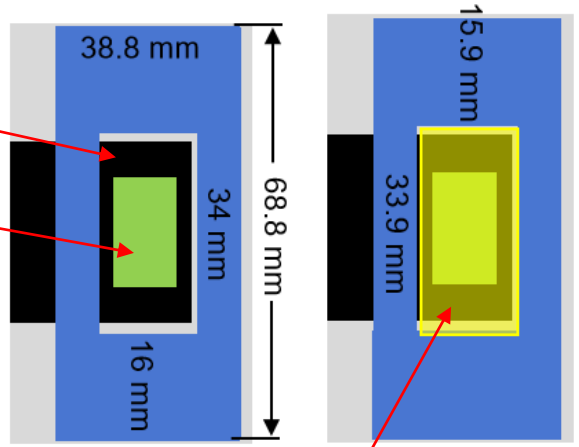
3. μ-CT for Supercapacitor Failure Research

Step 2: measure the different components in image, compare with the component sizes in sample design, to identify the components.

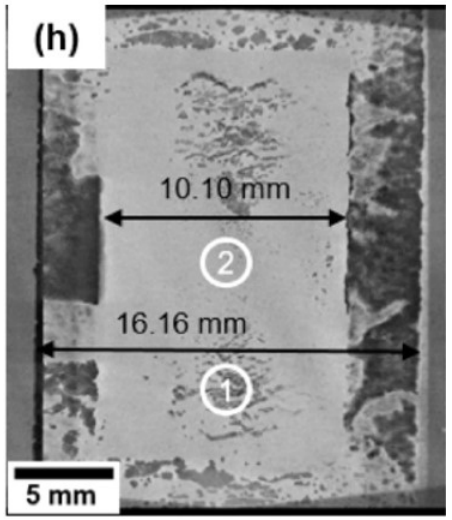
Current collector:
30 mm x 30 mm

Electrode:
18 mm x 10 mm



Separator:
15.9 mm x 33.9 mm

Dimensions of supercapacitor componnts in design



The 10.10 mm wide component is electrode.

The 16.16 mm wide component is separator (cellulose paper)

However, separator, electrode, current collector are stacked.
The lowered density of stacked material can be caused by the loss of material any of them, and/or the interface.

So, where the failure exactly? And what failure?

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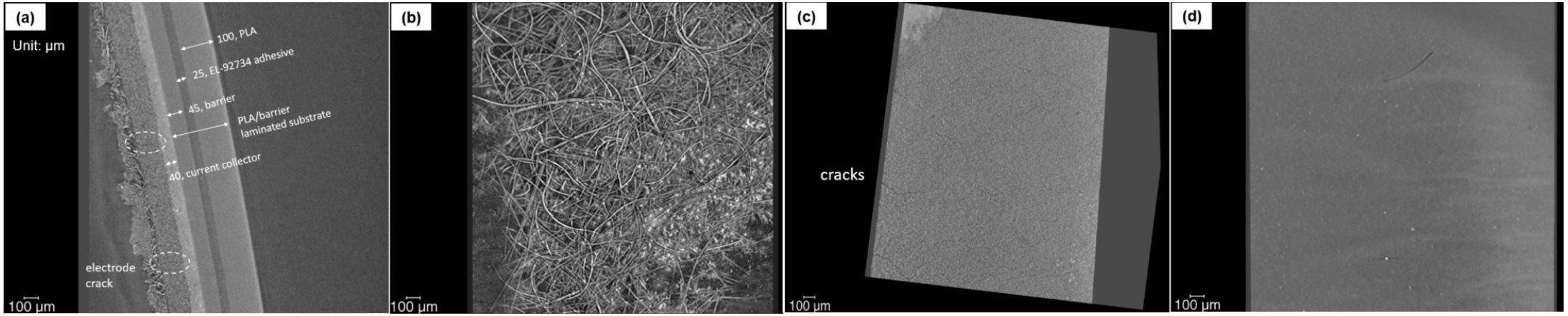
3. μ-CT for Supercapacitor Failure Research

Step 3: to check side view of stacked components to check interface failure, and check each layer of the supercapacitor, to define the exact location and mechanism of failure.



No interface failure

Fiber structure of **separator** is not damaged.

Electrode: crack failure

Current collector: no failure

Result: there is no structural failure in separator and current collector, no interface failure, only electrode crack failure

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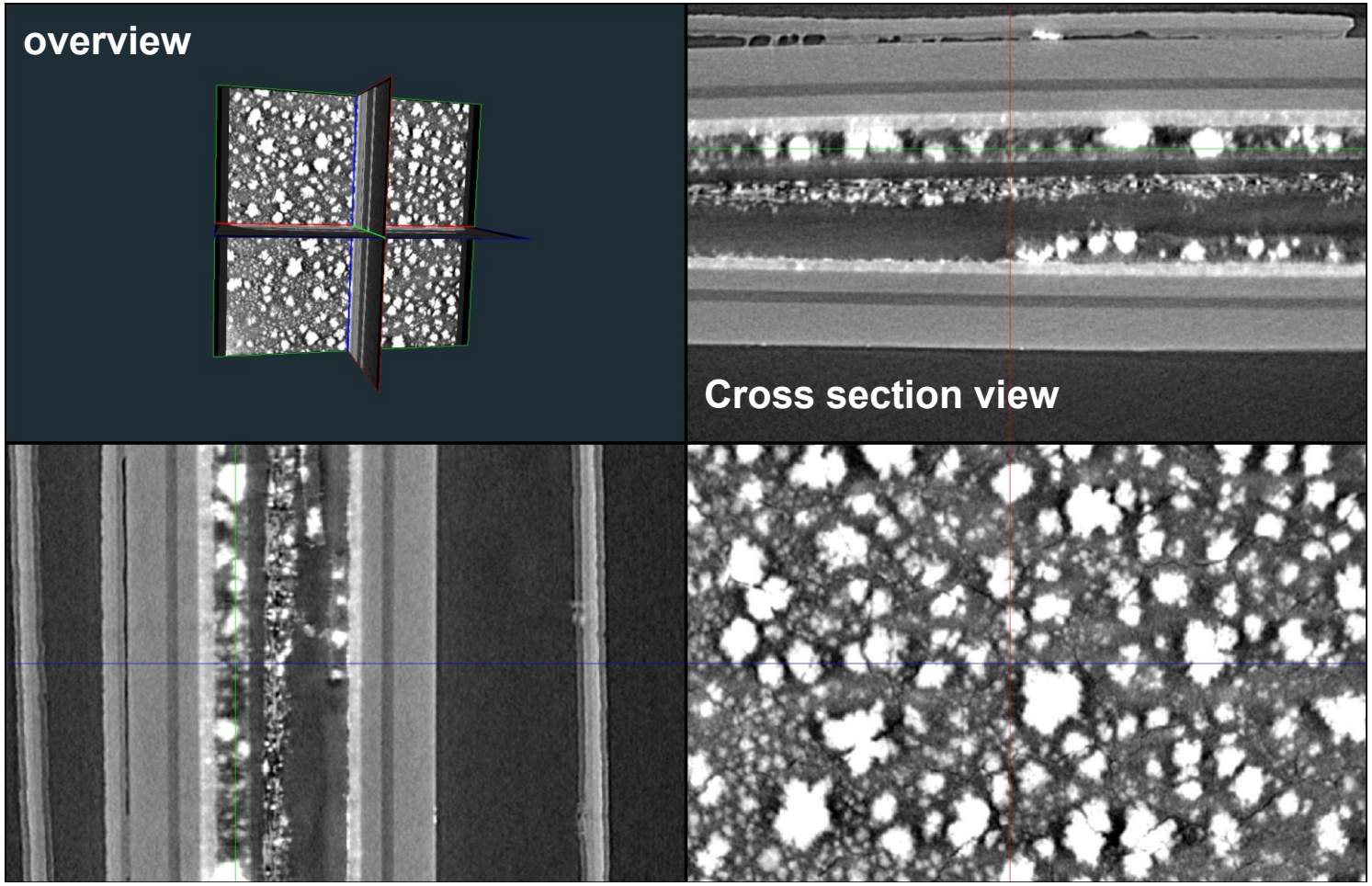
4. X-Ray μ-CT (Imaging)

It enables to observe each layer of the sample from whatever angle!

From side view (cross section view), the material stacking issue and material structural failure can be detected.

Then, one can choose the interested layer to check its top/front view to find the detailed failure.



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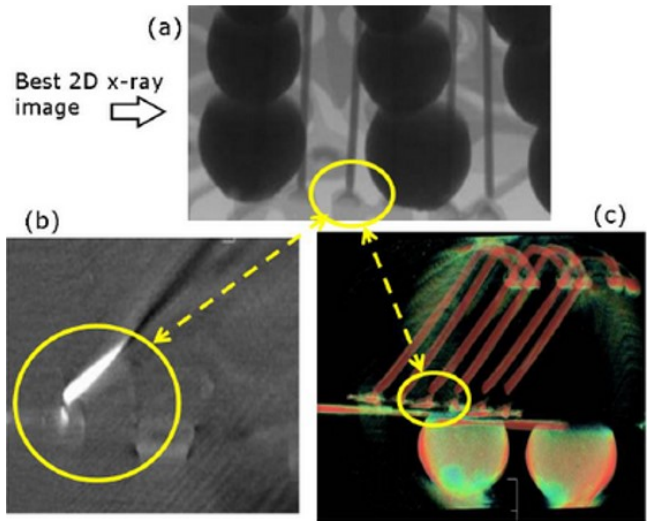
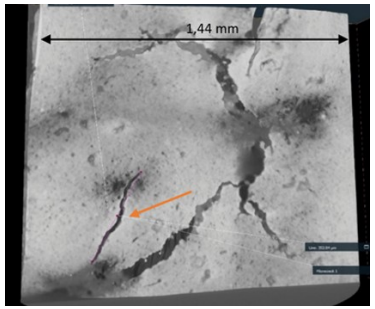
5. µ-CT Application Cases

Structure failure

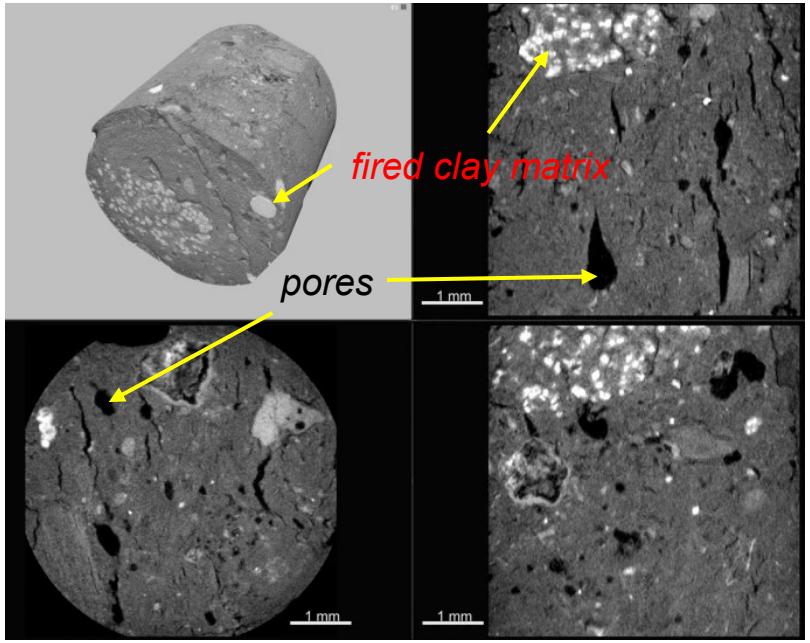


Wire breakage in microelectronics



Crack in painting

Materials porosity

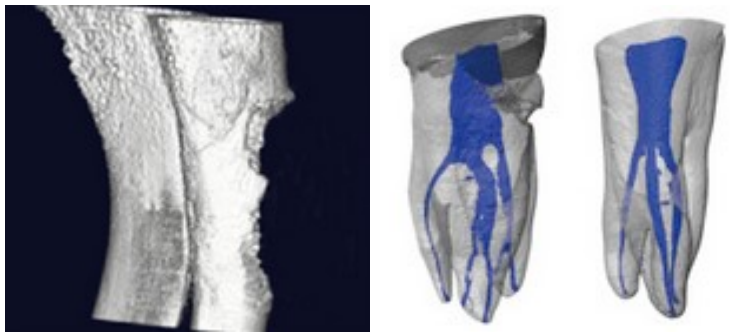


C.L. Reedy, C.L.Reedy. High-resolution micro-CT with 3D image analysis for porosity characterization of historic bricks. *Herit Sci* **2022**. 10, 83.

M. Pacheco and D. Goyal, "*X-ray computed tomography for non-destructive failure analysis in microelectronics*," 2010 IEEE International Reliability Physics Symposium, Anaheim, CA, USA, 2010, pp. 252-258.

K. Præstholm. (30.3.2022). Micro-CT scanning for verification of microcracks in paintings, available: https://kglakademi.dk/blog-collection-care/micro-ct-scanning-verification-microcracks-paintings

Biological applications



human bone root canal

C. Shuai, *et. al.*, *Mater. Des.* **2019**, 185:108275.
A. Kato, *et. al.*, *J. Oral Biosci.* **2016**, 58(3): 100-111.

Great potential for failure analysis of printed electronics, as printed electronics always involves stacking of materials and interface, which may involve failures.

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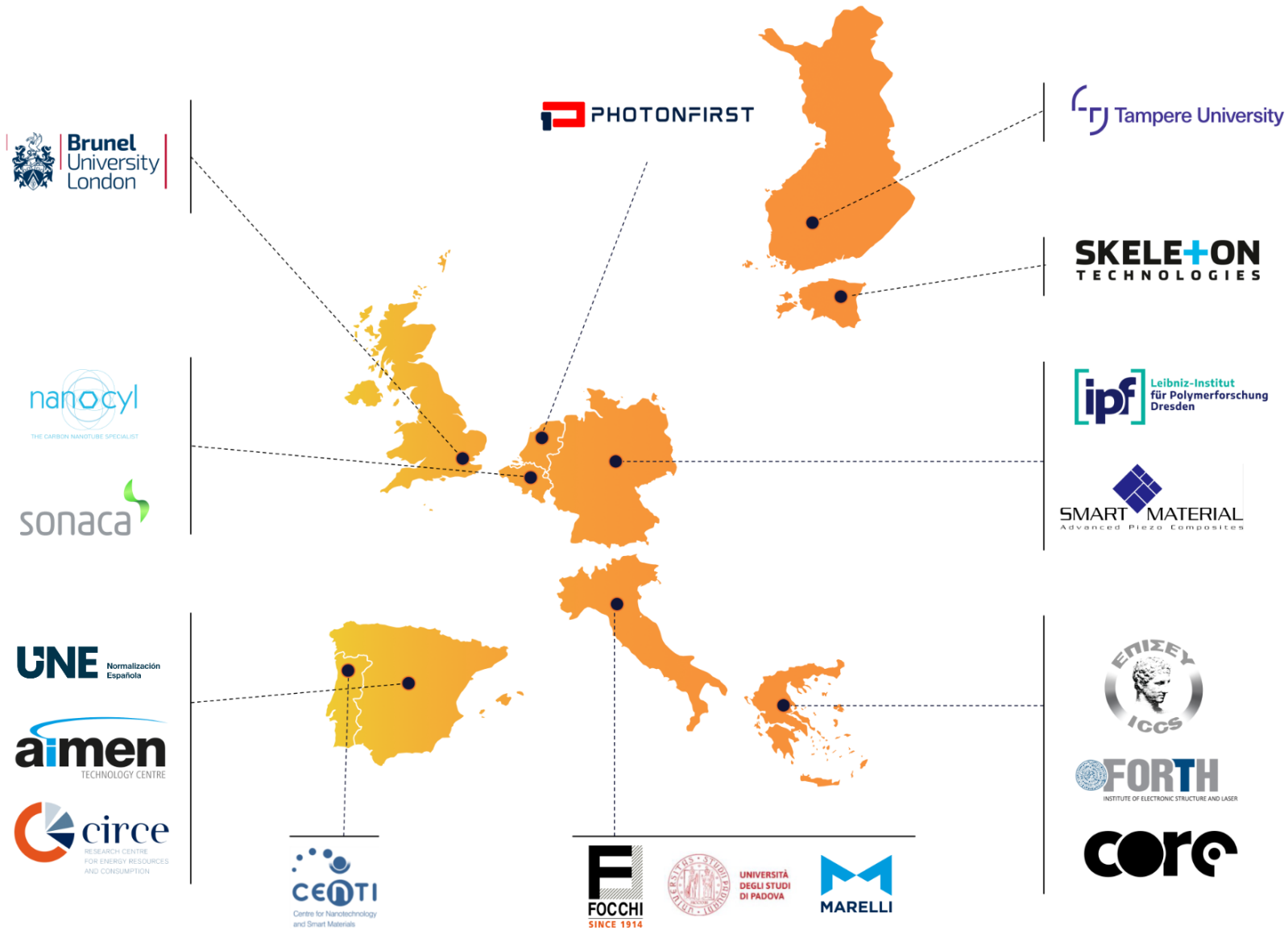
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Thank you

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State of material:
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