Aalto University
School of Chemical
Engineering

Materials research using XCT at Otaniemi

XCT Launch meeting 9.11.2017 GTK

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Case studies

Information on the opportunity of the XCT was distributed within Aalto research groups

Performance of the instrument was explored using cases raising from Aalto CHEM research during last summer

Case study 1: 3D printed poly trimethyl carbonate scaffold

Case study 2: Water–ice system in freeze crystallization

Some other important potential XCT applications are identified

Case study 1:
3D printed PTMC scaffold

- Poly (trimethylene carbonate) scaffolds are used in tissue engineering
- Patient-specific 3D-printed bio-gradable implants are targeted
- The materials are often biofunctionalized; for example, in orthopedics by mixing nano-hydroxyapatite in the resin to make the implants osteo-promotive
- However, variations in material composition may not only change the implant's biological properties, but can also influence its manufacturing processing

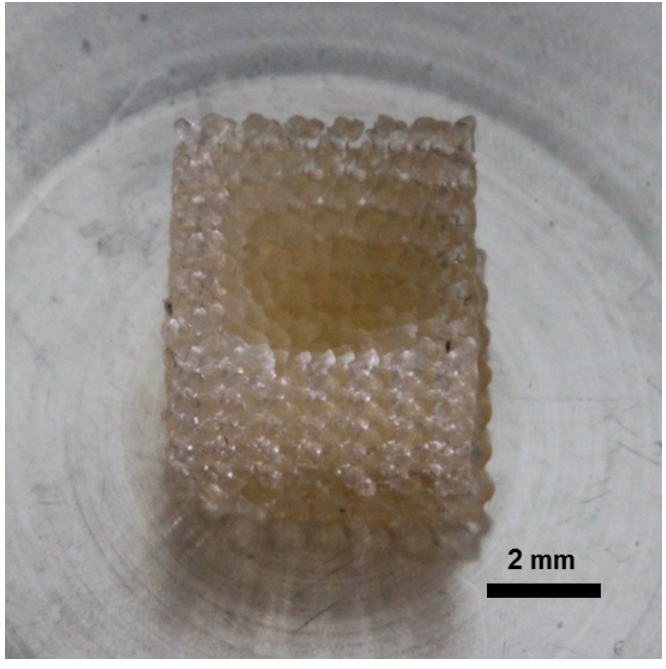
Case study 1: The sample

Sample from
prof. Jukka Seppälä's group

m =0,0601 g

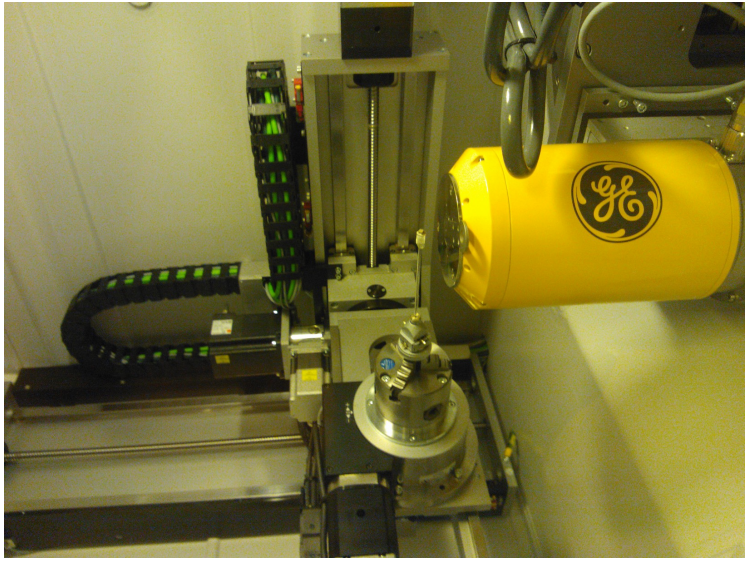
ρ = 1,2 g/cm^3

=> V = 50 mm^3

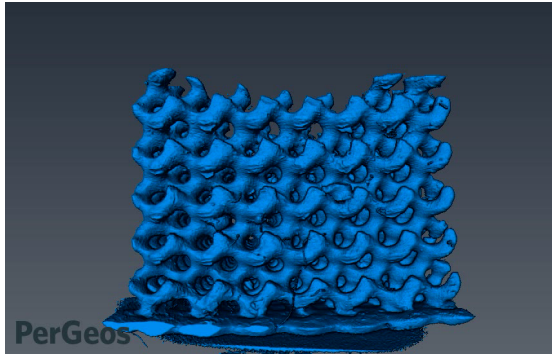


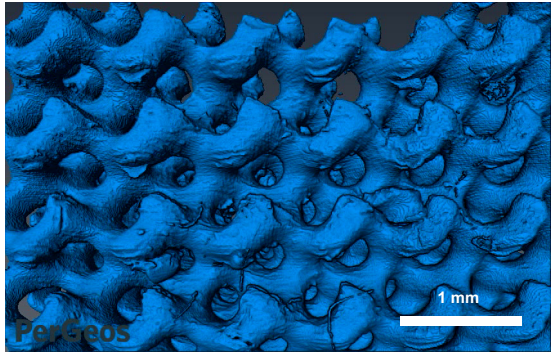
Case study 1: X-ray measurements

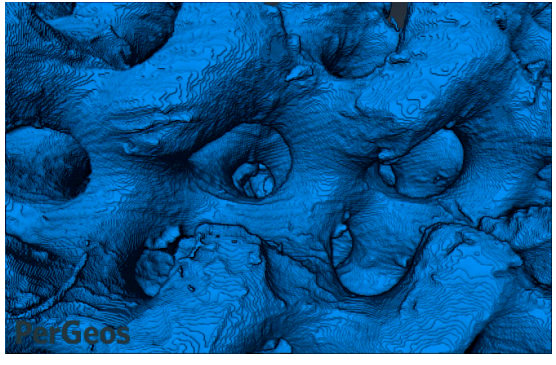
- Voltage 35 kV
- Current 500 μA
- Images 2100
- Total time > 7 hours

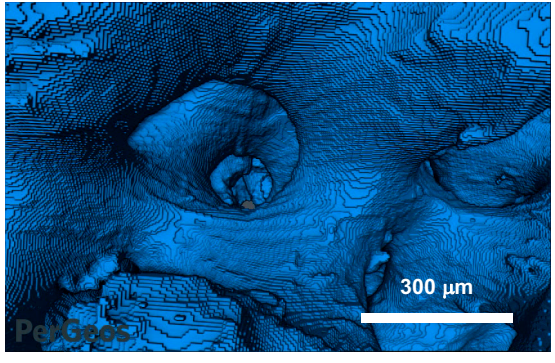


Case study 1: 3D reconstruction



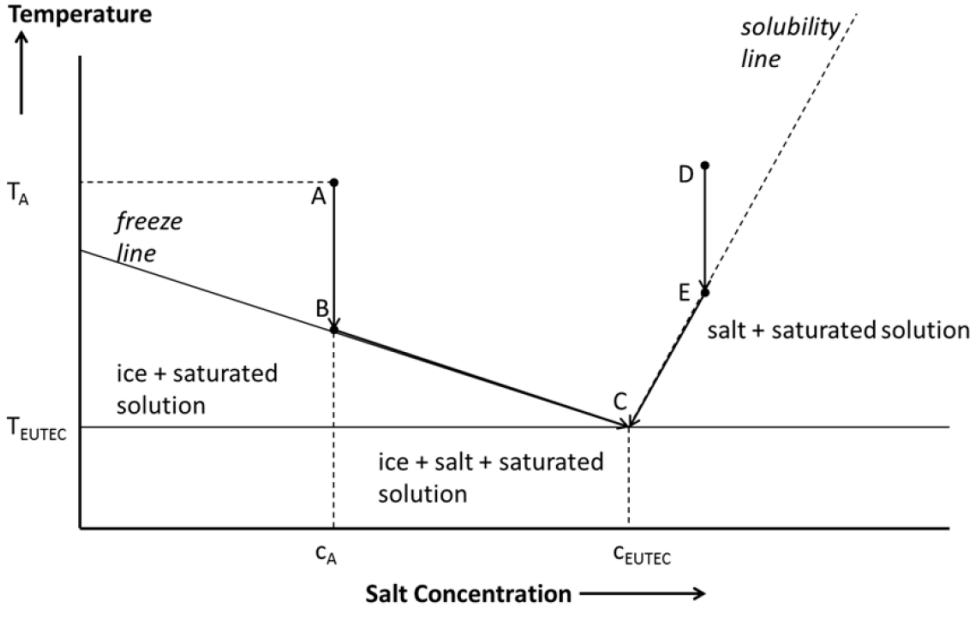






Case study 2: Water ice system

- **Freeze crystallization is a purification process that is based on solubility differences between solid and liquid phases**
- **Prof. Marjatta Louhi-Kultanen**



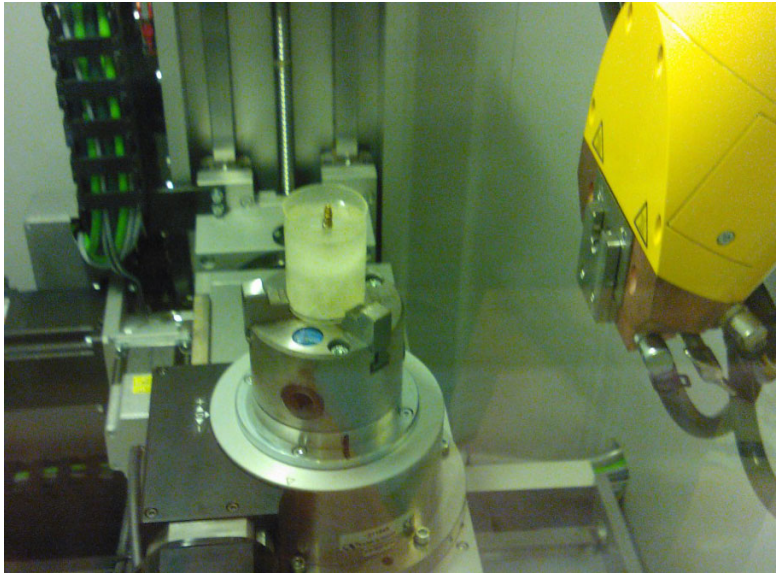
Case study 2: Experimental test setup

- Plastic cup
- Ice and water on the top
- Brass screw in the middle

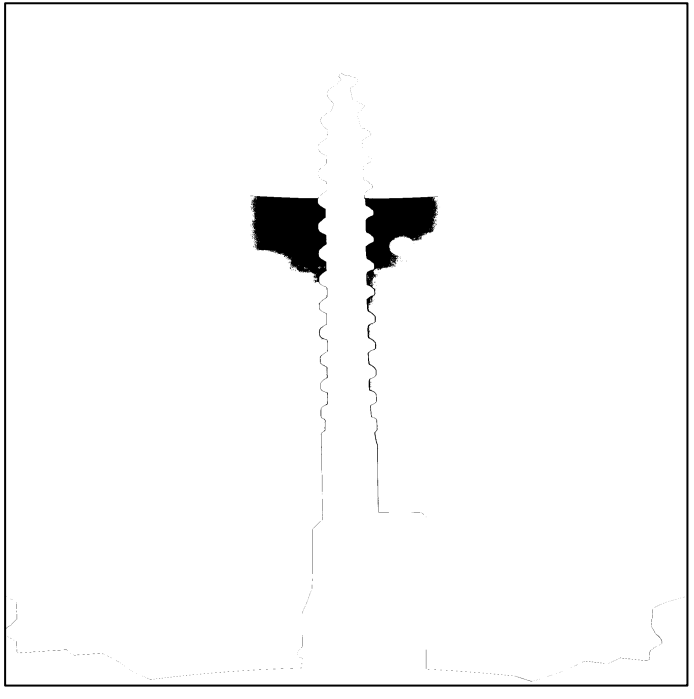
- Room temperature ->
  Melting over the time

Case study 2: X-ray measurements

- Voltage 50 kV
- Current 500 μA
- Images 100
- Exposure 131 ms
- total time 13 s



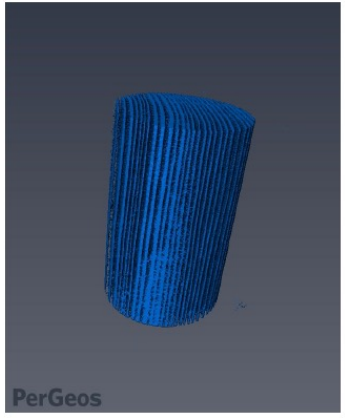
Case study 2: Segmentation of a single radiograph



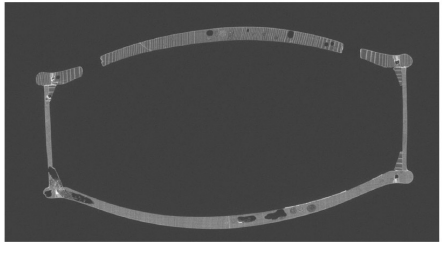
Expected materials science applications

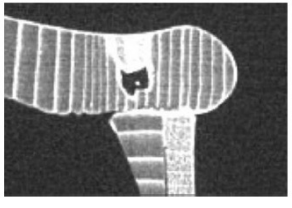
- Studies of wood and wood composites
- Battery studies
- Studies of metal-slag systems
- Atomic layer deposition of nanolaminates in composites

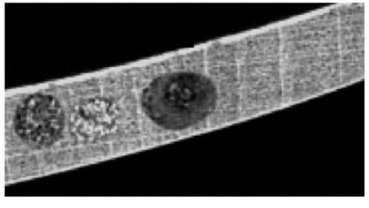
Studies of wood and wood composites



- Failure modes of (bio)fiber reinforced composites in compression and tension
- Structural failure of wood at different length scales
- Layer adhesion in engineered wood products
- Fluid flow in porous wood structures
- Occurrence of (microstructural) damage in “old” wood in terms of its structural reliability.







N. Sodini et al., Journal of Cultural Heritage 27S (2017) S88–S92

Battery studies

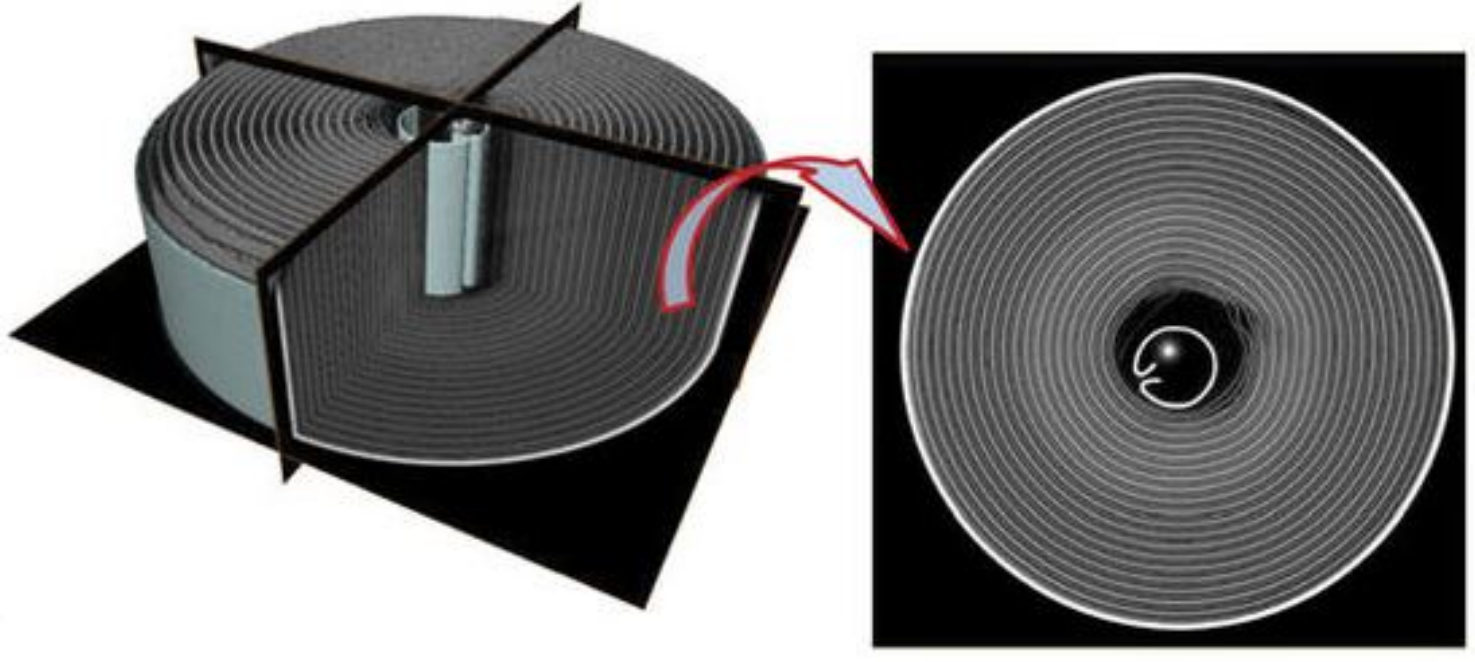
Advantages of XCT

Enables non-destructive testing : Same sample can be analysed using different imaging modes number of times.

Facilitates internal imaging in 3D: Internal structure of opaque materials can be images easily in a three-dimensional space

Vacuum is NOT required: Allows in-situ and in-operando imaging of large samples of anisotropic materials in ambient and controlled environments

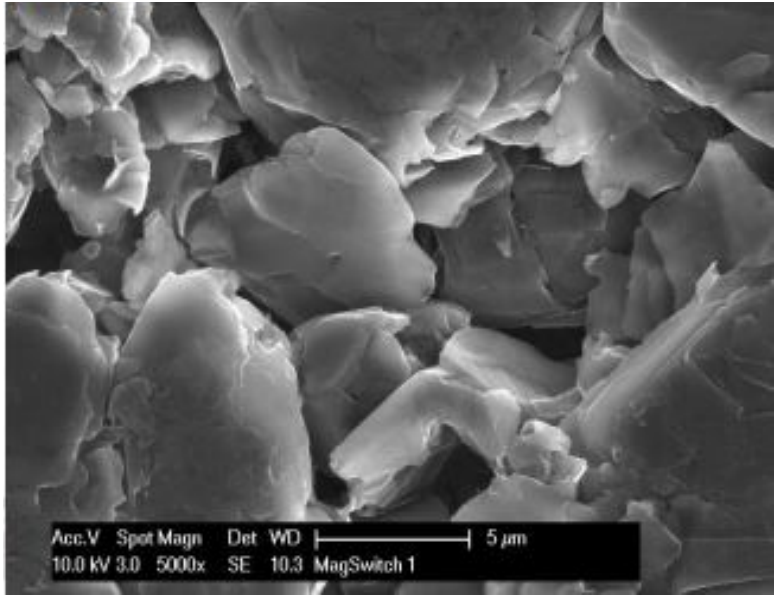
3D reconstruction with orthoslices in the *XY*, *YZ* and *XZ* planes of a battery cell with isolated *XY* slice



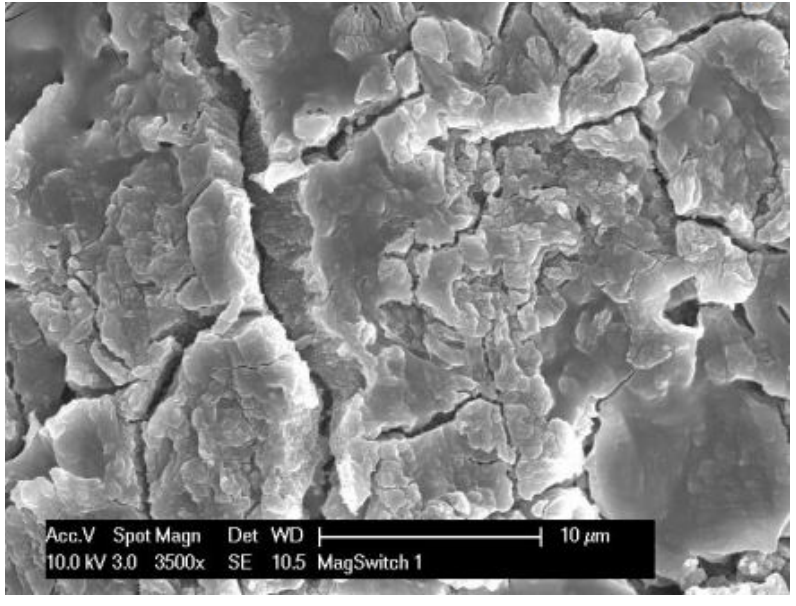
Source: Finegan et al, Nature Communications, 2015

Estimating battery ’end-of-life’ using XCT

Solid electrolyte interphase growth and morphology change visible at the end of life



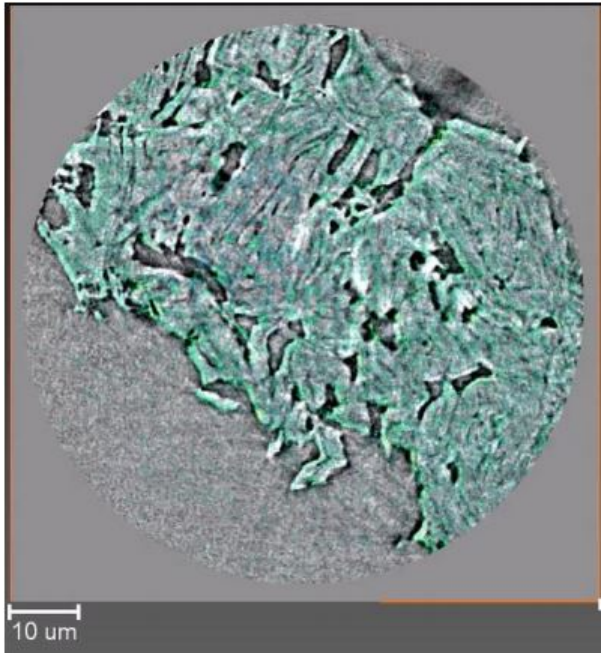
At the ’Beginning’ of Life



Near ’End’ of Life

Source: http://muri.materials.cmu.edu/wp-content/uploads/2015/06/Litster-3D_Summer_School.pdf

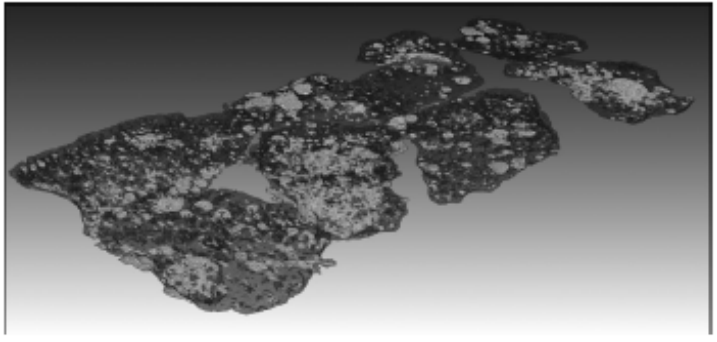
Absorption contrast imaging for inorganic SEI signal



Excessive solid electrolyte interphase film coating and blocked internal pores visible through XCT images for a porous electrode

Source: http://muri.materials.cmu.edu/wp-content/uploads/2015/06/Litster-3D_Summer_School.pdf

Studies of metal-slag systems

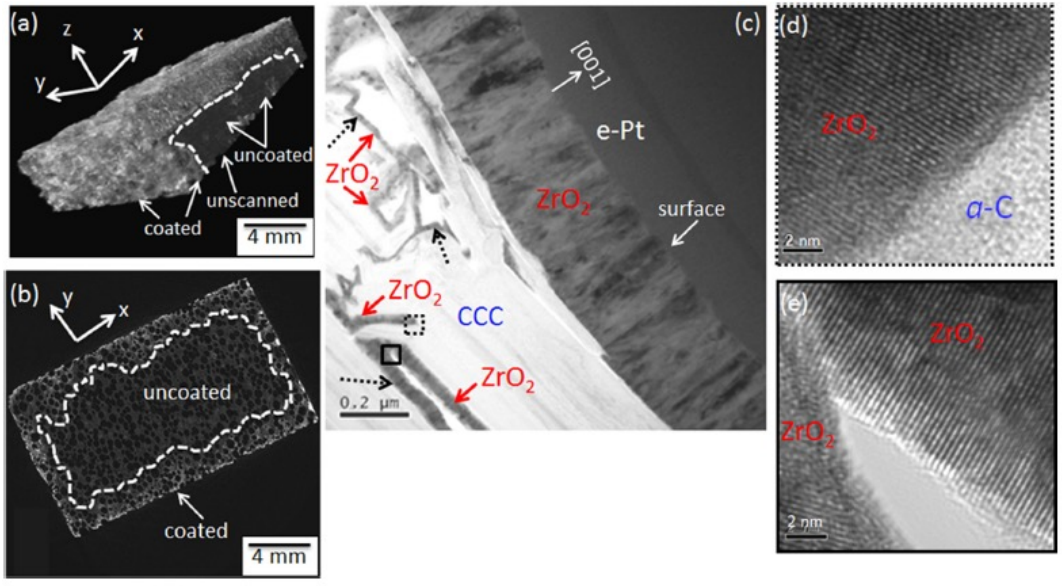


3D segmentation with semi-transparent slag phase in order to see the dispersion of metal droplets (light grey).

S. Spooner et al., 2016 ISIJ

- **Phase distribution**
- **Structure**
- **Time evolution**

Example: Atomic layer deposition of $ZnO_2/Al_2O_3/ZrO_2$ nanolaminates for improved thermal and wear resistance in carbon-carbon composites



H. Mohseni et al., J. Vac. Sci. Technol. A 30(1), Jan/Feb 2012

Concluding remarks

- Several potential research fields were identifield
- Aalto has obtained initial experience on XCT to enable new groups to apply the technology as part of RAMI infrastructure
- Future XCT use is based on Aalto research group activities