Novel characterization techniques in Metallurgy

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Introduction

• Characterization in metallurgy:
  – Mechanical
  – Microstructural
    – Physical, chemical...

• What can be regarded as “novel” in the world of characterization techniques?

• Mechanical:
  – Indentation, local measurements, simulation

• Microstructural
  – Progress in microscopy, fields measurement
  – 3D imaging: novel, potential development
Why is 3D imaging useful for metallurgy:

- Microstructure can have a very complex nature
- Standard procedure = 2D + stereology
- Some cases: not enough
- 3D images to
  - Qualify (to make nice renderings)
  - Quantify
    - Fraction, Size, Shape
    - Connectivity
    - Percolation, coalescence
- Some are non destructive: evolution during loading (in situ)
  - A lot of new infos for mechanical, physical and chemical metallurgy
Little quizz to convince you further

How many wires?  How many pores?
The answer can only be given by the 3D image:

Only one wire

Fully connected porosity

(Martin et al. Scripta Mat 2000)
Outline

1) A general panorama of 3D imaging methods
   1) Destructive (serial sectioning, FIB, APT)
   2) Non destructive (electron, Neutron, X ray Computed tomography)

2) X Ray CT
   1) In situ experiments
   2) XRCT in metallurgy, Modeling based on the microstructure
   3) Digital Volume Correlation
   4) Future of XRCT
3D imaging methods

Destructive methods

Successive abrasion of layers
- mechanical polishing
- ion milling

+ Imaging
ex situ: OM, SEM
or in situ: SEM, ion, atomic probing

Selective dissolution

Non destructive methods

Transmission + rotation:
electrons or X rays

Computed reconstruction

Reflexion, emission or scattering
Acoustic waves, magnetic field, X rays
1. Selective dissolution, deep etching

Destructive methods

Successive abrasion of layers
- mechanical polishing
- ion milling

+ Imaging
ex situ: OM, SEM
or in situ: SEM, ion, atomic probing

Selective dissolution
2. Serial sectioning

2.1 Mechanical polishing

2.2 Ion milling
Existing devices to do mechanical polishing for serial sectioning automatically

- Serial sectioning + optical microscopy
Example: ferrite martensite + cavities

DP 11%

Fracture surface
~80 μm

PhD C Landron
Collaboration
TU Vienna
Ion milling

- Can be done like serial sectioning
- New devices: dual beam
  - Ion milling
  - SEM imaging
    - EDX
    - SEI, BEI
    - EBSD
Duplex stainless steel
JY Maetz, S Cazottes, C Verdu, X Kléber

Images: FIB ZEISS du CLYM
Yttria stabilized zirconia results

Reconstruction and smoothing by Sukbin Lee
Lee, Dillon, Rollett, Rohrer, Microscopy and Microanalysis 2007
Ion milling : Tomographic Atom Probe
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Non destructive methods for 3D imaging
Scanning acoustic microscope
Confocal microscopy of transparent objects

- Send optical light
- If the object is transparent, images of different focal planes can be sequentially obtained (contrast = colour)
- Used a lot for colloids
- Food industry (ice cream)
- Biology samples
Nuclear Magnetic Resonance applied to materials

Not many published studies
Transmission / Rotation Techniques

Non-destructive methods

Transmission + rotation: electrons or X rays

Computed reconstruction

Reflection, emission or scattering
Acoustic waves, magnetic field, X rays
In radiography you record the sum of $\mu$. Ideally one would like to decompose $\mu$ along the path.

**Tomography by projection/rotation** (+reconstruction)
Tomography

- Combines the information of MANY radiographs to reconstruct the 3D map of \( \mu \)

View from the top:
Tomography

- Combines the information of MANY radiographs to reconstruct the 3D map of $\mu$

View from the top:
There is a COMPUTED step (reconstruction)

The result is a 2D map of $\mu$
Electrons

- Transmission electron Microscopy
- Some problems:
  - Absorption
    - Thin samples
  - Diffraction
    - HAADF
    - Energy loss imaging
  - Missing angles
- Cf P. Midgley, T. Epicier
Neutrons

- Penetrate interact and are not absorbed
- Imaging: radio/tomo
- Low flux of the sources
- Low resolution, detectors not so much developed
- Very useful for highly absorbent low resolution
X ray imaging

- Limited in terms of resolution (less than SEM although 20 nm is the upcoming target)
- But interesting because non destructive bulk information
  - Transmission of X-rays THROUGH opaque materials

- Access to information IN THE BULK
- Of particular interest for
  - Damage studies (because damage is different at the surface)
  - Architected materials materials
X ray tomography in metallurgy

• Non destructive
• 3D
• “Easy” to access
  – Lab tomographs
  – Synchrotron beam lines
• 16 years of background
In situ experiments in X-Ray tomography

- Restricted to non-destructive methods
- Best way to understand the microscopic mechanism involved during your
  - Deformation
  - Transformation
- Apply a stimulus to your material *in situ* (in the tomograph)
  - Mechanical, thermal, electrical...
- + Analyze the change in the microstructure of a similar region of your material
1. Deformation
In situ testing

- Tension, compression

- Stepping motor
- Reductor
- F and disp recorded
- $10^{-5}$ – 1 mm/s
- Several Force sensors: 50 – 5000 N
- Grips adapted for different geometries

Buffière et al Exp Mech 2009
A lot achieved so far in the interrupted mode (15 years at the ESRF)

- Al/SiC
- TiSiC
- Al alloys
- Polymers, Composites
- Steels
  - DP
  - Trip
  - TWIP
- Co, Cu, Ti

- Model materials
  - Cu sheets
    - Mc Master University
- Industrial
  - Al alloy
    - 5xxx
CoCr Biomedical material
For a lab tomograph:
Hydrostatic compression

For the collapse of polymer foams in sea water
J. Lachambre, D. Choqueuse

(collaboration IFREMER)
Indentation

- PhD P Clément, S Meille
Double torsion INSA

« Stable » crack propagation Ceramic materials
Faster (fatigue device)

- 50 Hz
- Tension only
- We have done ex situ compression of metal hollow spheres too
- JY Buffière

- Cracks initiate at the pore/surface intersection
2. Temperature
Cooling
Cryostat INSA

Reduced diameter of the tube to fit to the lab tomograph + computed control of the temperature
Heating

- The problem of missing views vanishes
- Different technologies for heating
  - Lamps
  - Induction
  - Standard resistors
- The sample rotates in the furnace which is fix and equipped with windows for the X rays
Talk by Michel Suery
In situ solidification of an Al-Cu alloy

ESRF
ID 15
Scan 15 s
res ~ 3 μm

L. Salvo (INPG)
M. Di Michiel (ESRF)
3. Both Temperature + Deformation
Tension test in the semi solid state

Talk by Michel Suery et al. Cavitation in liquid films
Add ons to the technique

Microstructure calculation
Strain fields measurements
Fe/TiB2 composite (arcelormittal)
Local tomography for multiscale analysis

Highly loaded but not broken
No intermets
PhD Tao Zhang / Luc Salvo
A supplementary tool for the analysis: Digital volume correlation

Strain maps local DVC: M Bornert

- No deformation bands for these foams (EPFL)
Incremental strain fields

Strain maps Global DVC: F. Hild S Roux

Compression axis Z
Prospects
Improve spatial resolution using KB mirrors

Use a conical beam on synchrotron to magnify

Spot size down to 20 nm
Damage in Fe TiB$_2$ composites during tension (ex situ observation)

Voxel size = 50 nm !!!
Temporal resolution

Using ID15 beamline (pink beam = high flux)
Resolution 1.5 μm
See previous movies
Drastic reduction of the exposure time!
Time for one scan:

5 minutes

20 seconds

0.16 second: tensile test with $d\varepsilon/dt=10^{-3} \text{ s}^{-1}$

500 microns

63/75
DCT on a Ti alloy. PhD M Herbig
JY Buffière – W Ludwig

Metastable β-titanium alloy
‘Timet®21S’
Chemical composition:
15 wt% Mo, 3 wt% Nb
1008 grains
Laminography

- For large objects (sheets)
- T Morgeneyer CDM
Challenges

- A bit of incremental development to bring the latest developments to routine
- Ready for metallurgy
- Spread the info
- Have access to beam time
- A “metallurgy” beamline
  - High energy
  - Diffraction + imaging
  - Hermès Equipex project (on ID15)
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