IN-SITU STRAIN MEASUREMENTS

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When stretching/compressing a material, competition between:

- Orientation mechanisms
- Damage mechanisms

Orientation should dominate to ensure a high durability

→ real-time identification of these mechanisms as a function of temperature, strain rate, formulation, processing…..
PRINCIPLES

• Load range: 0N - 5000 N (precision +/- 1 N)
• Minimum specimen length: 20 mm
• Maximum specimen width: 10 mm
• Strain rate ranges: + 0.1 µm/s to +20 µm/s
  - 0.1 µm/s to -20 µm/s
• Temperature ranges: room temp. up to 800° (vacuum)
  or room temp. to 300° (ambient)
• Maximum displacement: +/- 6 mm (symmetrical)
• Stage dimensions: 220 mm x 150 mm x 55 mm
• Weight: 2.4 kg
• Analysis in transmission is possible (e.g. on XRD)
• The miniature tensile stage provides:
  • the monotonous tensile or compressive behaviour
  • the cyclic tensile/compressive behaviour

• of the following materials:
  • metals
  • ceramics
  • plastics
  • glass ceramics or galvanic surface coatings
  • soldering and welding interfaces
  • minerals
  • wood and organic materials

• Advantage

• The area of interest does not move (symmetric displacement of the two crossheads)
Coupling of a miniature tensile machine with characterisation tools for real-time in-situ investigations

PRINCIPLES

Miniature tensile stage

SAXS/WAXS Lab (pseudo-real time)

SAXS/WAXS synchrotron (real-time)

→ gain information about objects scattering x-rays with sizes ranging from 0.1 to 100 nm (chains, crystals, voids, nanoparticles....)
Coupling of a miniature tensile machine with characterisation tools for real-time in-situ investigations

**Miniature tensile stage**

**SEM Lab (real time)**

→ Imaging of sub-micron to micron objects (crystals and voids) as well as imaging of surface topography changes
EXAMPLE 1

Damage kinetics in HDPE by SAXS

Lamellar morphology of HDPE

Time-resolved SAXS pattern (synchrotron)

Lamella scattering

Additional scattering due to voids
EXAMPLE 1

Damage kinetics in HDPE by SAXS

Scattering by lamellae
Onset of voiding
Void Development
Void Recovery
Void Recovery
EXAMPLE 1
Damage kinetics in HDPE by SAXS

VOID scatttering = overall scatting - lamella scatting

Lamella scatttering: Supposed to be a linear function of strain

Lamella scattering:

loading  unloading  recovery

INTEGRATED INTENSITY, (A.U. x pixel²)

0.0  2.0x10⁷  4.0x10⁷  6.0x10⁷  8.0x10⁷  1.0x10⁸  1.2x10⁸  1.4x10⁸

TIME, t (s)

0  200  400  600  800  1000  1200  1400  1600  1800  2000
EXAMPLE 1

Damage kinetics in HDPE by SAXS

→ Identification of damage kinetics during loading, stress unloading and strain recovery:
  → Void fraction was not stable during stress unloading and strain recovery

Addiego et al., Polym Int, 2015
EXAMPLE 2

Damage kinetics in HDPE by SEM

Surface spherulitic morphology of HDPE (in-situ testing)

→ No voids although SAXS indicated 5 vol.% of voids at a strain of 9 %!!
EXAMPLE 2

Damage kinetics in HDPE inside SEM

Core spherulitic morphology of HDPE (postmortem testing)

LFD mode: strain 9%

→ Voids at the core of material:
   → in-situ mechanical testing may not be relevant for some cases due to skin/core effect!

STEM mode: strain 9%

F. Addiego et al., Polym Int, 2015
EXAMPLE 3

Stretching of coated fabrics inside SEM

The coating withstands the fabric’s deformation (no damage)

Collab. with Bianca Rita Pistillo, unpublished results
EXAMPLE 4

In-situ materials testing: micro-tensile machine inside SEM

- The area of interest does not move (symmetric displacement of the two crossheads)
- Area of breakage can be followed easily

Visualization of sample damage as a function of stretching

Increase in elongation

Micro-tensile machine

100nm scale bars
Current Developments

- Mechanical testing at different temperatures (micro-tensile machine + SEM / XRD)
- Coupling of AFM and micro-tensile module
- Local strain measurements coupled with Digital Image Correlation system ARAMIS 3D (optical extensometer replacement)
- Coupling of Raman microscopy and micro-tensile module
Thank you for your attention!