Colloque National Mécamat *, Aussois, Janvier 2017*

Support of dissipated energy measurements for the fatigue design of short fibers composites and elastomers

Y. MARCO , *et al.*

Institut de Recherche Dupuy de Lôme

et al.!

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Bertrand HUNEAU, Habibou MAITOURNAM, Pierre CHARRIER , Ida RAOULT, Gilles ROBERT,

Antoine LAUNAY, Loïc JEGOU, Isaure MASQUELIER, Leonell SERRANO, Clément CHAMPY,

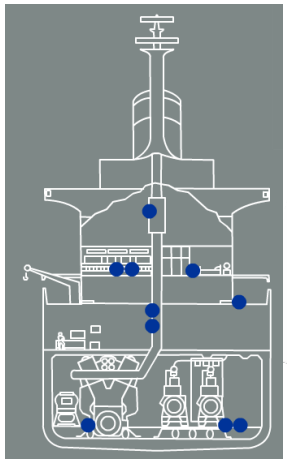
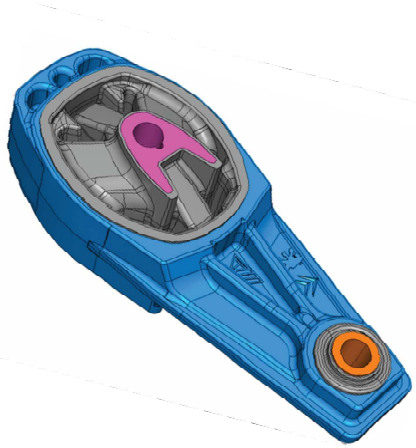
Louis LEVEUF, Thomas GLANOWSKI,

…

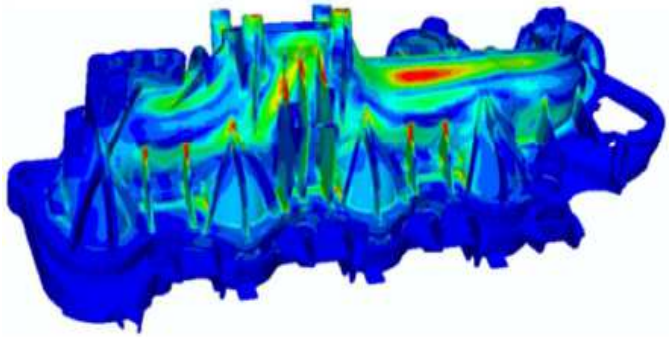
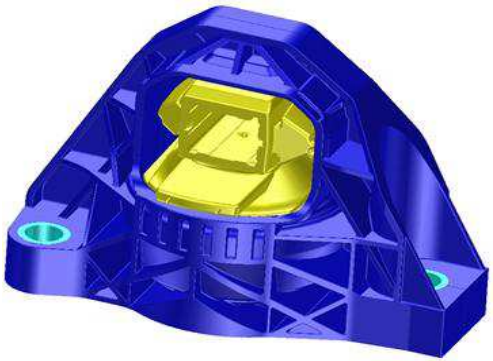
Introduction: Industrial and scientific contexts

DURABILITY : FATIGUE and AGEING

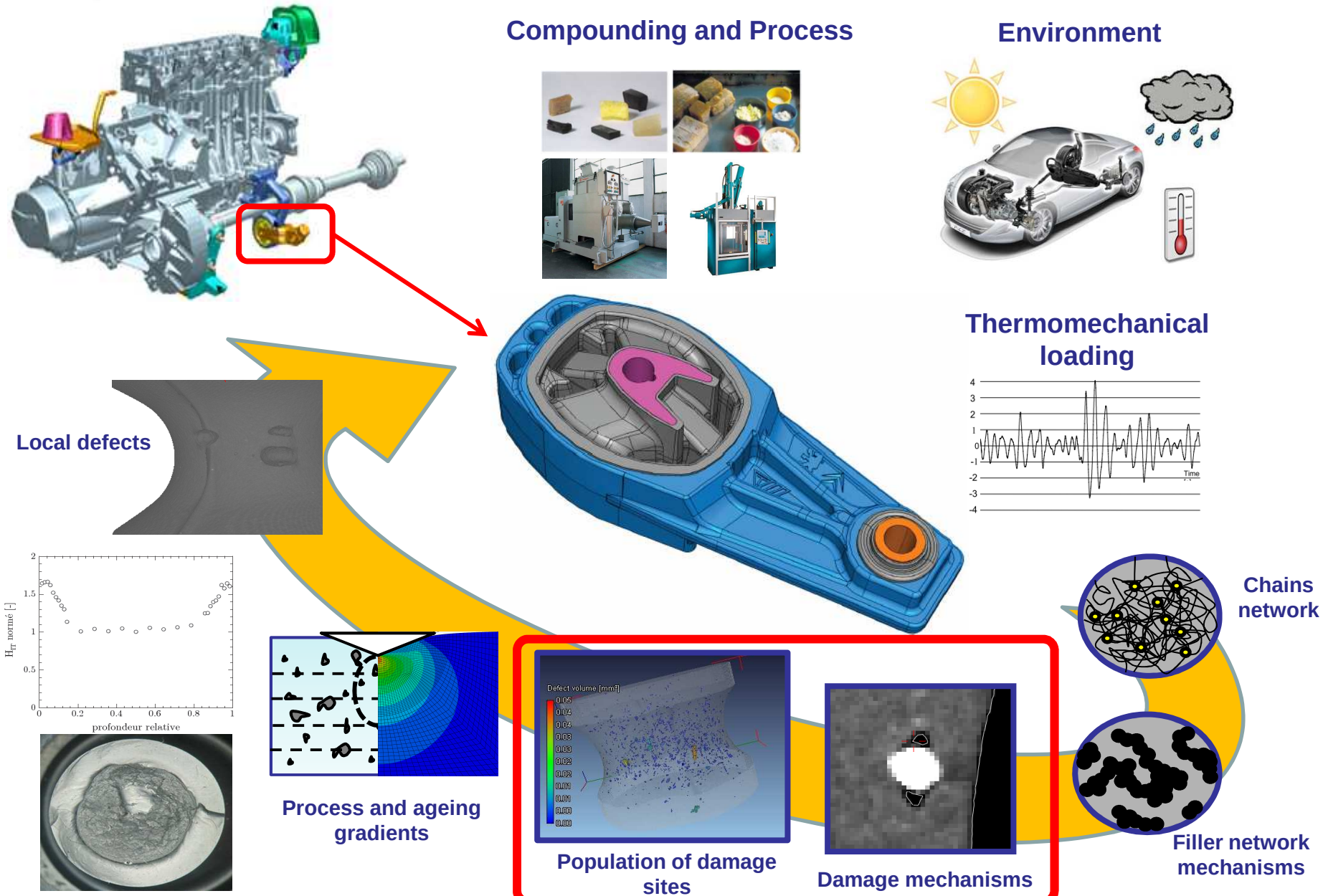
Elastomeric materials

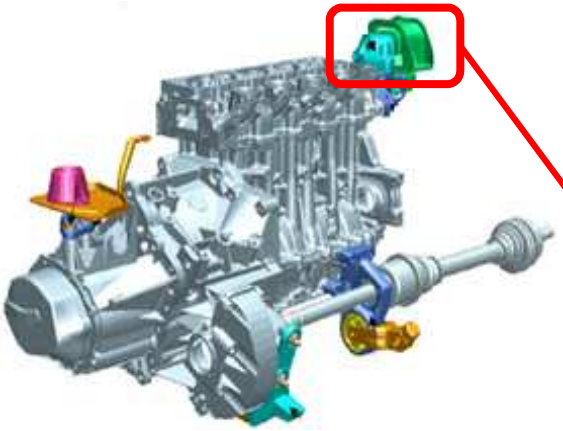

Short Fibres reinforced plastics and continuous fibers composites


Introduction: Industrial context for elastomers

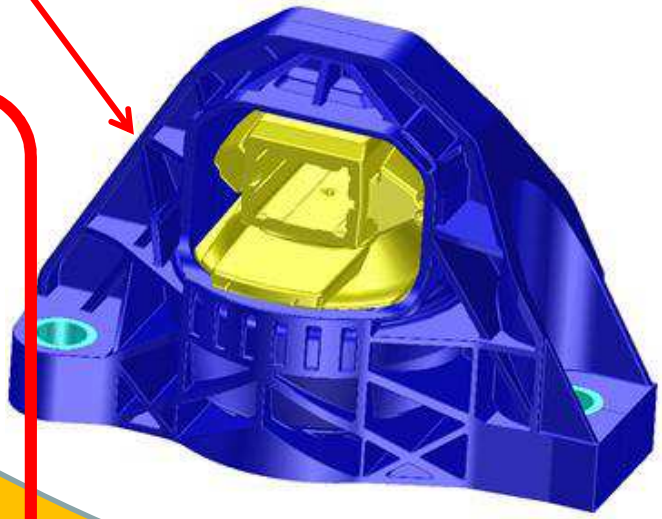


Introduction: Industrial context for Short Fibers Reinforced Plastics (SFRP)

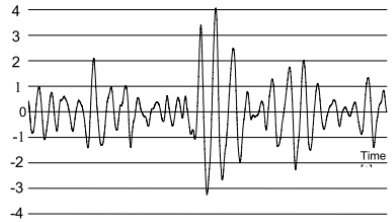


Processing

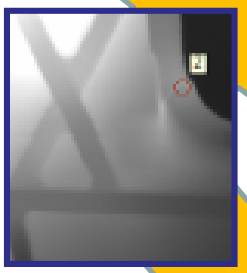
Environment

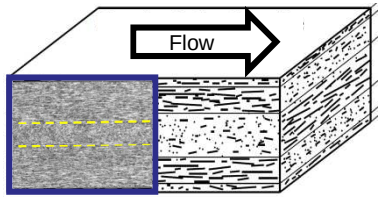


Thermomechanical loading

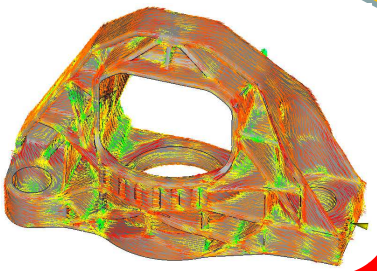


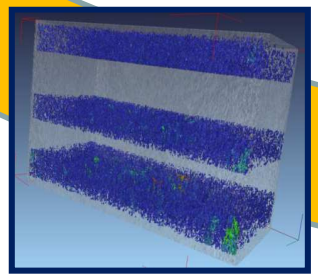
Structural scale



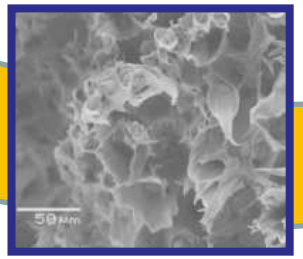


Gradients induced by the process and/or the environment



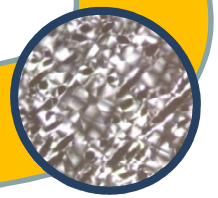


Fatigue defects population



Fatigue mecanisms

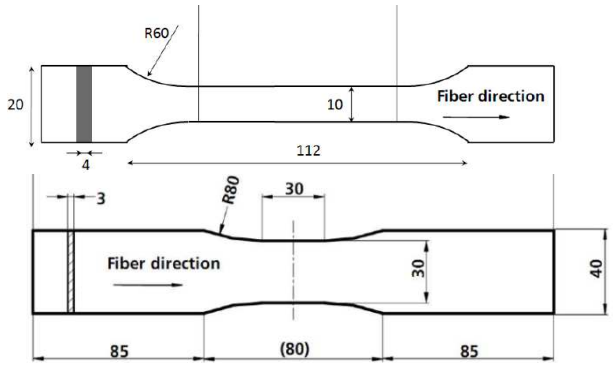
Macro-molecules



Semi-cristalline microstructure

Introduction: Industrial context for Short Fibers Reinforced Plastics (SFRP)

Characterization on samples



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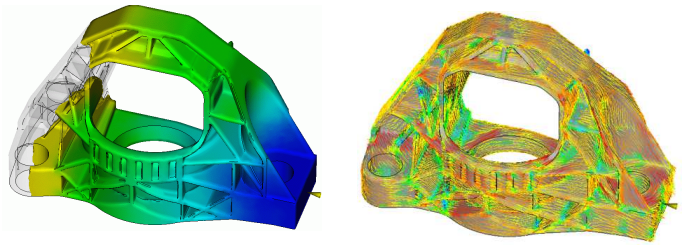
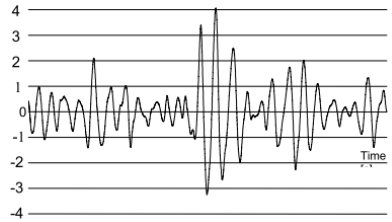
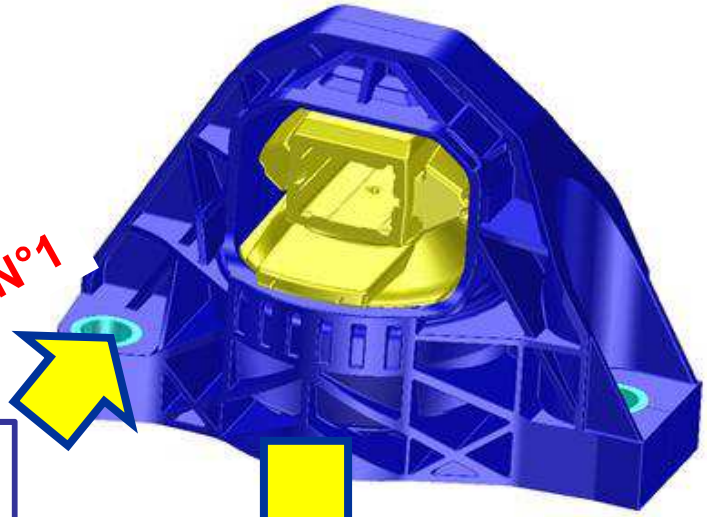


Environment

Thermomechanical loading



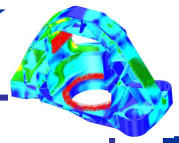


DIFFICULTY N°1

Complex mechanical tests

Cyclic constitutive law

Support of thermal measurements ??



Wöhler Curves

DIFFICULTY N°2

initiation criterion

Local strain, stress and energy fields

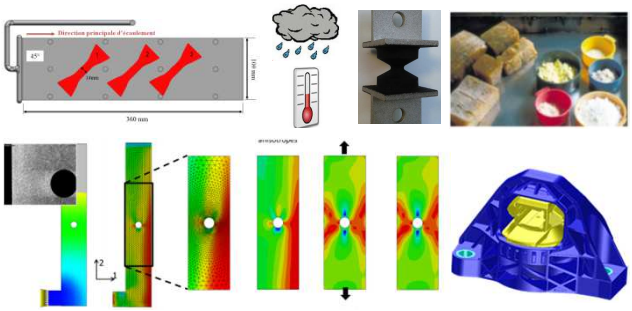
DIFFICULTY N°3

Validation of the prototype

Outline of the presentation

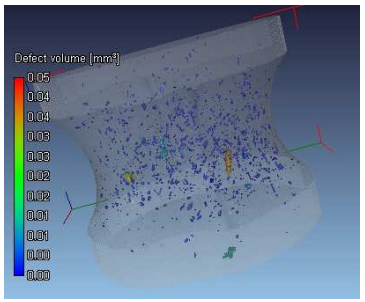
1. Toolbox and Evaluation of dissipation from thermal measurements

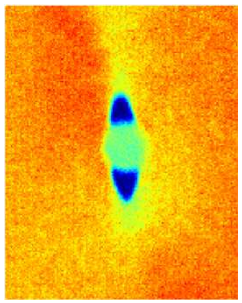
2. Some answers to industrial expectations



- Supply data to feed or validate design computation loops
- Fast screening of fatigue properties for various parameters
- Fast diagnostic on structural samples and parts

3. Questions, limits and tracks to move further





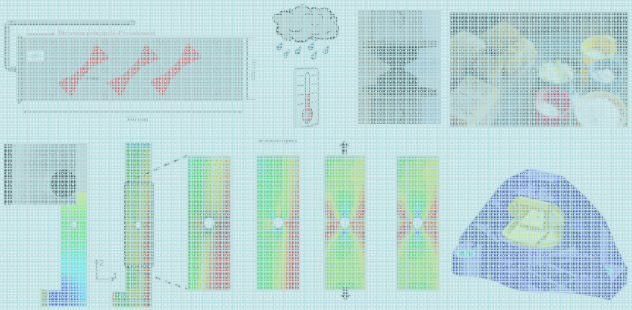
- Illustration of some questions and limits
- Analysis based on constitutive modeling
- Analysis based on the evaluation of defects populations
- Thermomechanical evaluation at the microscopic scale

4. Conclusions

Outline of the presentation

1. **Toolbox and Evaluation of dissipation from thermal measurements**

2. Some answers to industrial expectations



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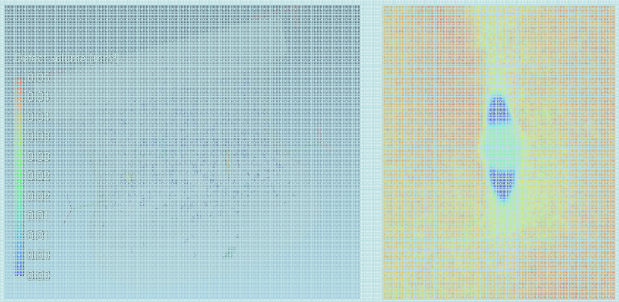


- Illustration of some questions and limits
- Analysis based on constitutive modeling
- Analysis based on the evaluation of defects populations
- Thermomechanical evaluation at the microscopic scale

4. Conclusions

Thermal measurements ?

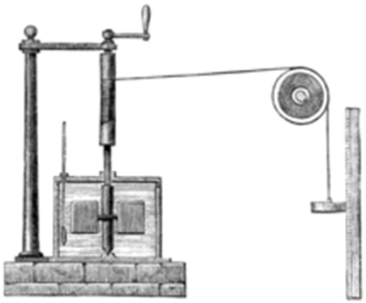
Defects detection

Physical constants

Microscopic field measurements

Acoustic sollicitation, Lock-In thermography, Laser pulse, …

Thermo elasticity and Quantitative calorimetry under mechanical loading



Joule (1850)

Taylor and Quinney (1934)

Anthony (1942)

Treloar (1975)

A. Chrysochoos *et al.*

D. Rittel

GDR CNRS 2519 « Mesures de champs et identification en mécanique des solides » et « Calorimétrie quantitative en mécanique des matériaux »

Evaluation of dissipation from thermal measurements: the toolbox

Infrared Camera
Lens 50mm and "G1"

Testing machines

GeM
Institut de Recherche en Génie Civil et Mécanique

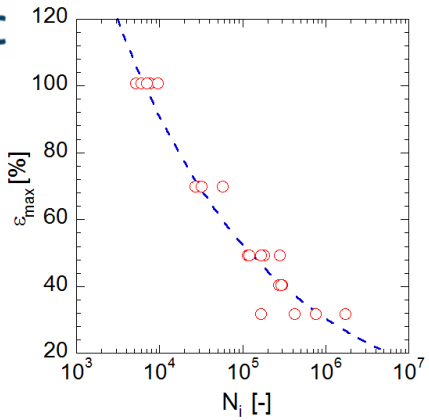
X-ray Micro Tomograph

SEM with EDS

Vibracoustic

PSA GROUPE

SOLVAY
asking more from chemistry®



Initiation criterion

Classical and interrupted fatigue tests

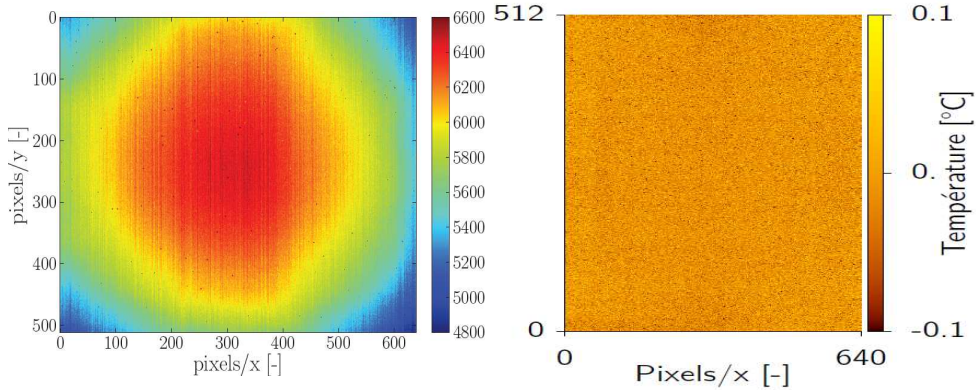
Environmental testing conditions

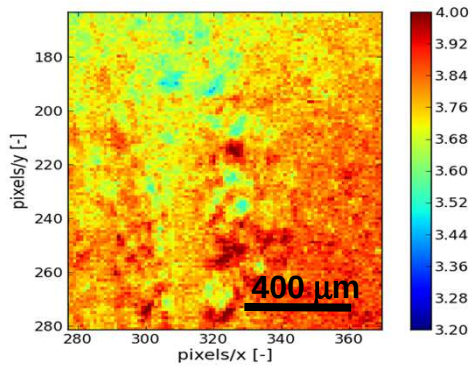
Various temperatures and humidities

Evaluation of dissipation from thermal measurements: the toolbox

Homemade pixelwise calibration

[*Belenos*®, V. Le Saux]



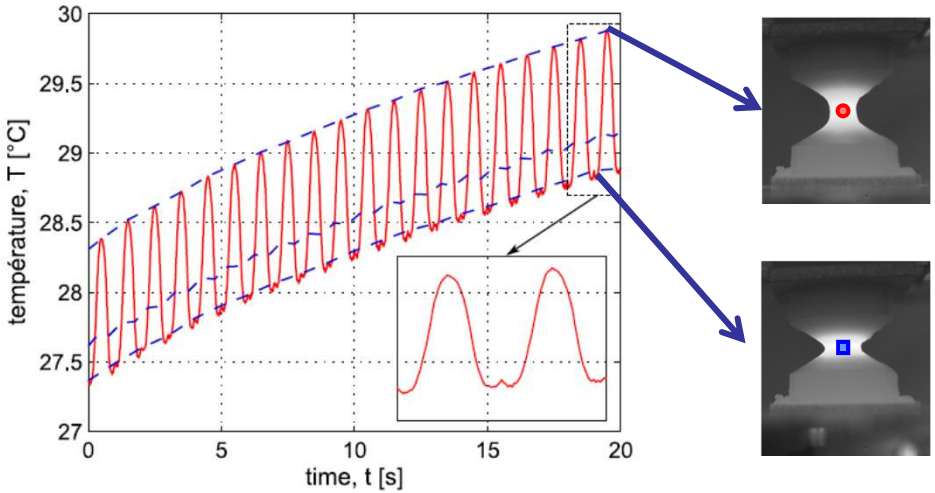


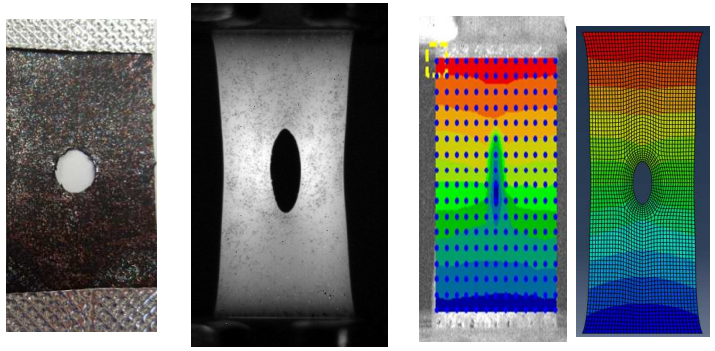
Thermal resolution < 10 mK differential measurements

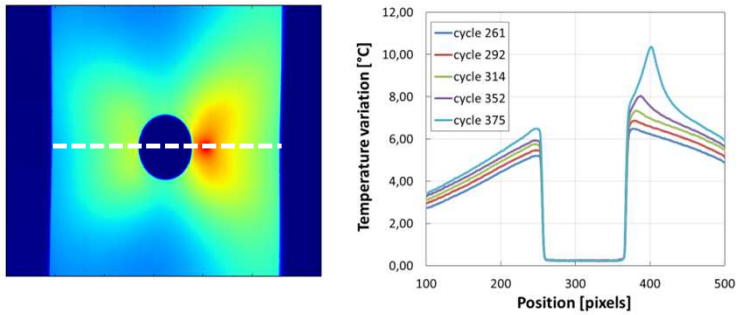
Spatial resolution 1 pixel ~ 15 µm

Measuring accuratly temperature is fine …

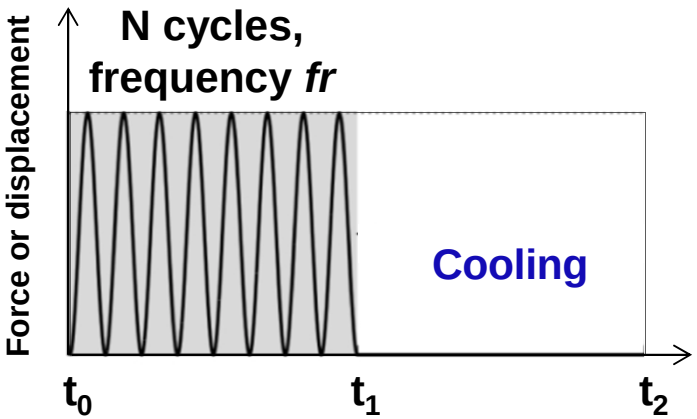
but here, we seek dissipation!

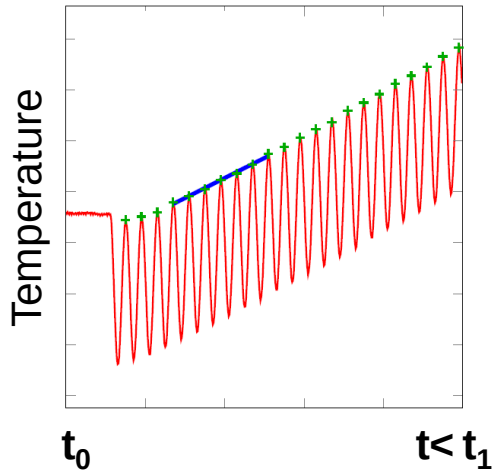


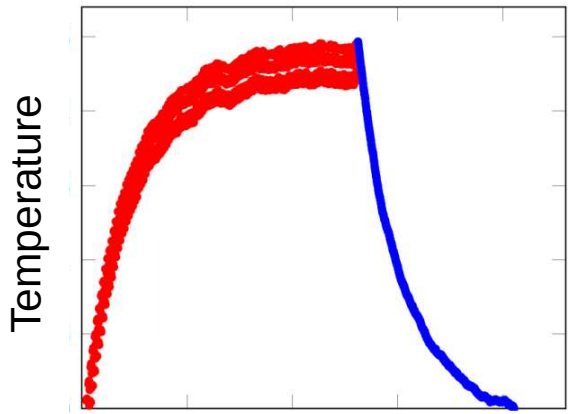




Evaluation of dissipation from thermal measurements: the approaches







$$\rho c\dot{T} + div(\vec{q}) = \Delta + r + \rho T \frac{\partial^2 \Psi}{\partial V_k \partial T}.\dot{V}_k + \rho T \frac{\partial^2 \Psi}{\partial \varepsilon^e \partial T} : \dot{\varepsilon}^e$$

Evolution along time	Exchange and diffusion	Intrinsic dissipation	Reflection from external sources	Couplings between internal variables V_k	Thermo-elastic couplings

Local equation.
It is mandatory to consider the same volume

For large displacements, the convective term of the time derivative can be important

Surface measurements vs volumic sources:

ill conditioned problem

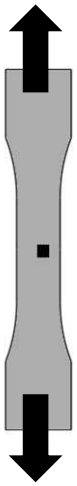
Evaluation of dissipation from thermal measurements: the approaches

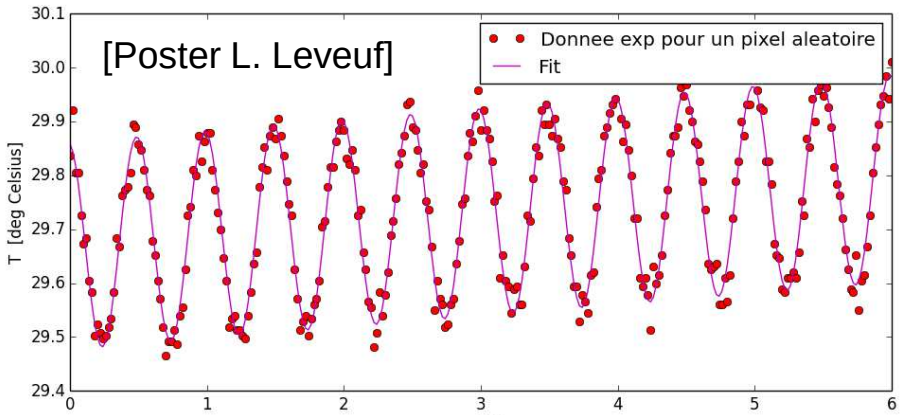
$$\rho c\dot{T} + div(\vec{q}) = \Delta + r + \rho T \frac{\partial^2 \Psi}{\partial V_k \partial T}.\dot{V}_k + \rho T \frac{\partial^2 \Psi}{\partial \mathfrak{E}^e \partial T} : \dot{\mathfrak{E}^e}$$

Evolution along time | Exchange and diffusion | Intrinsic dissipation | Reflection from external sources | Couplings between internal variables V_k | Thermo-elastic couplings

Fit of thermal functions

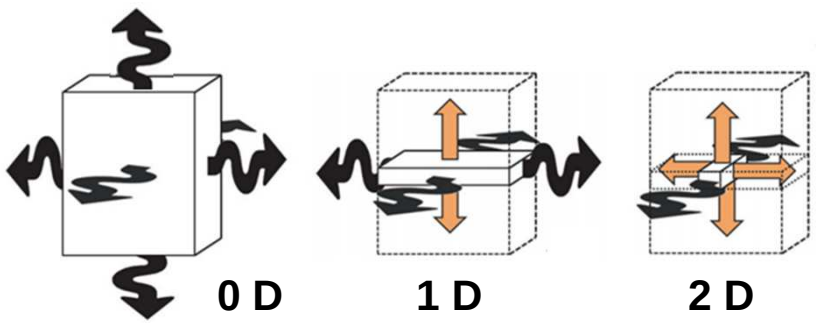
- Direct numerical fit
- Decomposition in functions (usually Fourier's)
- Assumptions on the function shape, based on mechanical and thermal hypotheses





$$\theta = T - T_{ini} = \dot{\theta}_0 \times t + \Delta\theta_c \times sin\left(\frac{f_{méca}}{f_{thermo}} t + \phi\right)$$

Hypotheses on spatial sources dissipation



Hypotheses on temporal evolution

- Stationary thermal state
- Analysis at the limits of transient states
- Adiabatic
- Average analysis over a cycle
- Constant cyclic source

Evaluation of dissipation from thermal measurements: the approaches

$$\rho c\dot{T} + div(\vec{q}) = \Delta + r + \rho T \frac{\partial^2 \Psi}{\partial V_k \partial T}.\dot{V}_k + \rho T \frac{\partial^2 \Psi}{\partial \varepsilon^e \partial T} : \dot{\varepsilon}^e$$

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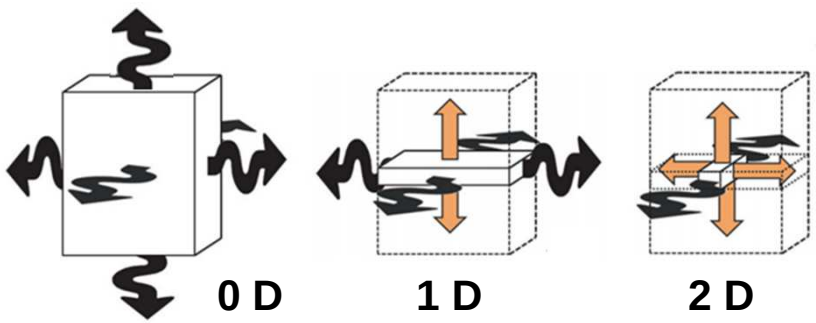
Sources assumptions

- Spatial distribution of S(t)
- Assumptions on the function shape, based on mechanical and thermal hypotheses
- Thermomechanical model based analysis

Direct experimental evaluation of the sources from thermal fields

Hypotheses on spatial sources dissipation



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Evolution along time | Exchange and diffusion | Intrinsic dissipation | Reflection from external sources | Couplings between internal variables V_k | Thermo-elastic couplings

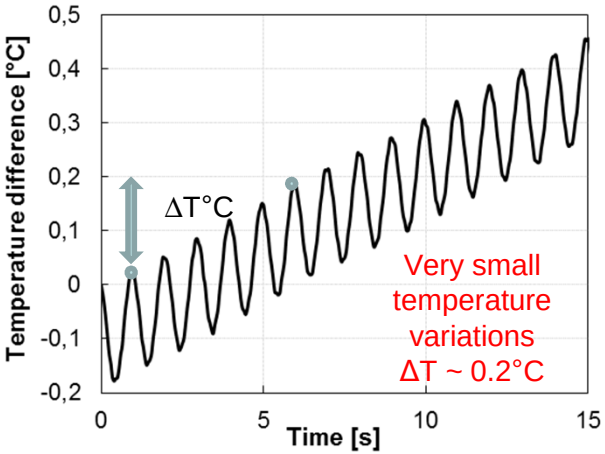
Considering $\theta = T - T_{reference}$

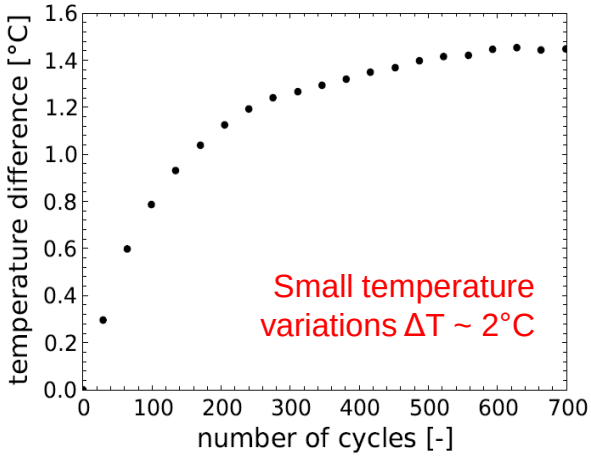
H The external radiation supply is considered constant

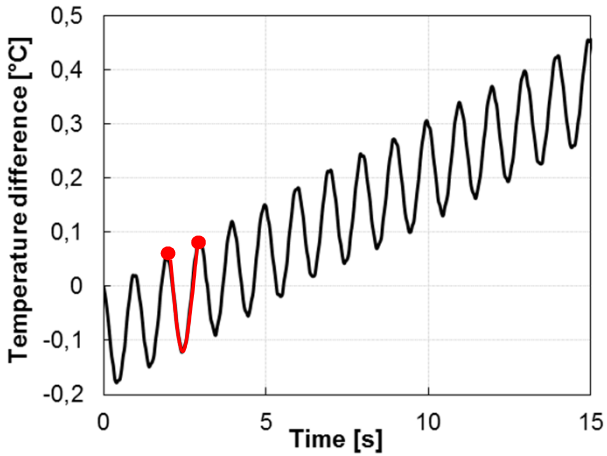
$$\rho c \dot{\theta} + div(\vec{q}) = \Delta + r + \rho \theta \frac{\partial^2 \Psi}{\partial V_k \partial \theta} . \dot{V}_k + \rho \theta \frac{\partial^2 \Psi}{\partial \mathcal{E}^e \partial \theta} : \dot{\mathcal{E}}^e$$

H **Low dependency of V_k on temperature variations**

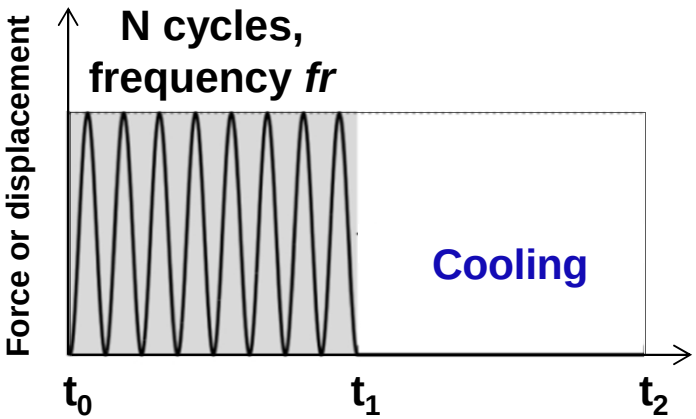
H **The thermo-elastic couplings compensate over a mechanical cycle**

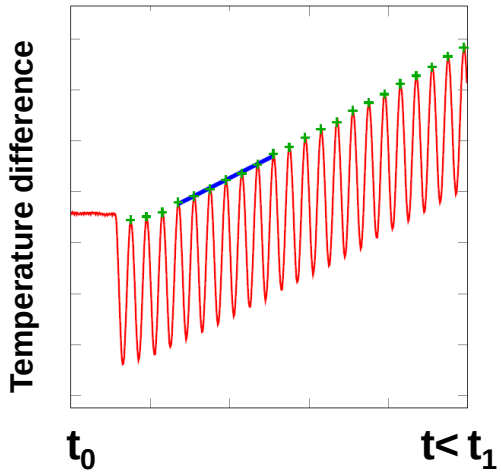


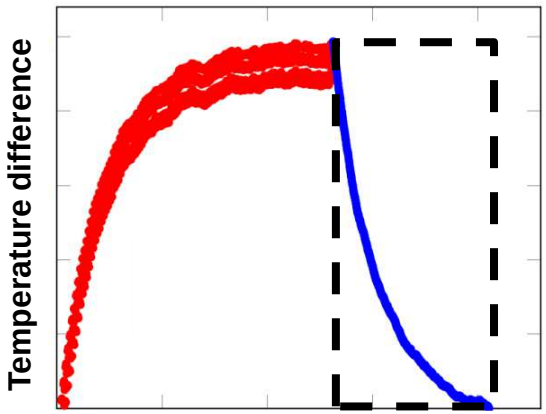




Evaluation of dissipation from thermal measurements: the approaches







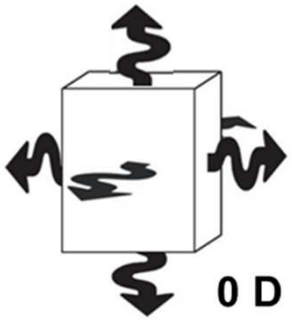
Use the stationary state

Sources assumptions

- Spatial distribution of S(t)
- Assumptions on the function shape, based on mechanical and thermal hypotheses
- Thermomechanical model based analysis

$$\rho c \dot{\tilde{\theta}} + div(\vec{q}) = \Delta = fr.\ \Delta^*$$

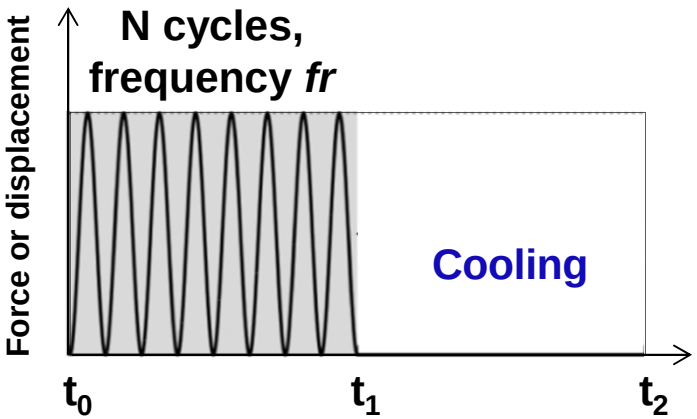
H the mean dissipation per cycle is constant

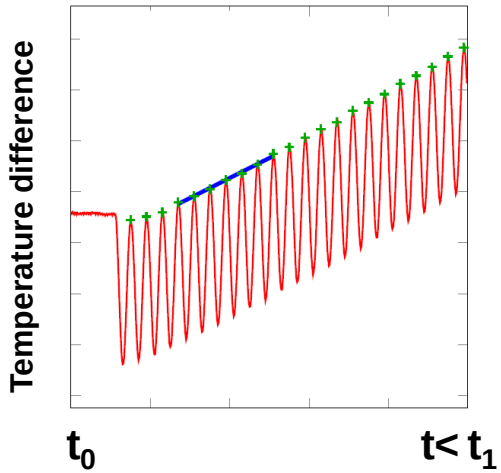


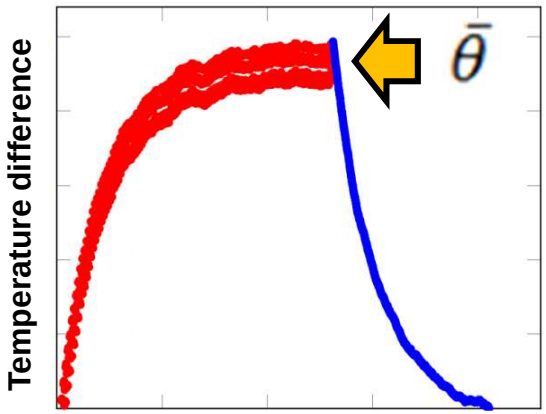
$$\dot{\theta} + \frac{\theta}{\tau_{eq}} = \frac{fr\ \Delta^*}{\rho c}$$

1 Identify τ_{eq}

Evaluation of dissipation from thermal measurements: the approaches





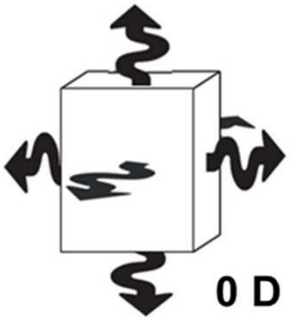


Use the stationary state

Sources assumptions

- Spatial distribution of S(t)
- Assumptions on the function shape, based on mechanical and thermal hypotheses
- Thermomechanical model based analysis

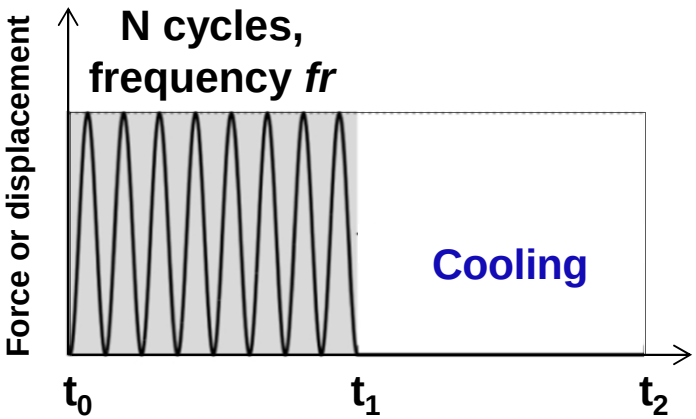
$$\rho c \dot{\tilde{\theta}} + div(\vec{q}) = \tilde{\Delta} = fr.\ \Delta^*$$

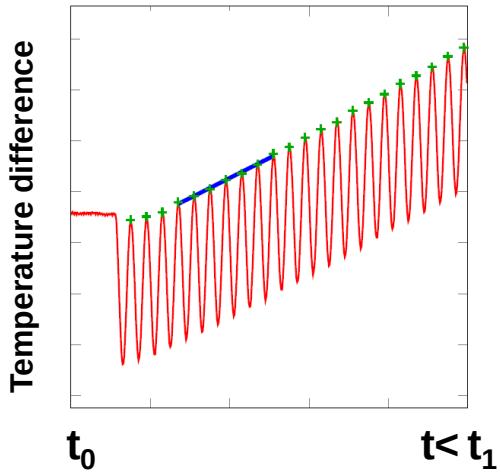


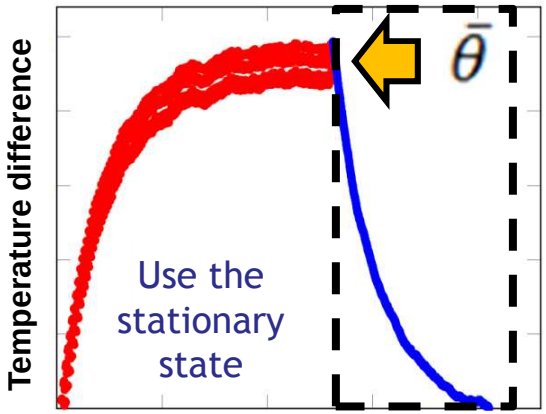
$$\dot{\theta} \leftarrow \frac{\theta}{\tau_{eq}} = \frac{fr\ \Delta^*}{\rho c}$$

1. Identify τ_{eq}
2. $\Delta^* = \dfrac{\rho c\ \bar{\theta}}{fr\ \tau_{eq}}$

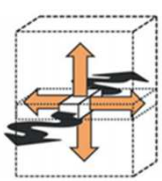
Evaluation of dissipation from thermal measurements: the approaches







[Poster T. Glanowski]

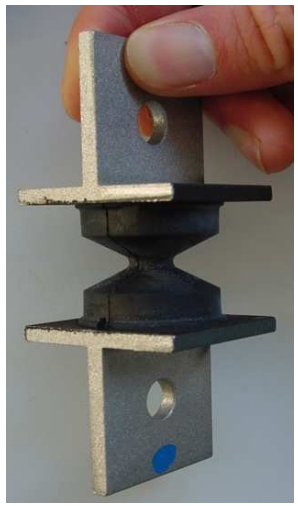


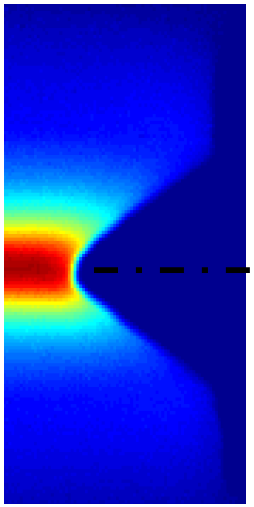
2 D

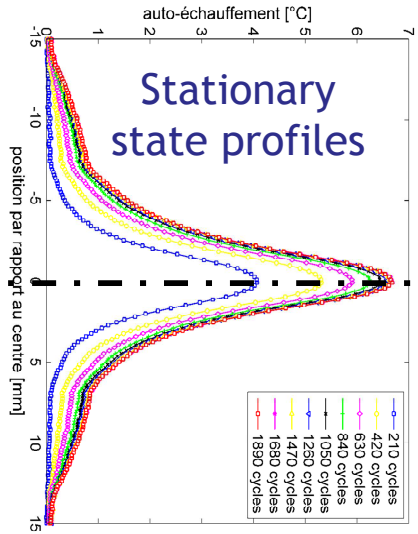
Sources assumptions

- Spatial distribution of S(t)
- Assumptions on the function shape, based on mechanical and thermal hypotheses
- Thermomechanical model based analysis

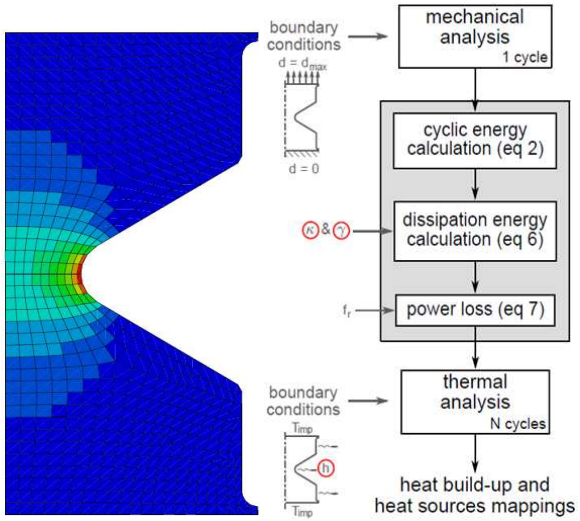
$$\rho c \dot{\tilde{\theta}} + div(\vec{q}) = \tilde{\Delta} = fr.\ \Delta^*$$



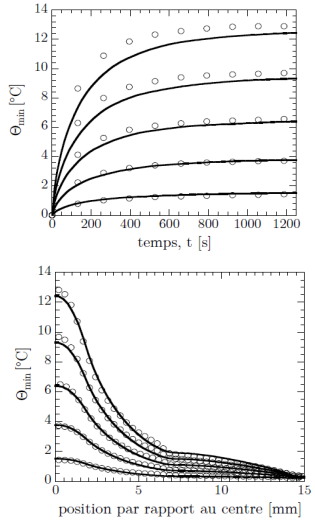




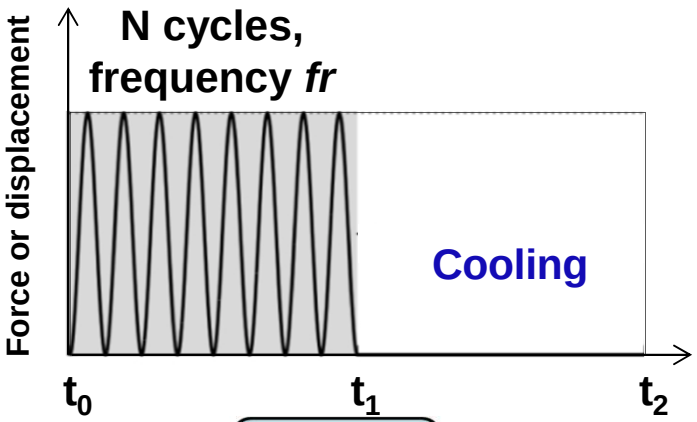
Inverse identification by FE

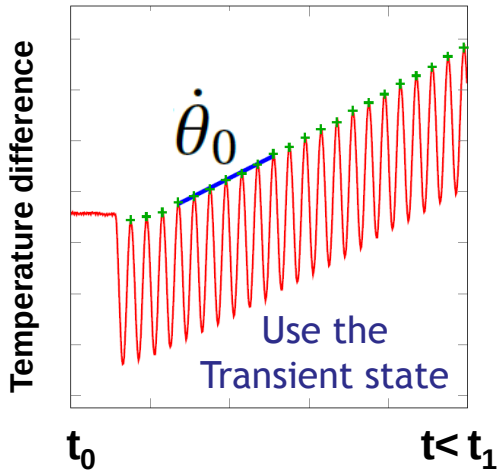


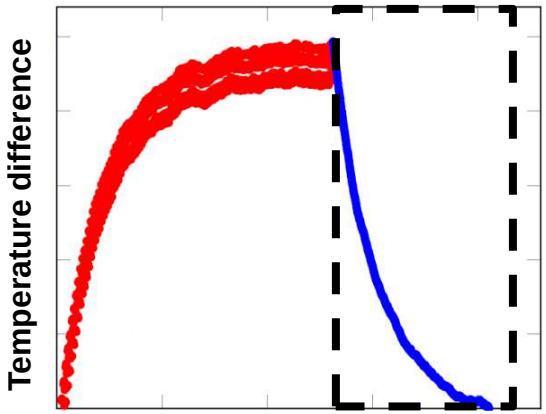
Validation



Evaluation of dissipation from thermal measurements: the approaches

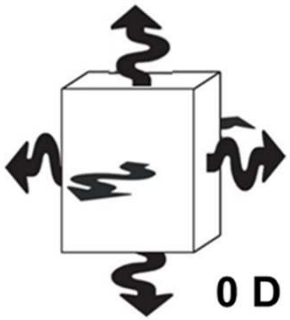






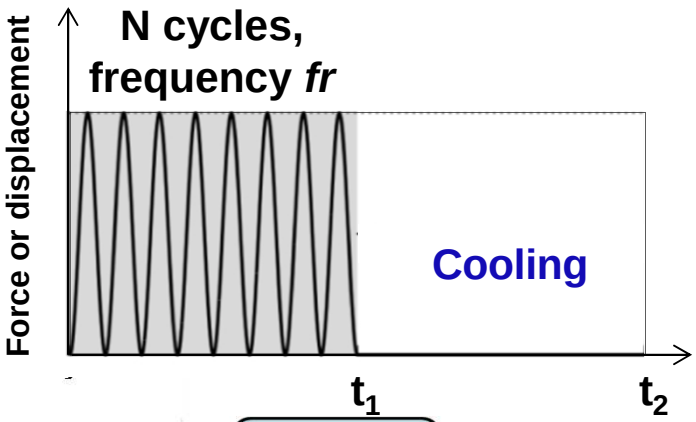
Direct experimental evaluation of the sources from thermal fields

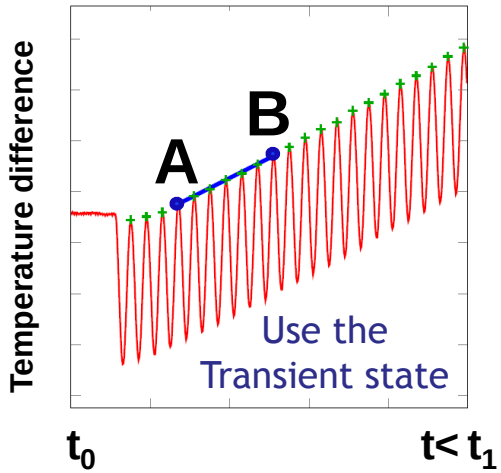
$$\rho c\dot{\tilde{\theta}} + \cancel{div(\tilde{q})} = \tilde{\Delta} = fr.\ \Delta^*$$

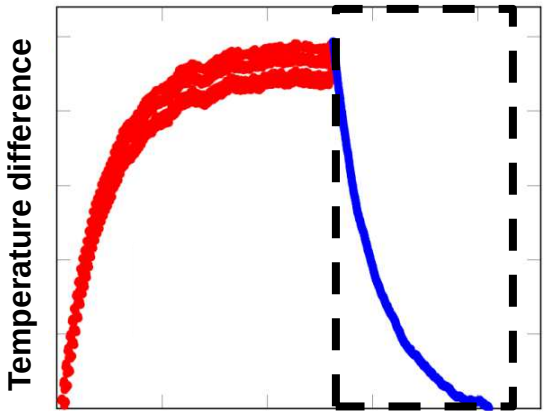


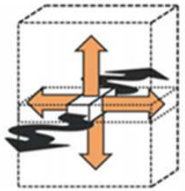
$$\Delta^* = \frac{\rho C \dot{\theta}_0}{f}$$

Evaluation of dissipation from thermal measurements: the approaches





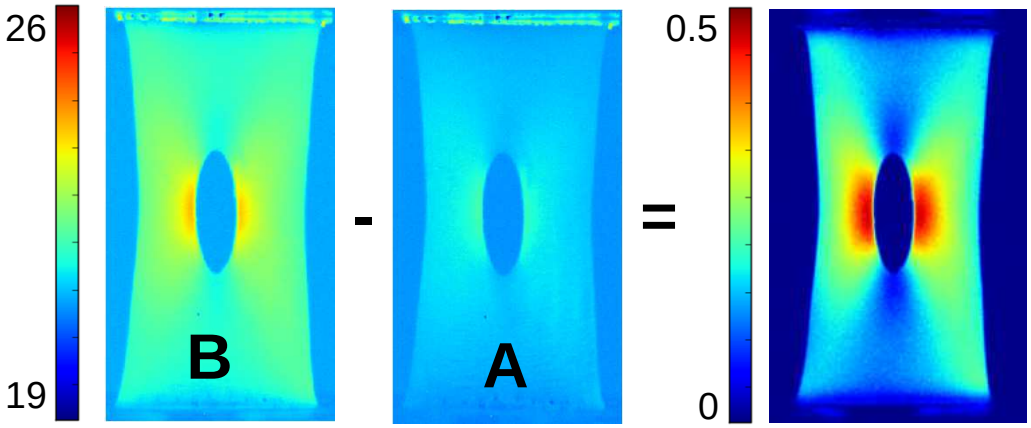




2 D

Direct experimental evaluation of the sources from thermal fields

$$\rho c \dot{\tilde{\theta}} + div(\tilde{q}) = \tilde{\Delta} = fr.\ \Delta^*$$

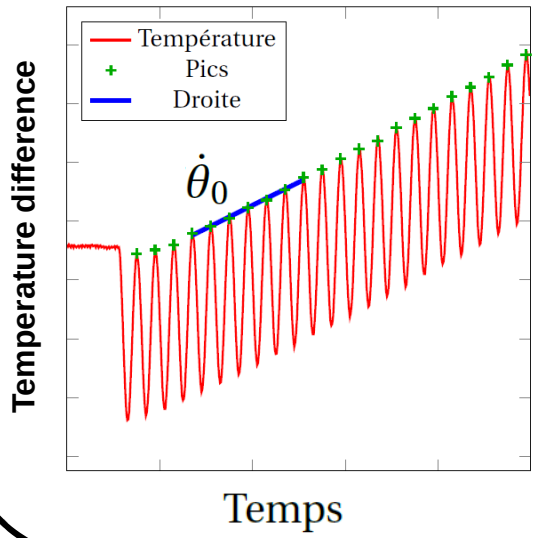


Direct mapping of the dissipated energy Δ*

Over a given thickness depending on thermal exchanges vs mechanical frequency

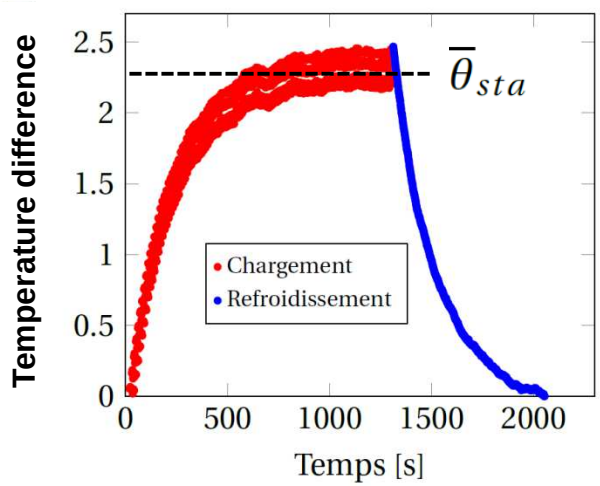
Evaluation of dissipation from thermal measurements: the approaches

Evaluation from the transient state

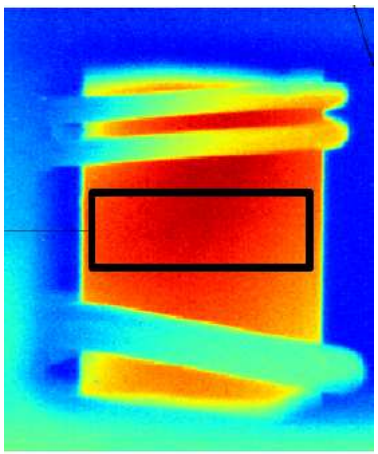


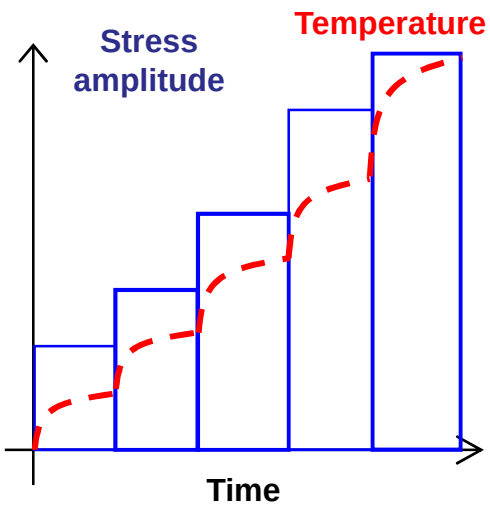
$$\Delta^* = \frac{\rho C \dot{\theta}_0}{f}$$

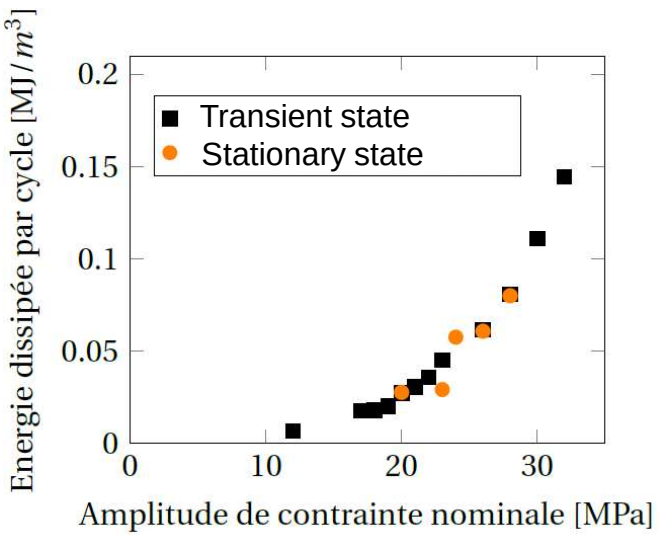
Evaluation from the stationary state



$$\Delta^* = \frac{\rho C \overline{\theta}_{sta}}{f \tau_{eq}}$$



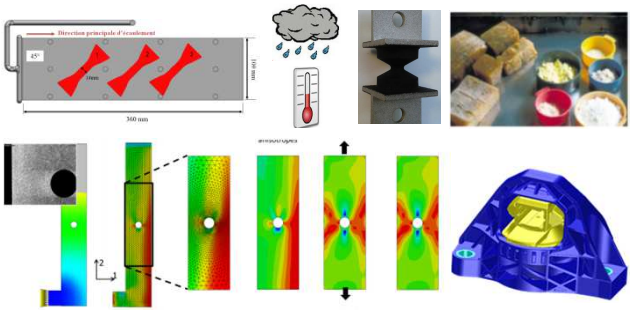




Outline of the presentation

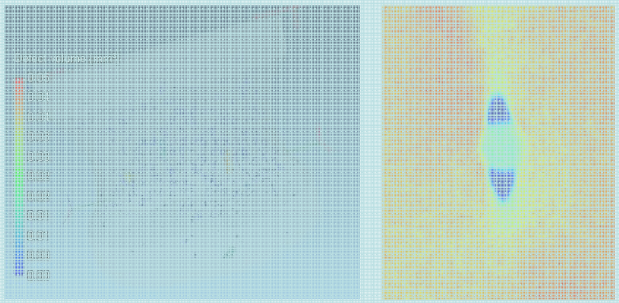
1 Evaluation of dissipation from thermal measurements and heat build-up curve

2 Some answers to industrial expectations



- Supply data to feed or validate design computation loops
- Fast screening of fatigue properties for various parameters
- Fast diagnostic on structural samples and parts

3 Questions, limits and tracks to move further

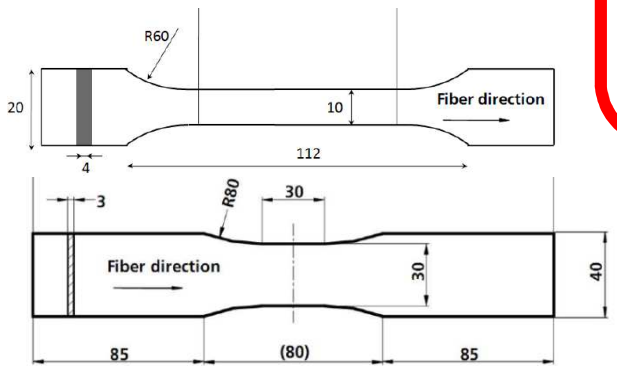


- Illustration of some questions and limits
- Analysis based on constitutive modeling
- Analysis based on the evaluation of defects populations
- Thermomechanical evaluation at the microscopic scale

4 Conclusions

Overview for SFRP

Characterization
on samples

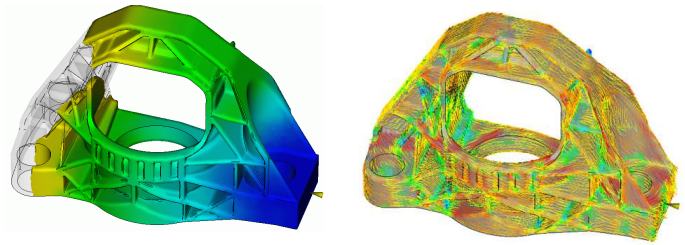


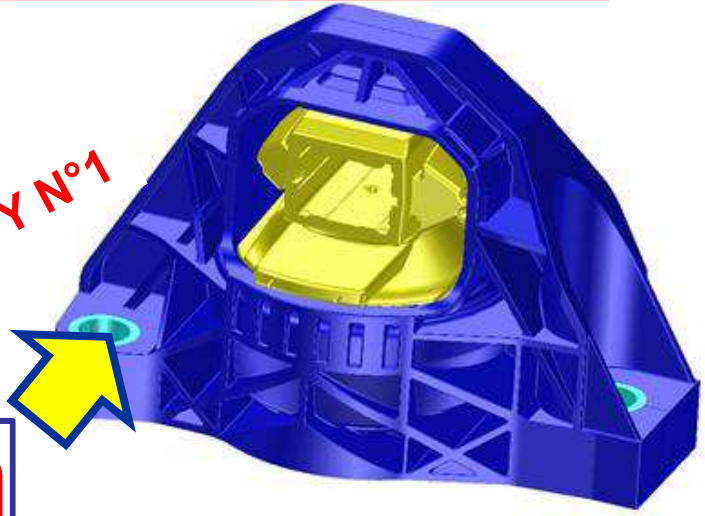
Complex mechanical tests

Cyclic constitutive law

DIFFICULTY N°1

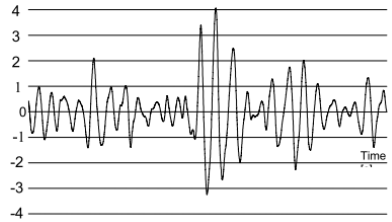
Processing





Environment

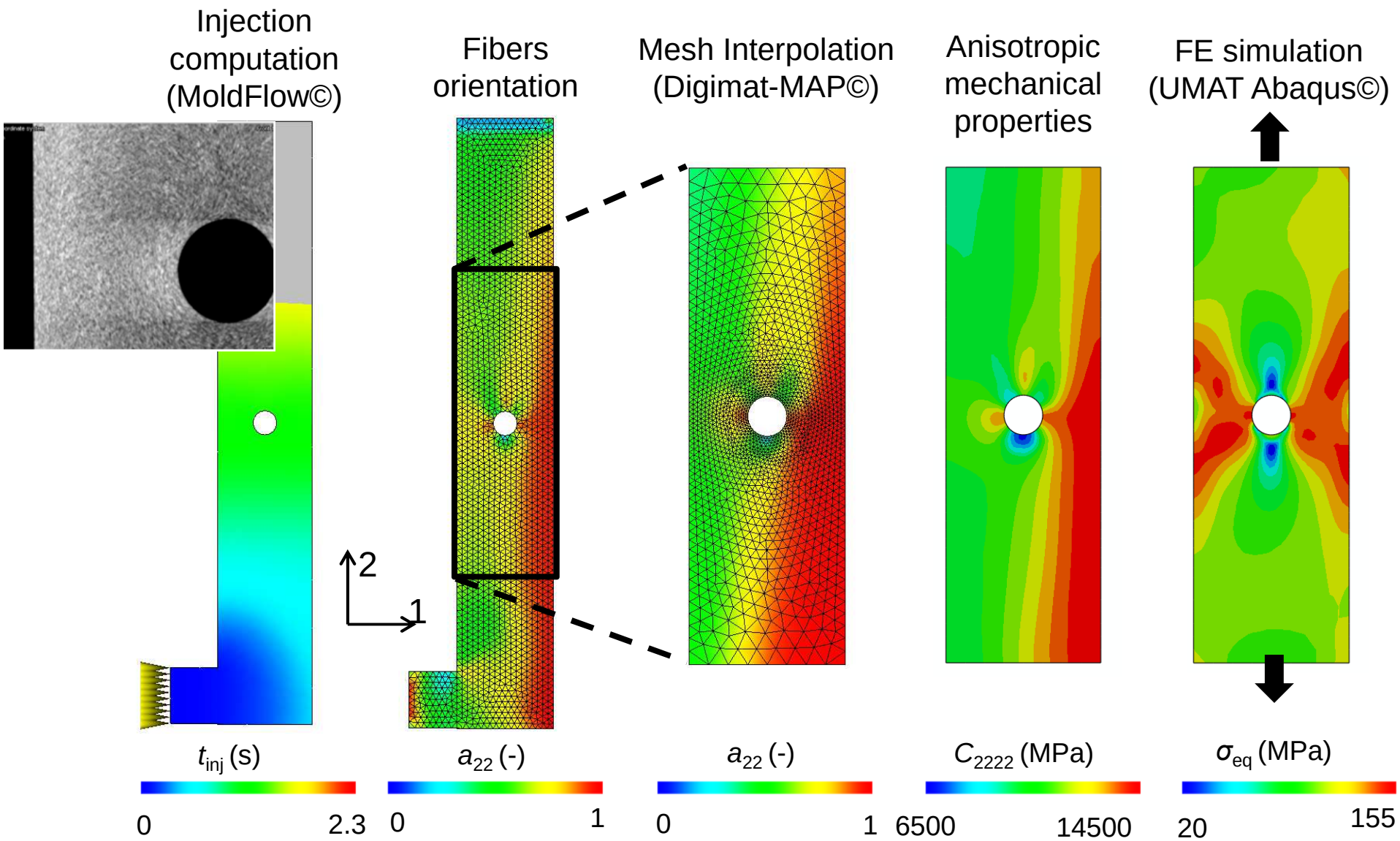
Thermomechanical loading



Goal: to take into account the local microstructure
to evaluate the local mechanical properties
and to predict both the global stifness and local fatigue criterion

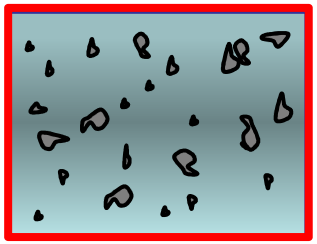
Constitutive model for SFRP

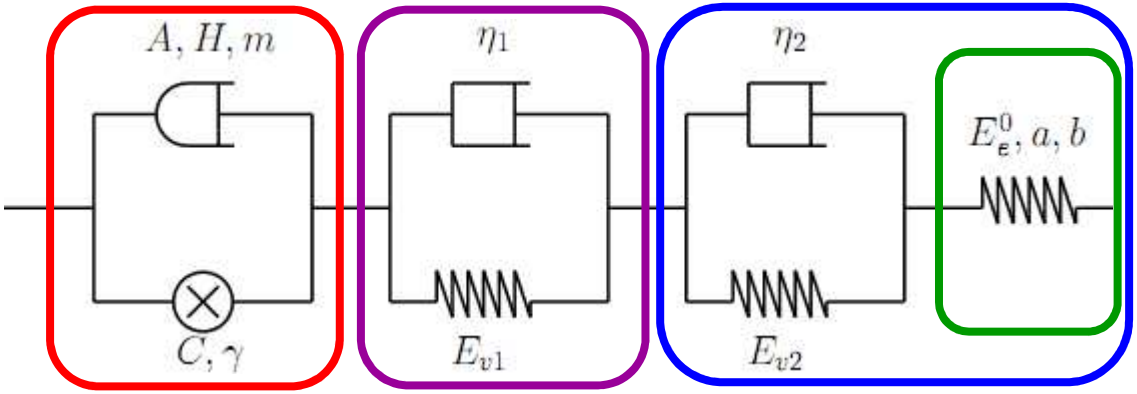
- Anisotropic model to account for the gradients induced by the process



Constitutive model for SFRP: model proposal and identification

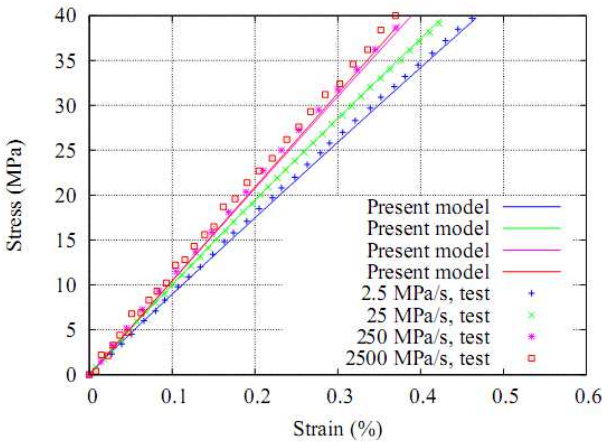
- Phenomenological modeling





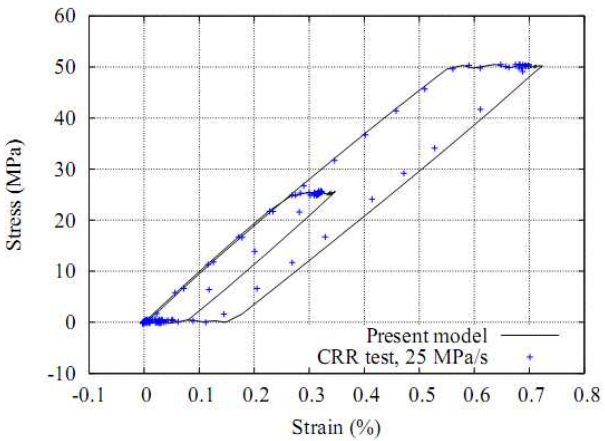
1. Short term visco-elasticity

E_e^0, E_{v2}, η_2



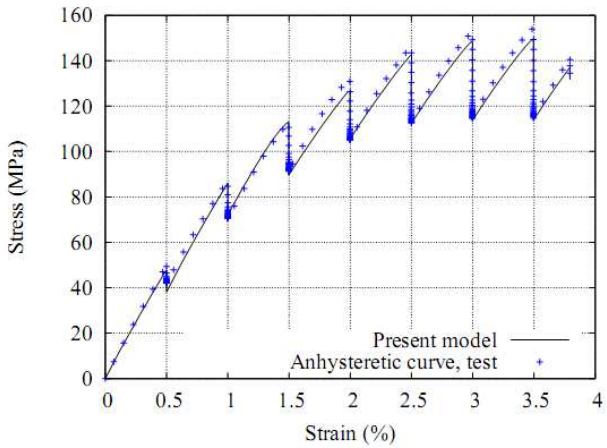
2. Long-term Visco-elasticity

E_{v1}, η_1



3. Non-linear Visco-plasticity

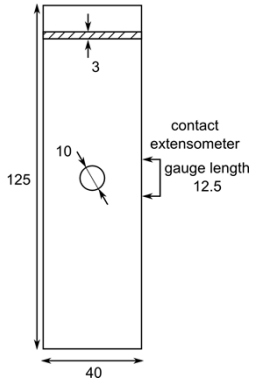
A, H, m, C, γ

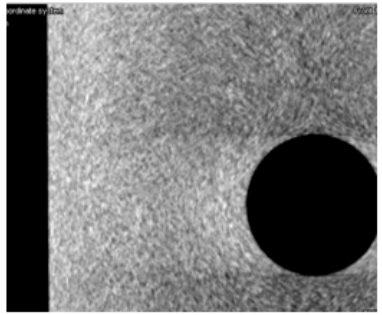


4. Softening parameters a, b

Constitutive model for SFRP: challenge on experiments on structures

- Correlation between simulations and experiments





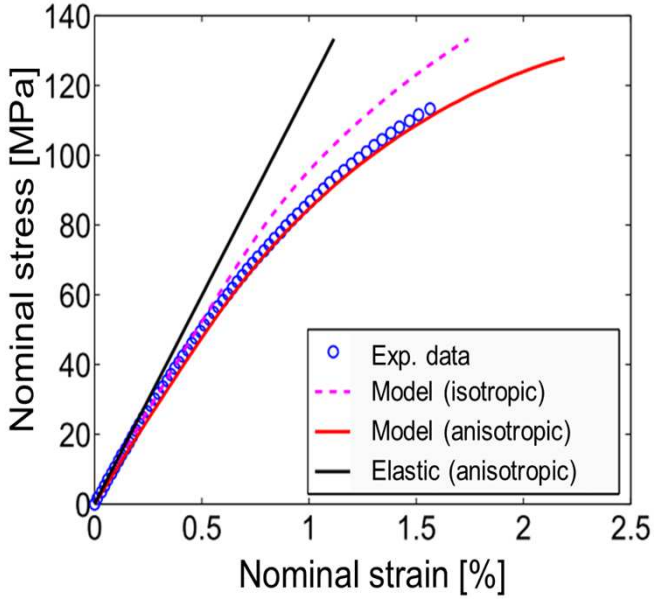
viscoelasticity viscoplasticity softening

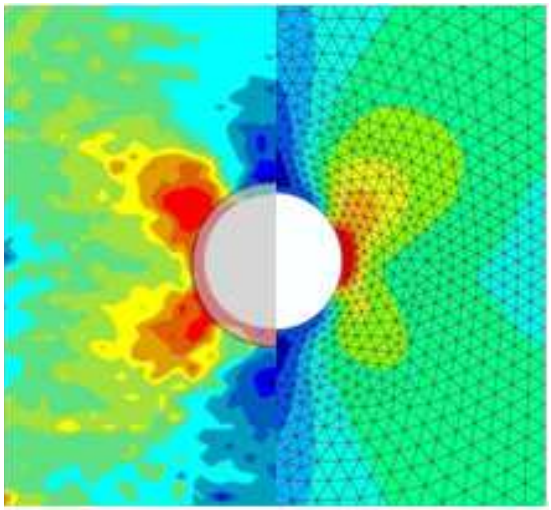
$$\mathscr{D}_1 = \underline{\underline{\mathcal{A}}}_{v1} : \underline{\underline{\dot{\varepsilon}}}_{v1} + \underline{\underline{\mathcal{A}}}_{v2} : \underline{\underline{\dot{\varepsilon}}}_{v2} + \underline{\underline{\mathcal{A}}}_{vp} : \underline{\underline{\dot{\varepsilon}}}_{vp} + \underline{\underline{X}} : \underline{\underline{\dot{\alpha}}} + \mathcal{A}_\beta \cdot \dot{\beta}$$

Sources assumptions

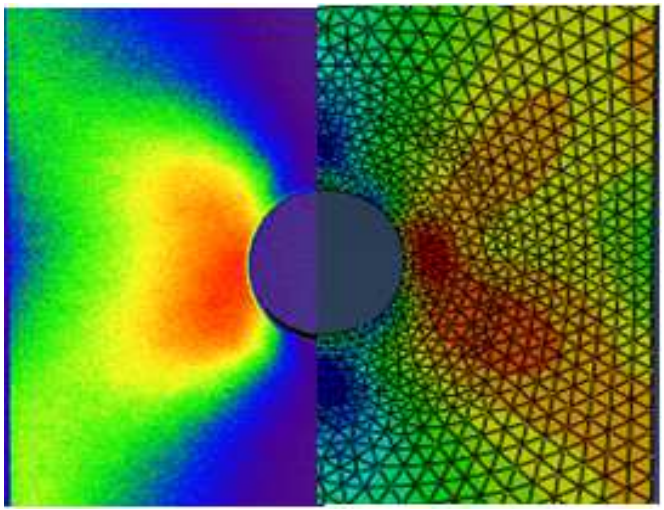
- Spatial distribution of S(t)
- Assumptions on the function shape, based on mechanical and thermal hypotheses
- Thermomechanical model based analysis

$$\Delta^\star = \frac{1}{f_r} \int_{cycle} \mathcal{D}_{int} \, \mathrm{d}t$$



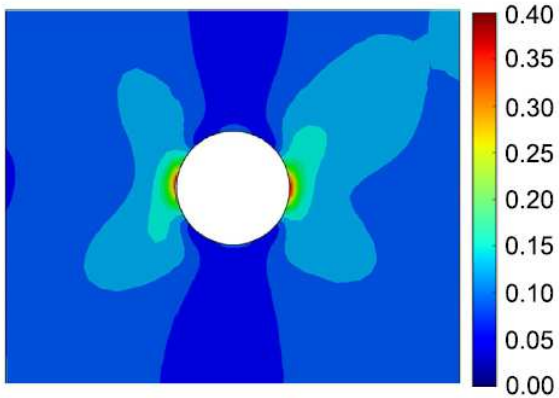


Strain fields

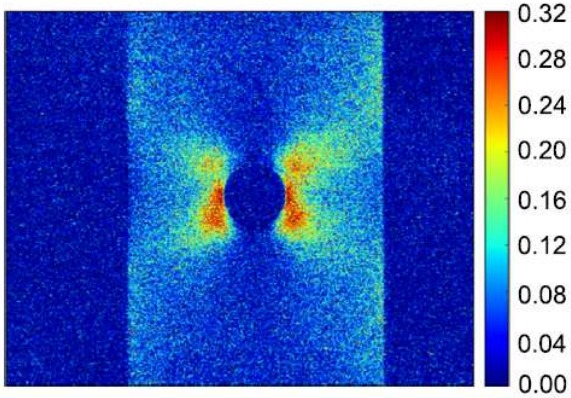


Dissipation fields

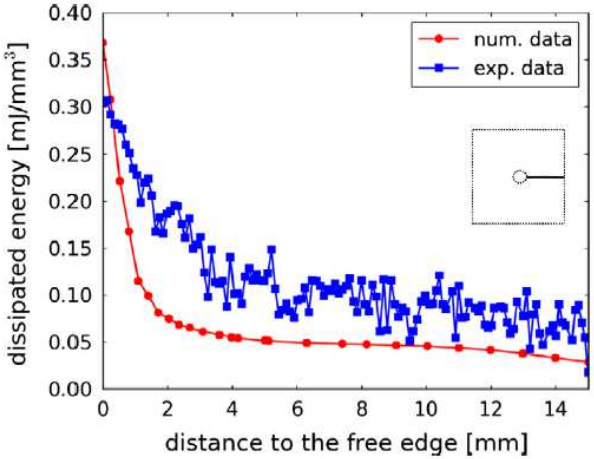
Constitutive model for SFRP: challenge on experiments on structures

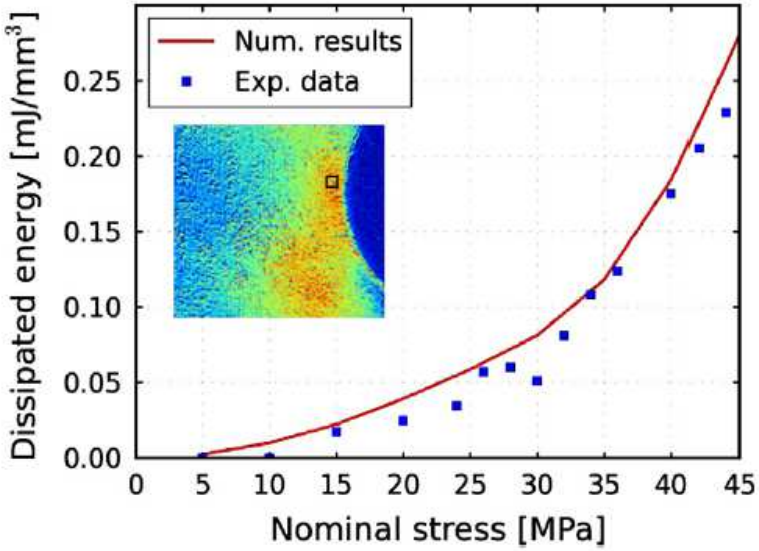


Predicted Dissipated energy (mJ/mm³)

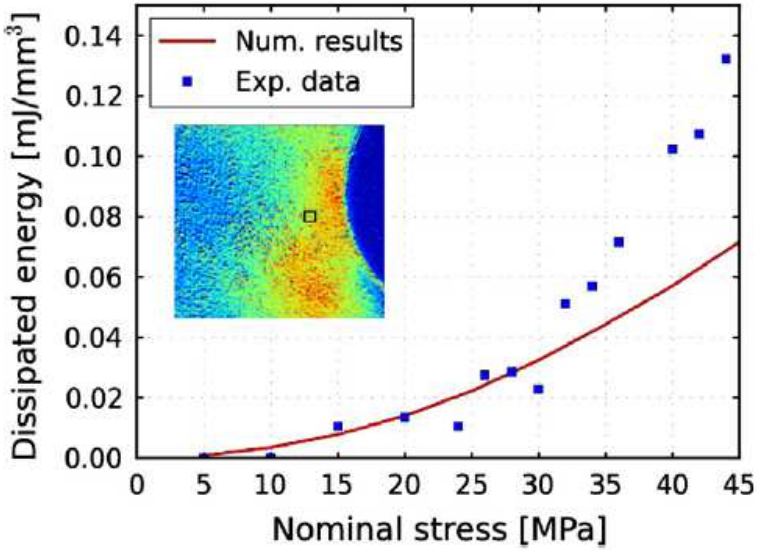


Experimental Dissipated energy (mJ/mm³)





(a) zone 1

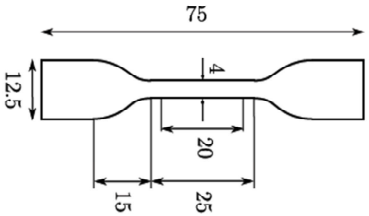


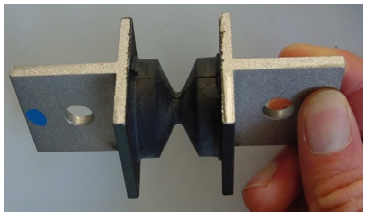
(b) zone 2

Next step: inverse analysis but here more confidence is needed on the microstructure !

How to evaluate fastly the fatigue properties ???

Characterization on samples

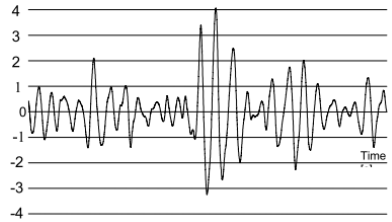


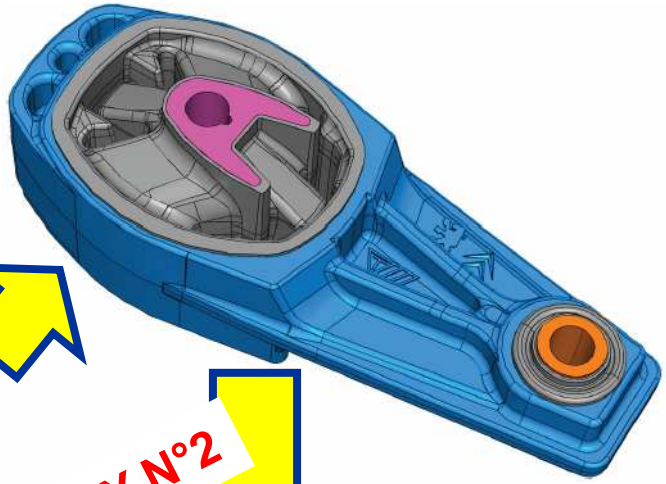


Compounding and Processing

Environment

Thermo-mechanical loading





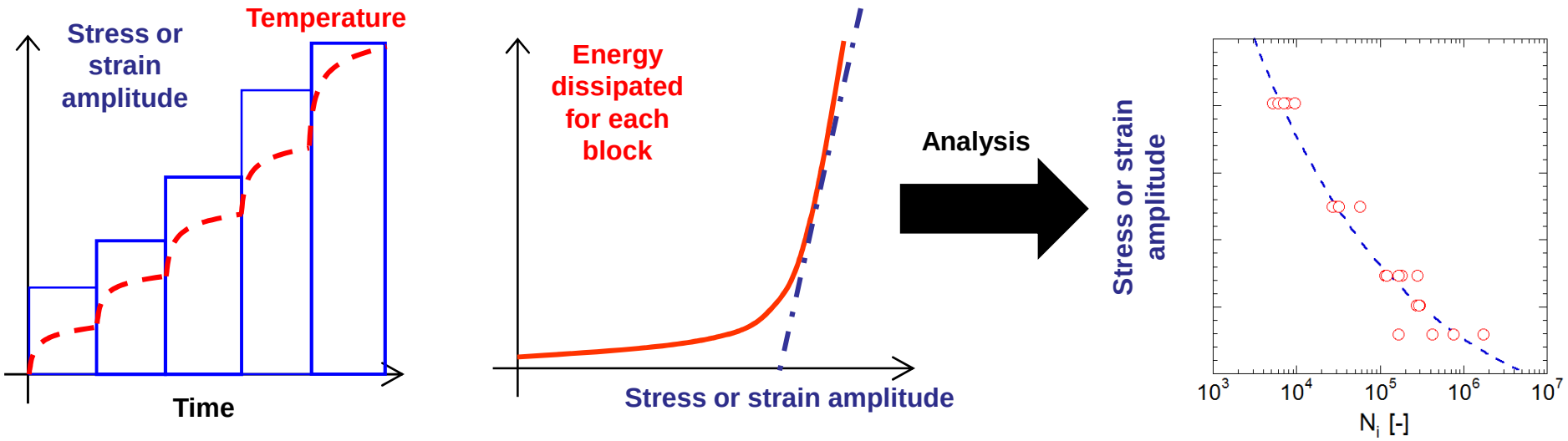
Complex mechanical tests | Cyclic constitutive law

DIFFICULTY N°2

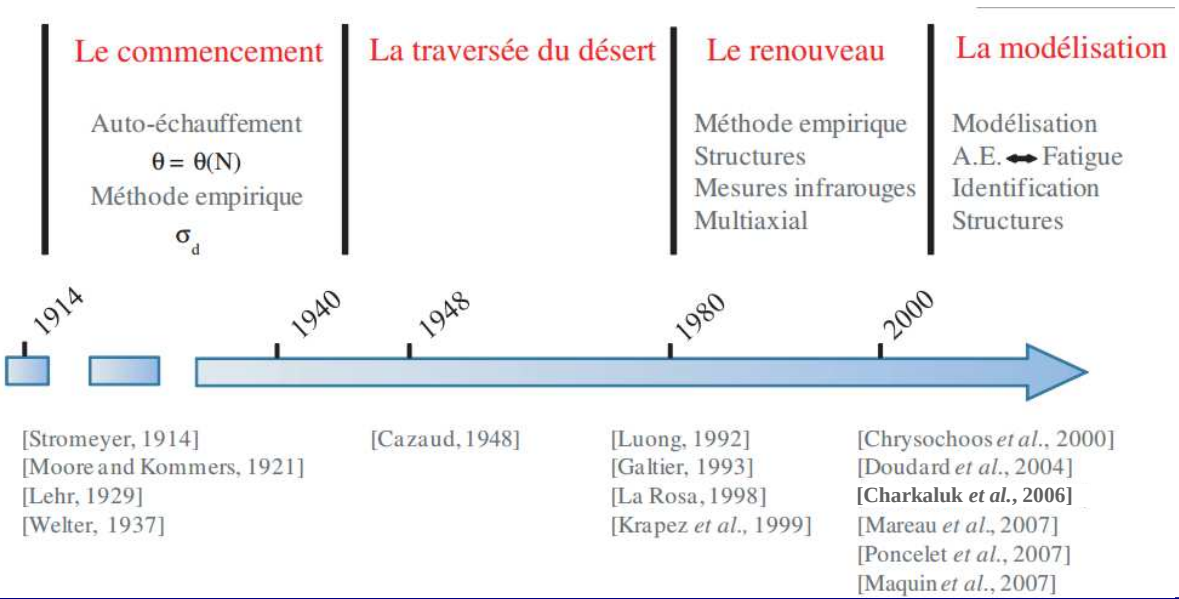
Wöhler Curves | Fatigue initiation criterion | Local strain, stress and energy fields

1. Fatigue approach based on a stabilized cycle
2. Energy based criterion
3. Very fast evaluation

How to evaluate fastly the fatigue properties: Heat build-up protocols



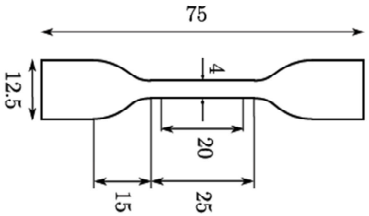
Principle and history of heat build-up tests

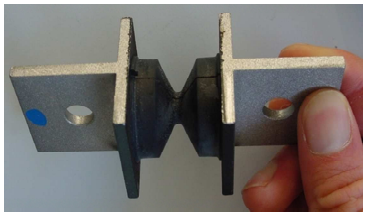


[source: Munier]

How to evaluate fastly the fatigue properties of rubber compounds ???

Characterization on samples

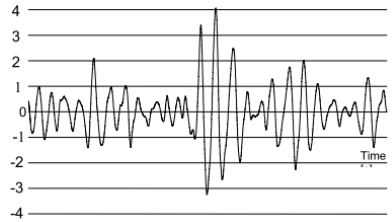


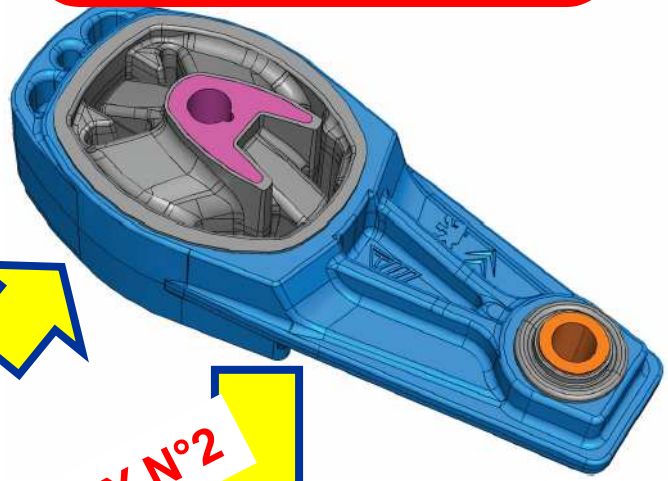


Compounding and Processing

Environment

Thermo-mechanical loading





Complex mechanical tests

Cyclic constitutive law

DIFFICULTY N°2

Wöhler Curves

Fatigue initiation criterion

Local strain, stress and energy fields

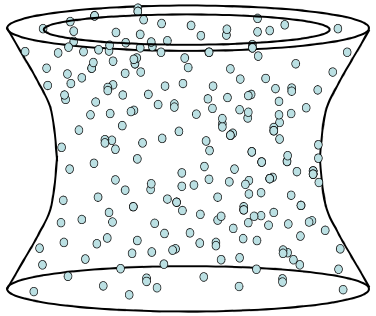
1. **Fatigue approach based on a stabilized cycle**
2. **Energy based criterion**
3. **Very fast evaluation**

How to evaluate fastly the fatigue properties of rubber compounds ???

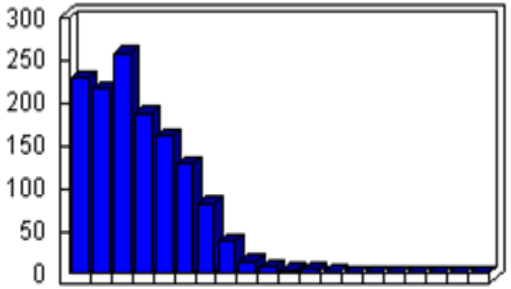
Given compound and process

Given geometry

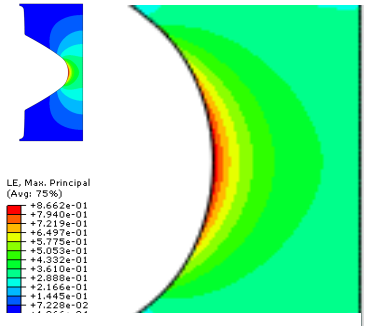
Testing conditions



Potential sites location



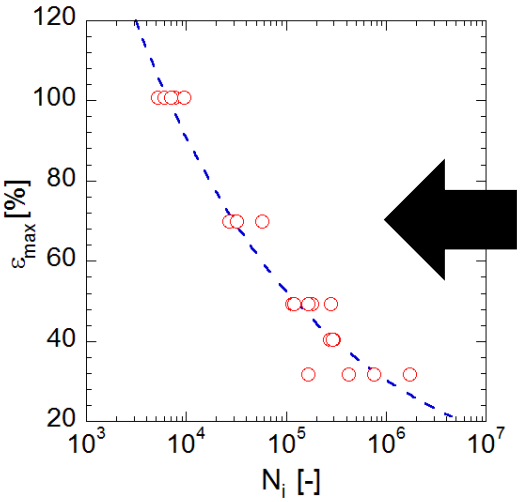
Inclusions size scaterring



Local mechanical loading

Fast evaluation

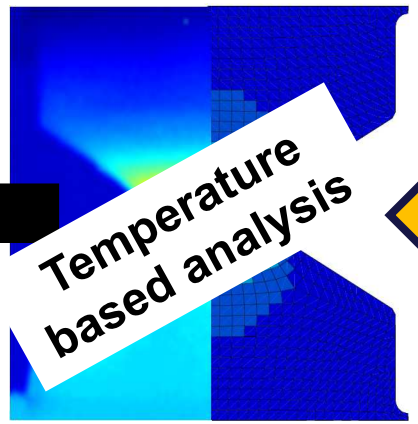
Initiation criterion at a local scale



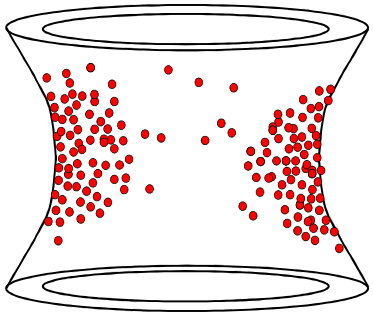
Fatigue properties

$$\Delta^*.N^b = C$$

Energy based fatigue criterion



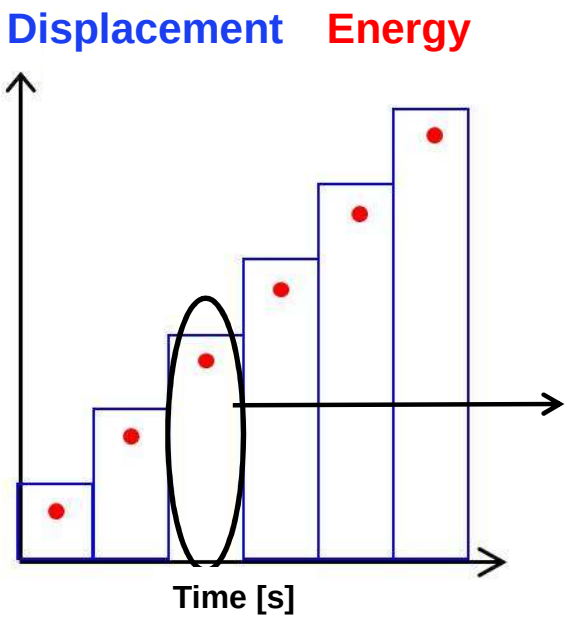
Map of dissipated energy (experimental or numerical)

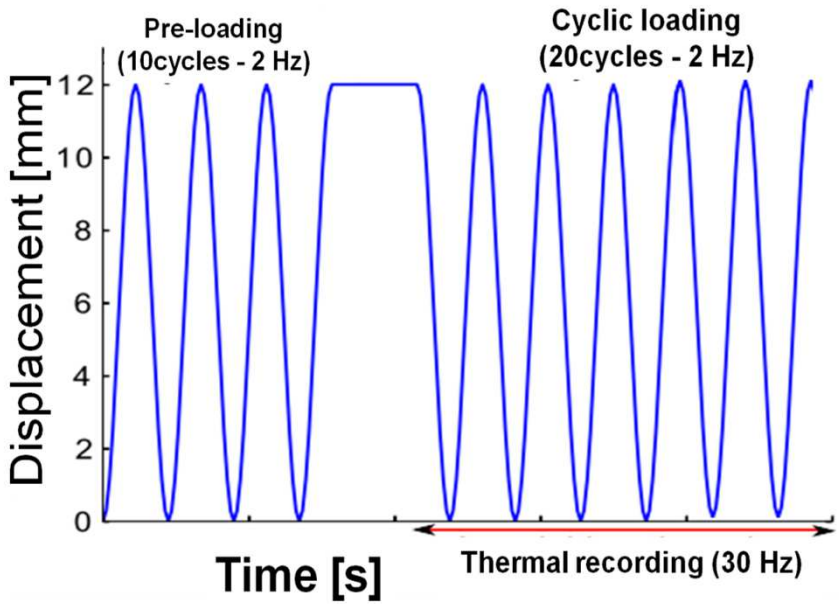


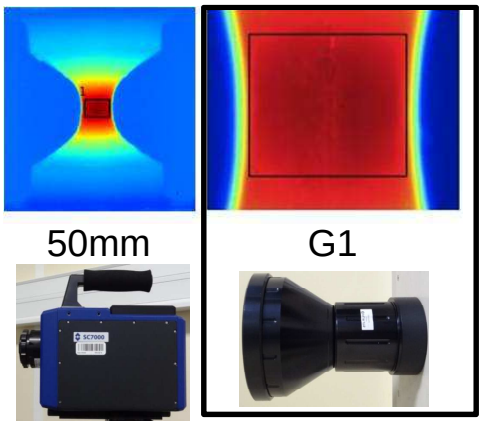
Activated sites locations

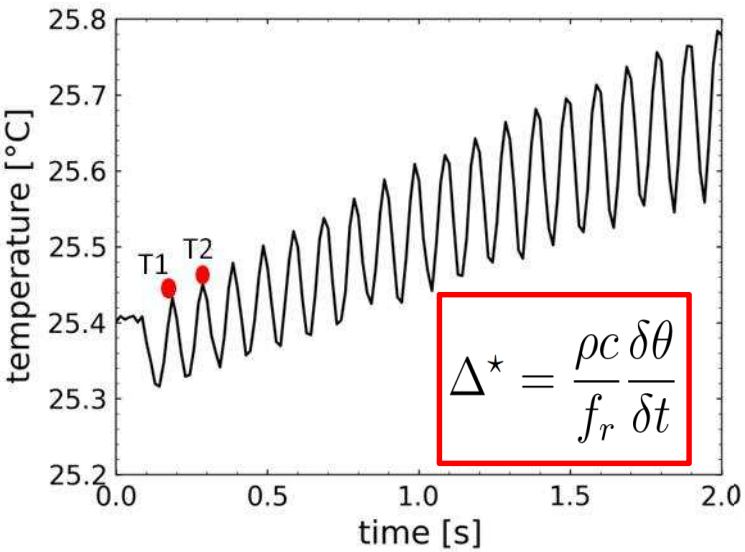
Heat build up protocol

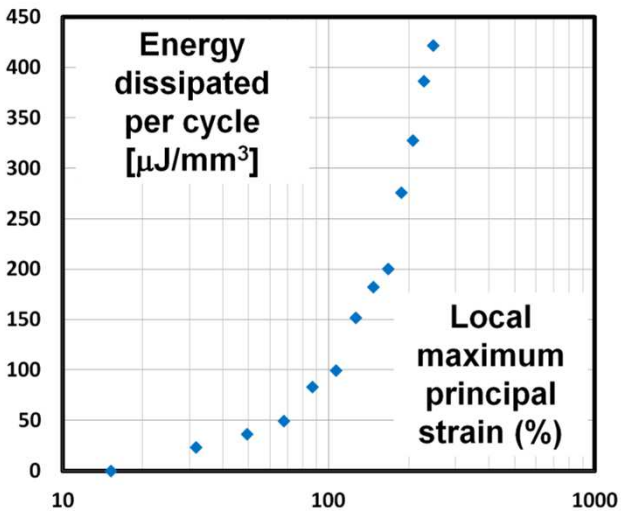
NR 43 CB N550







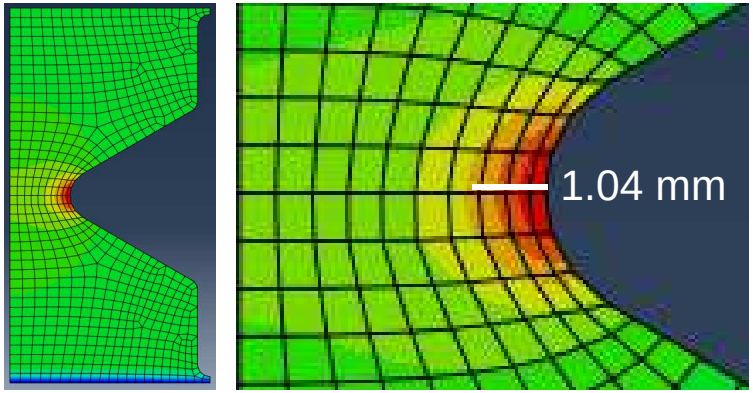


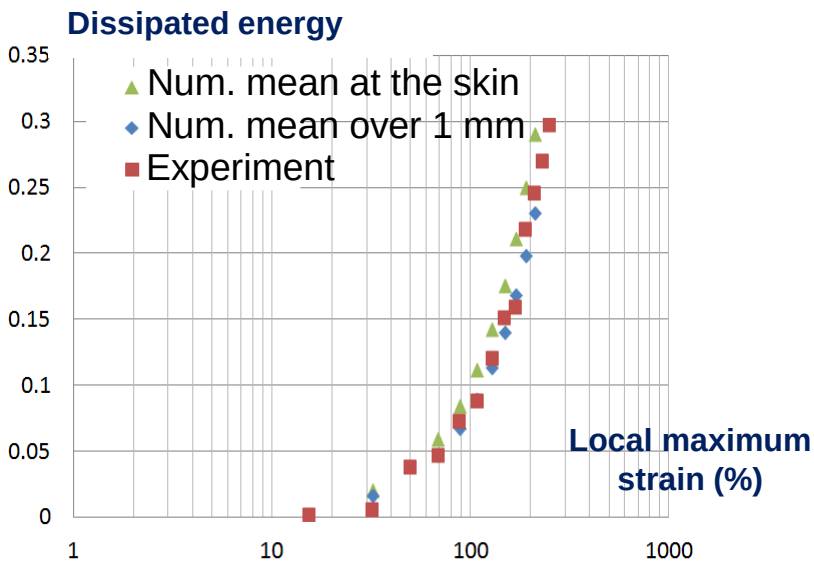


Questions on the heat build-up curve

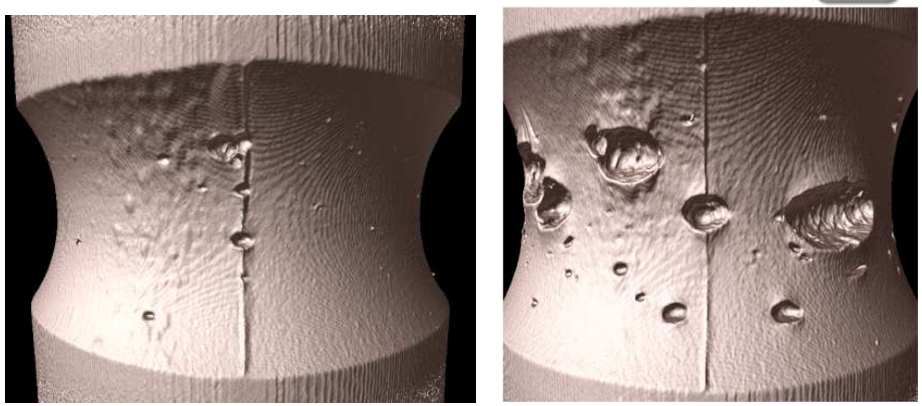
Evaluation over which volume ?

$$e = \sqrt{\frac{\rho C_p}{\lambda \pi f}} = 1.06 \text{ mm at 2Hz}$$

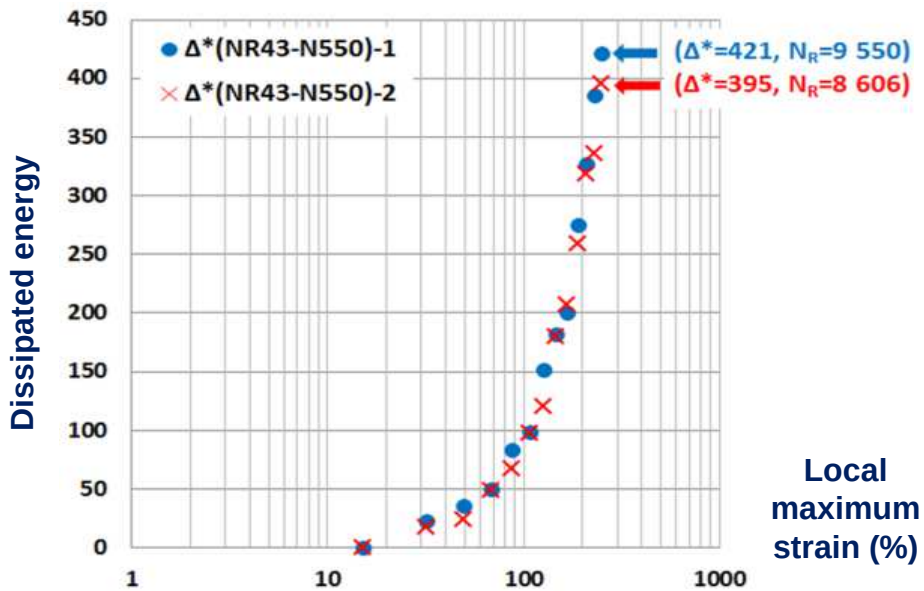




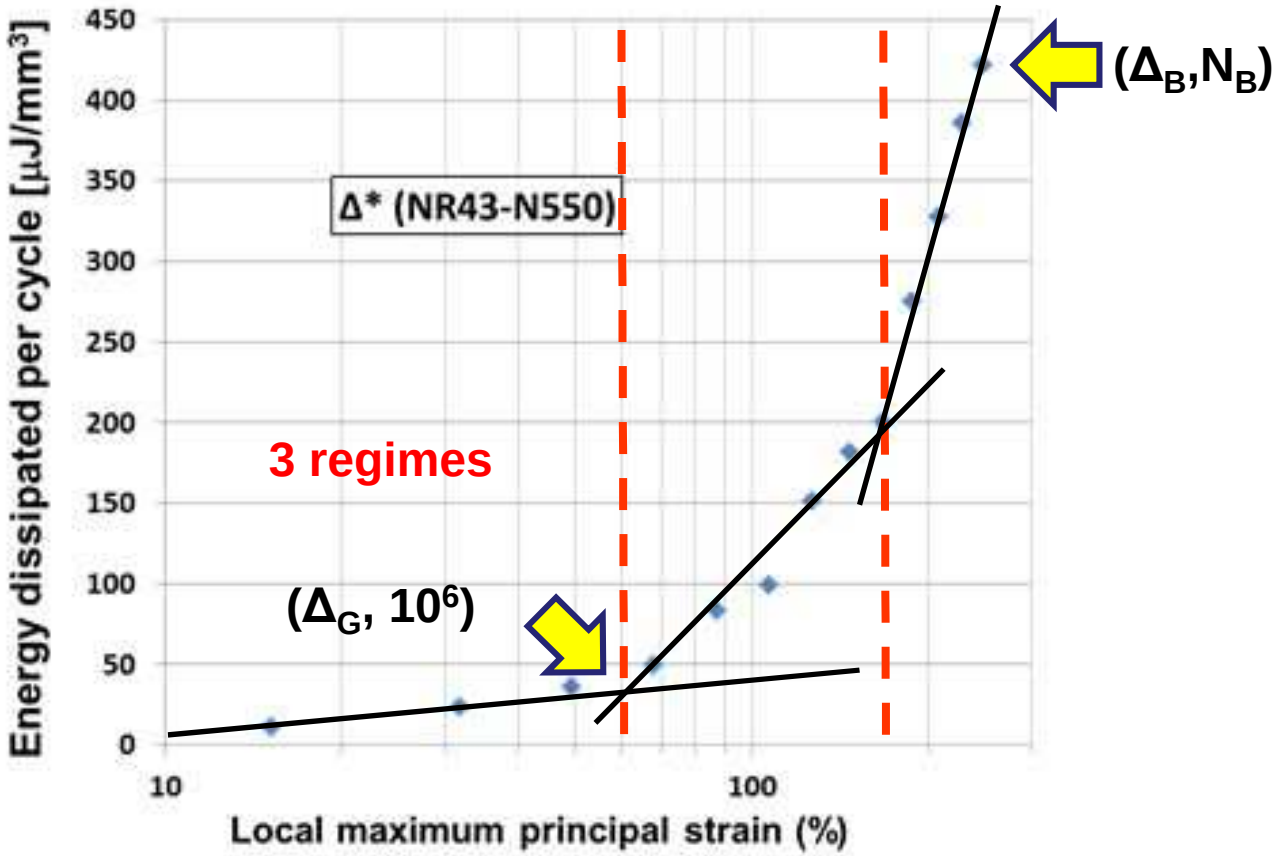
Consistent with damage location ?



Repeatable ?



Fast identification of an energy based criterion



1- First pair
(Δ^*_B,N_B)

Strain / energy assumed to lead to a 10^6 cycles lifetime

2- Second pair
(Δ^*_G,10^6)

Determination from a graphical evaluation

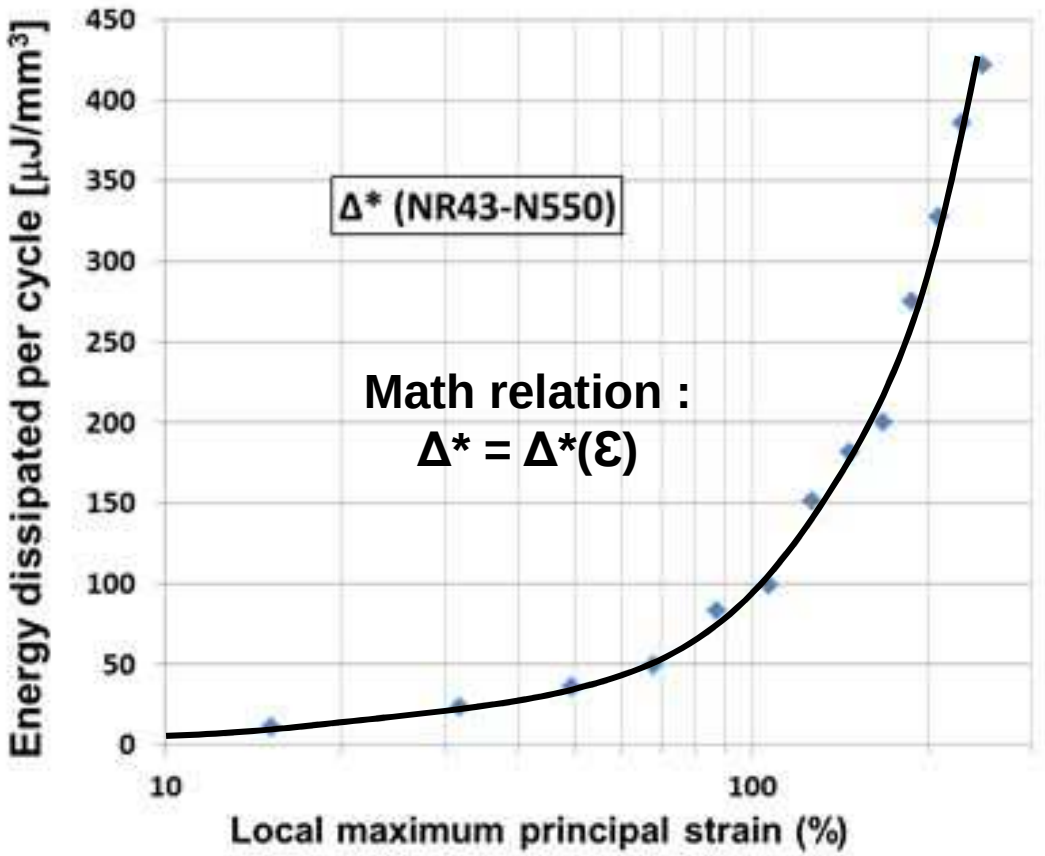
Energy model

$$\Delta^* \cdot N^b = C$$

Two pairs (Δ,N)

Analytical identification of the 2 model parameters
b = 0,467
C = 14437

Fast identification of an energy based criterion



Energy model

$$\Delta^* \cdot N^b = C$$

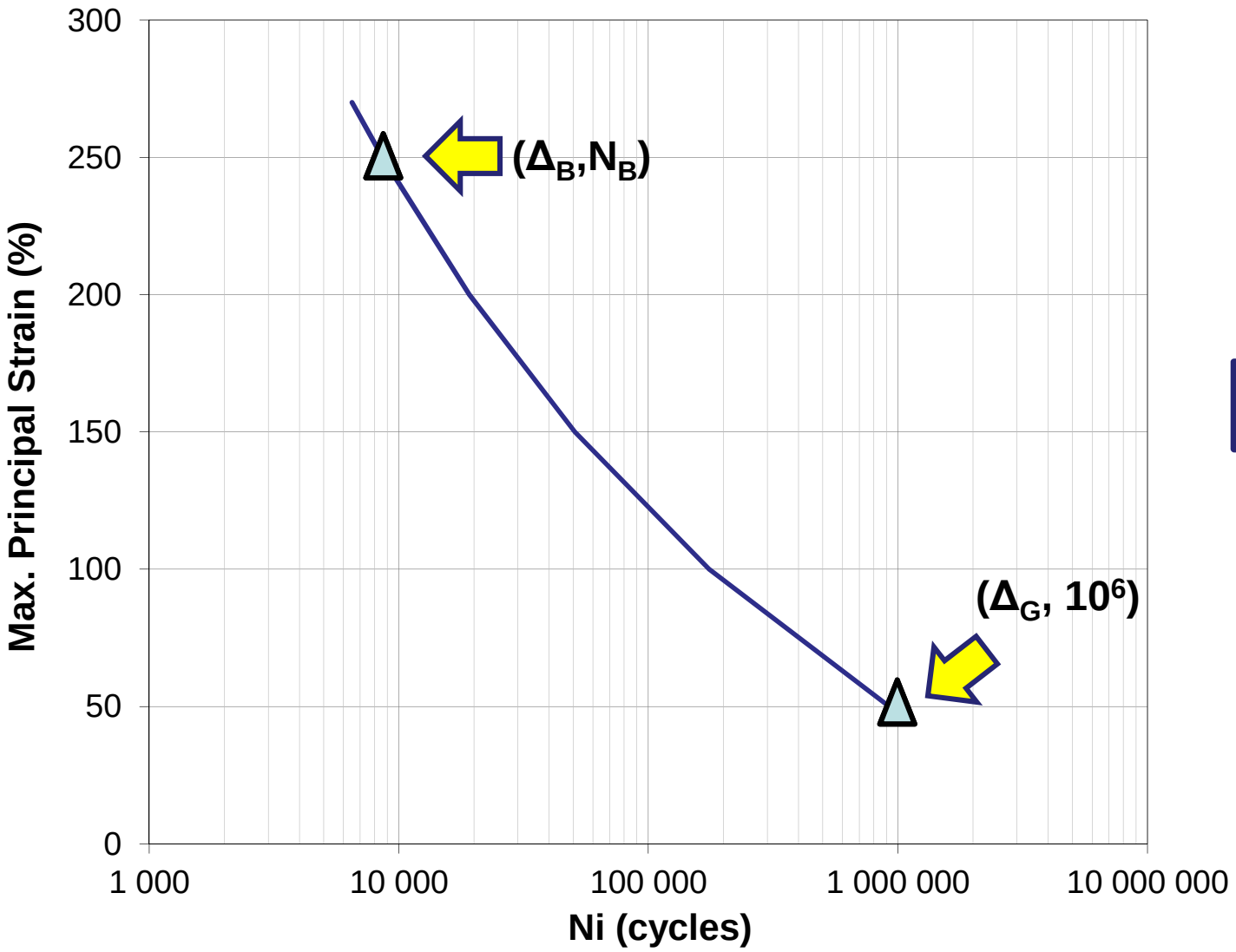
b = 0,467
C = 14437

$$\Delta^*(\varepsilon) \cdot N^b = C$$

$$N = f(\varepsilon)$$

How to evaluate fastly the fatigue properties of rubber compounds ???

Proposal and challenge of an energy-based fatigue criterion



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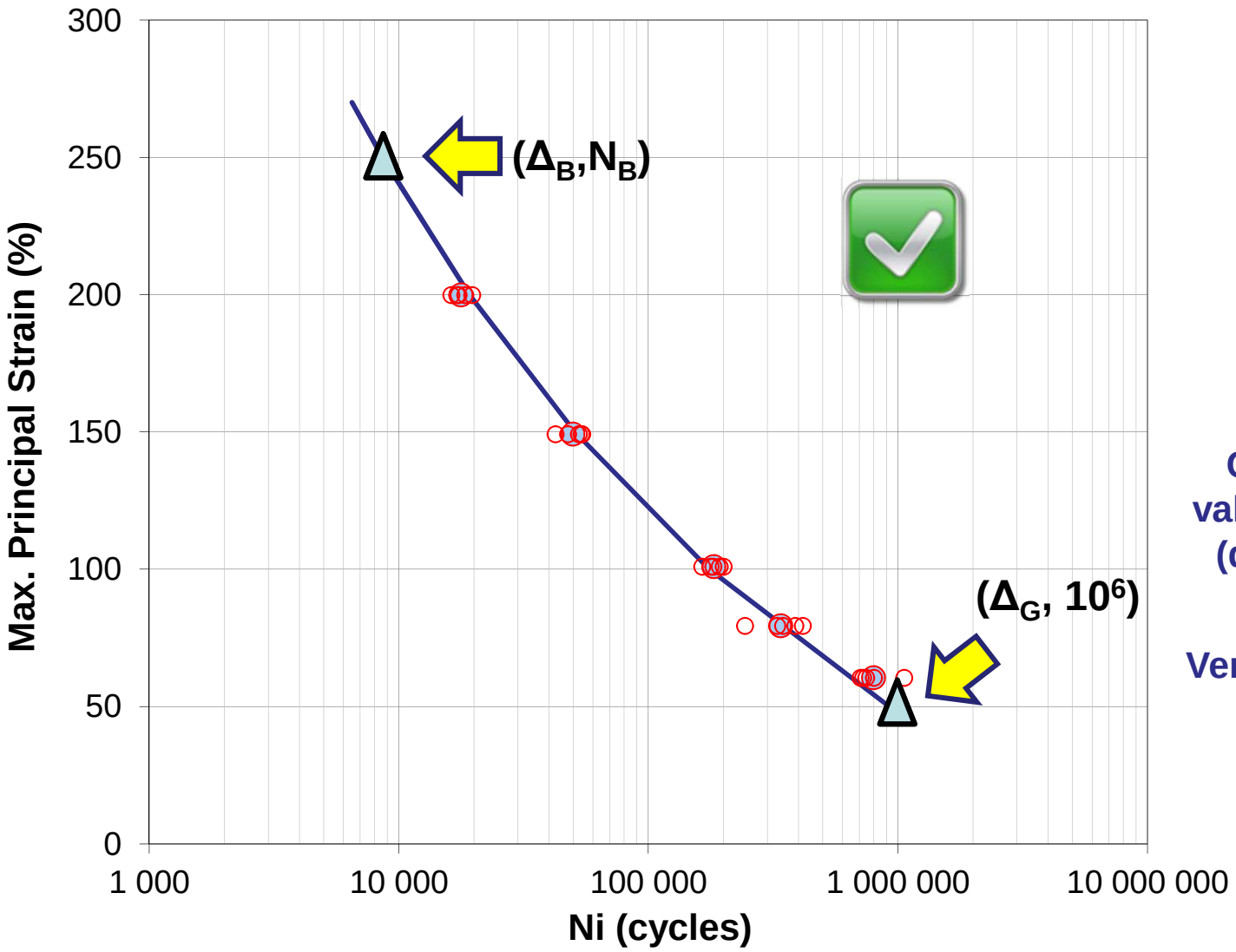


Energy model

$$\Delta^* \cdot N^b = C$$

b = 0,467
C = 14437

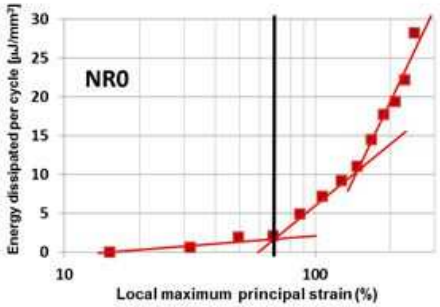
Clear identification, validation on the whole (deterministic) curve satisfying

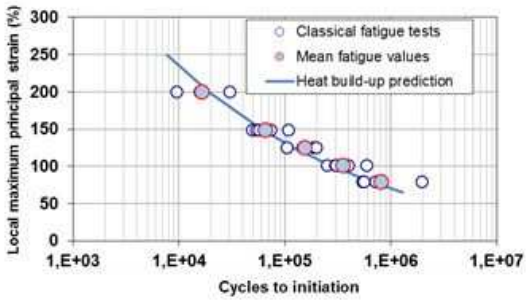
Very quick evaluation of the criterion

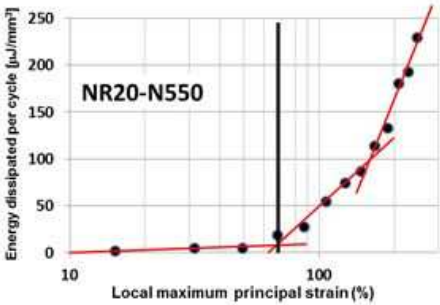
(2 days, 1 sample)

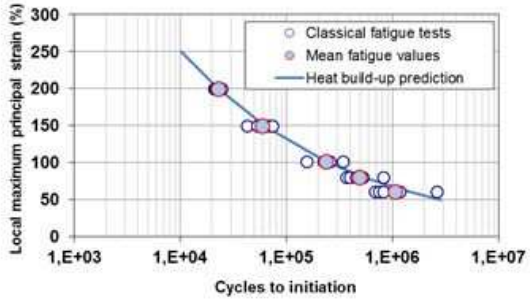
Challenge of the approach on other compounds

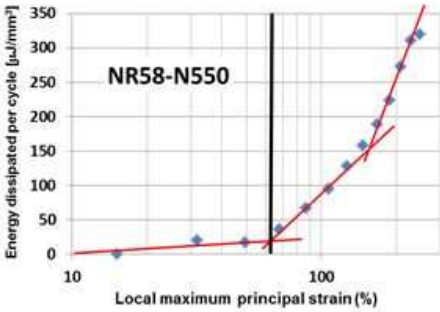
Variation of the fillers ratio:

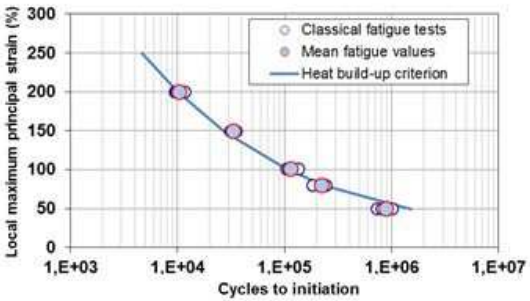




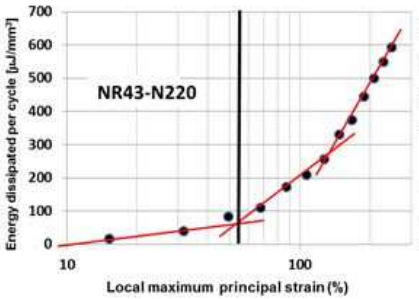


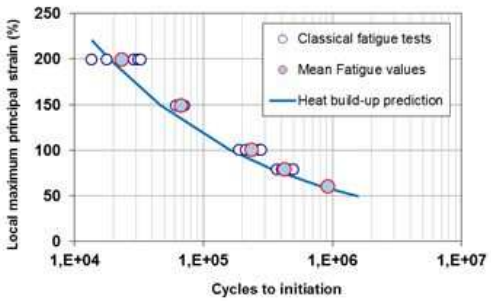


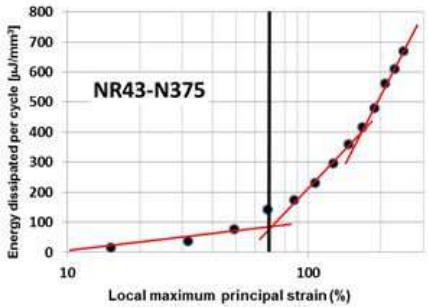


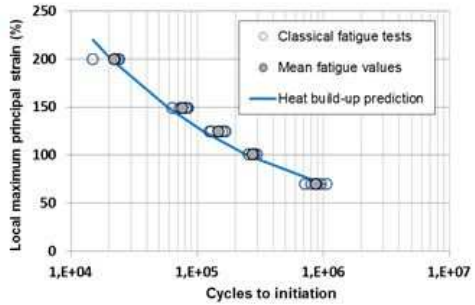


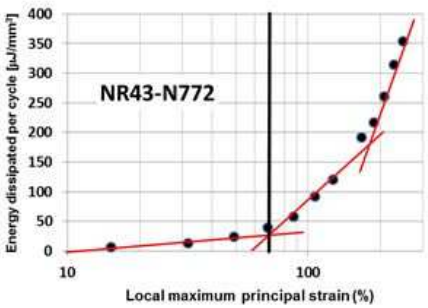
Variation of the fillers type:

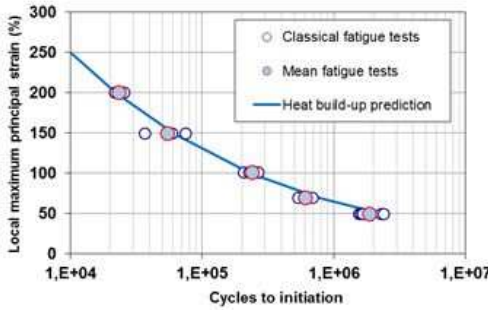






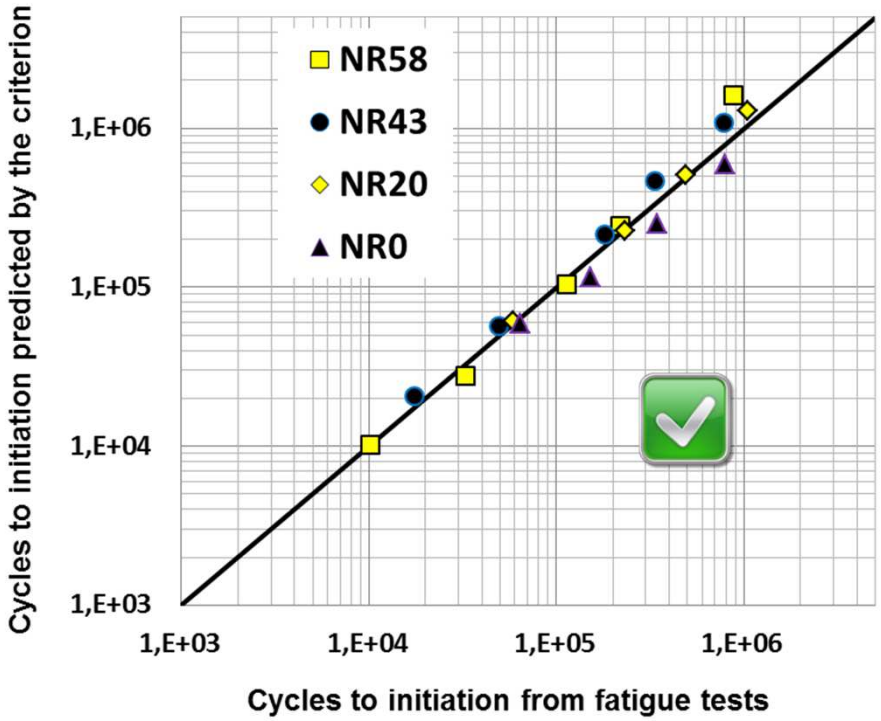


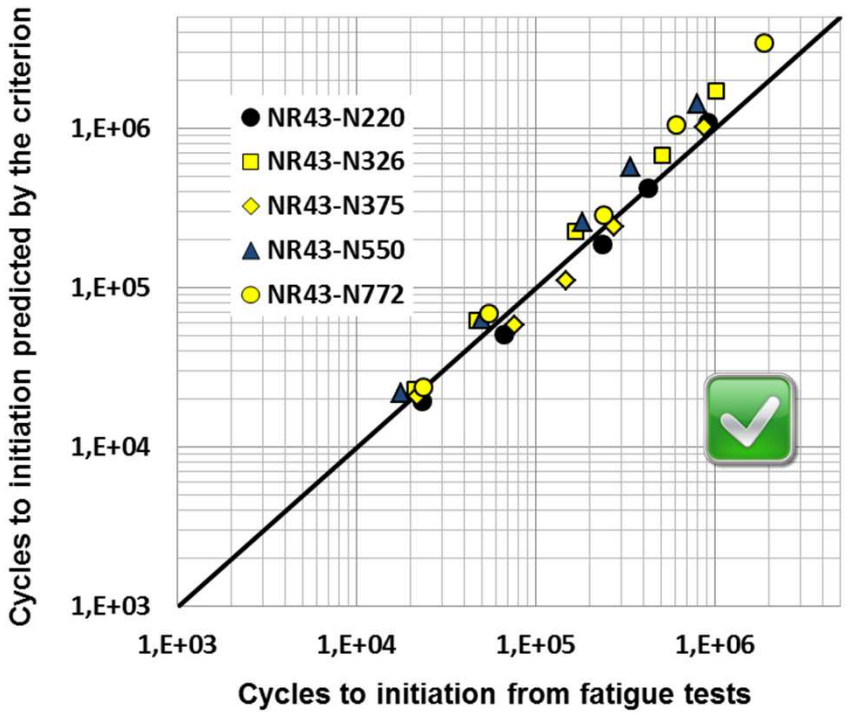




Challenge of the approach on other compounds

Global comparison

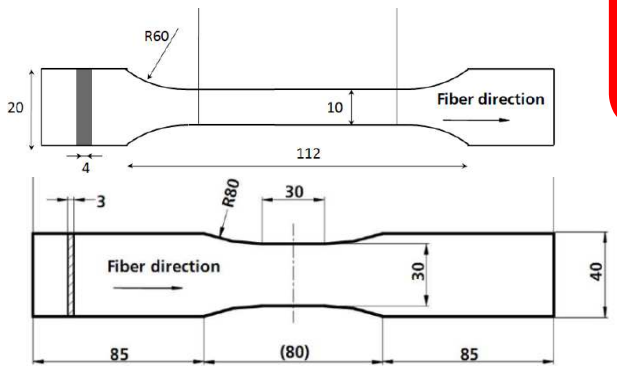




→ **Very fast and efficient prediction of the fatigue curves for various compounds**

How to evaluate fastly the fatigue properties of SFRP ???

Characterization on samples



Complex mechanical tests

Cyclic constitutive law

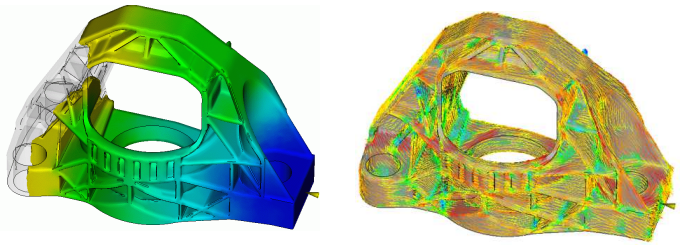
Wöhler Curves

Fatigue initiation criterion

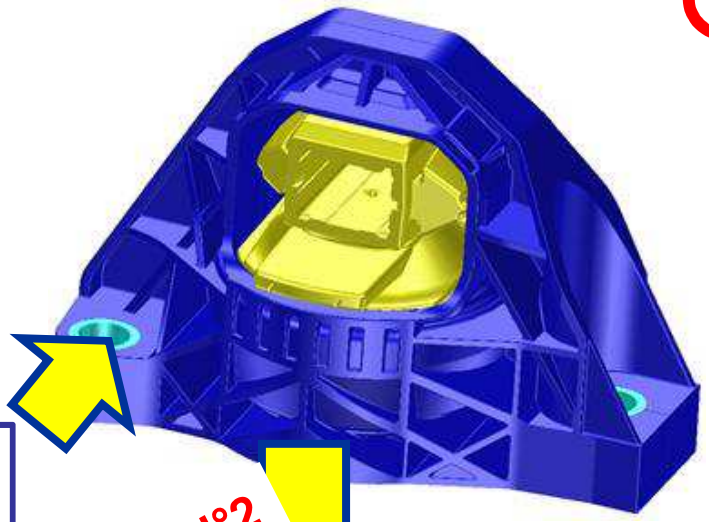
DIFFICULTY N°2

Local strain, stress and energy fields

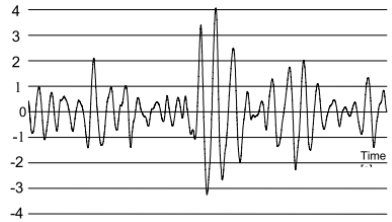
Processing

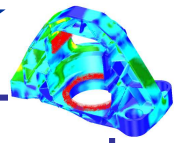


Environment



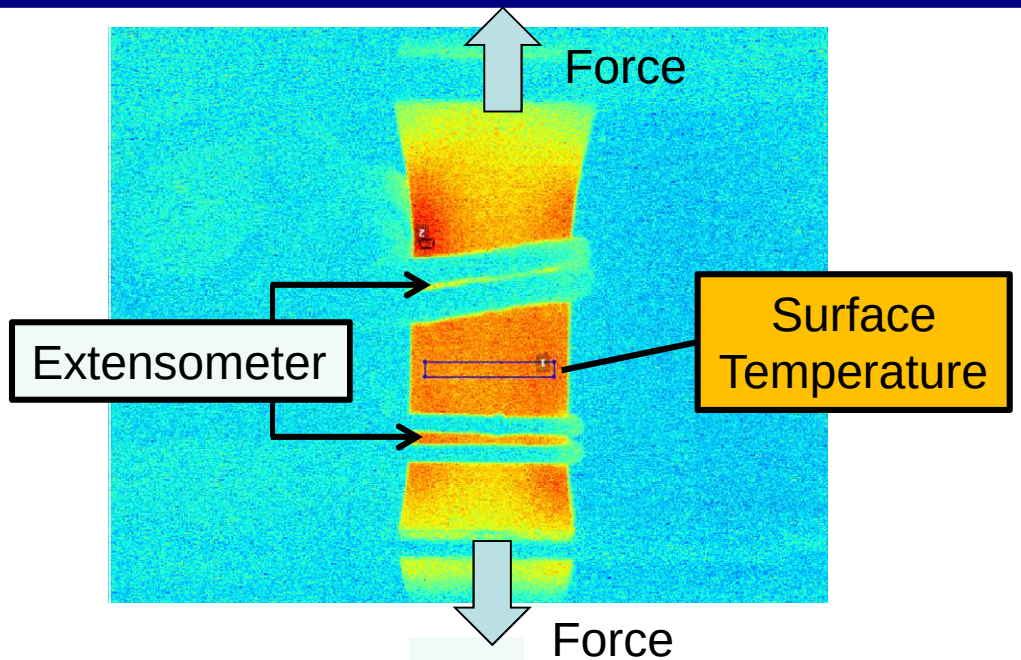
Thermomechanical loading

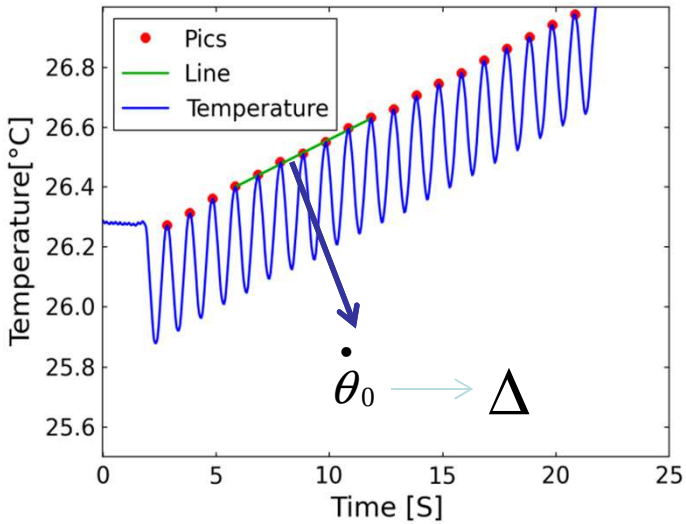




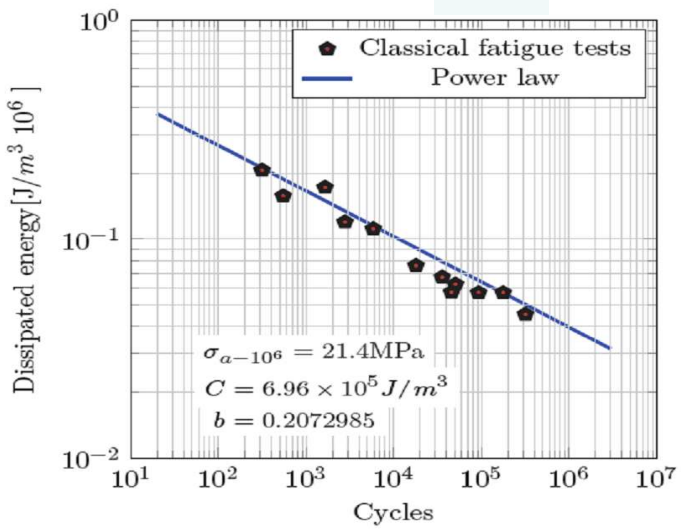
1. **Fatigue approach based on a stabilized cycle**
2. **Energy based criterion**
3. **Very fast evaluation**

Application to SFRP





❑ Mean dissipated energy

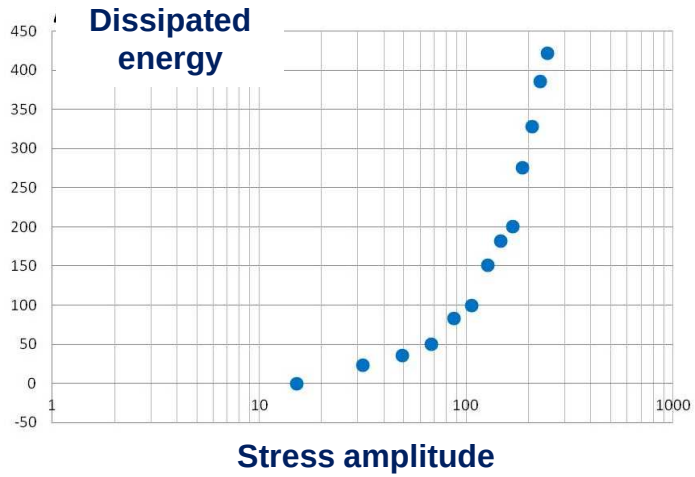


❑ Fatigue lifetime

Dissipated Energy criterion

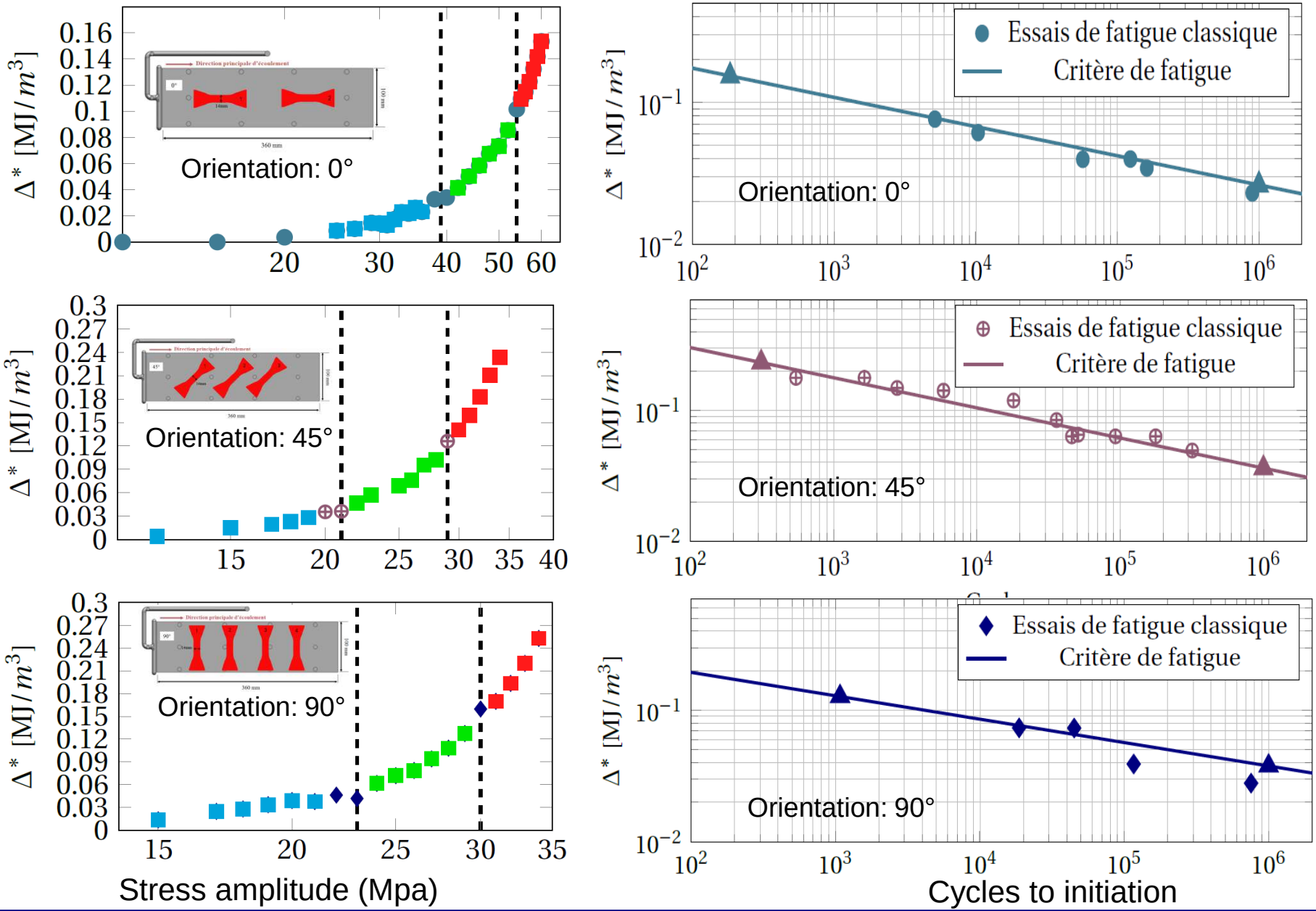
$$\Delta \cdot N^b = C$$

1 sample
1 day



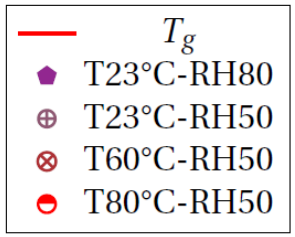
❑ Heat build-up curve

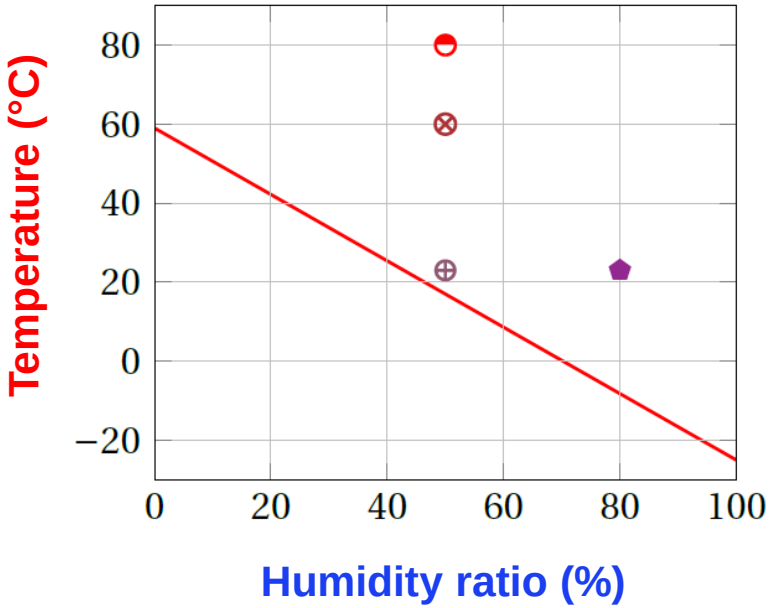
Application to SFRP: Dogbone samples, various orientations

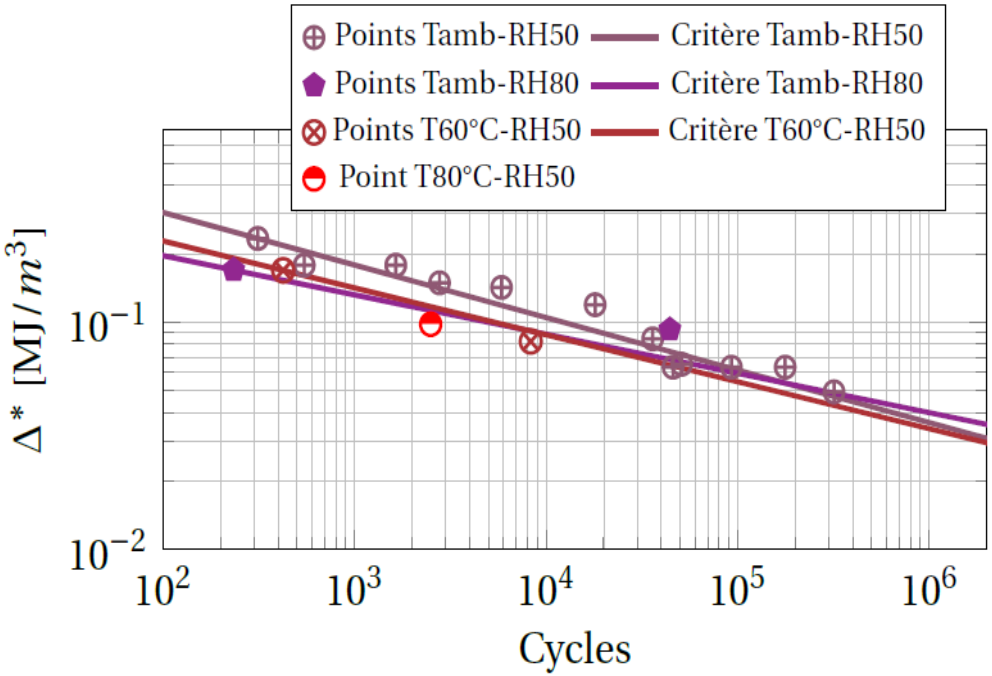


Application to SFRP: Dogbone samples, various environments (T°C, RH)

$T - T_g$ >0







Overview for SFRP

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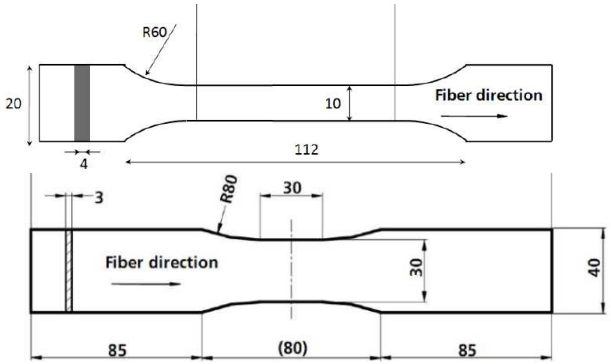
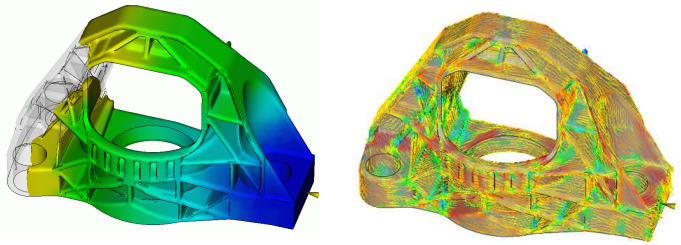
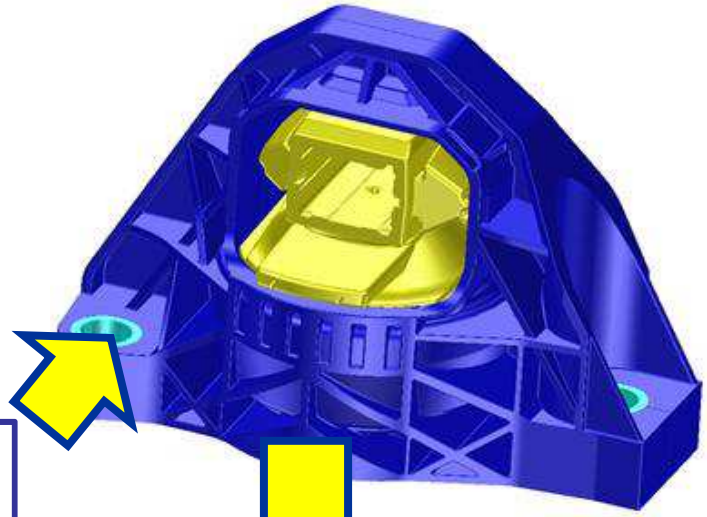


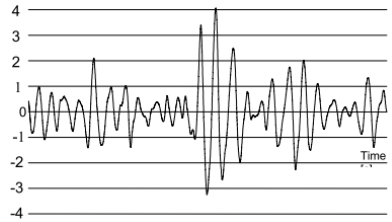
Processing



Environment



Thermomechanical loading



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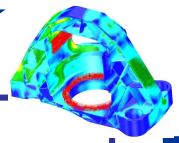
Wöhler Curves

Fatigue initiation criterion

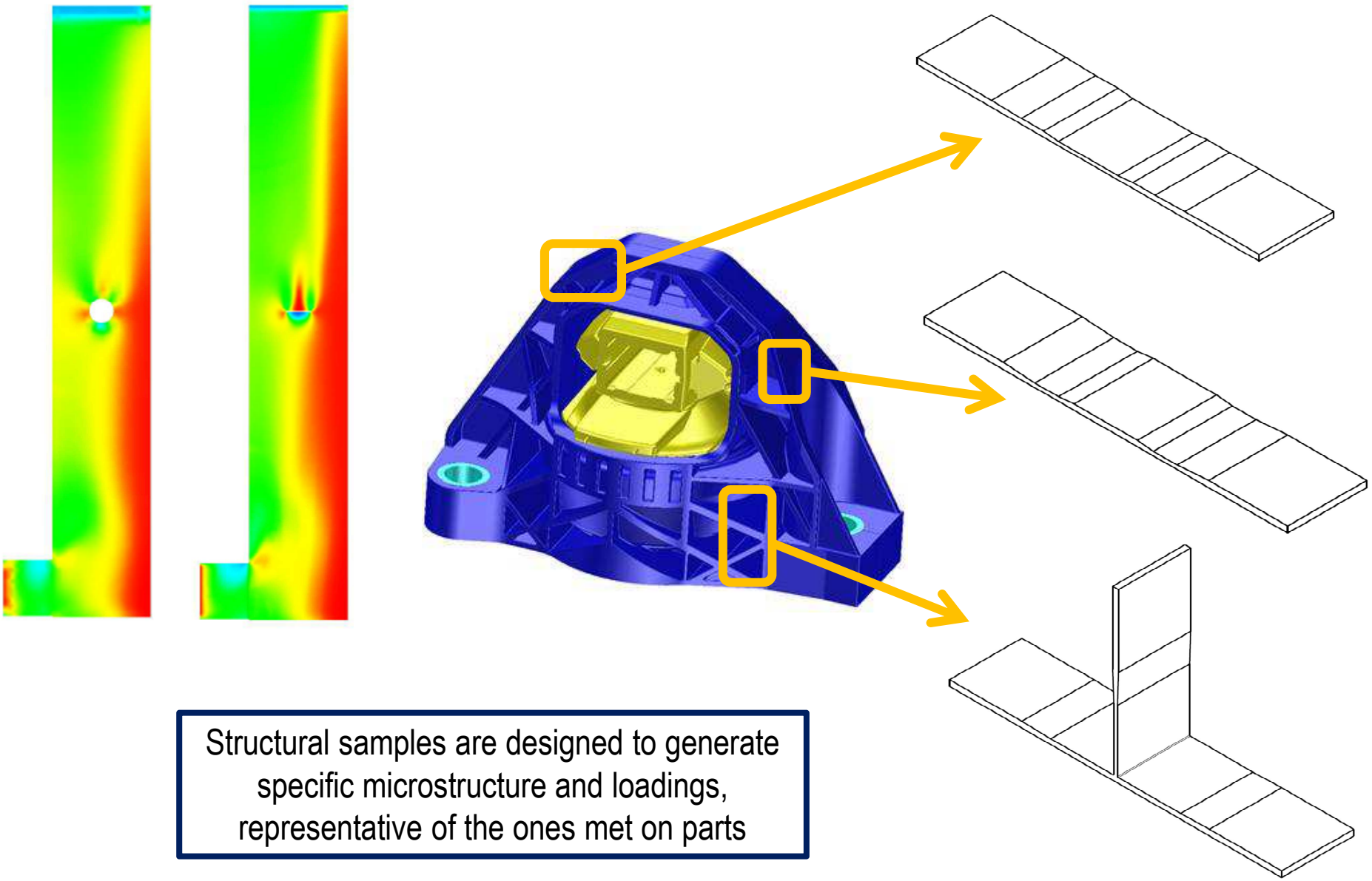


Local strain, stress and energy fields

DIFFICULTY N°3

Validation of the prototype

Application to SFRP: Structural samples and parts

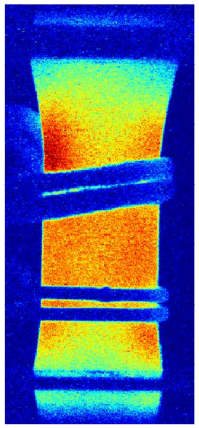


Structural samples are designed to generate specific microstructure and loadings, representative of the ones met on parts

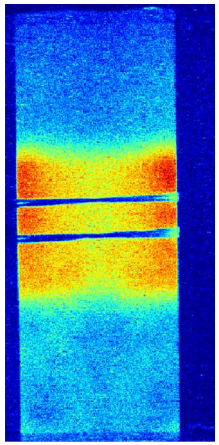
Application to SFRP: Structural samples and parts

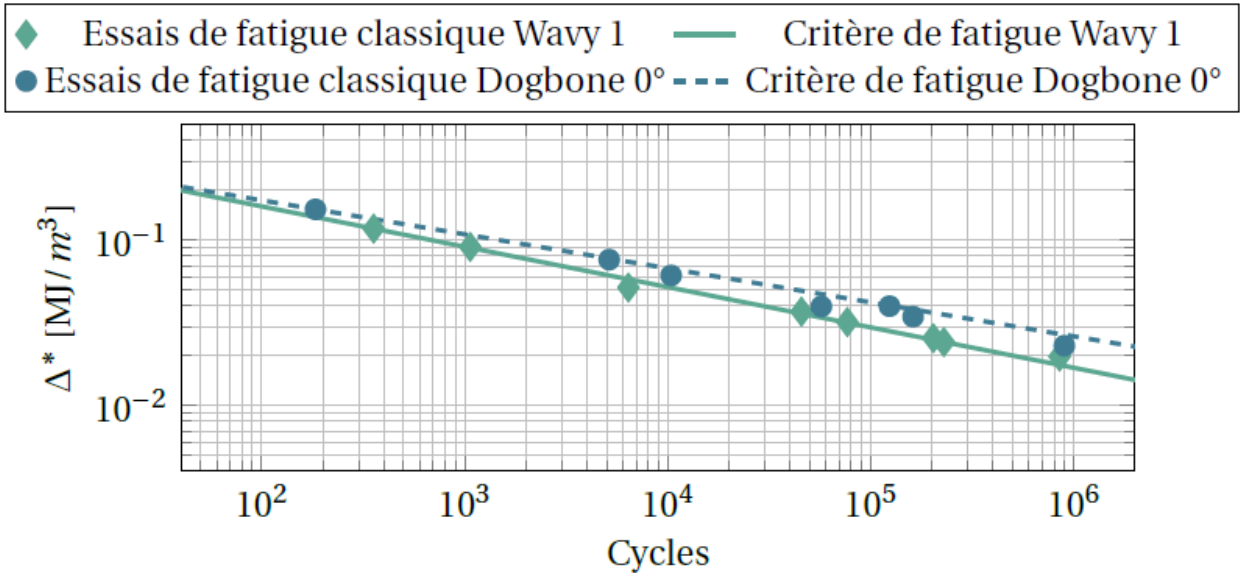
f=1Hz, R=0, RH50, Ambient.

Dogbone 0°

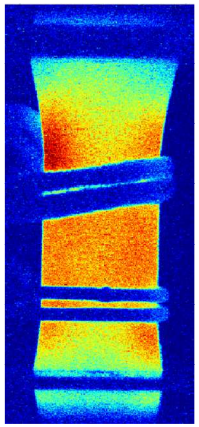


Wavy 1

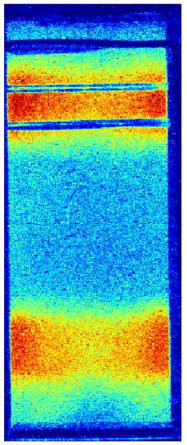


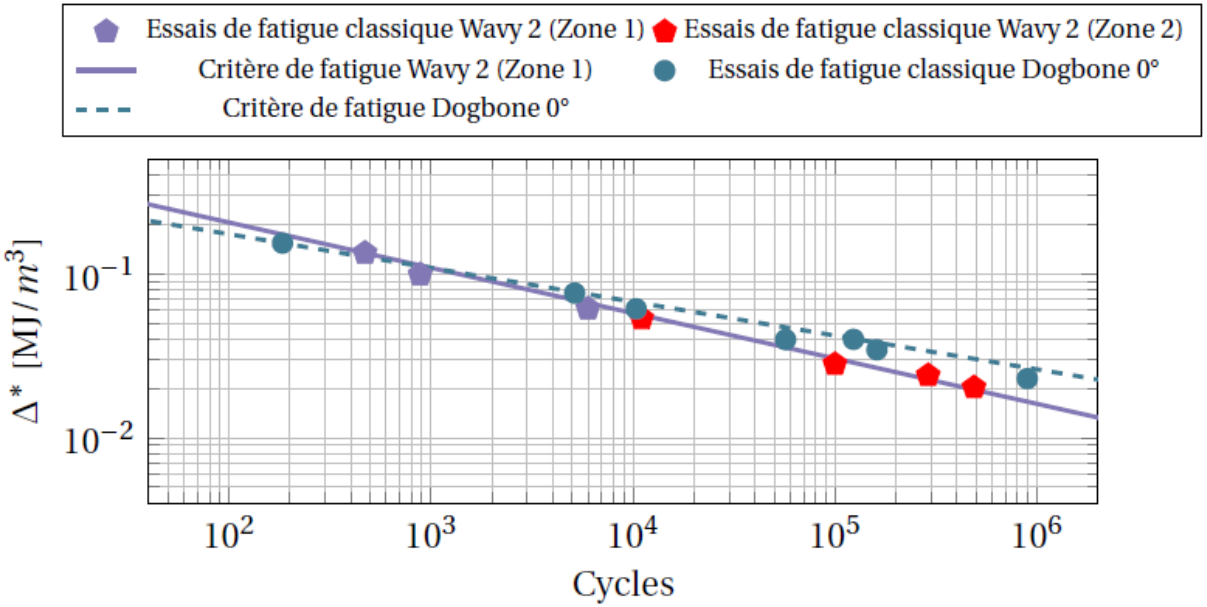


Dogbone 0°



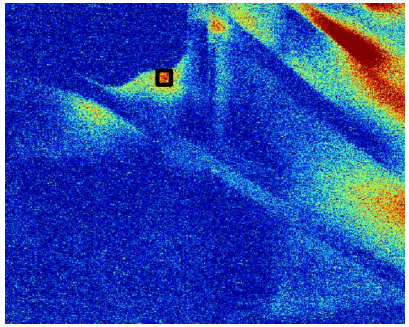
Wavy 2

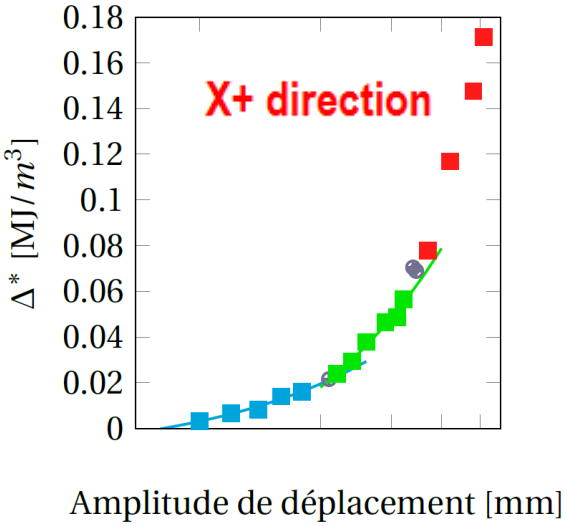


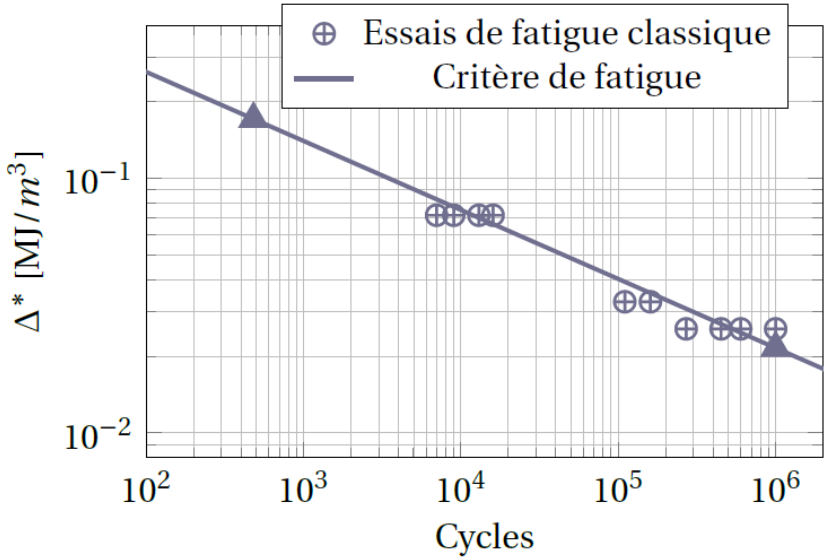


Application to SFRP: Structural samples and parts

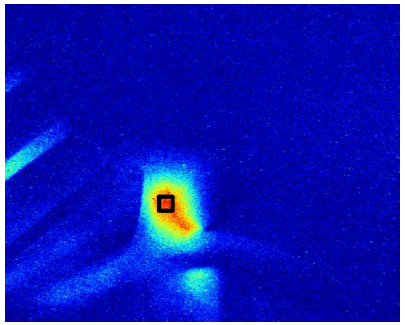
X+ direction

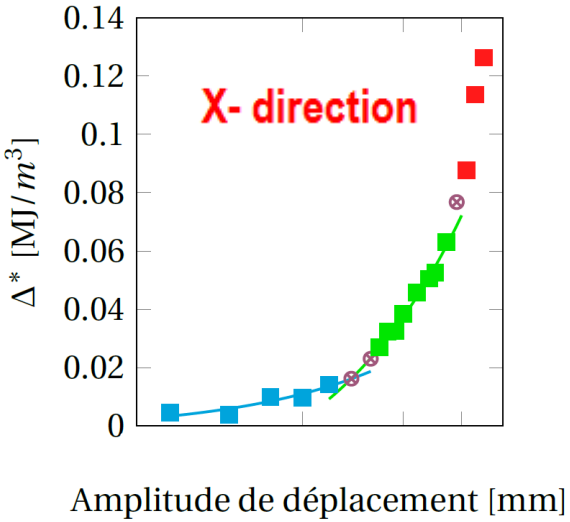


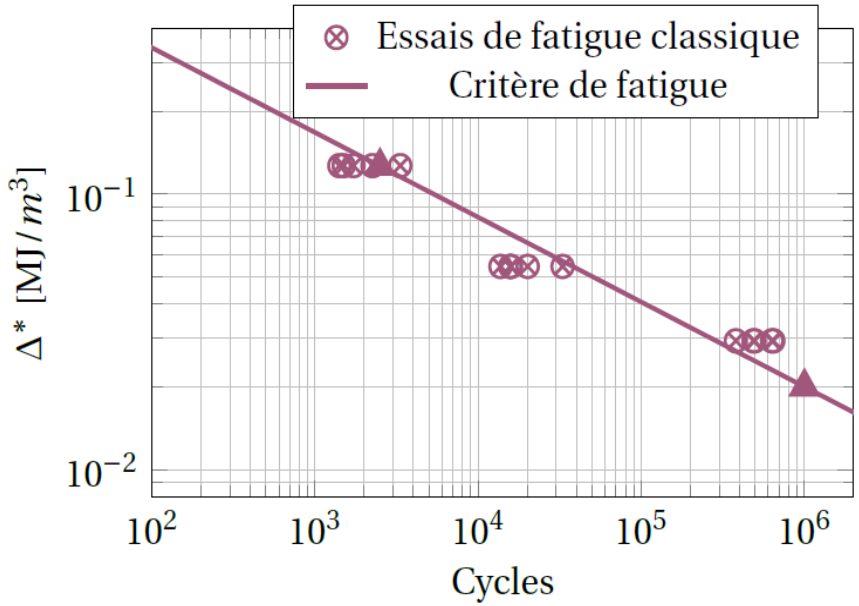




X- direction

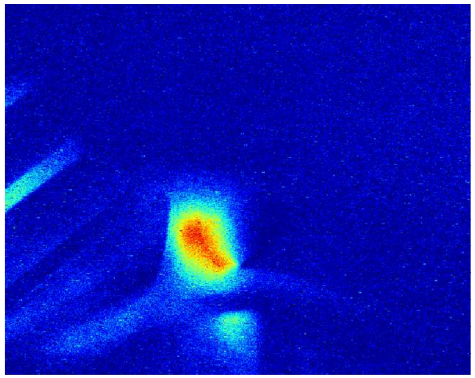




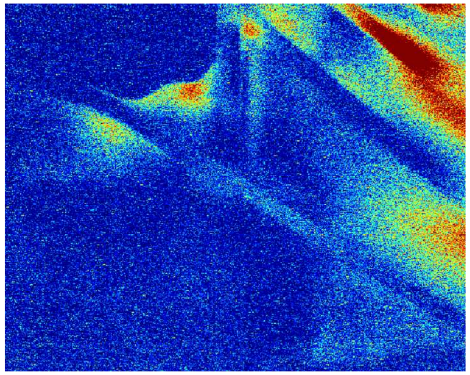


Application to SFRP: Structural samples and parts

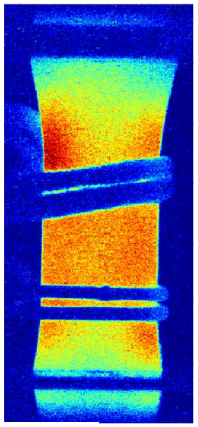
X- direction

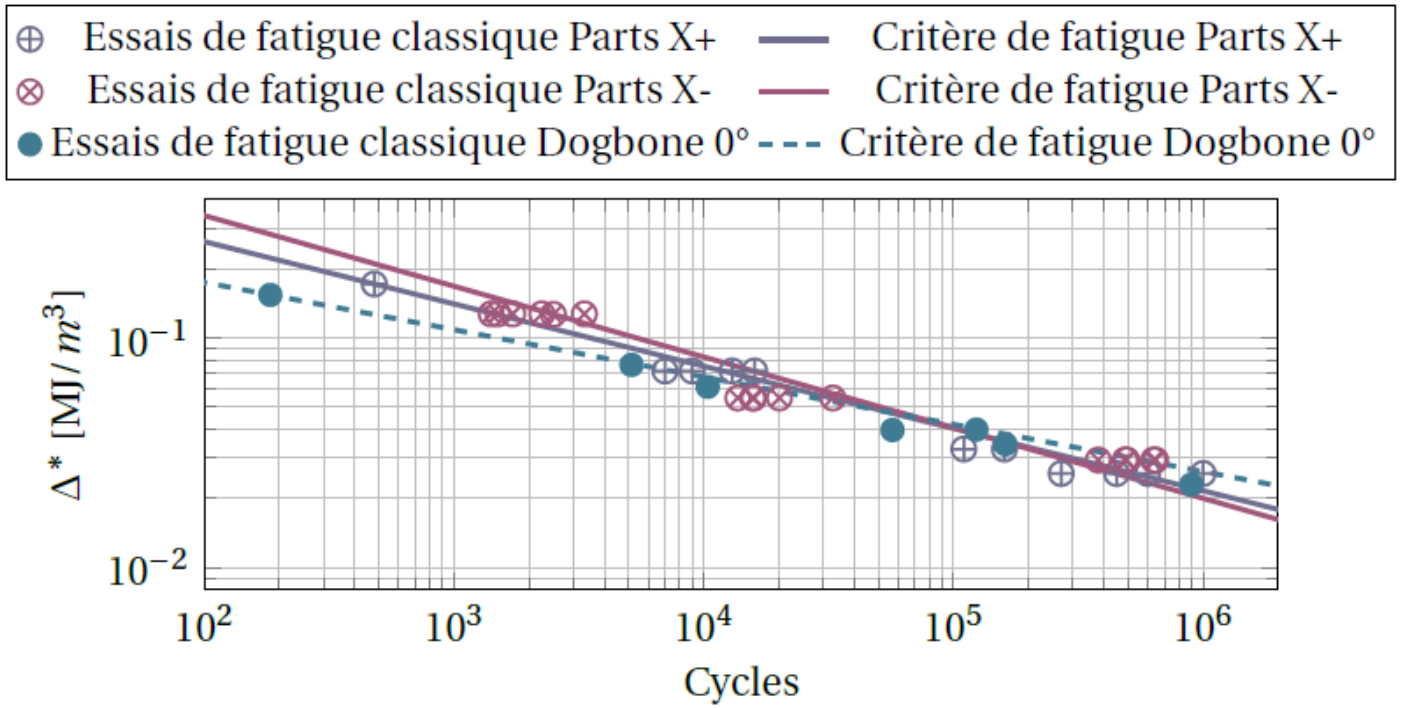


X+ direction



Dogbone 0°

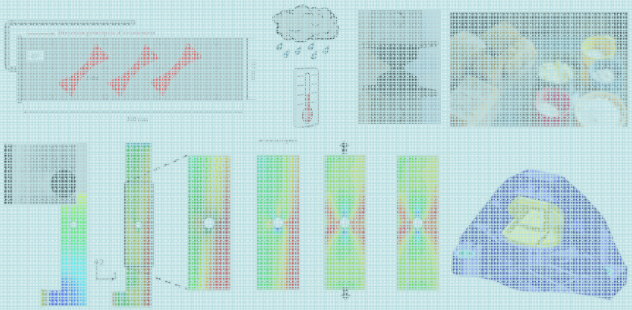




Outline of the presentation

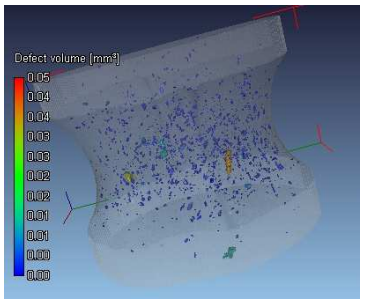
1 Evaluation of dissipation from thermal measurements and heat build-up curve

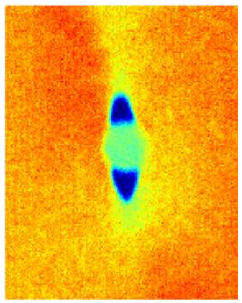
2 Some answers to industrial expectations



- Supply data to feed or validate design computation loops
- Fast screening of fatigue properties for various parameters
- Fast diagnostic on structural samples and parts

3 Questions, limits and tracks to move further





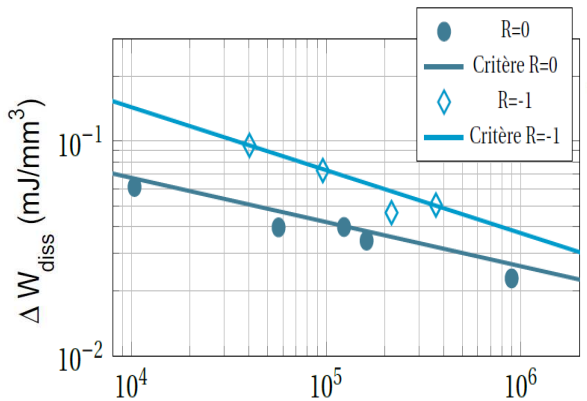
- Illustration of some questions and limits
- Analysis based on constitutive modeling
- Analysis based on the evaluation of defects populations
- Thermomechanical evaluation at the microscopic scale

4 Conclusions

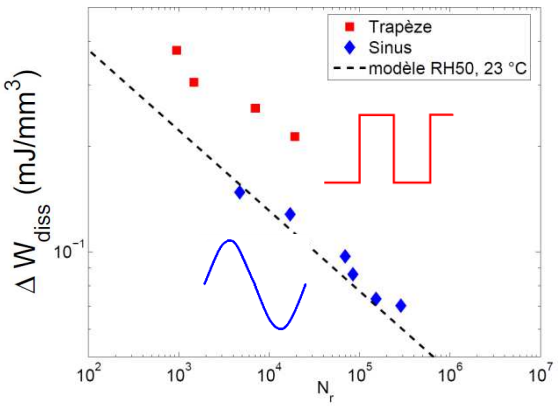
Questions, limits and tracks to move further

1 **Energy based criterion is good but can be improved**

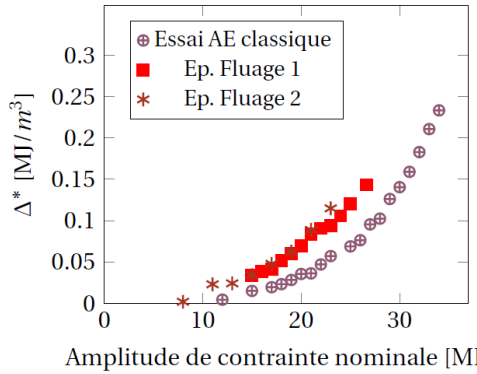
$$(\Delta W_{\text{diss}} + \alpha P_{\max}) \cdot N_a^b = C$$ [Amiable, Launay]

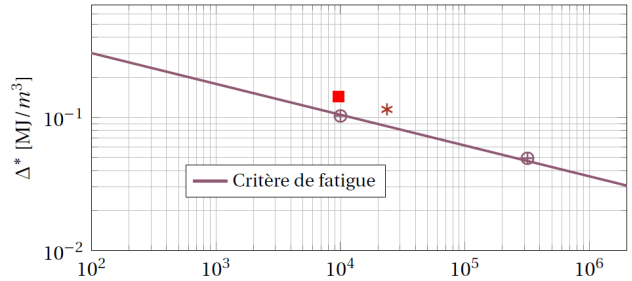


Unification OK for several R> 0, but not if R<0



Dependancy on the loading shape





Dependancy on the loading history (creep+fatigue, here)

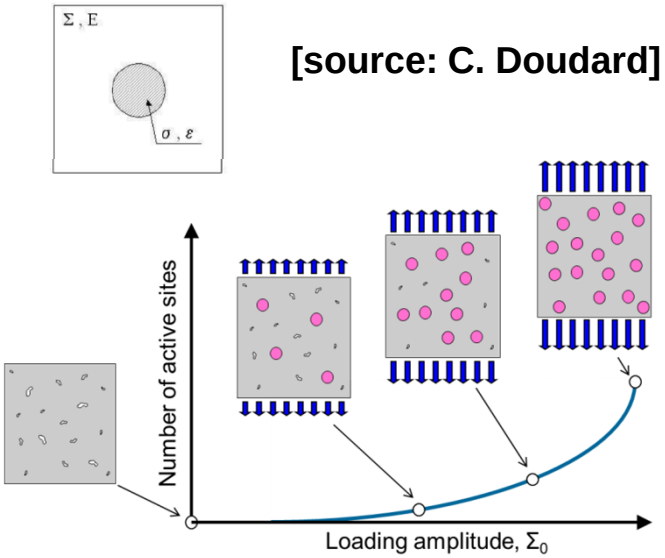
2 **Describe the fatigue scattering**
Improve the prediction of the mean curve

Constitutive models + Probabilistic approach

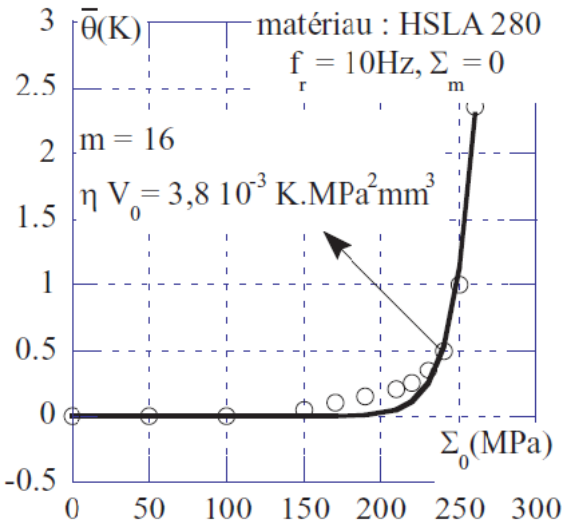
Use microstructural data
Defects population
Link between dissipation and fatigue mechanisms

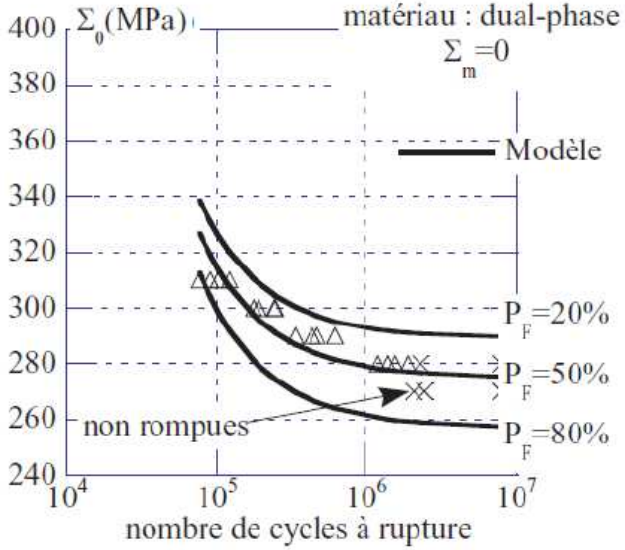
3 **Go beyond a threshold analysis for these materials exhibiting no clear fatigue limit**
Validate the extrapolation over 10^6 cycles

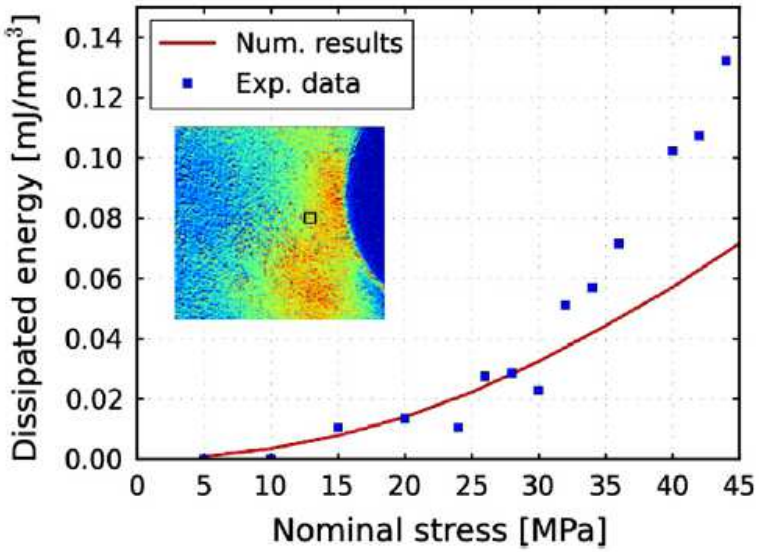
Tracks to move further: Analysis based on constitutive modeling



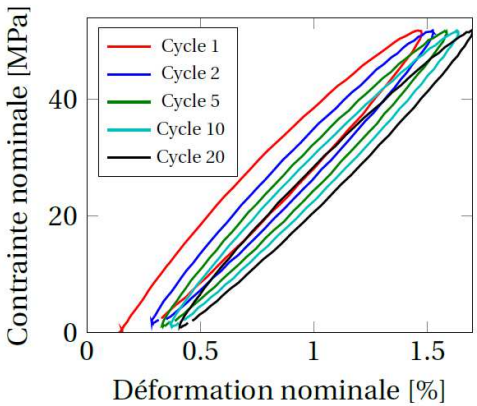
[source: C. Doudard]



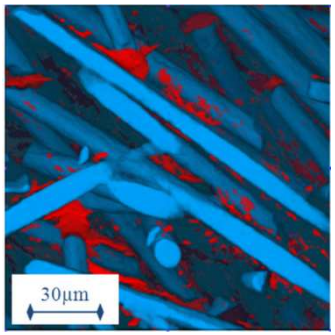




1 **Better description of the hysteresis**



2 **Better link to the damage mechanisms**



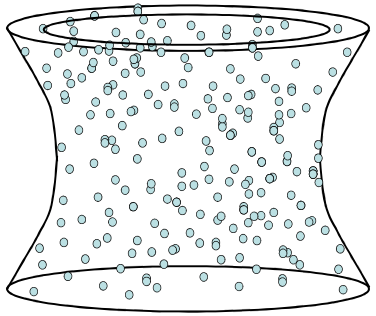
[source: H. Rolland]

Tracks to move further: Analysis based on the evaluation of defects populations

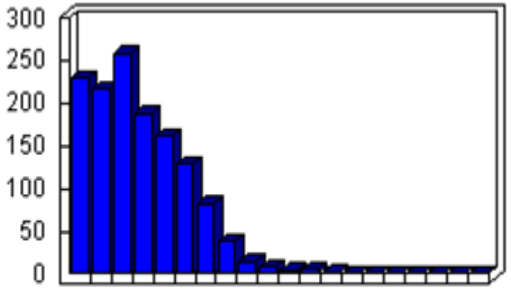
Given compound and process

Given geometry

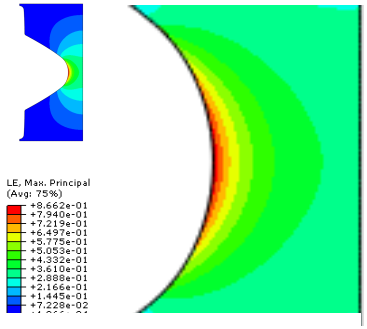
Testing conditions



Potential sites location



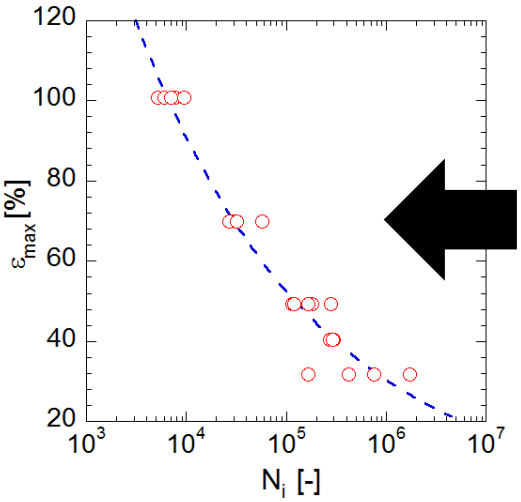
Inclusions size scaterring



Local mechanical loading

Fast evaluation

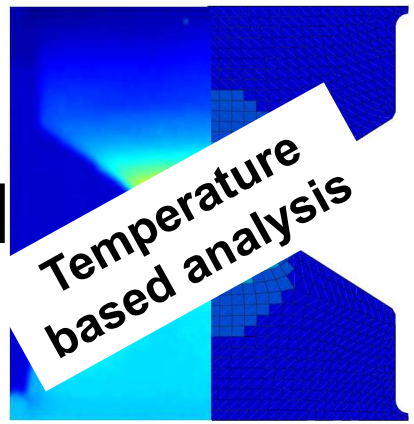
Use microstructural data
Defects population



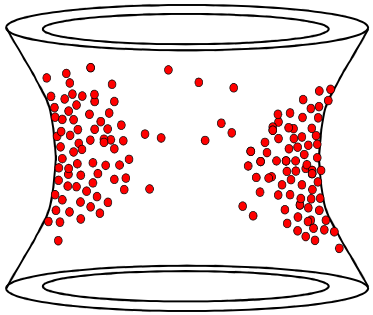
Fatigue properties

$$\Delta^*.N = C$$

Energy based fatigue criterion

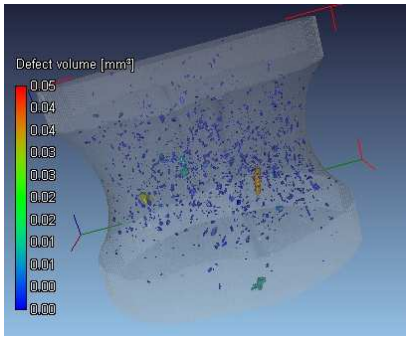


Map of dissipated energy (experimental or numerical)

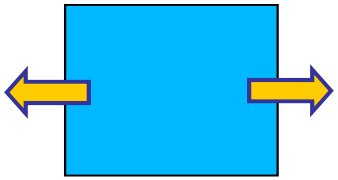


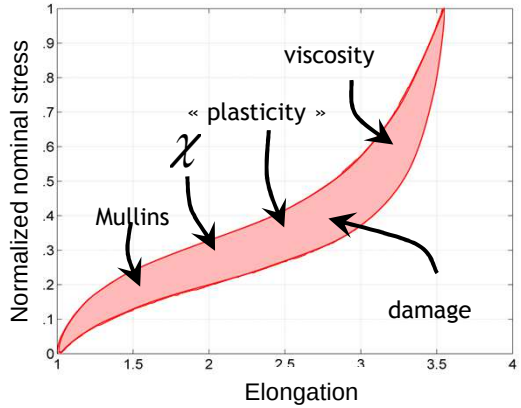
Activated sites locations

Tracks to move further: Analysis based on the evaluation of defects populations

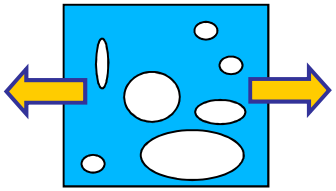


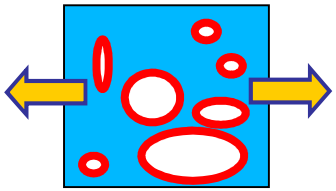
A given volume under a given load





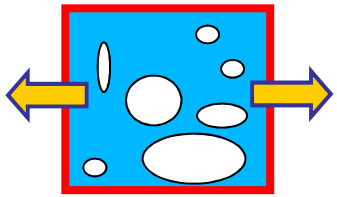
A given defects population under a given load





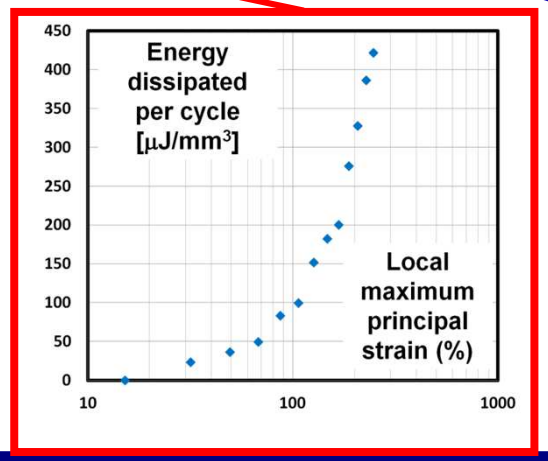
$$E_D = \int_0^{N_i} E_{diss,f}(\varepsilon, N)dN$$

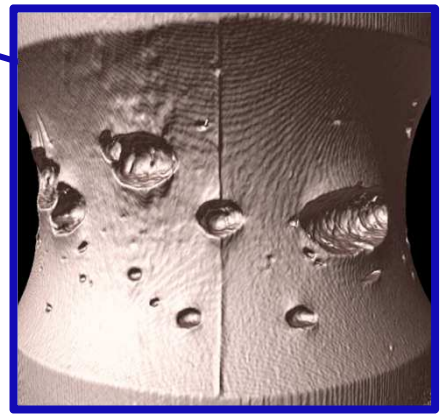
$$E_{diss,f} = \Delta^\star(\varepsilon).\omega_d(\varepsilon, N)$$



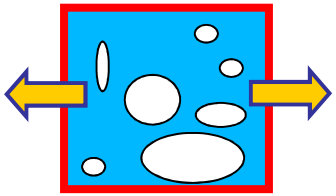
$$E_D = \int_0^{N_i} \Delta^\star(\varepsilon)\,\omega_d(\varepsilon, N)dN$$

$$\cancel{E_{fatigue,diss/cycle} = \sum_{defects} E^d_{diss/cycle}}$$



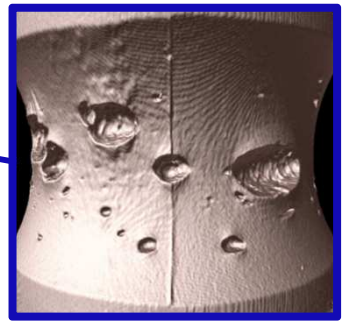


Tracks to move further: Analysis based on the evaluation of defects populations

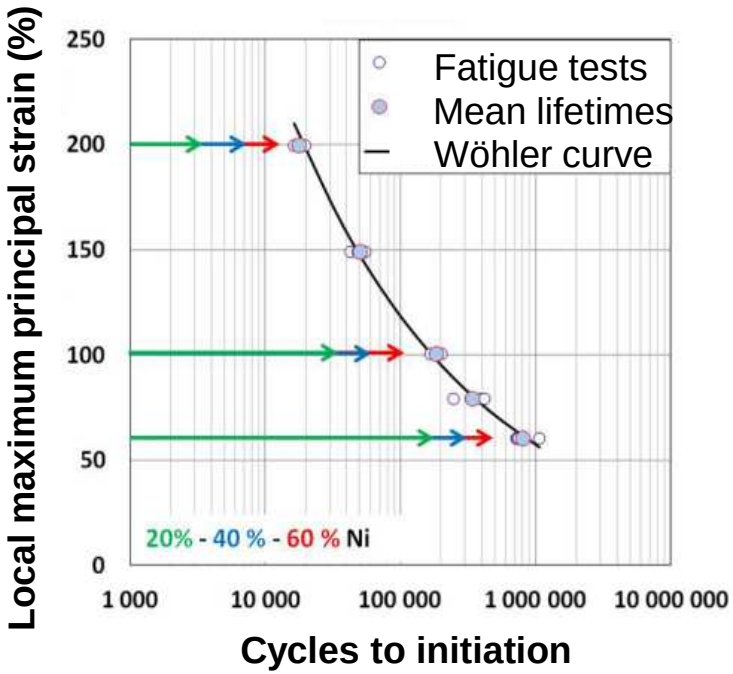


$$E_D = \int_0^{N_i} \Delta^\star(\varepsilon)\, \omega_d(\varepsilon, N)\, dN$$

Surfacic defects density



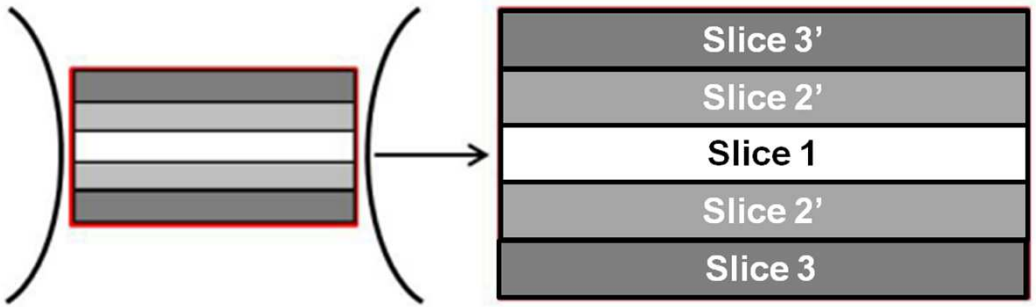
Interrupted fatigue tests



SEM analysis for several zones

3 percentages of life time
3 level of displacement } 9 samples

For each sample, 3 local max. principal strain studied

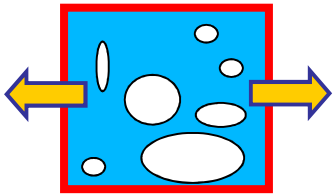


Evaluation of the defects surfacic density for Areas 1, (2, 2′), (3, 3′)

Vibracoustic

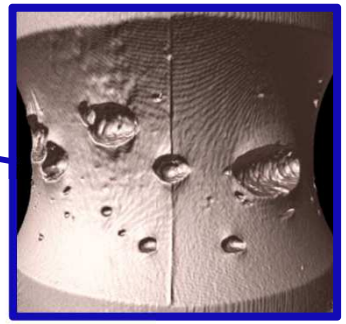
GeM Institut de Recherche en Génie Civil et Mécanique

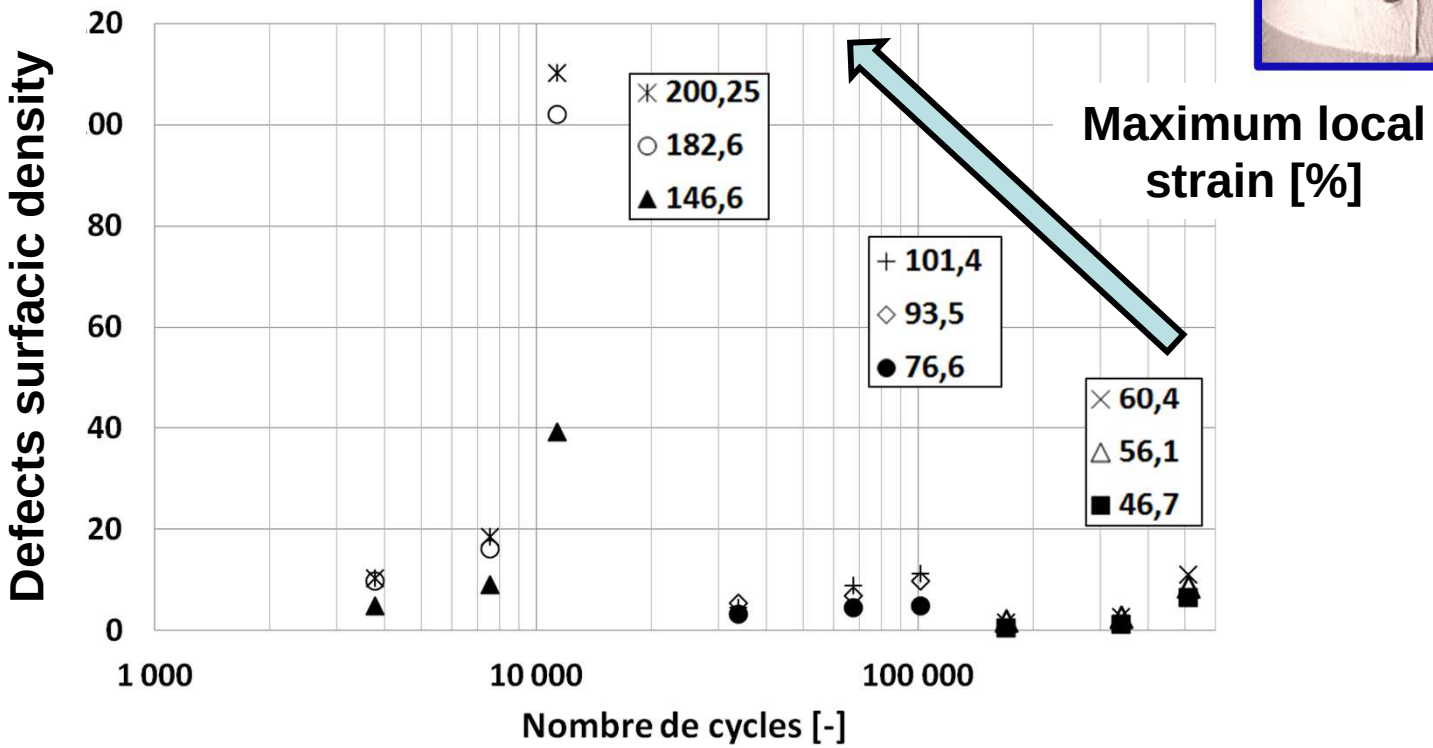
Tracks to move further: Analysis based on the evaluation of defects populations



$$E_D = \int_0^{N_i} \Delta^\star(\varepsilon)\,\omega_d(\varepsilon, N)\,dN$$

Surfacic defects density

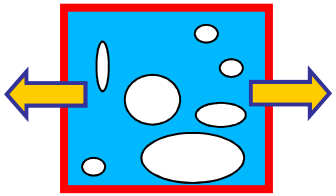


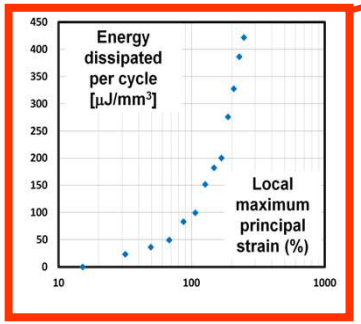


Observations: ϖd **depends both on N and on ε**

ε is the first order factor

Tracks to move further: Analysis based on the evaluation of defects populations





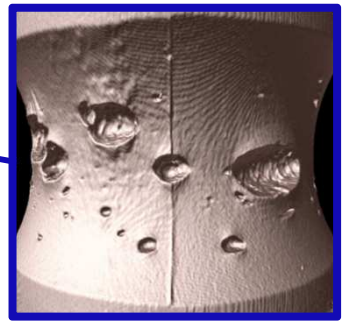
$$E_D = \int_0^{N_i} \Delta^\star(\varepsilon)\,\omega_d(\varepsilon, N)\,dN$$

$$E_D = \Delta^\star(\varepsilon) \int_0^{N_i} K(\varepsilon).\,N \quad N = C$$

$$E_D = \Delta^*(\varepsilon).K(\varepsilon) \int_0^{Ni} N.dN$$

$$E_D = \Delta^*(\varepsilon).K(\varepsilon).\frac{Ni^2}{2} = C$$

Surfacic defects density

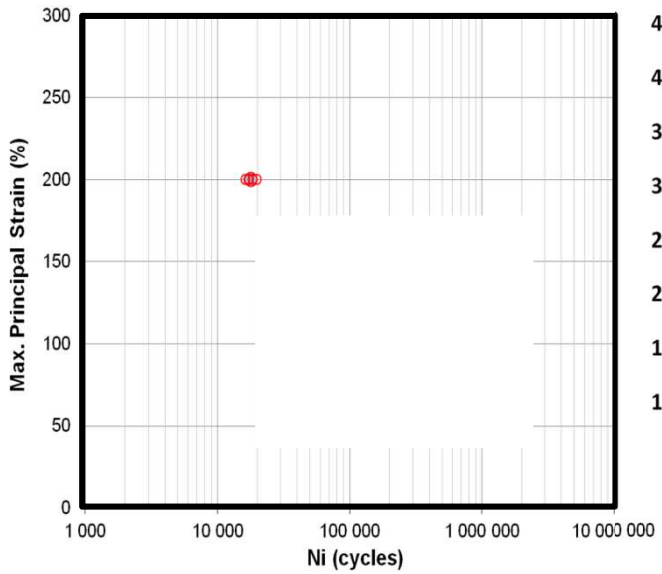


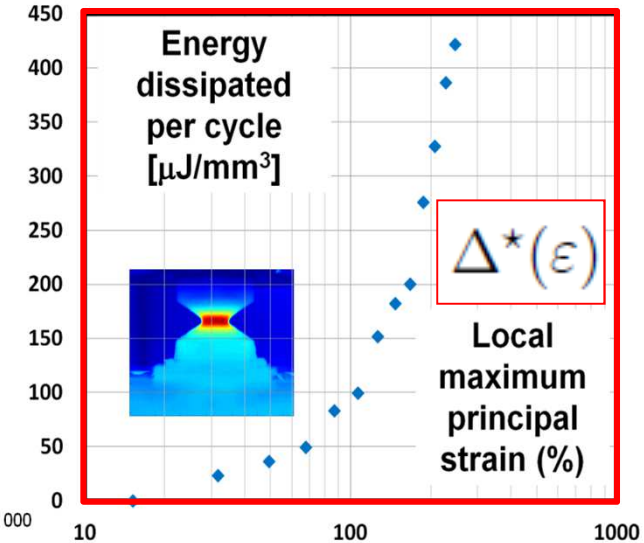
$$\varpi d(\varepsilon, N) = K(\varepsilon).N$$

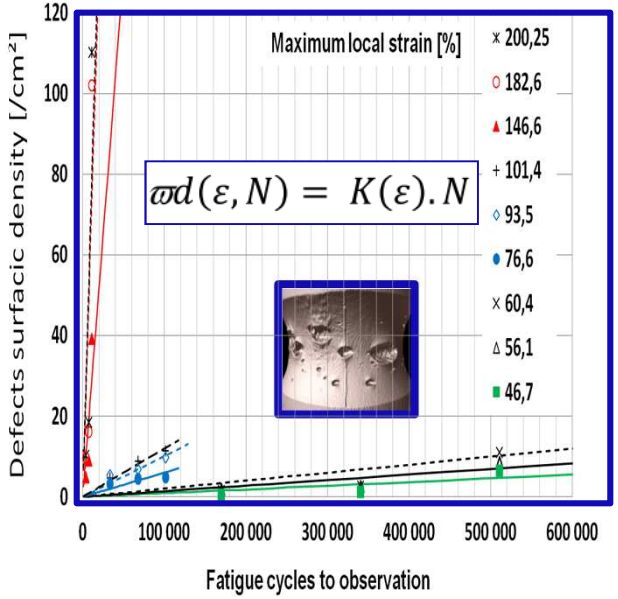
Challenge of the approach: can E_D be considered as a constant ?

ε	N_i	K(ε)	Δ*(ε)
90%	250 000	$1.43.10^{-4}$	83
140%	58 000	$1.28.10^{-3}$	167.4
190%	22 000	$5.79.10^{-3}$	277.3

Tracks to move further: Analysis based on the evaluation of defects populations

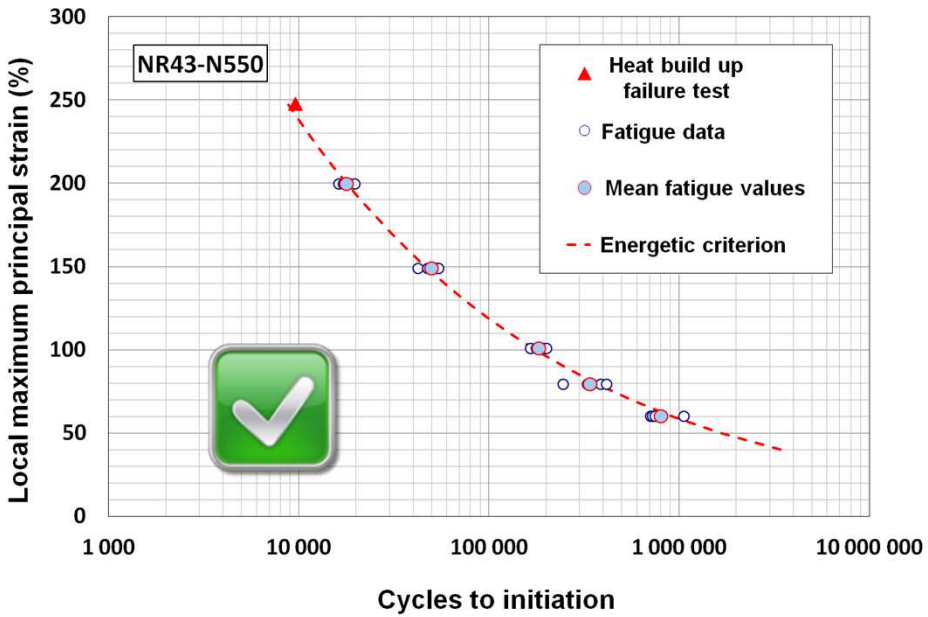






E_D = 3.8.10^8 $= C$

$$E_D = \Delta^*(\varepsilon).K(\varepsilon).\frac{Ni^2}{2}$$

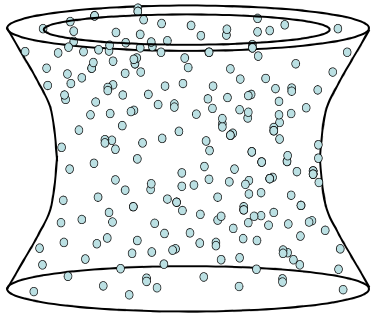


Tracks to move further: Thermomechanical evaluation at the microscopic scale

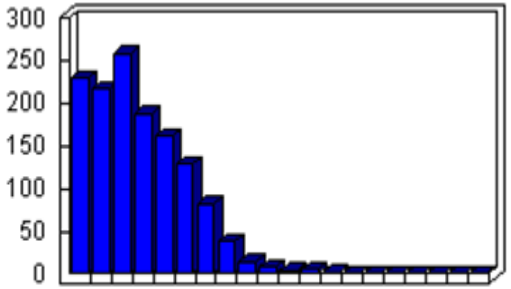
Given compound and process

Given geometry

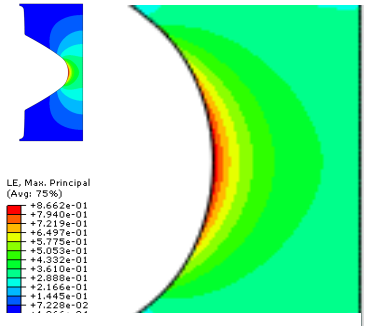
Testing conditions



Potential sites location



Inclusions size scaterring

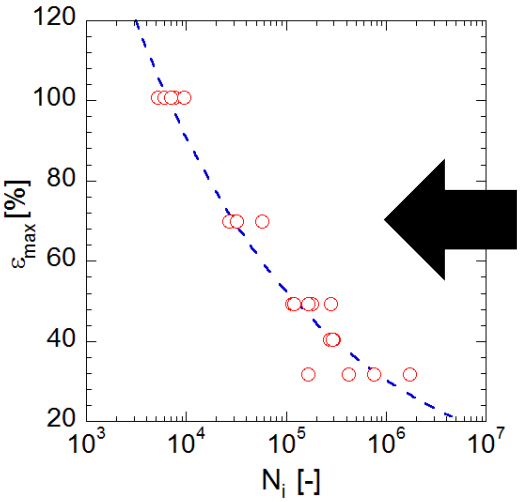


Local mechanical loading

Fast evaluation

Link between dissipation and fatigue mechanisms

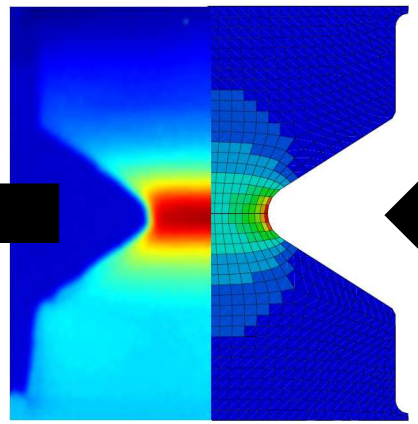
Initiation criterion at a local scale



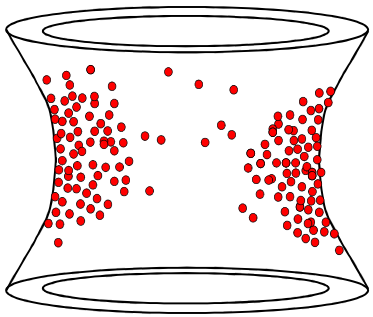
Fatigue properties

$$\Delta^*.N = C$$

Energy based fatigue criterion



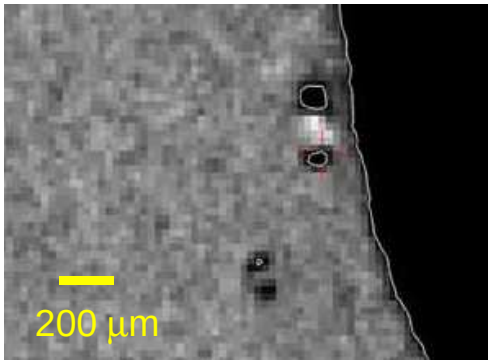
Map of dissipated energy (experimental or numerical)



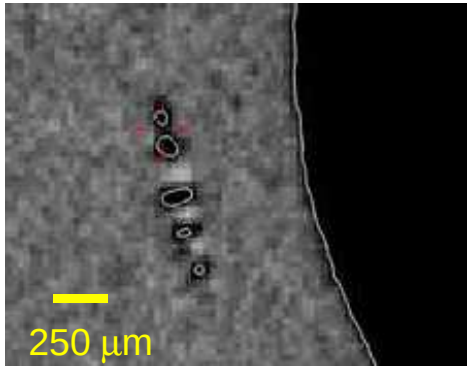
Activated sites locations

Tracks to move further: Thermomechanical evaluation at the microscopic scale

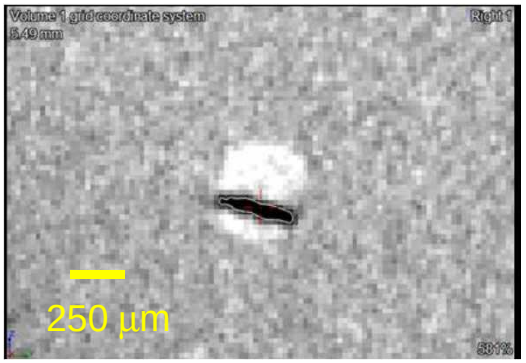
Local description of initiation sites for filled rubbers: tomo and SEM views



Polar cavities

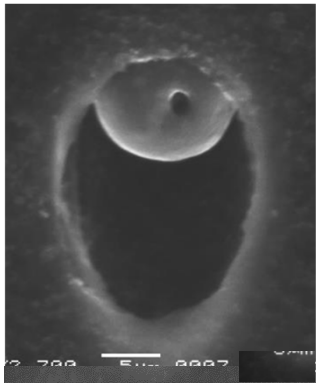


Cavitation between close inclusions

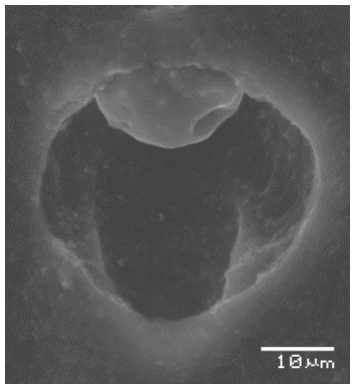


Break of fillers agglomerates

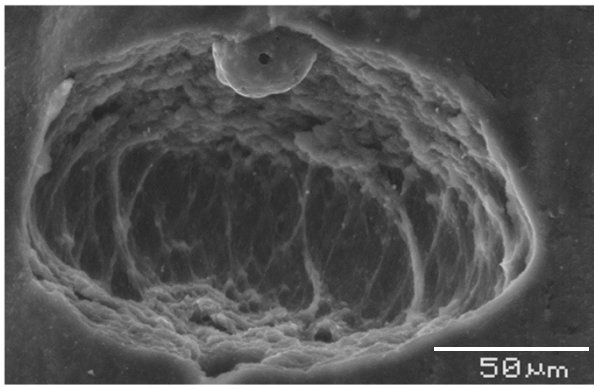
Precious results … but providing only a kinematic view !



Decohesion at one pole



Decohesion on the sides



Propagation on the surface and in the volume

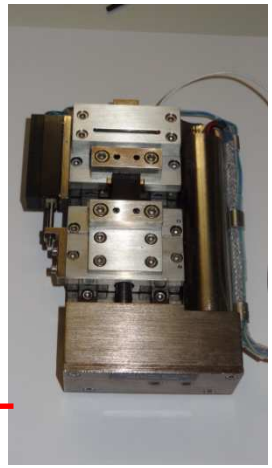
Tracks to move further: Thermomechanical evaluation at the microscopic scale

Infrared Camera
Lens 50mm and "G1"

Electro-dynamical testing machine Bose 3.2kN

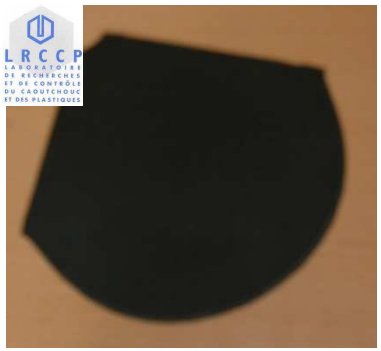
SEM with EDS

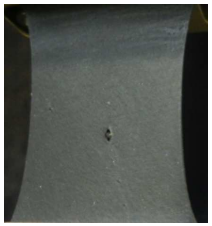
Micro tension compression device



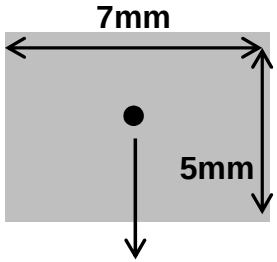
Optical microscope with extension device

Very thin films (200 µm), well or badly mixed compounds



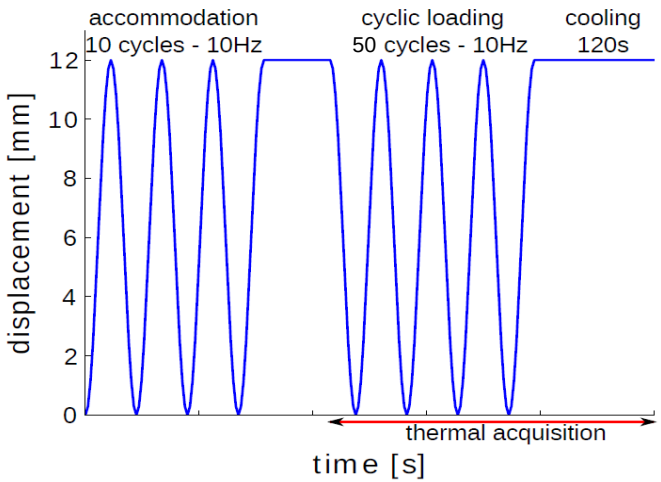


Tracks to move further: Thermomechanical evaluation at the microscopic scale

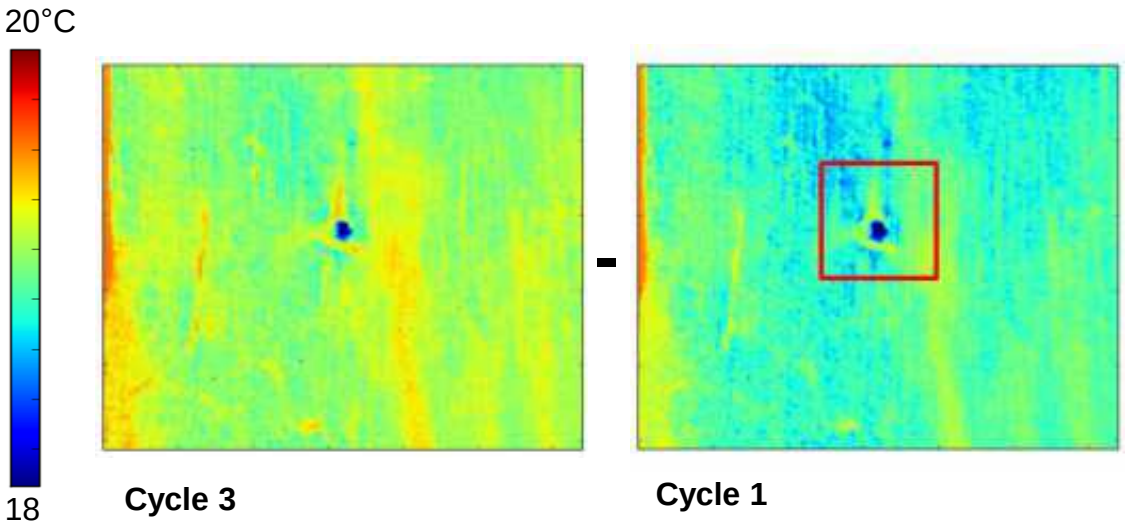


Lens "G1"

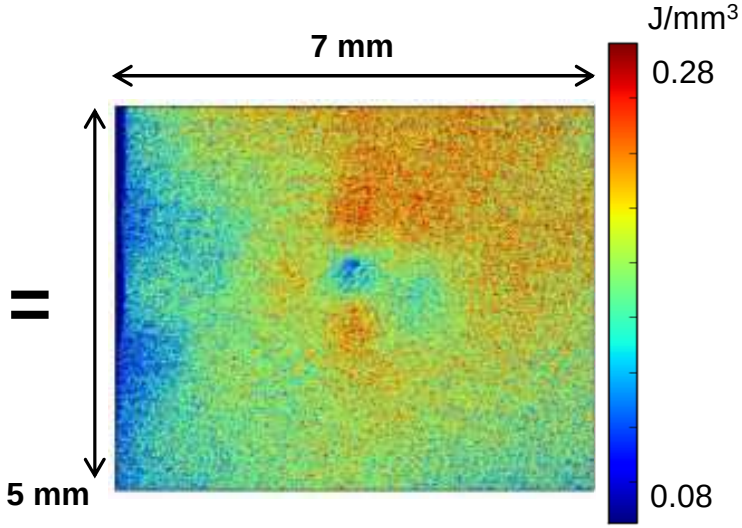
Thermal acquisition at 70Hz.



Mechanical protocol

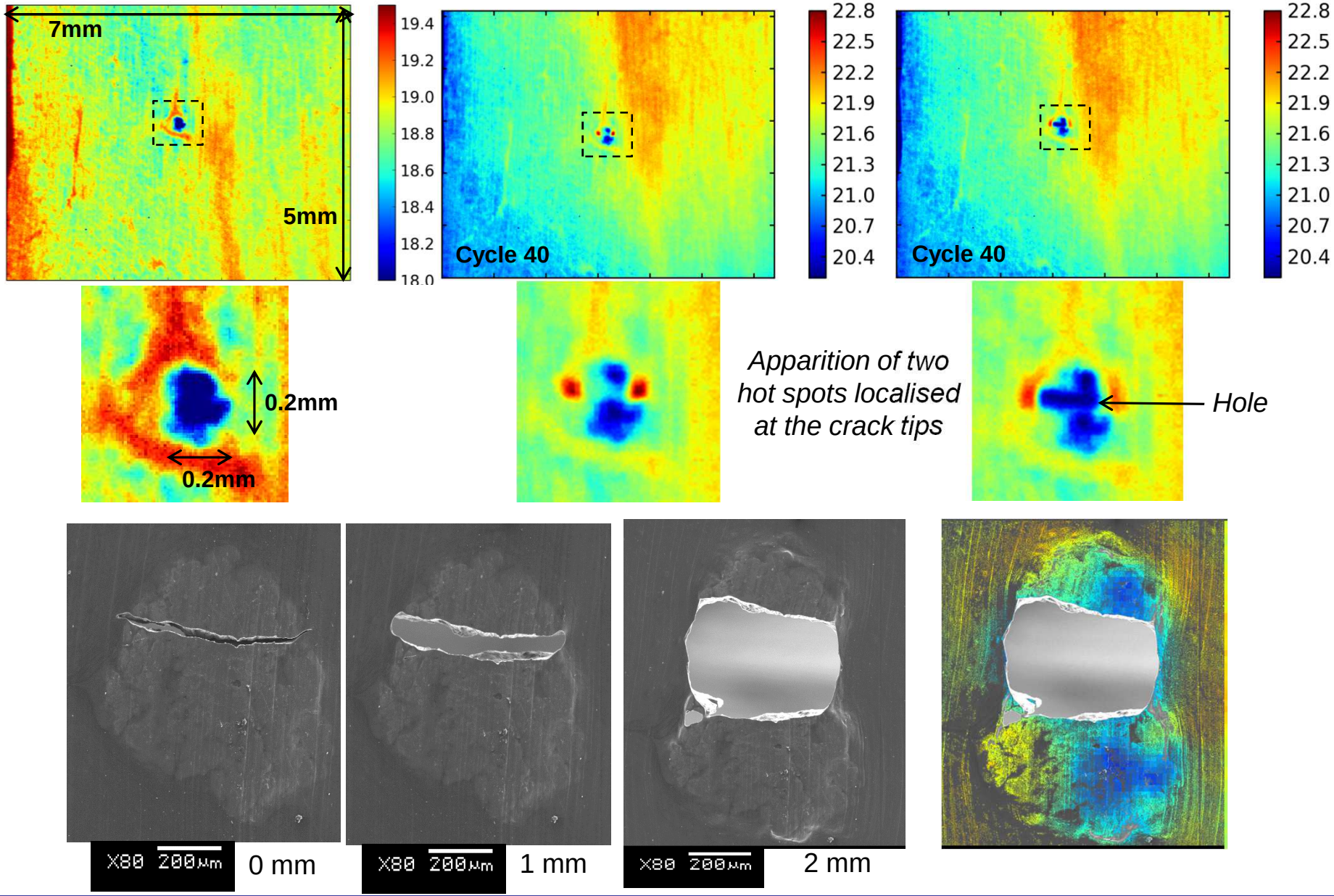


Temperature fields



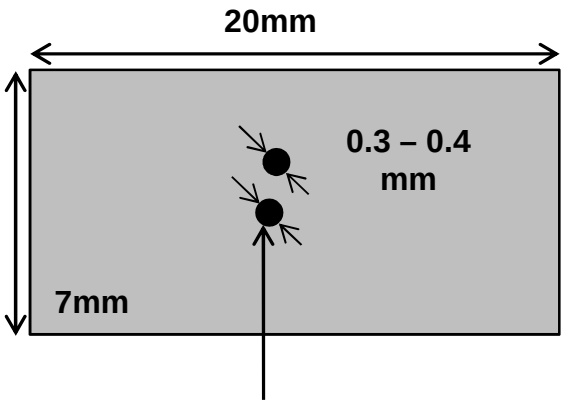
Dissipated energy fields

Tracks to move further: Thermomechanical evaluation at the microscopic scale

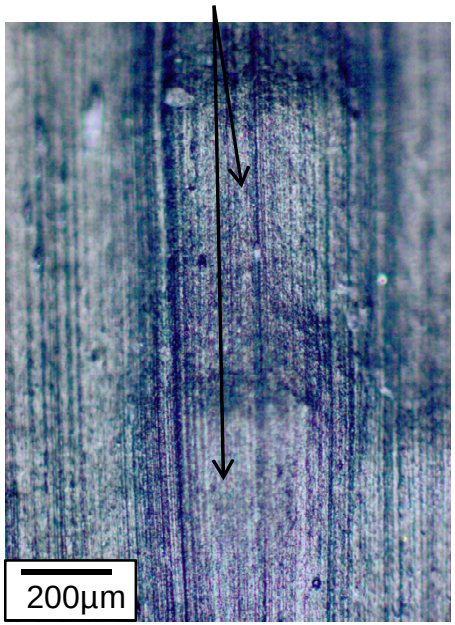


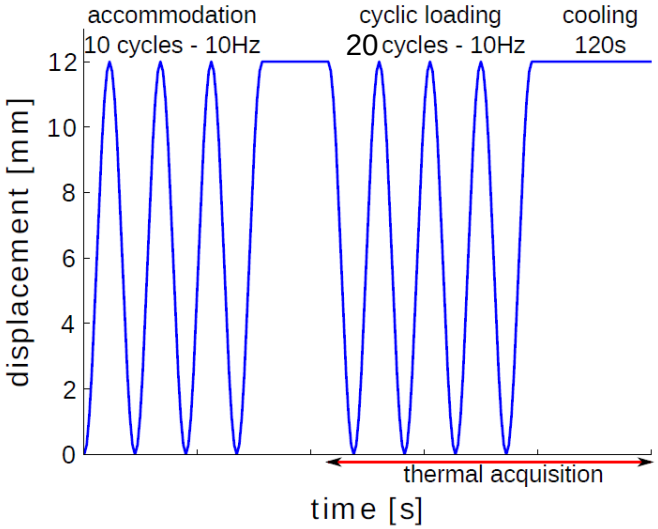
Tracks to move further: Thermomechanical evaluation at the microscopic scale

Geometry of the sample



CB agglomerates embedded by matrix

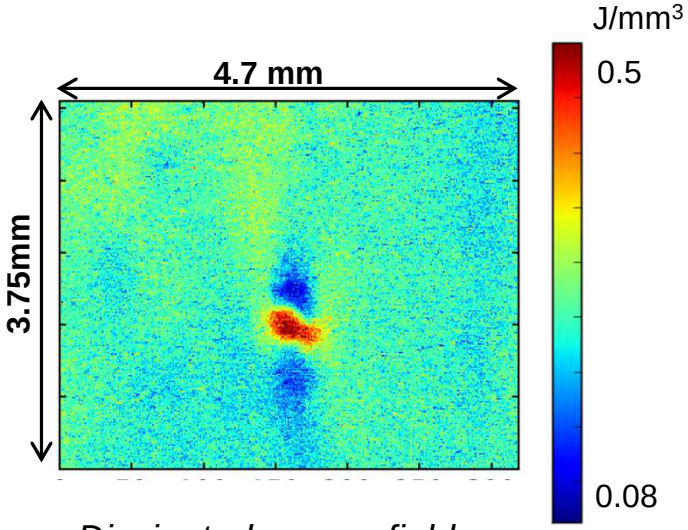




Thermal acquisition at 100Hz with reduced observed zone.

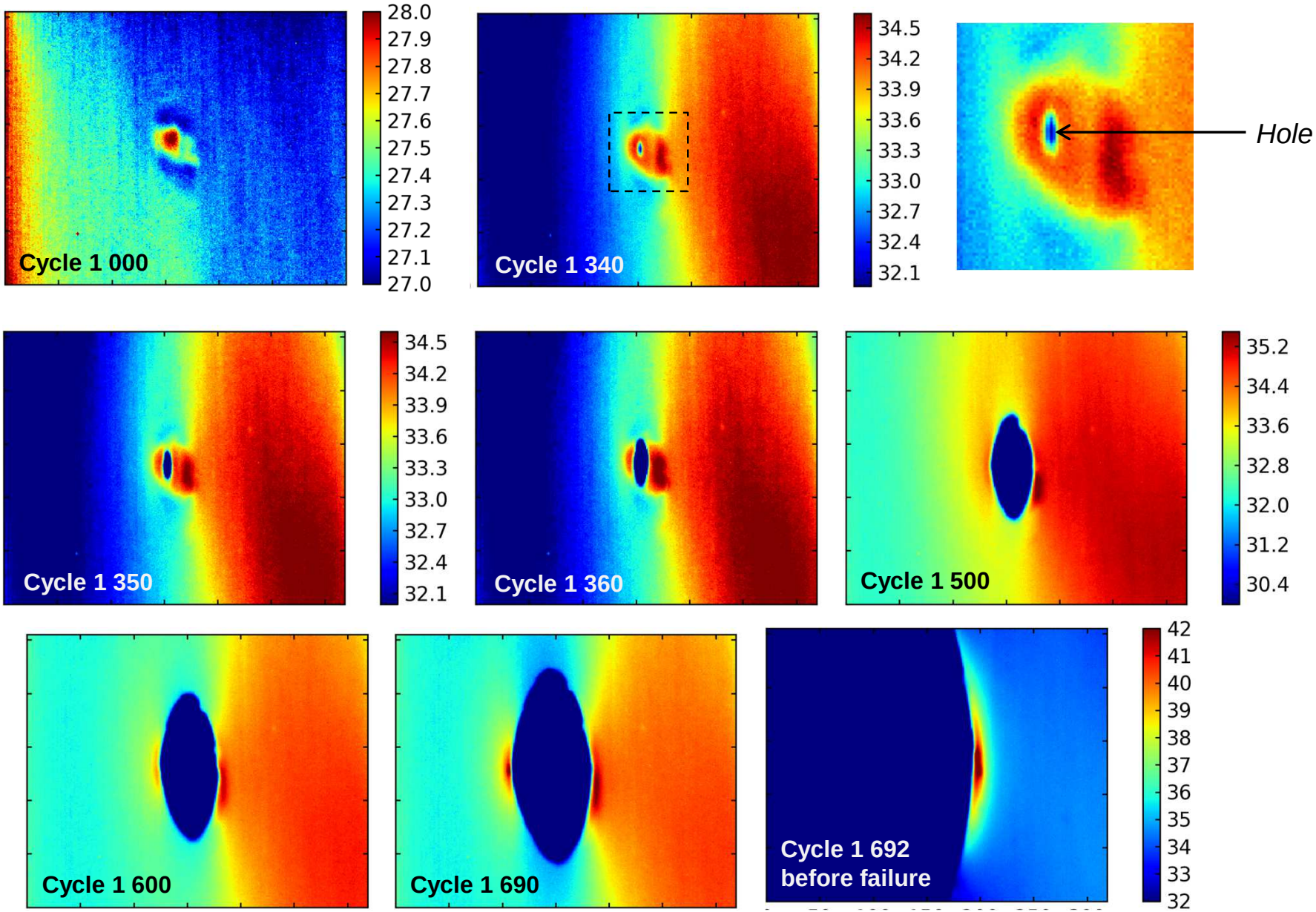
Lens "G1"

Global mean strain of 200%



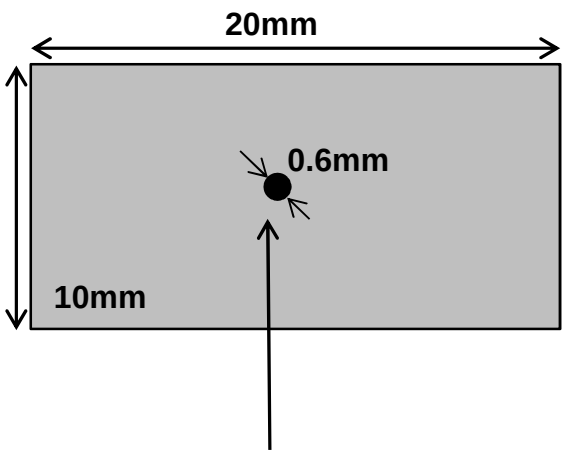
Dissipated energy fields

Tracks to move further: Thermomechanical evaluation at the microscopic scale

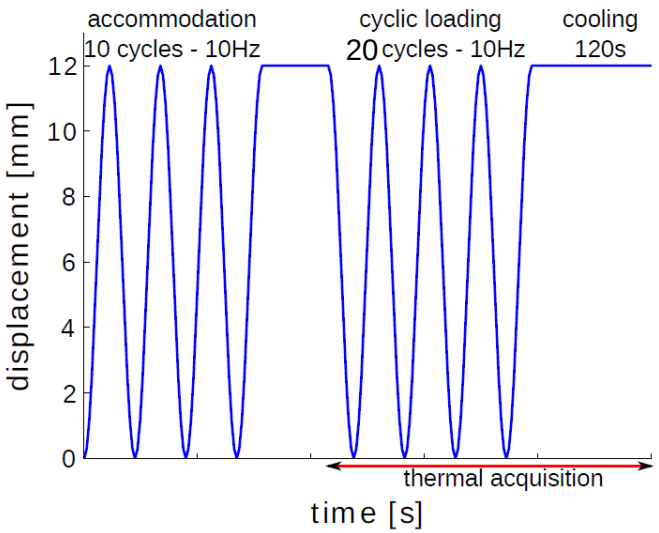


Tracks to move further: Thermomechanical evaluation at the microscopic scale

Geometry of the sample



CB agglomerate

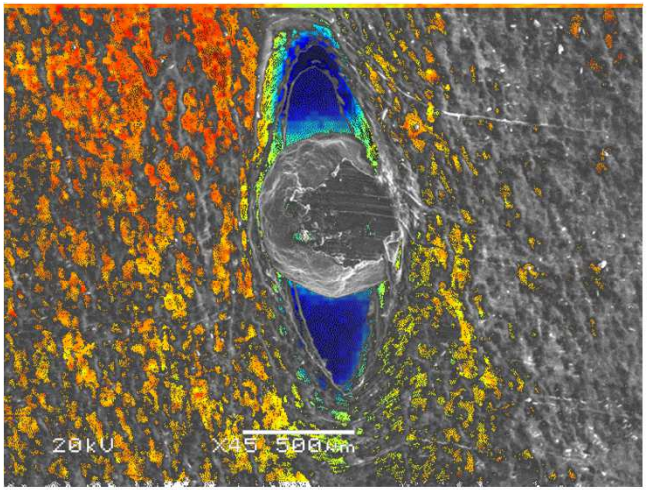


Mechanical protocol

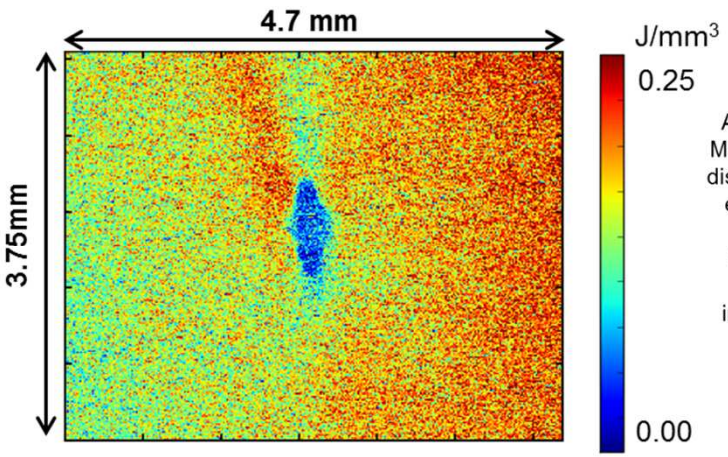
Thermal acquisition at 100Hz with reduced observed zone.

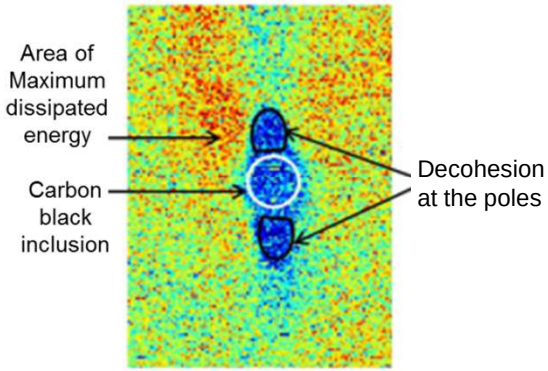
Lens "G1"

Global mean strain of 120%



Comparison of the geometries : IR and SEM pictures

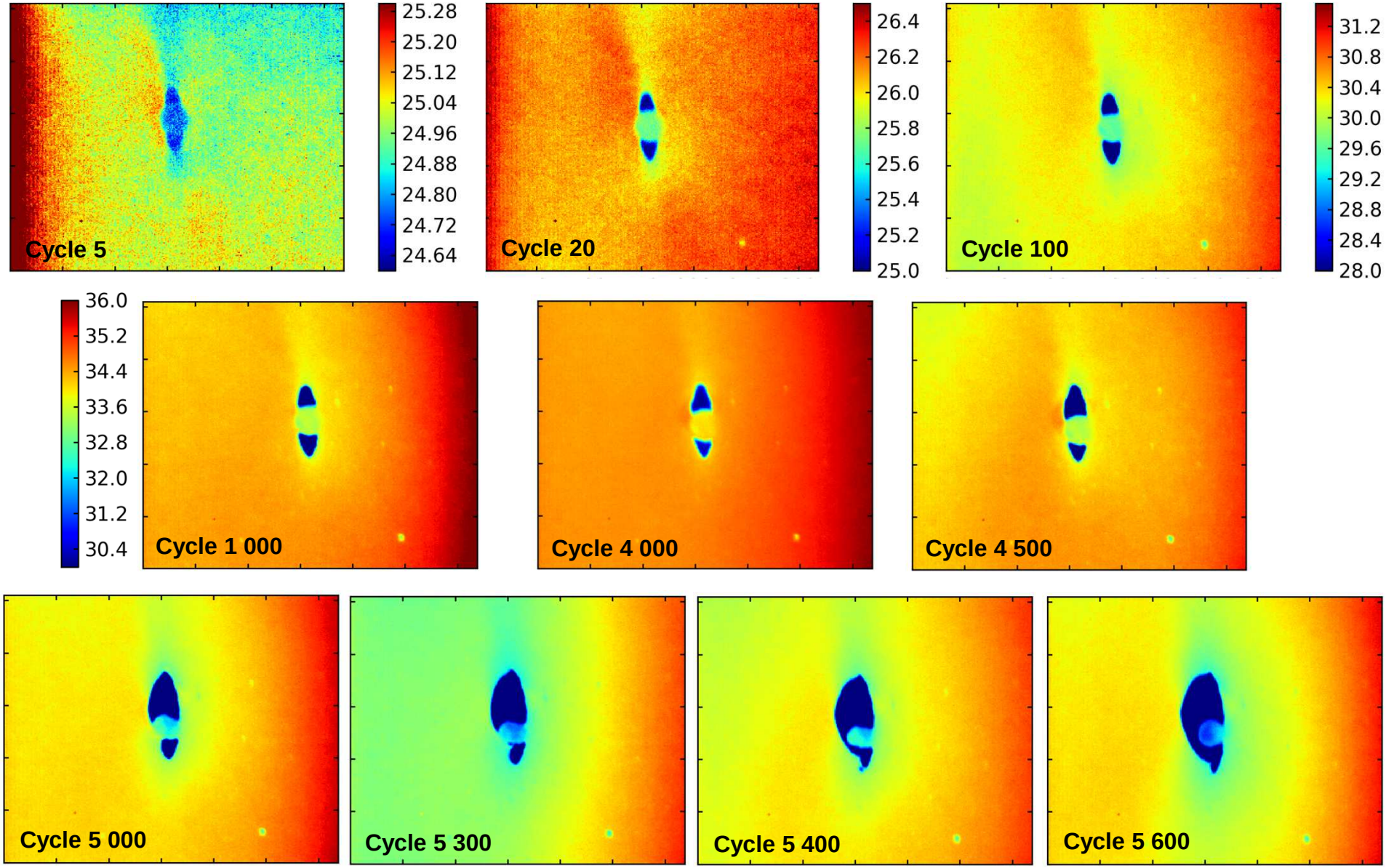




Dissipated energy fields

Tracks to move further: Thermomechanical evaluation at the microscopic scale

Temperature fields

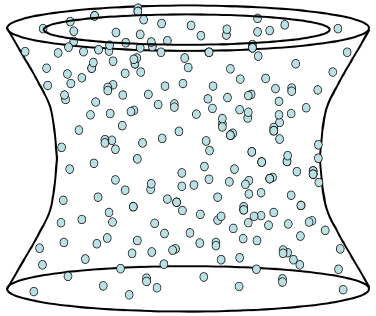


Tracks to move further: Thermomechanical evaluation at the microscopic scale

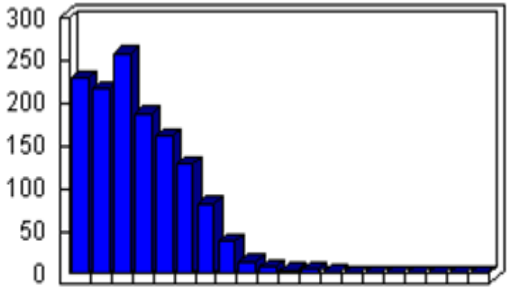
Given compound and process

Given geometry

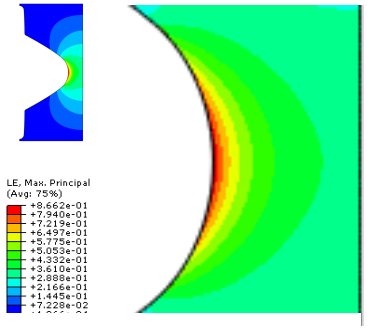
Testing conditions



Potential sites location



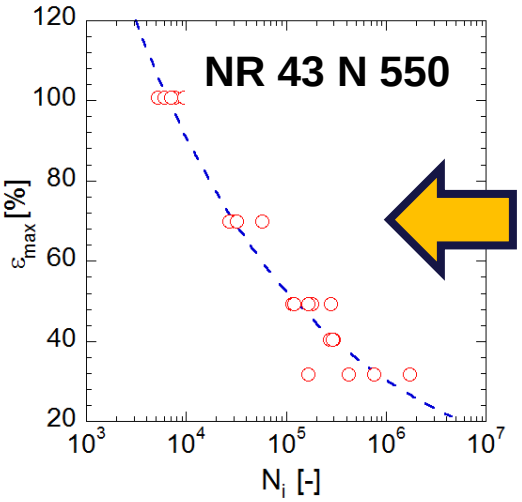
Inclusions size scattering



Local mechanical loading

Fast evaluation

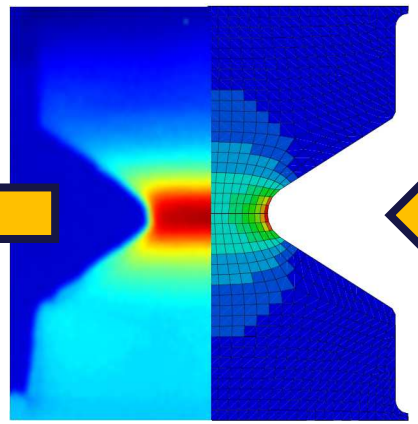
Initiation criterion at a local scale



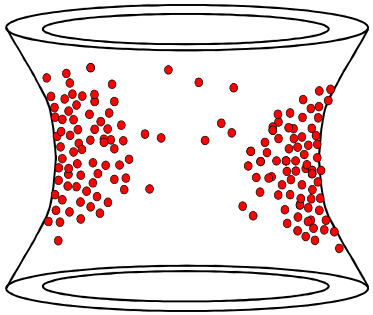
Fatigue properties

$$\Delta^*.N^b = C$$

Energy based fatigue criterion



Map of dissipated energy (experimental or numerical)

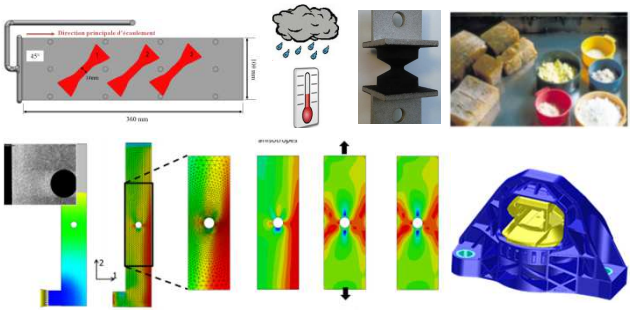


Activated sites

Outline of the presentation

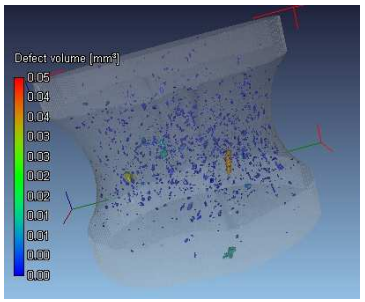
1. **Evaluation of dissipation from thermal measurements and heat build-up curve**

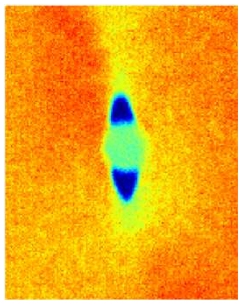
2. **Some answers to industrial expectations**



- Supply data to feed or validate design computation loops
- Fast screening of fatigue properties for various parameters
- Fast diagnostic on structural samples and parts

3. **Questions, limits and tracks to move further**





- Illustration of some questions and limits
- Analysis based on constitutive modeling
- Analysis based on the evaluation of defects populations
- Thermomechanical evaluation at the microscopic scale

4. **Conclusions**

Thank you for your attention

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Références

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