New possibilities for characterising elastomers using Dynamic Mechanical Analysis (DMA) at high strain rates.

Brice TAILLET

RIEG ATDM - March 13, 2020

Metravib

SUMMURY

- Acoem Group presentation
- DMA background and products
- Measurement examples
- New software version – New functionalities for strain sweep test
- Conclusions and contact

ACOEM Group

- ACOEM Group around the world
- METRAVIB Business unit

ACOEM Group – Around the world

670
employees

€100 M
turnover

17
subsidiaries and offices

150
distributors worldwide

ACOEM Group – our activities

Environment

Prevent and control air, noise and vibration pollution

Maintenance

Increase the productivity and reliability of industrial machinery

Design

Contribute to the development of effective, robust & noiseless products

Defence

Protect soldiers, sites and vehicles in military theaters of operation

ACOEM Group – Design

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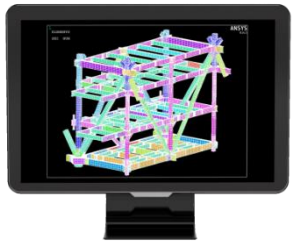
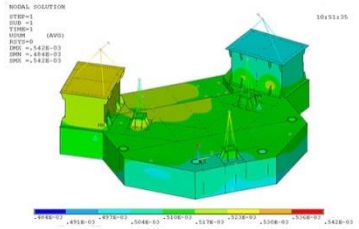
Noise & Vibration Engineering





- Design and produce effective, robust & noiseless products ;
- Expertise in the field of noise and vibrational engineering.

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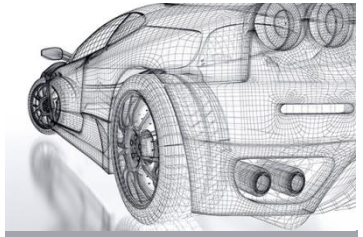
Material testing

- Provide manufacturers and laboratories DMA instruments for the characterization of the dynamic mechanical properties of materials

Transport

Aerospace

Oil & Gas



Engineering

Energy

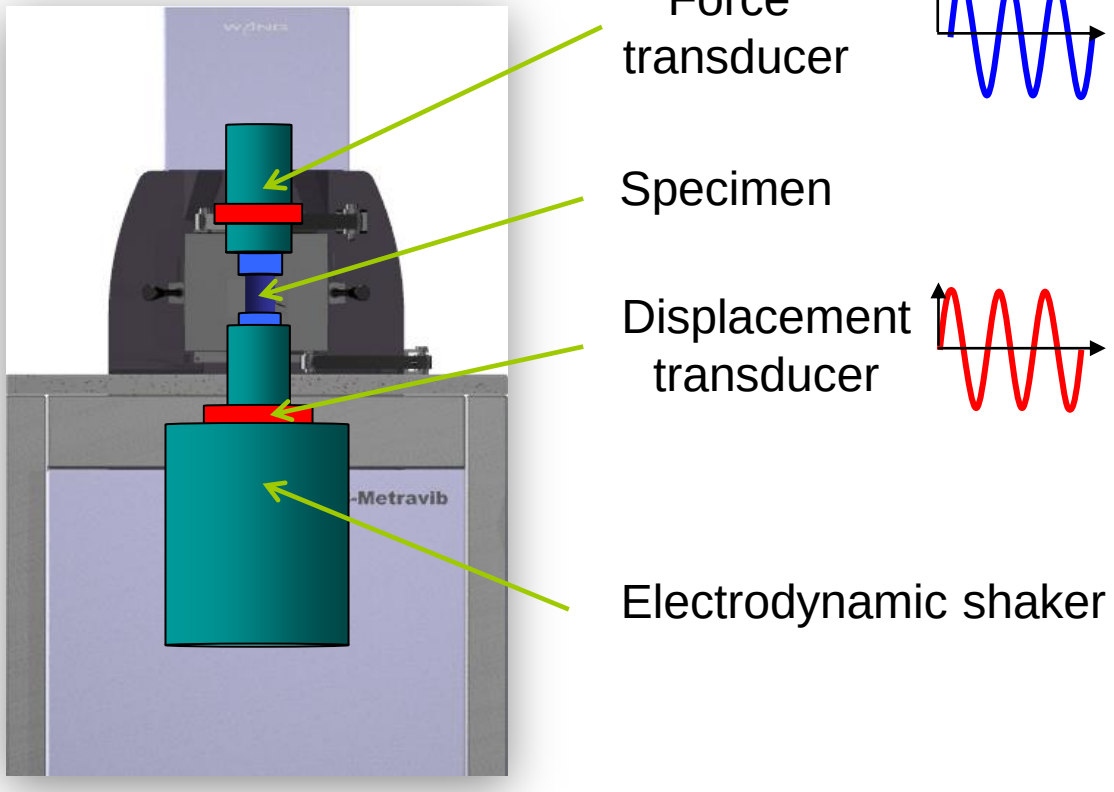
DMA background and products

- Background
- Range of products
- Focus on Xpander

DMA background

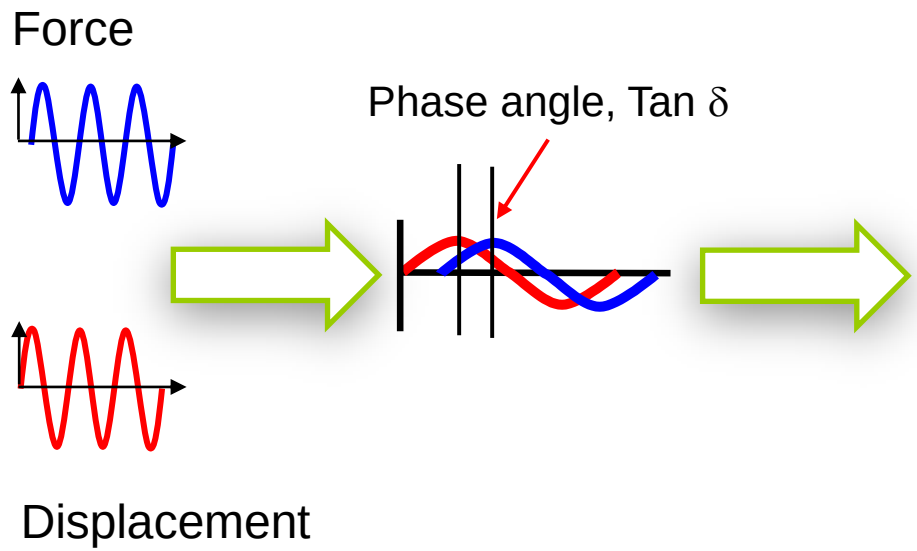
DMA principle:

- Dynamic Mechanical Analysis (DMA) is the most accurate technique for measuring the viscoelastic properties of materials.
- DMA consists in applying a dynamic forced excitation to a specimen of material, measuring the applied force and the resulting deformation.



DMA background

- Displacement and Force signal processing leads to the accurate determination of the complex stiffness ;
- From the specimen shape and dimensions and the excitation mode the complex modulus of material can be precisely calculated



- Stiffness: K* = F / D
- Modulus: E* or G*

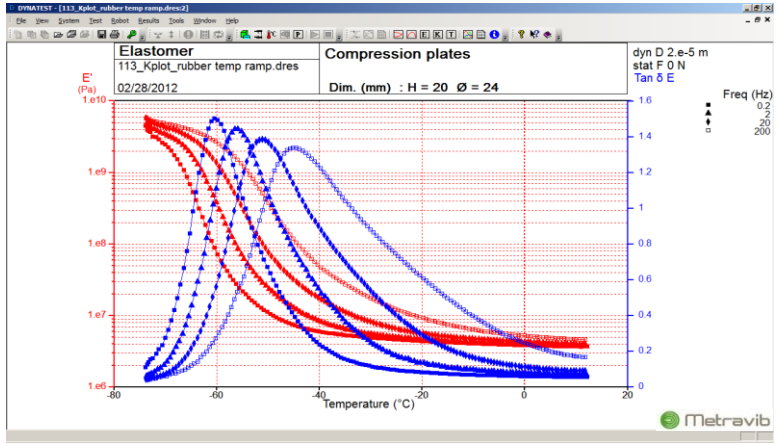
  E = Shape factor . (Length/Surface) . K

  Tan δ = K” / K’

DMA background

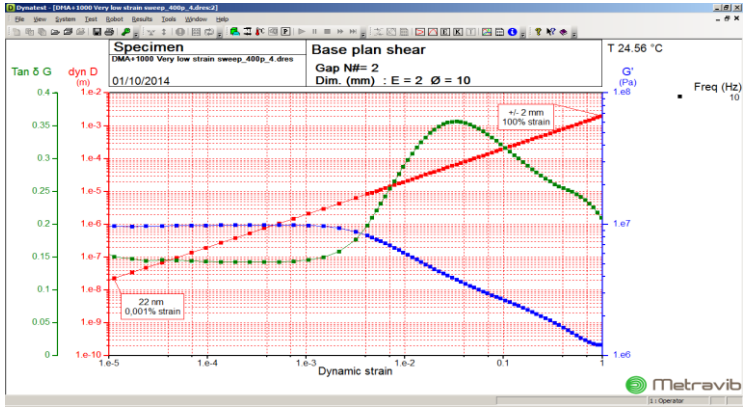
What is the usefulness of a DMA ?

Determination of glass transition of polymers



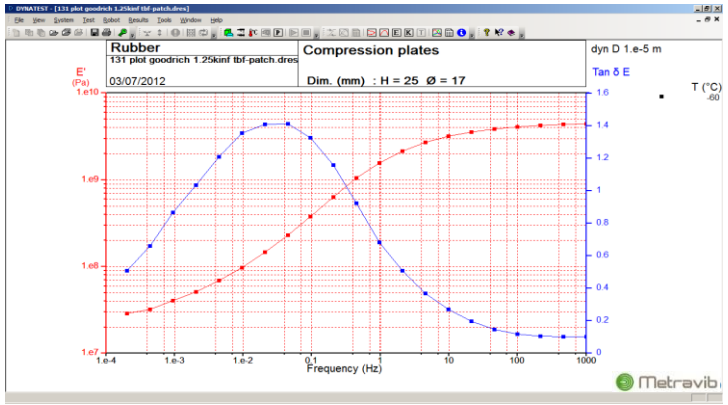
Multiple frequency sweep

Characterisation of linear/non linear behavior of elastomers



Strain sweep from 0,001% up to 100%

Characterisation of the frequency dependence of the viscoelastic properties



Frequency sweep from 0,001Hz to 1000Hz

- Creep and relaxation test
- Master curves computation
- TTS (Time Temperature Superposition)

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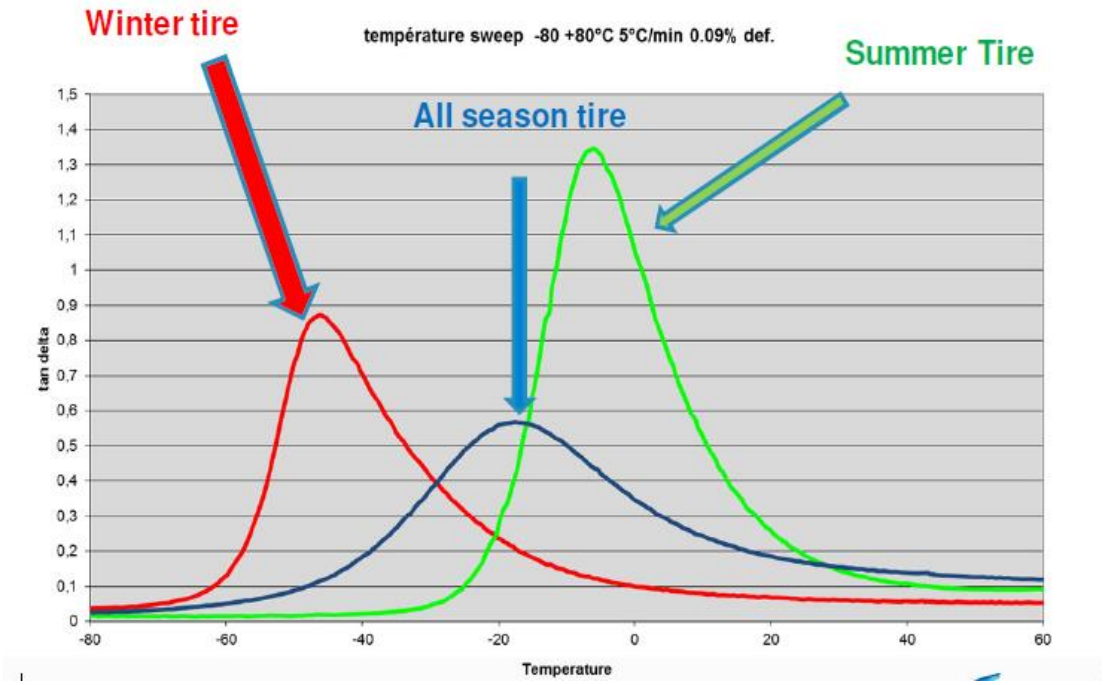
DMA background

What is the usefulness of a DMA ?

Example for tire industry



Benefits

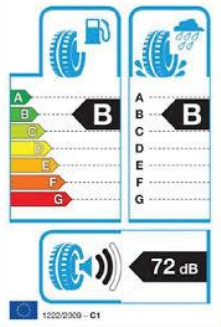
- Tire performance optimization
- Relation between tan delta and grip and rolling resistance properties of tires

Temperature ramp

Temperature ramp -70 to 120°C
Low strain amplitude

Comparison Carbon black and Silica filler for tire tread

Data courtesy SOLVAY



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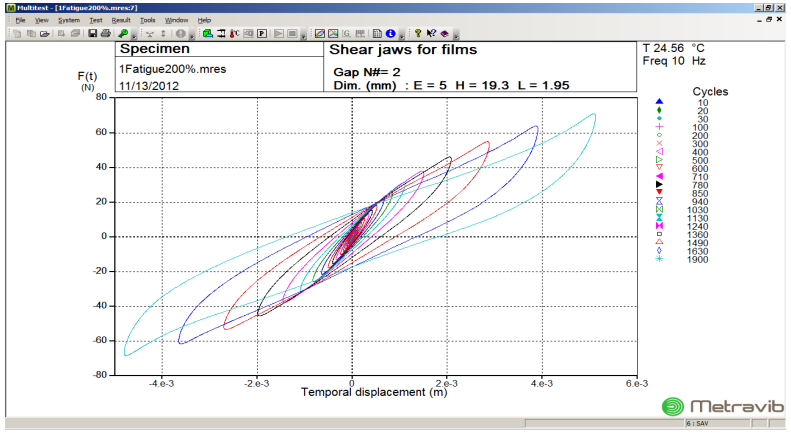
DMA background

What is the usefulness of a DMA ?

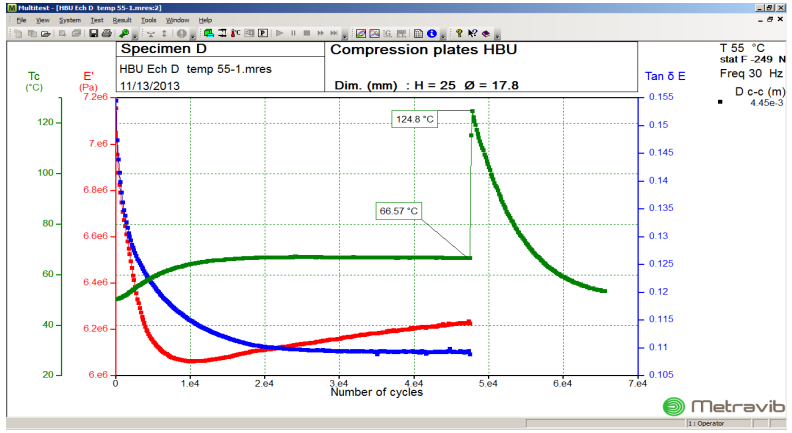
But also

Fatigue testing



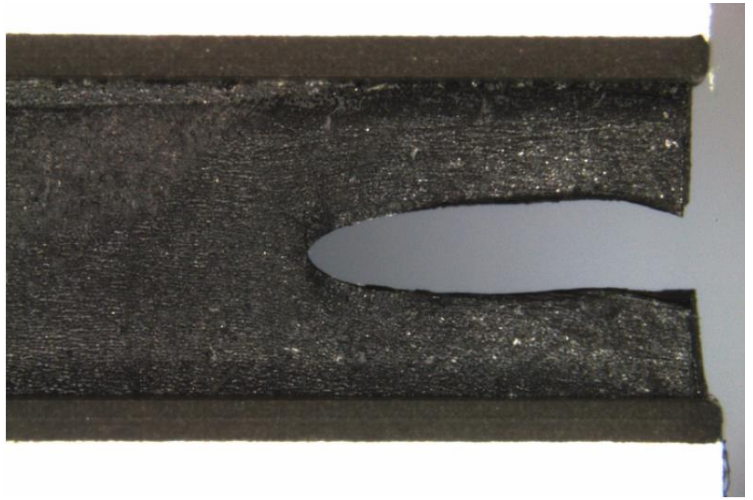
- Strain sweep
- Lissajous curves
- Possibility to take onto account multiple harmonics for measurements

Heat-build up



- Measurement of temperature of large specimen during test

Crack-growth



- Analysis of crack propagation in rubber specimen
- Control of strain, stress or tearing energy
- Waveform control for similar excitation than real life

DMA background

To Sum-up

DMA helps researchers and engineers to:

- Understand the relationships between the material's molecular structure and its mechanical properties ;
- Predict how the material will perform ;
- Select the right material to obtain products expected performances ;
- Control the key performances of manufactured products in their conditions of use and ensure their manufacturing quality and lifetime.

Michelin UPTIS Pneu Airless

Sole shoe (SALOMON)

Electric engine mount : need of rubbers with lower stiffening at high frequencies (example Mini Electric)

METRAVIB – Range of products

- Leading manufacturer of High-End DMA instruments
- 50 years plus experience in DMA

Desktop series	DMA+ series			HF Analyzer
Low force DMA	High force DMA	Automated DMA	Fatigue	High Frequency
DMA25 DMA50	DMA+300 DMA+1000 DMA+2000	Xpander Compression Tension Shear	HBU Crack Growth testing	VHF104

METRAVIB – Range of products

Focus on Xpander : fully automated DMA

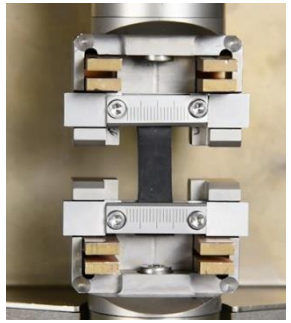
- 6 axis industrial and collaborative robot arm ;
- Storage carrousel ;
- Up to 400 specimens ;
- Up to 12 removable storage racks.

Benefits

- H24 testing
- Improved data repeatability/reproducibility
- Fully compatible with DMA+1000/2000
- Keeps all original DMA+ capabilities
- Fast conversion between excitation modes
- Operator alert

Shear

Compression

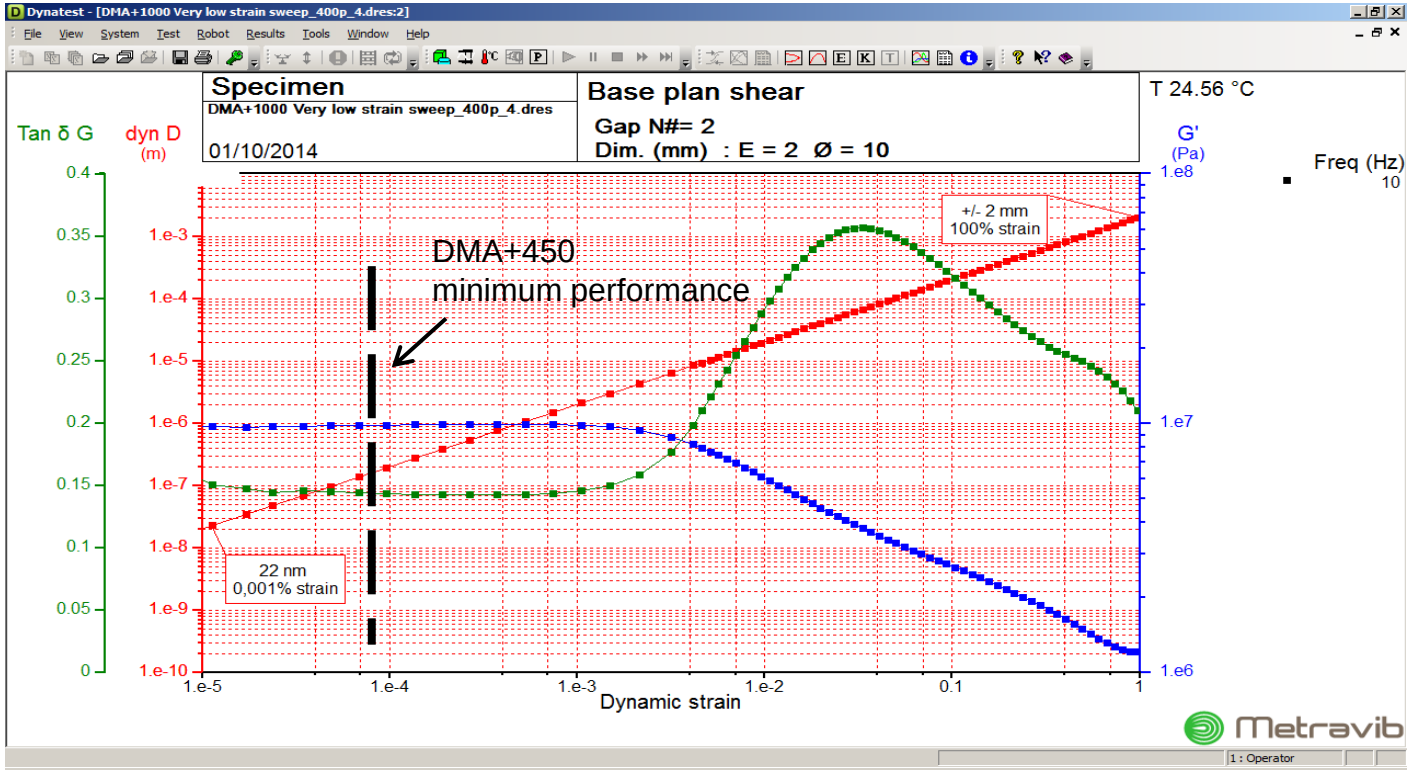


Tension

Measurement examples

- DMA typical tests
- Fatigue capabilities
- HBU test
- Crack growth module
- High frequencies test

METRAVIB – Material testing



Dynamic strain sweep

Strain sweep from very low to high strain

0,001% up to 100%

Shear for solids

Glued or molded sample
(video)

Benefits

- Payne effect characterization
- Very low strain achievable thanks to high resolution
- High strain achievable thanks to high force of instrument

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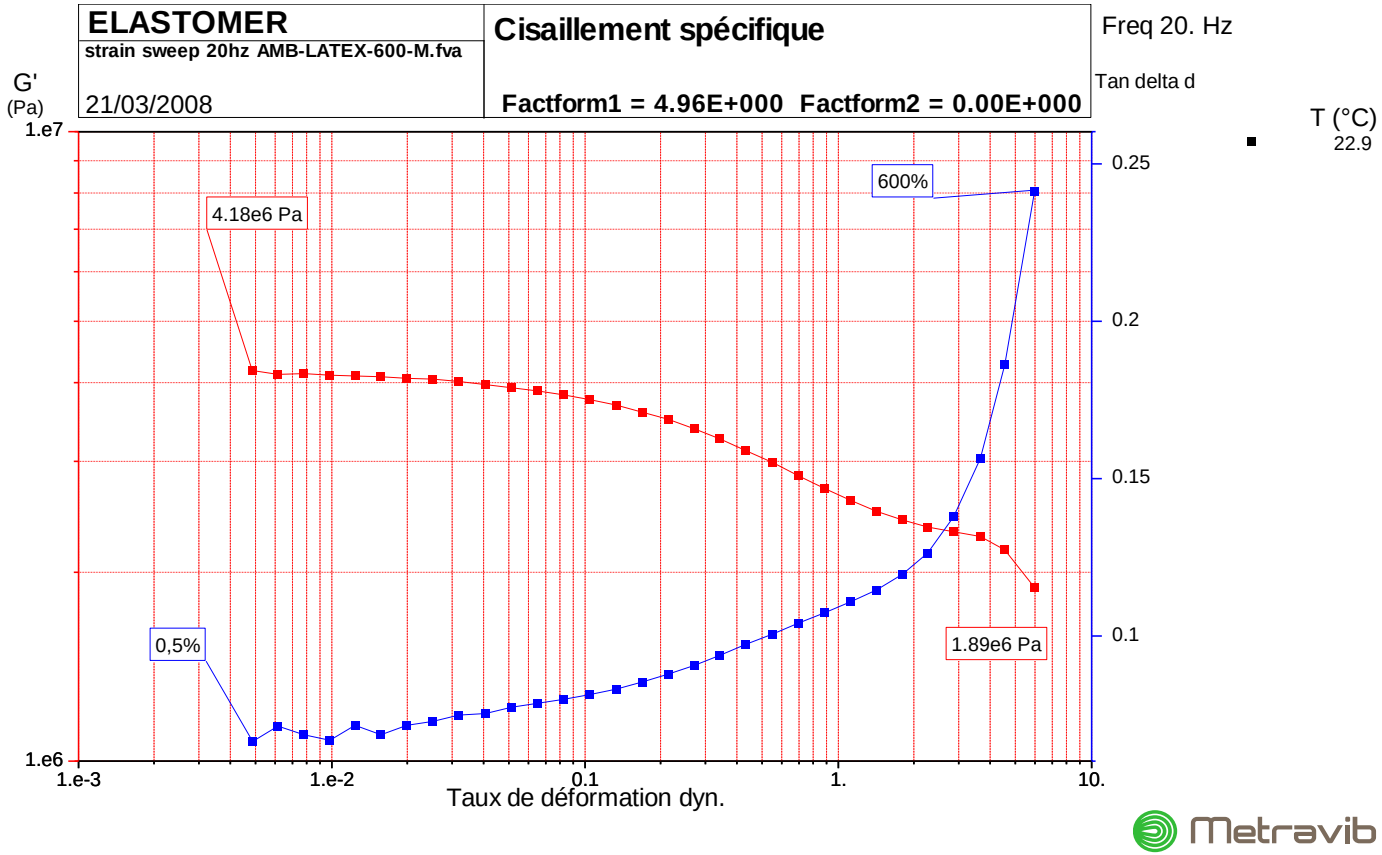

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Dynamic strain sweep

Strain sweep from very low to high strain on latex glove

0,5% up to 600%

Shear for solids

Latex is a very particular material in the nonlinear materials family, able to support huge strain deformation, without breaking.

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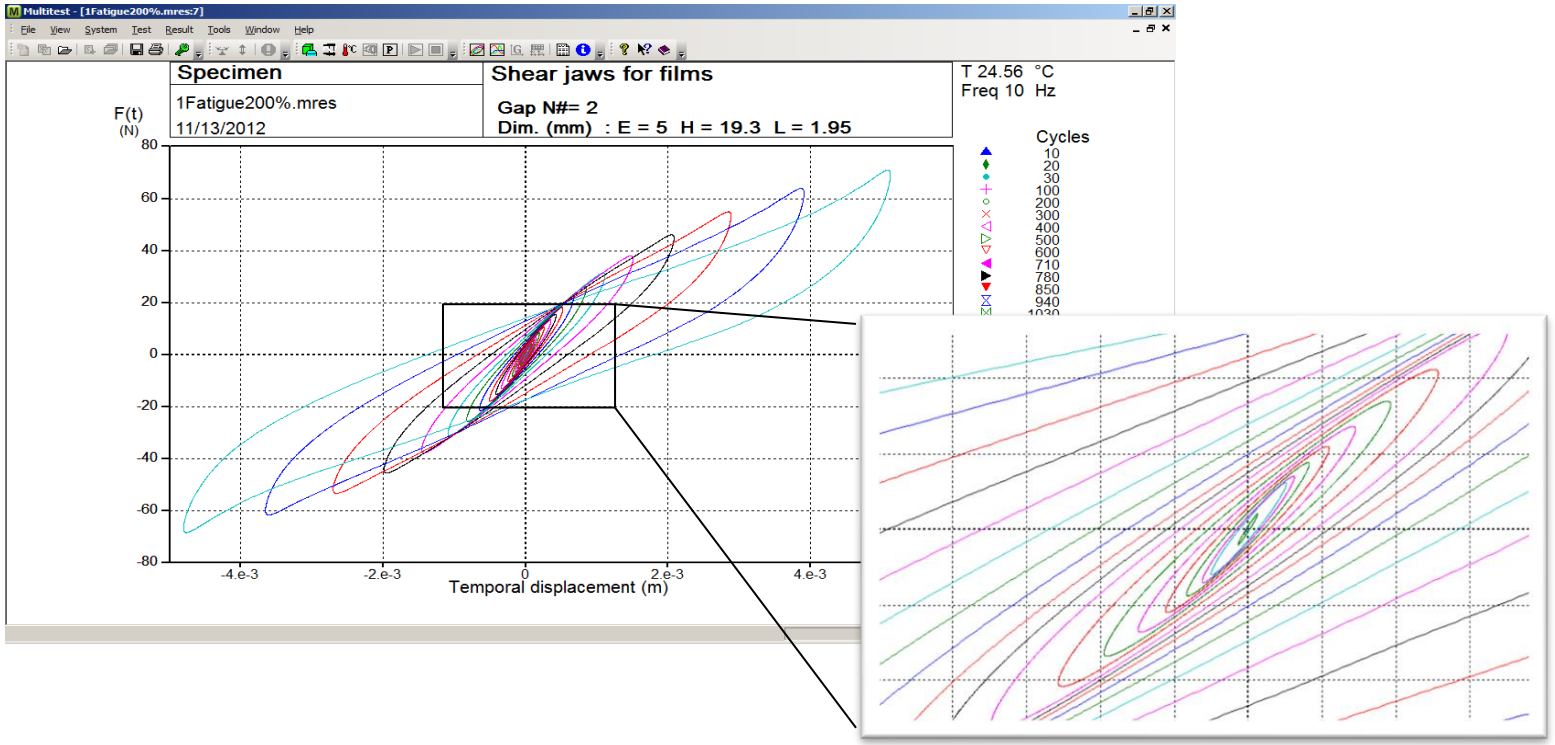

Fatigue testing

- Waveform control
- Sine, Haversine, Pulse, Triangle, Square..
- Customized wave forms
- Multiple harmonics control
- Lissajous curves
- Heat build up capabilities
- Crack growth module

Strain sweep - Lissajous curves

Possibility to take onto account multiple harmonics for measurements

Time data available: force and displacement signals

Benefits

- More accurate analysis of non linear materials
- Energy calculation
- Extend capabilities of analysis

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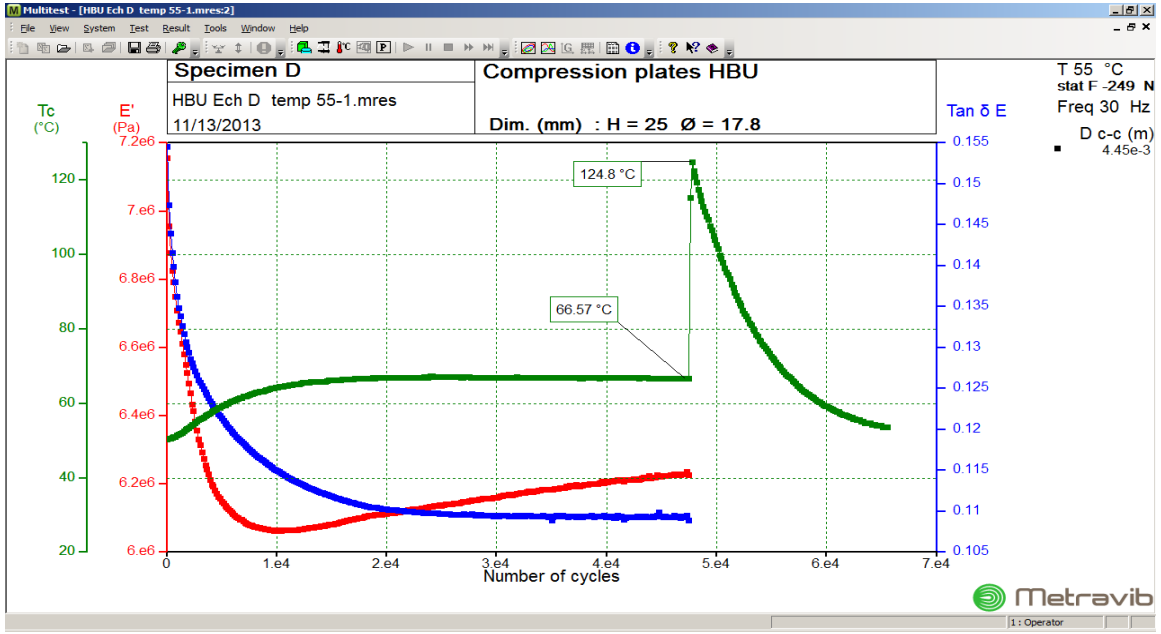
Specific specimen holder for HBU test (*video*)

- Evolution of E’ and Tan δ during the fatigue test according the number of cycles ;
- During the test : temperature surface is about 66°C ;
- At the end : the internal temperature reaches almost 125°C.



Heat Build-up

Fatigue testing with high strain rate

Temperature probe included in the superior plate (continuous temperature measurement during fatigue testing)

Additional needle temperature probe introduced inside the rubber specimen by a pneumatic command (instantaneous temperature in the heart)

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Fatigue crack growth module

- Motorized microscope equipped with a CCD video camera
- Software module dedicated to crack growth testing
- 4 cracks can be followed in the same time

Video

Pure shear specimen up to 80mm wide

The full test includes 3 separates test sequences :

1/ Accommodation : a preliminary accommodation test is applied to the un-cracked specimen (limit the Mullins effect).

2/ Characterisation : a preliminary characterization test is applied to the un-cracked specimen to determine the relationship between the applied energy and the strain amplitude.

3/ Crack growth :The crack growth test is performed automatically without operator's intervention, at given energy. From the successive crack images and detected crack tip positions, it determines crack growth rate and related data.

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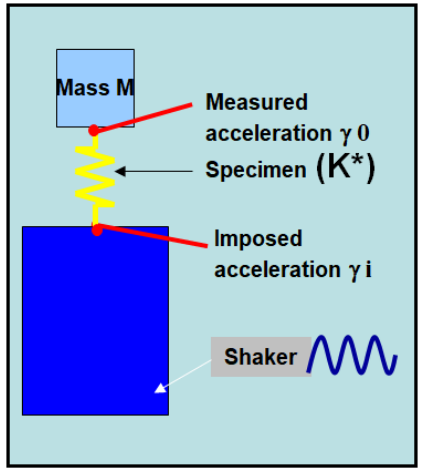
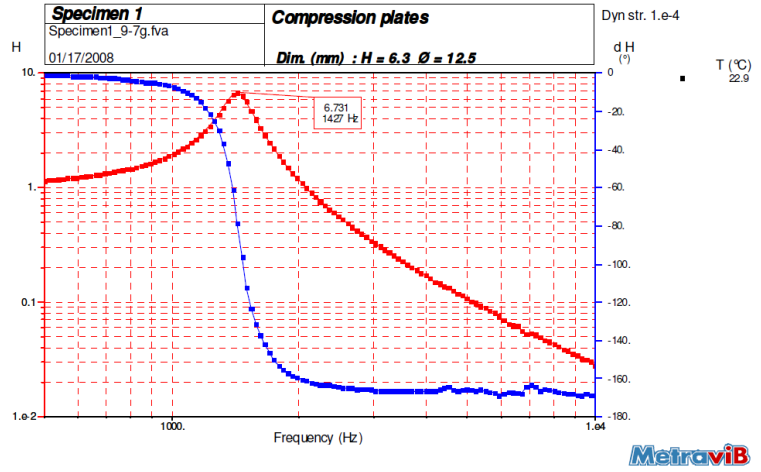
Focus on VHF measurements - principle

- A unique high frequency mechanical analyzer to accede to the direct experimental analysis of material viscoelastic properties over a high frequency range: 100 Hz up to 10 kHz.
- Characterisation of a damped element through a resonant analysis:
  - Measurement of both accelerations (γ0, γi) over an imposed sweep of frequency.
  - Calculation of the specimen's transfer function: **T = γ0 / γi**
- Calculation of the intrinsic material's characteristics E, Tan δ by F.E.M.

- Typical test condition:
  - Cylindrical Shape.
  - Wave form: sine.
  - Additional mass: 10g.
  - -50°C up to 110°C.





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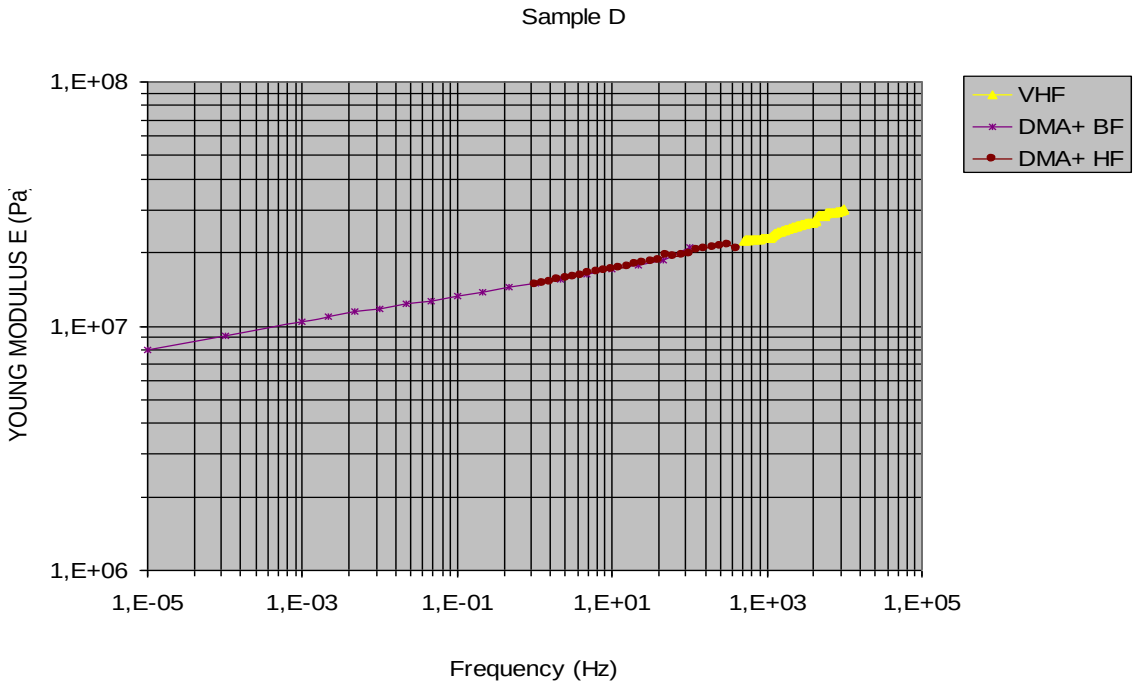
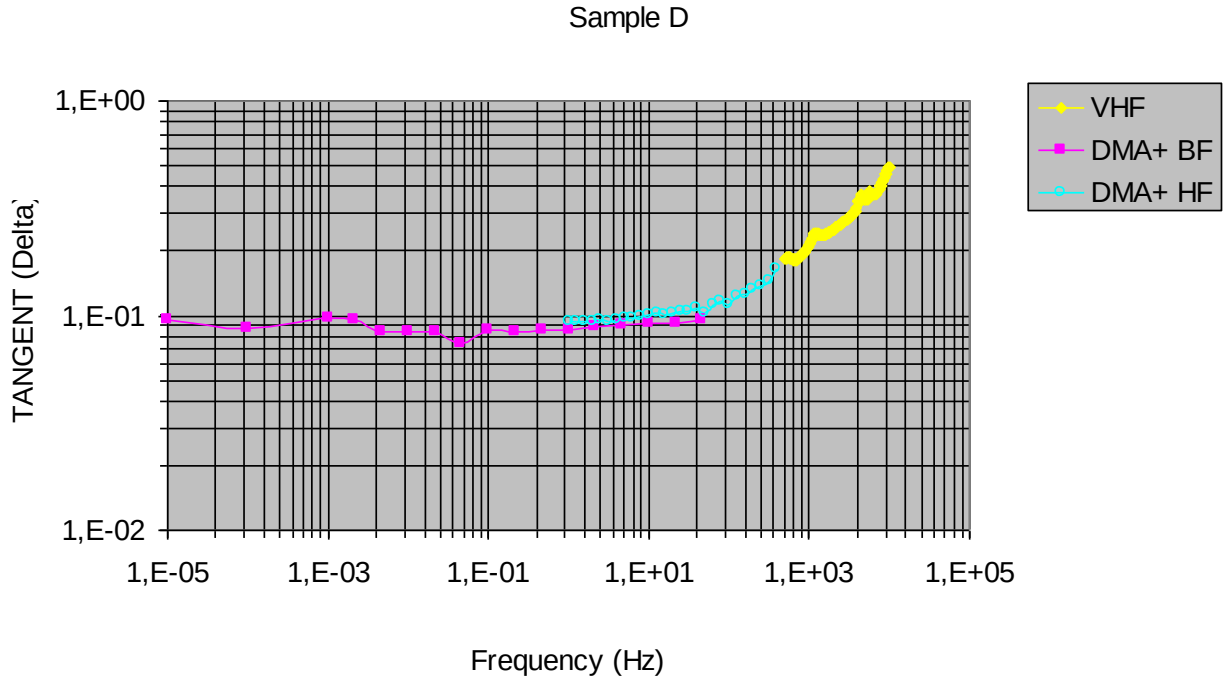
Focus on VHF measurements – Comparison with DMA+

Data superposition DMA+ & VHF104





- Perfect continuity between DMA and VHF104 ;
- VHF104 allows to go further in frequency analysis.

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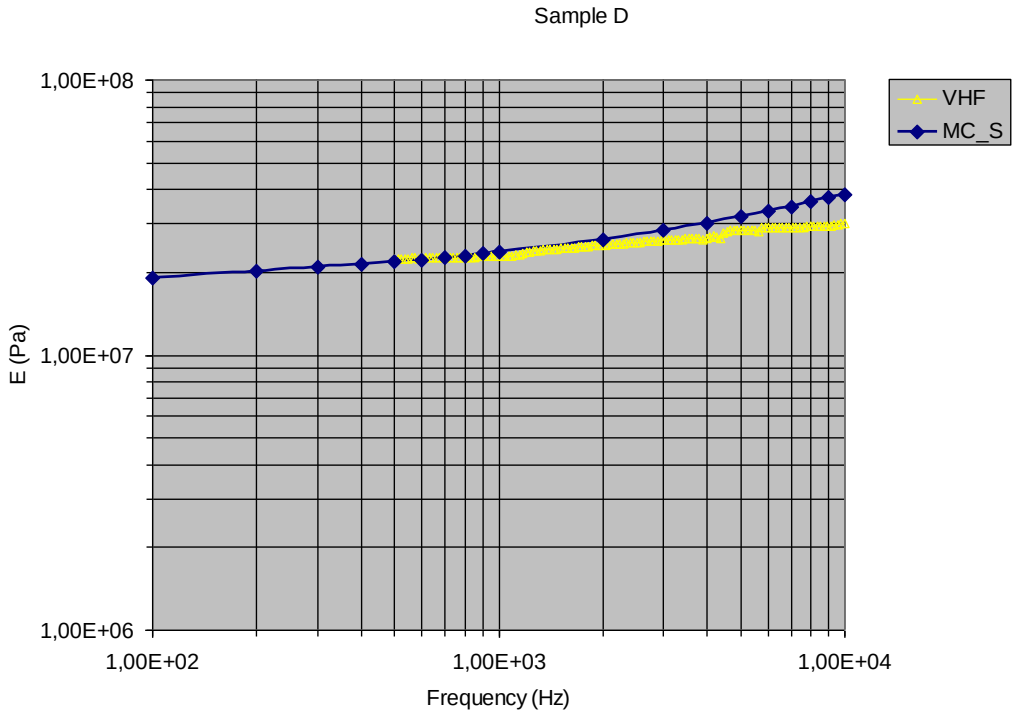
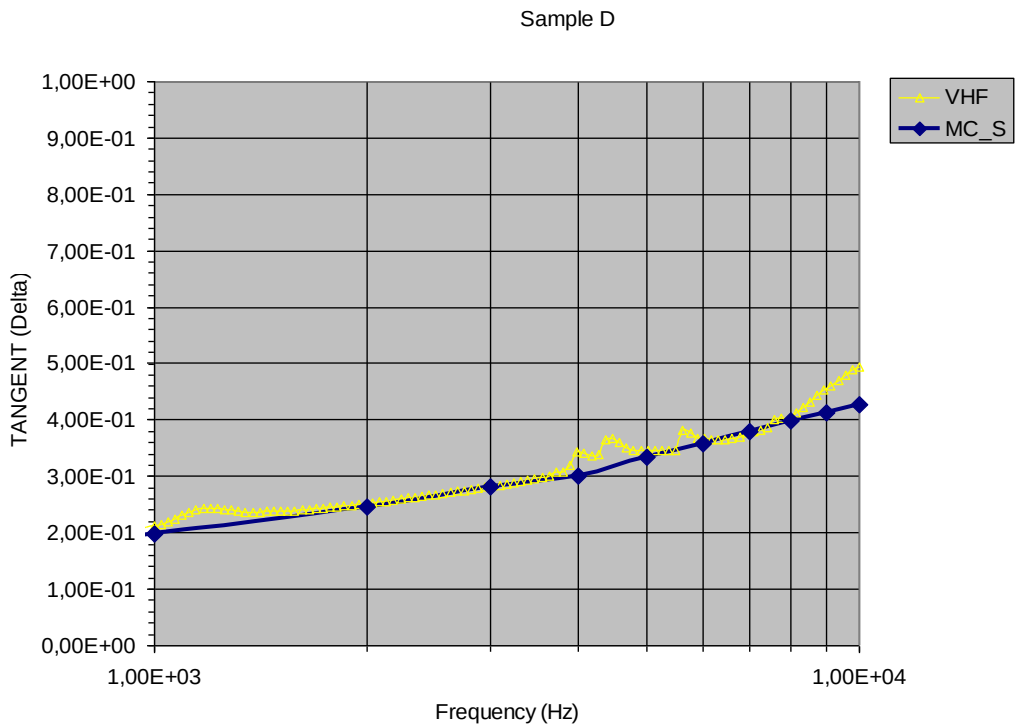
Focus on VHF measurements – Comparison with DMA+

Data superposition DMA+ & VHF104: E'/tan d vs master curves





- Duration for test in order to compute master curves : 7 hours
- Duration for VHF104 test : 15min

DMASUITE 8.5 – New functionalities for strain sweep test

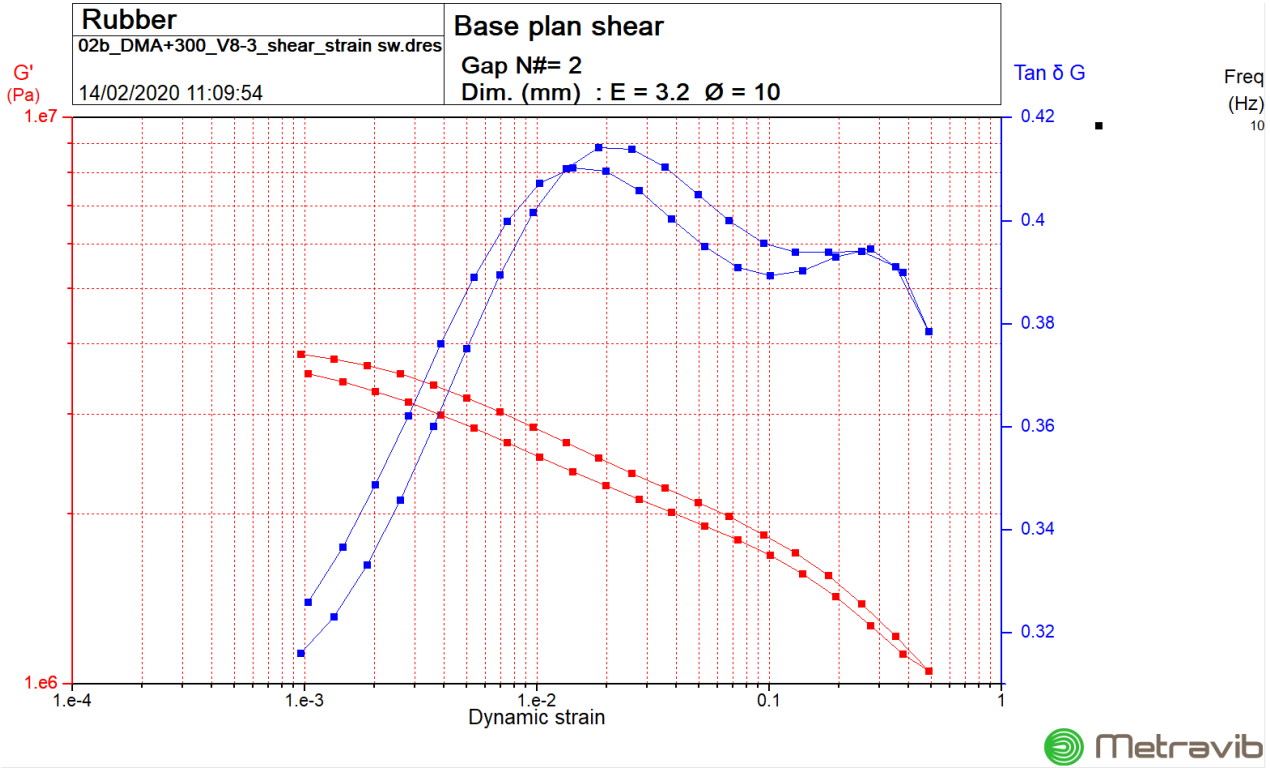
- Optimization of the test duration
- Storage of the temporal signals

New functionalities for strain sweep test

Optimisation of test duration

- New software version
  - More open software ;
  - Greater configuration possibilities.

- Experimental conditions
  - Mode : double shear
  - Sample : rubber (tire type)
  - Dynamic strain sweep : 0,1% up to 50% and 50% up to 0,1% ;
  - 20 measurement point;
  - Frequency : 10 Hz;
  - Room temperature



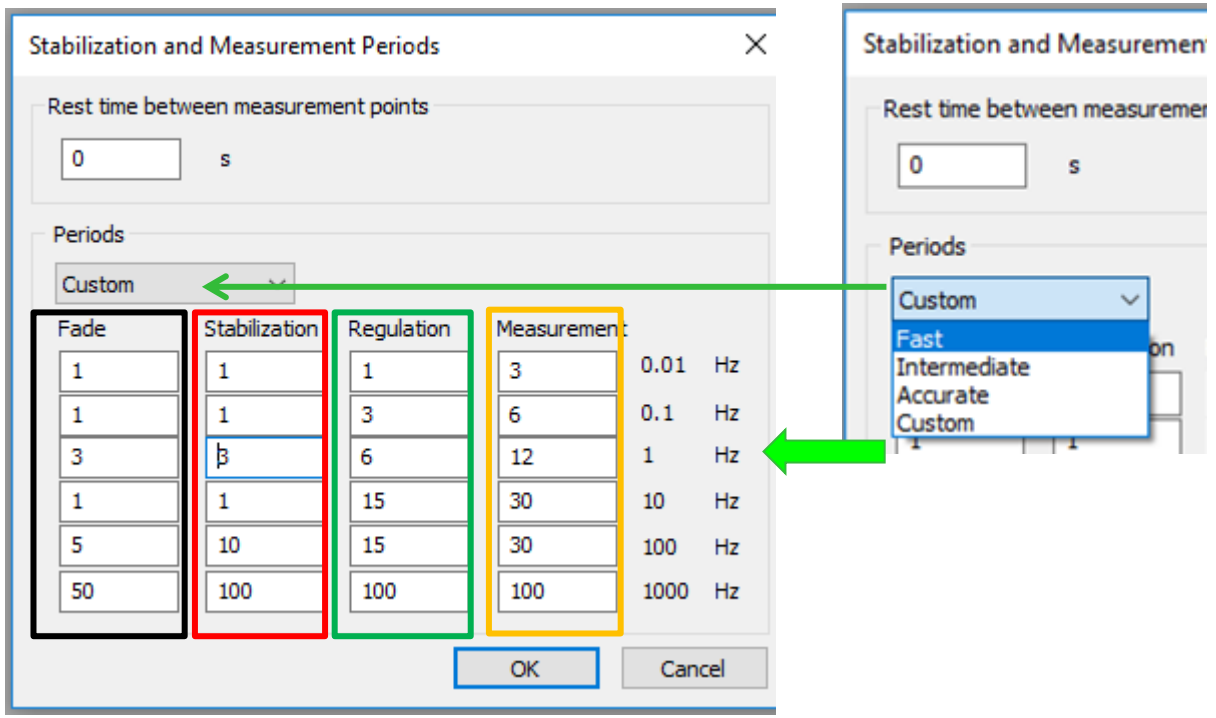
Duration test with old version : 9min

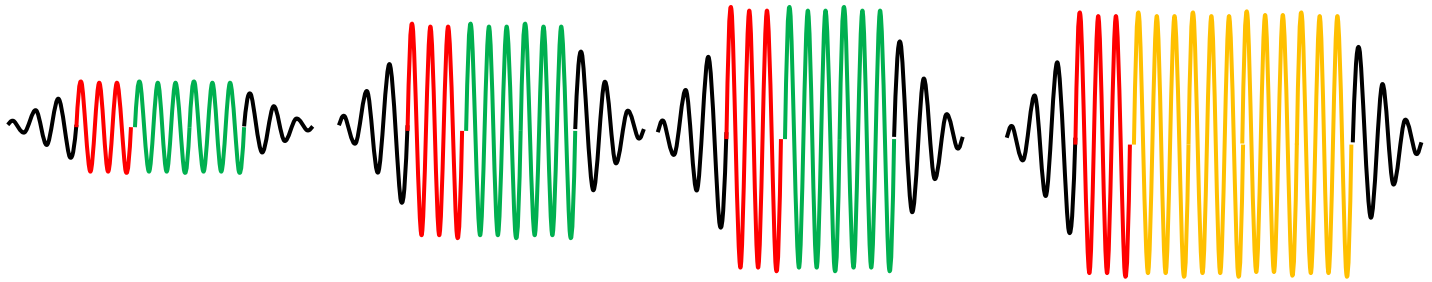
New functionalities for strain sweep test

Optimisation of test duration

One measurement point means several steps before :

1. Fade : transcient state, amplitude modulation to avoid overshooting (3 periods) ;
2. Stabilization : accomodation step in order to limit the Mullins effect (3 periods) ;
3. Regulation : acceptance tolerance (6 periods) ;
4. Measurement : average of 12 periods.





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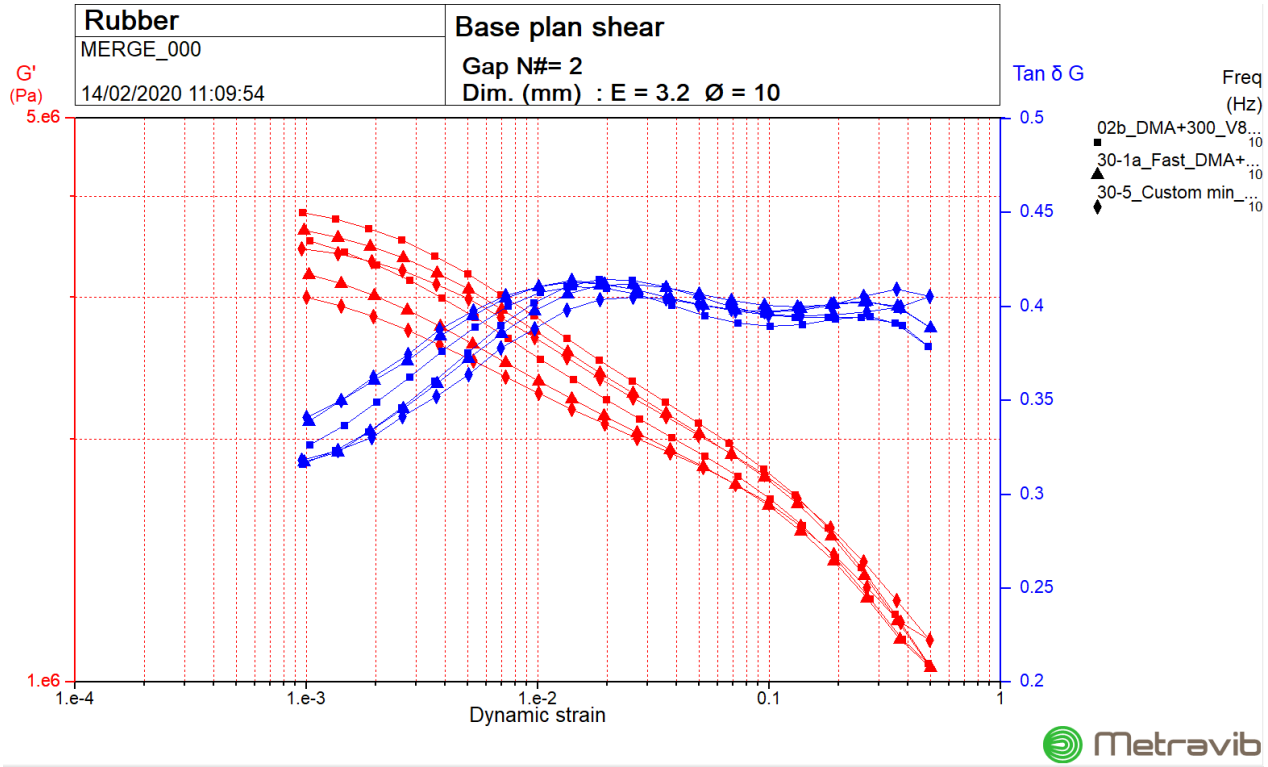
New functionalities for strain sweep test

Optimisation of test duration

Old version
- Parameters have been imposed (no user control)

New version :
- Creation of new menus (Fast, Intermediate, Accurate, Custom)

Menus	Test duration
Old version (no control)	9 min
Intermediate	5 min
Custom/Fast	2 min

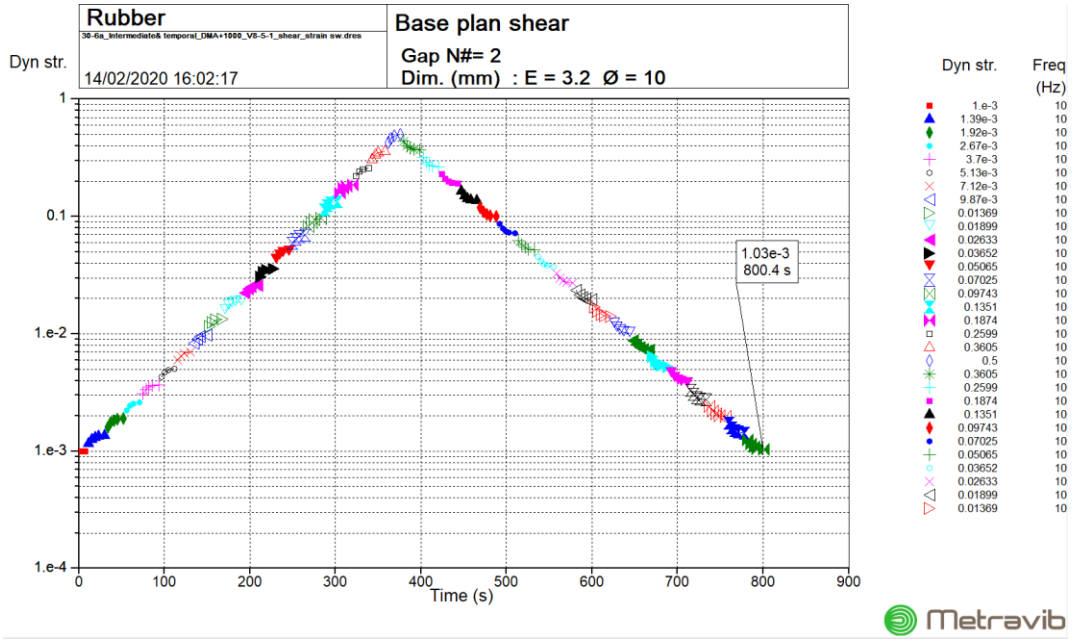


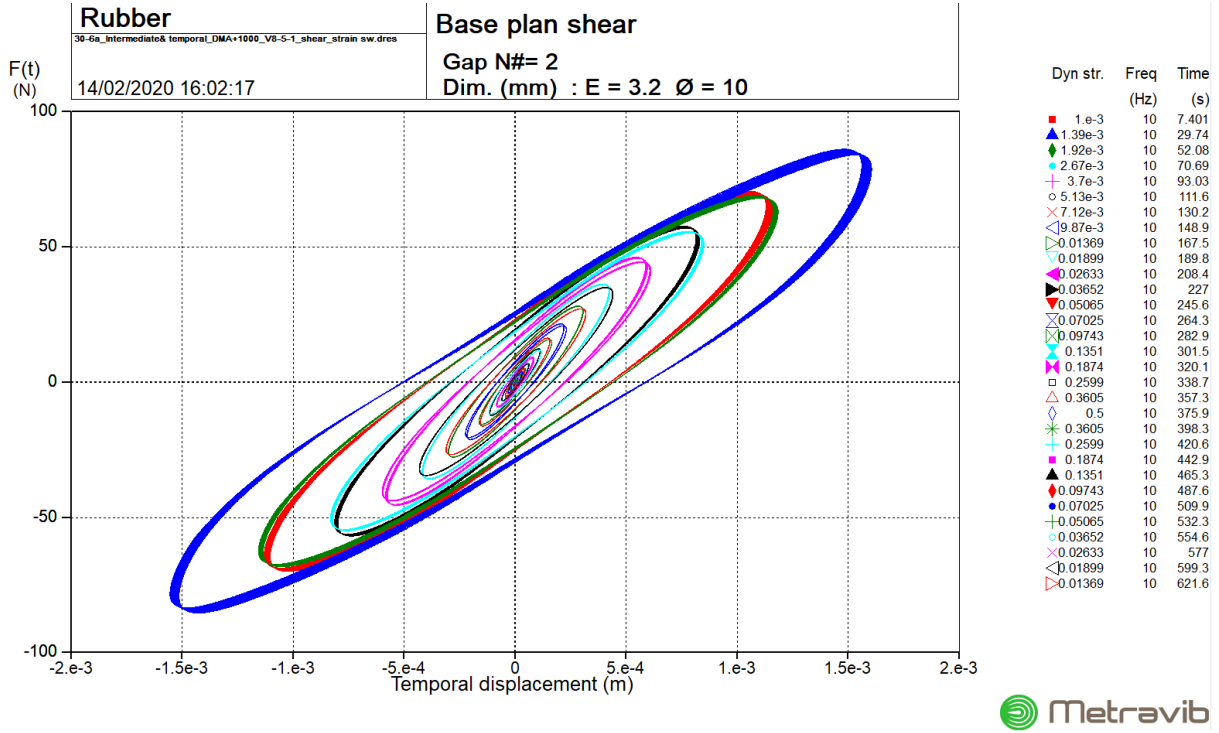
Benefits
- Control of excitation history during the whole test ;
- The energy injected into the material during the test is known and can be controlled (limitation of self-heating phenomena) ;
- The test duration can be reduced (time saving and productivity gain)

Results obtained in the different modes
→ the curves overlap

New functionalities for strain sweep test

Stores the temporal signals





Benefits

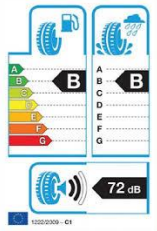
- More accurate analysis of non linear materials
- Energy calculation
- Extend capabilities of analysis

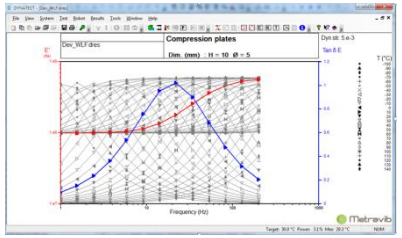
The shape of the loops :

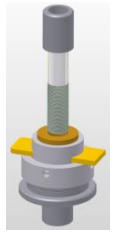
- Is representative of the viscous nature of the behavior (dissipation in thermal form) ;
- provides information on the nature of the damping (viscous behavior, mechanical damping, etc.).

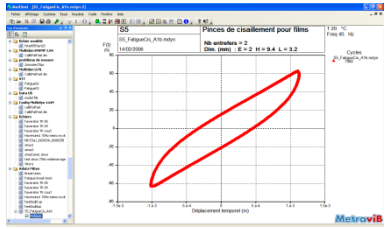
Conclusions

METRAVIB – Material testing









DMA

- E*, G*, Tan delta (Covering 6 to 7 decades)
- Glass transition
- Curing process
- Payne effect (High force capability)
- Mullins effect (High force capability)
- Frequency effect (High frequency capability)
- Static tests (Creep, stress relaxation, tensile…)
- Environment/immersion/humidity effect
- Automatic Master curves
- Automatic Long term creep prediction

FATIGUE

- Fatigue testing
- Waveform control
- Multi harmonics analysis

HBU/ FLEXOMETER

- Heat Build Up exceeding standard requirements

CRACK GROWTH

- Automatic crack growth testing , tearing energy …

- Automated DMA tests with Xpander in tension, compression and shear modes

METRAVIB – Material testing

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