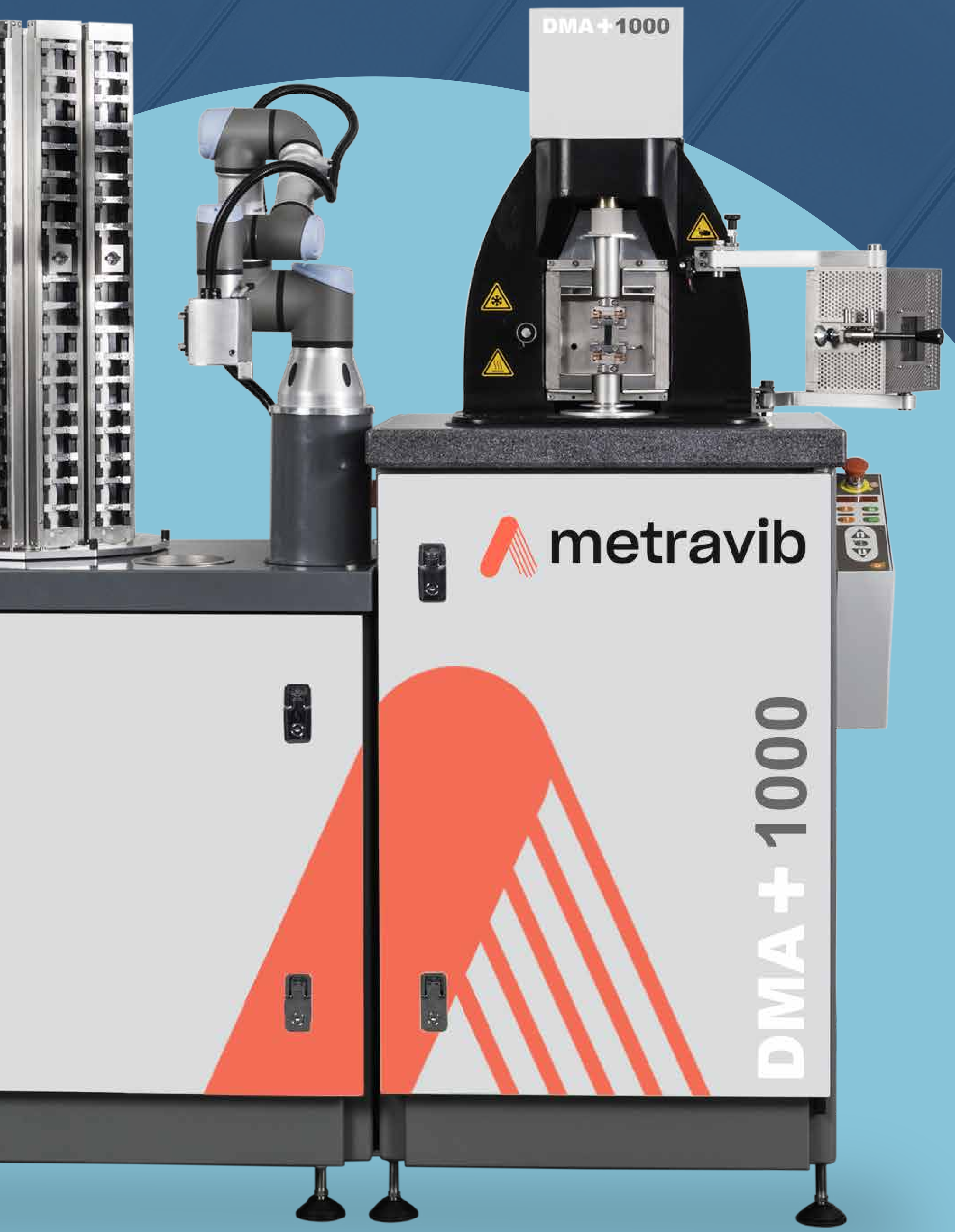
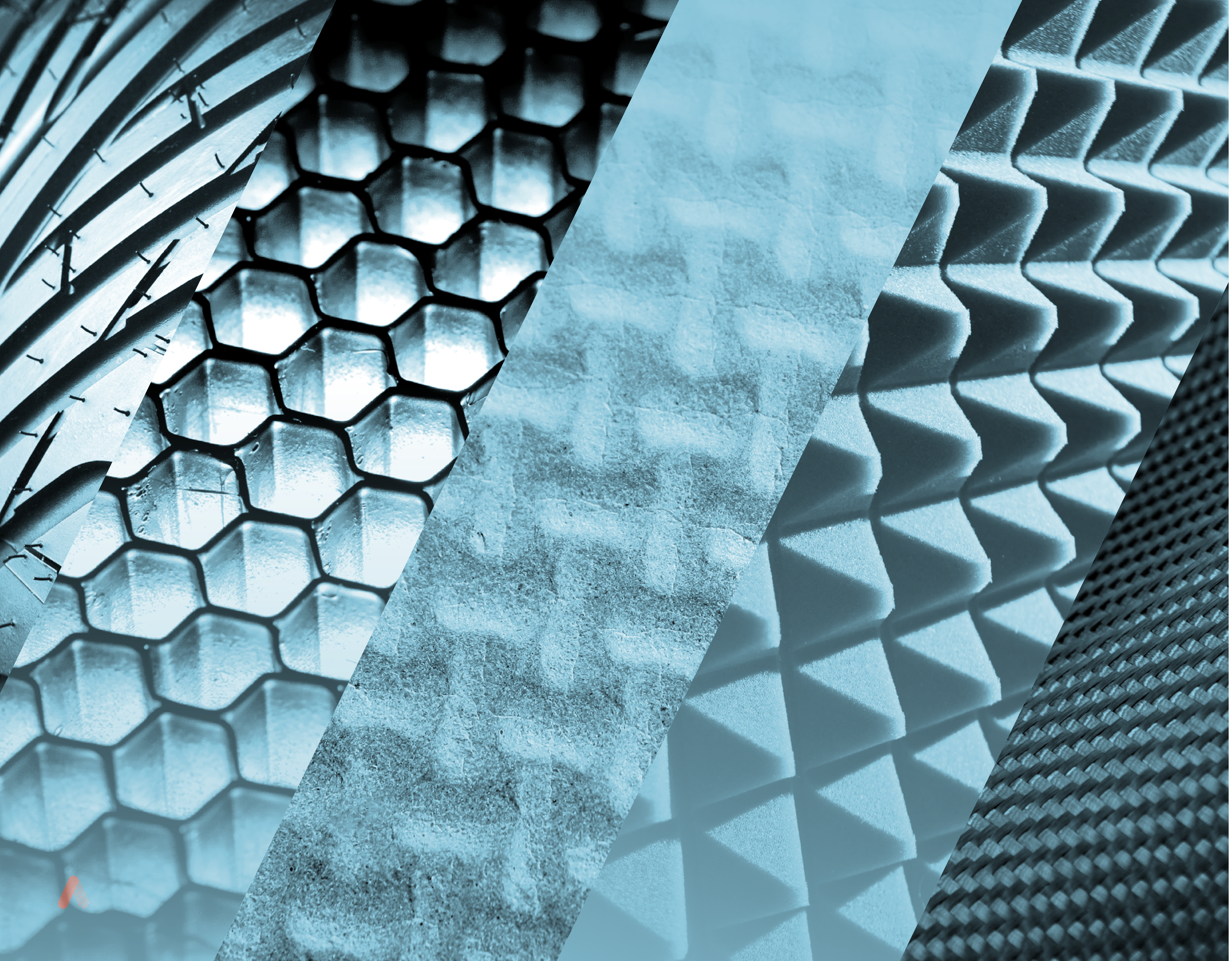
metravib
material testing



DMA+ series

DMA+300 / DMA+1000 / DMA+2000

ULTIMATE DYNAMIC MECHANICAL ANALYZERS
FOR ACCURATE DMA AND FATIGUE TESTING



A world of materials

The diversity of materials used for industrial needs is continuously expanding to address new challenges.

Advanced materials are designed to be stronger, lighter, more durable, more resistant to severe environmental conditions, and better performing.

In this material world, polymer-based materials such as thermosetting polymers, thermoplastic polymers, elastomers, composite materials, occupy a place of choice.

They are developed to meet specific requirements in extremely various fields such as automotive, aerospace, electronics, communication, medicine, energy infrastructure and even food.

Their complex research, development and production processes and quality control require advanced technologies to characterize and test their performance.

Within the required properties, elasticity, thermal dependence and durability are of major importance.

In a few decades, Dynamic Mechanical Analysis has become a major technique for characterizing, in a fine and relevant way, the thermomechanical behavior of materials.

In the materials development chain DMA is now a strategic link between the chemists who develop the materials and the engineers who use them to address specific functions requiring a strong expertise of their performances.

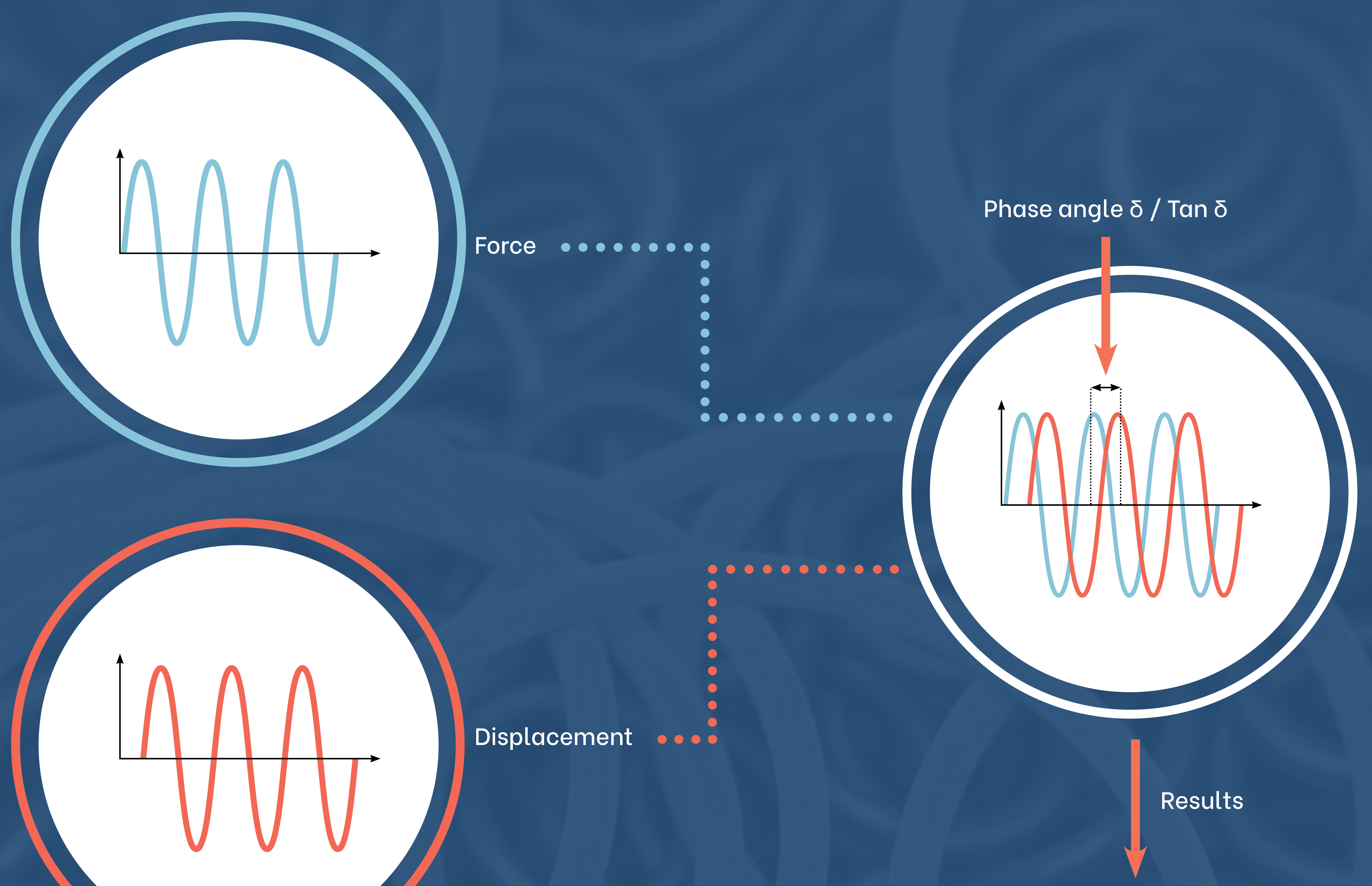
DMA technique

The Dynamic Mechanical Analysis (DMA) is a powerful technique to analyze the mechanical behavior of the material, and especially its viscoelastic properties.

A DMA test consists in applying a sinusoidal excitation to a material's specimen at controlled amplitude and frequency, out of resonance.

The measurement of applied force and resulting deformation (or displacement) allow to obtain:

- The Stiffness of the material's specimen, K which is the ratio of the applied force (N) divided by the resulting displacement (m)
- The phase angle δ between the applied force and the resulting displacement signals.

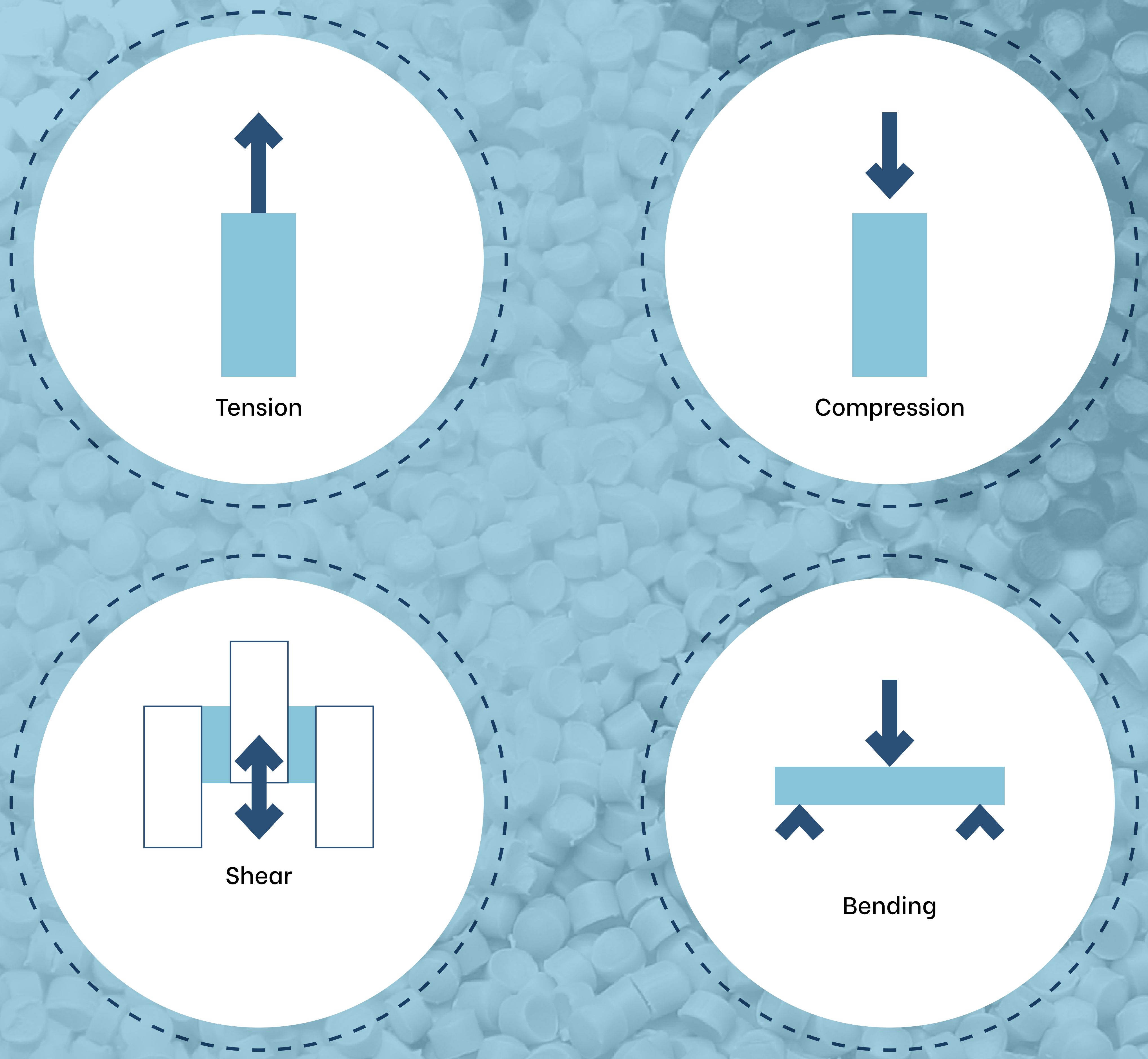


Stiffness: $K^* = F / D$

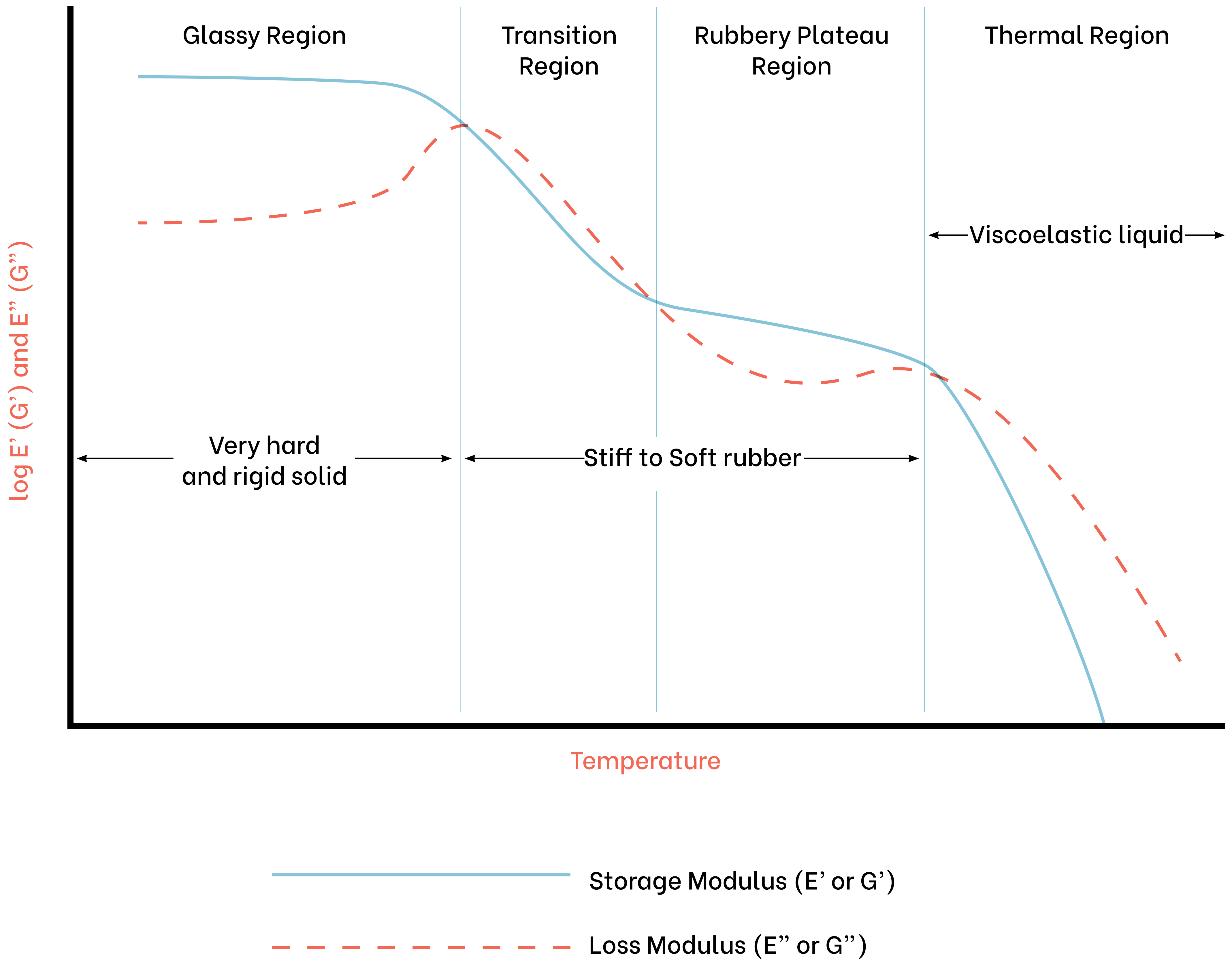
Modulus: E^* or G^*
E = Shape factor (Length/Surface). K

$\tan \delta = K'' / K'$

The linear excitation is applied to the material through various specimen holders whose design allows applying variable excitation modes to the specimen: tension, compression, shear, or bending.



Viscoelastic spectrum for a typical amorphous polymer versus temperature.



The geometry of the specimen shall respect drastic rules to ensure the accuracy of the analysis data (standard ISO6721 or equivalent).

From specimen dimensions, and the complex stiffness K* measurement, one can obtain a very accurate calculation of the complex Young modulus E* or Shear modulus G*.

- Tangent Delta (Tan δ) is calculated from the loss angle (δ).
- The real part (E' or G') and the imaginary part (E" or G") of the modulus express the elastic and the viscous behavior of the material respectively, in other words the ability of the material to store or to restitute energy.
- The temperature control during the dynamic test allows obtaining a complete picture of the material mechanical properties dependence to temperature. The DMA technique is especially the most accurate technique to determine the glass transition of materials (Tg).

The most capable instruments to measure the viscoelastic properties of materials and achieve the largest dynamic tests panel

Since 1968, Metravib is working in close cooperation with leaders of polymer and rubber industry to develop new testing instruments and continuous improvements meeting the expectations of scientists, compounders, research & development engineers.

The DMA+ series is the latest achievement of this strong and unique experience.

- Accurate technologies to measure low to high amplitudes of displacement and force
- Efficient and reliable environmental system
- Specimen holders to accommodate with a large variety of materials and tests
- Smart software to control routine and advanced tests
- Customizable to address specific testing needs

The DMA+ concept: Mechanical rigidity is the key!

In the DMA technique, the accuracy of the data depends strongly on the ability of the DMA instrument to measure the true stiffness value of the material's specimen. The rigidity of the DMA test frame is definitely a major feature that defines the performance of the instrument.

Metravib DMA+ series is based on a high rigidity test frame made in one piece cast frame.

This unique design has been elaborated to give superior testing capabilities and analysis performance:

- Accurate measurement of specimen deformation
- Soft to highly rigid specimen can be analyzed
- Large stiffness variations due to temperature effect may be accurately followed up
- High frequency measurement up to 1kHz can be performed without the need for calculating master curves

Additionally, the unique mechanical design offers unmatched ergonomics which facilitates access for specimen loading and adjustment required for positioning.

The retractable thermal chamber gives access to the specimen over 230° while the free standing design of the instrument offers a comfortable handling of the specimens by the operator.

Tailor your own DMA+ configuration among numerous options, accessories and capabilities extensions.

The configuration of your DMA+ can be tailored to suit your testing needs and budget!

In addition, a majority of options can be implemented later depending on the new needs of the test laboratory.

- Hardware and software testing capabilities extensions
- Specimen holders accessories
- Cold and environmental sources
- Specimen preparation tools
- Automation
- Customized adaptations

Regardless of the configuration, the DMA+ will be as easy to drive for expert engineers and scientists needing advanced test programming, as it is for first time users using predefined routine tests.

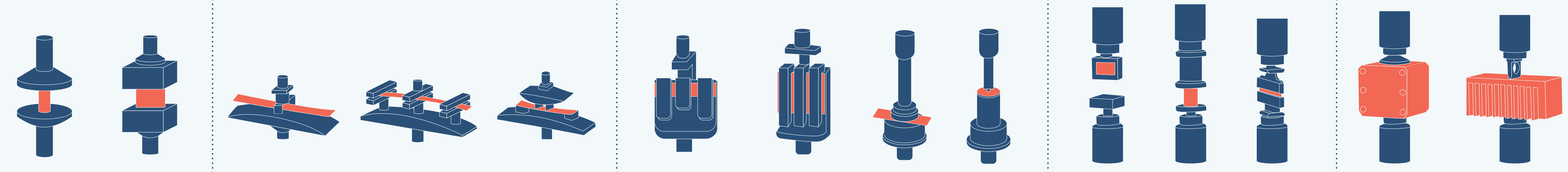
Adapt your DMA+ to your materials

A unique range of specimen holders allows to accommodate with the nature of the materials you need to test: solids, pastes, and even powders or liquids, and various shapes: rectangular, cylindrical specimens, films, fibers, cords...

The linear excitation can be turned into various test modes: Compression, Shear, Tension, Bending.

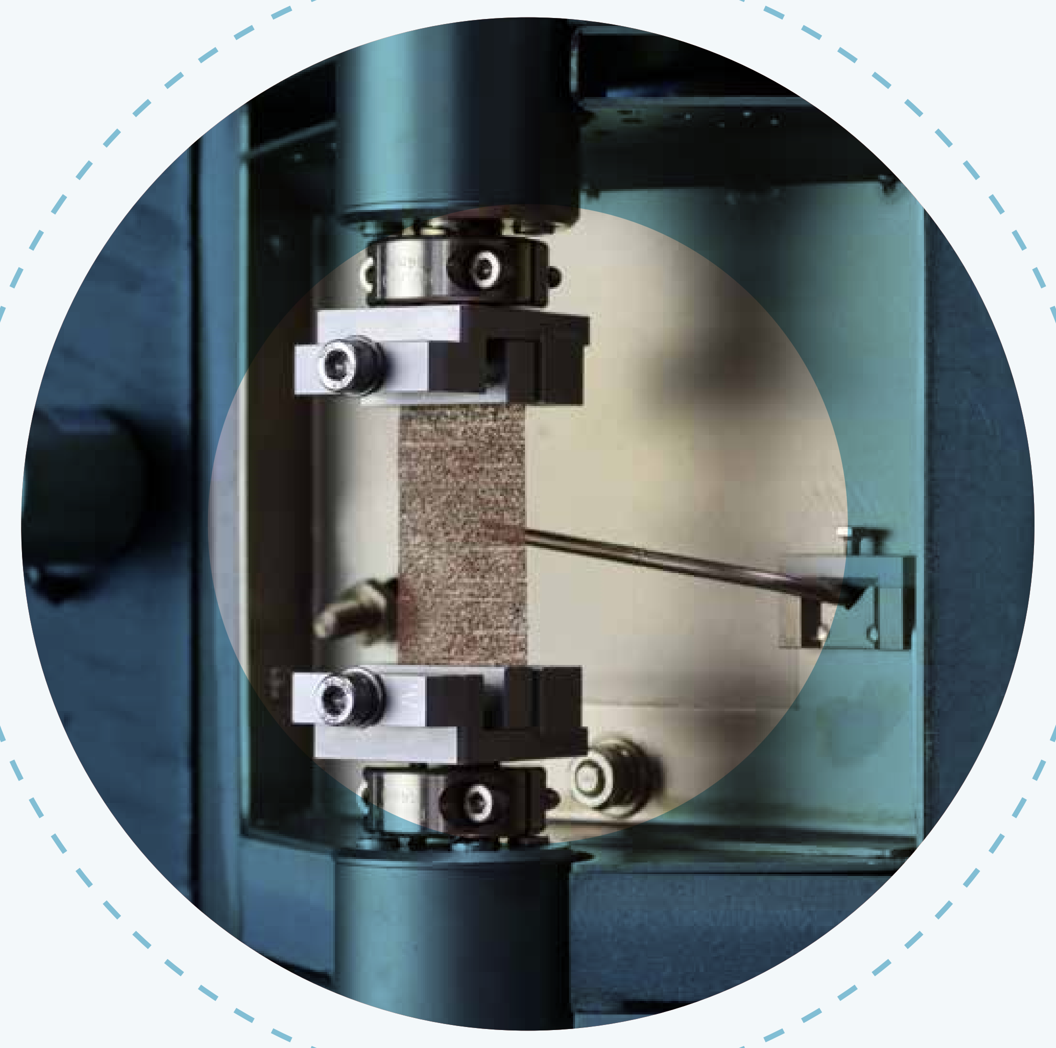
Dedicated specimen holders are designed to achieve special tests, such as polymerization, heat build up (HBU), matrix/ fiber interface, fatigue crack growth testing...

Most of specimen holders can be used as well to analyze the materials properties while immersed in a liquid with dedicated accessories.



	Tension compression		Bending			Shear				Specific devices for elastomers Pull out - HBU - Crack			Accessories for immersion tests	
Rubbers (cylinders)	●									●	●		●	
Rubbers (strips)		●				●	●					●	●	
Reinforced Polymers	●	●	●	●	●		●						●	●
Polymers (films)		●					●						●	
Polymers (fibers)		●											●	
Thermosets (cured)		●	●	●	●								●	●
Thermosets (uncured)								●	●					
Thermosets (prepregs)		●					●						●	
Bitumen, pastes								●					●	
Metals, alloys			●	●	●								●	●
Glass, ceramics			●	●	●								●	●

Specimen holders: Tension-Compression-Shear Testing



Universal tension jaws

The Metravib Universal tension jaws is the most versatile specimen holder.

Its smart design makes it suitable for films and fibers as well as for rectangular solid materials.

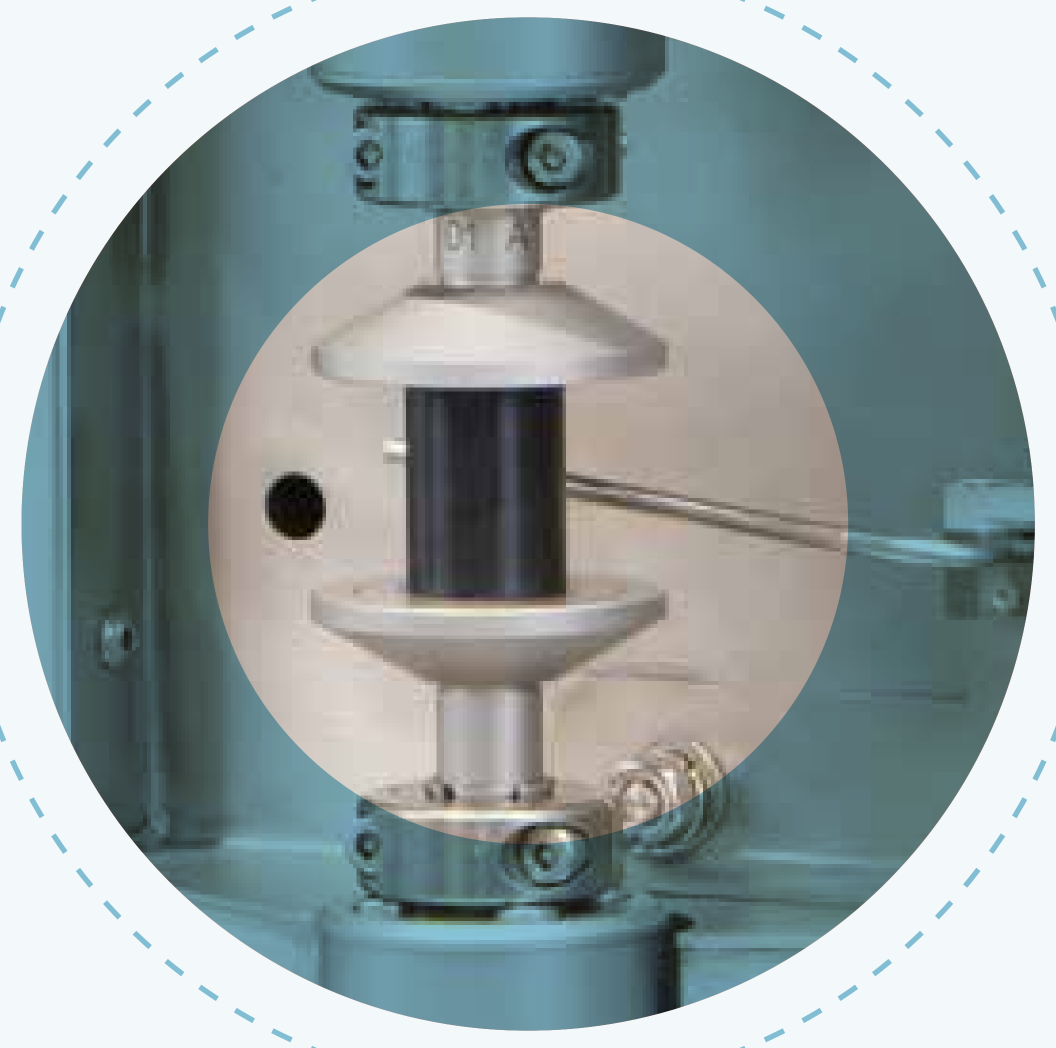
The 20 mm wide jaws are reversible with two faces of different shapes in order to suit to rigid materials and prevent indentation in soft materials.

The 4 removable jaws dedicated to fibers and cords avoid the risk of tearing the sample in the jaws.

They make possible to efficiently clamp the fibers and films by avoiding slippage and to position them precisely by managing the precise adjustment of the tension.

The jaw scale greatly facilitates the placement of the specimen and a centering tool facilitates the relative positioning of the upper and lower jaws and ensures precise adjustment of their angular positioning. Specimens up to 60 mm high can be tested.

Universal tension jaws are suitable for:
Films, including; resins, adhesives, and paper.
Fibers, including; fibers, glass fibers, hairs and cords. Bars, including rectangular or tubulars specimens. Thermoplastic polymers, thermosetting polymers, rubbers strips, paper, leather, food and more.



Compression plates

Parallel compression plates are best suited for soft solids and for moderate modulus solids. With a diameter of 40 mm they present concentric circles for an optimized specimen positioning. Specimens up to 60 mm high can be tested.

Bonding with the right glue can sometimes be a nifty way to give a solution to difficult materials.

Compression tests are recommended for all types of solid materials, thermoplastic polymers, thermosetting polymers, elastomers, bitumen, food and more.

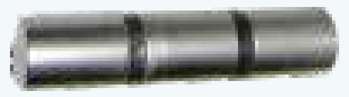
Shear for solids

Two identical specimens of the same material are bonded in between three cylindrical metallic supports. The resulting sandwich specimen is clamped inside three jaws. The central one provides the excitation, while the two lateral ones are fixed.

This geometry is currently used for rubber testing. In the tire industry, it corresponds to a standard test adopted by tire manufacturers, and their materials suppliers.

The geometry is suitable to cover a large strain range and to characterize the non linear behavior of elastomers (Mullins & Payne effects).

Combined to the high rigidity of the DMA+ mechanical frame, it leads to a precise viscoelastic properties measurement, including the glassy transition region where the specimen become very stiff.



Shear specimen

Specimen holders:
Shear testing

Shear for pasty materials

This specific holder is dedicated to the analysis of pasty materials.

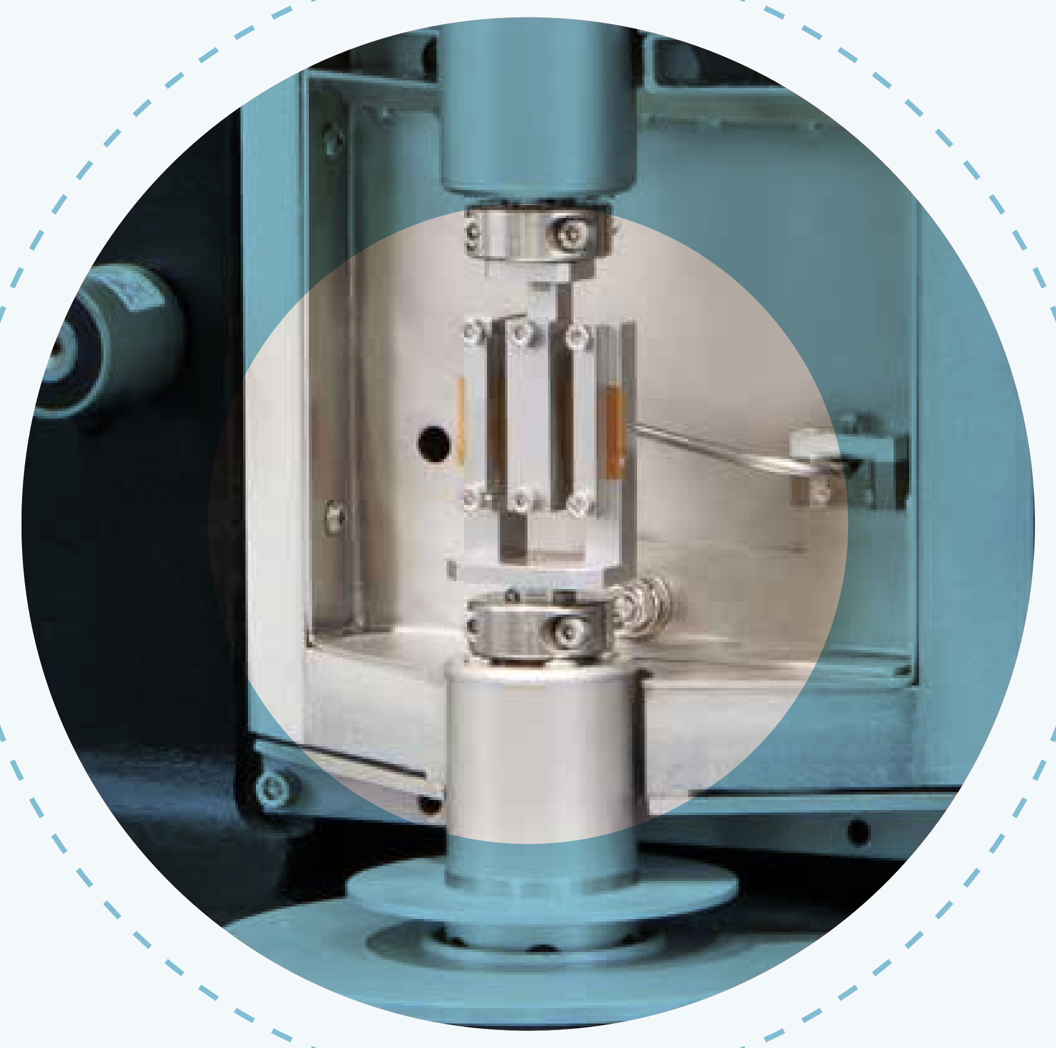
It is useful to analyze curing and polymerization processes.

It is especially suited for thermosetting polymers.

Large variations of viscosity can be measured in one single test.

A set of accessories allows accommodating with various viscosity or shear modulus ranges.

A material specimen volume of less than 1 cm3 only is required.



Shear for films

Polymer films are usually tested with tension holder. Nevertheless for materials exhibiting large dimensional variations under specific conditions of stress or temperature, the expansion can be too large to authorize a traditional tension test.

Then the shear films holder represents an efficient testing solution to achieve such test without making necessary to control a static stress or strain.

The shear films holder makes then possible to follow up large modulus variations over large strain and temperature ranges.

The specimen is mounted on the supports by precisely controlling its flatness.

A set of accessories allows accommodating with various specimen dimensions. Specimens surface of up to 30 x50mm can be tested.

Shear for liquids

This specific holder is dedicated to the analysis of liquid materials.

This holder is useful to analyze curing and polymerization processes.

It is especially suited for low viscosity materials.

Large variations of viscosity can be measured in one single test.

A set of accessories allows accommodating with various viscosity or shear modulus ranges.

A material specimen volume of less than 3 cm3 only is required.

Specimen holders:
Bending modes

4 points bending

In the 4 points bending test the specimen lays free on the 2 lateral supports while the opposite 2 supports in the middle of the specimen apply a combination of static load and dynamic excitation.

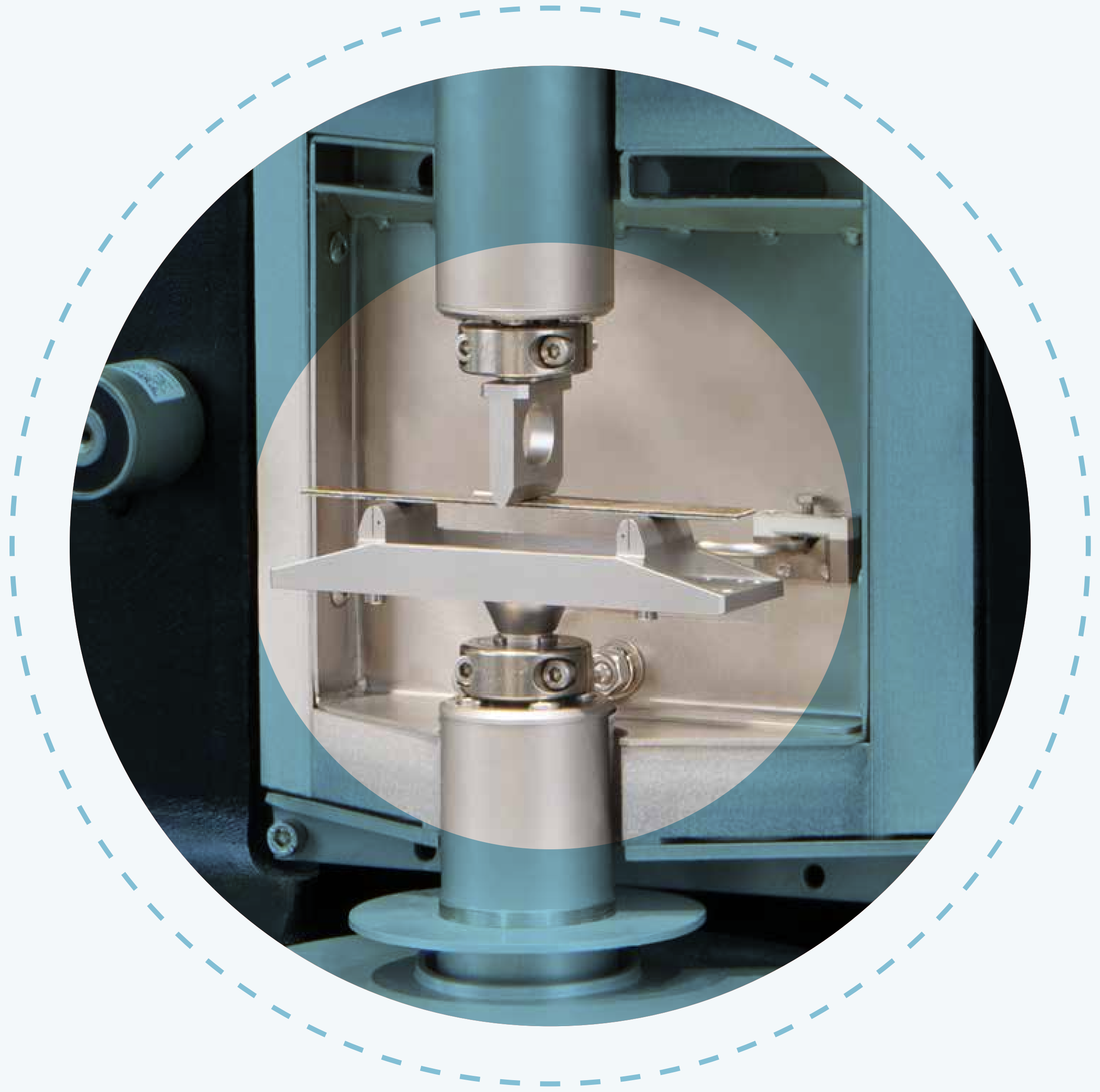
In the 4 points bending test configuration, the specimen is subjected to a bending stress that is distributed more evenly across the specimen compared to the test configurations in the 3 points bending mode. This results in reduced stress concentrations, which can lead to more accurate fatigue life predictions and a better understanding of the material's behavior under cyclic loading.

The span, adjustable from 32 mm up to 116 mm and the support width of 26 mm, enables a large range of materials and specimen dimensions to be accommodated.

Perfect alignment between upper and lower parts are made possible with a specialised tool.

The specimen graduation allows for easy and optimized positioning of a specimen.

4 points bending tests are applicable for high modulus materials such as thermoplastic, polymers, thermosetting polymers, metals, alloys, ceramics, glasses and more.



3 points bending

The 3 points bending specimen holder is known to obtain accurate measurement of the Young modulus of high modulus materials. The specimen lays free on the 2 lateral supports while the opposite support in the middle of the specimen apply a combination of static load and dynamic excitation.

The recommended shape factor make possible to get good accuracy and repeatability. The low friction on supports allows getting a good resolution of Tan Delta measurement.

The span, adjustable from 16 mm up to 112 mm and the support width of 26 mm, allows accommodating with a large range of materials and specimen dimensions.

Perfect alignment between upper and lower parts are made possible with a specialised tool.

The specimen graduation allows for easy and optimized positioning of a specimen.

3 points bending tests are applicable for high modulus materials such as thermoplastic polymers, thermosetting polymers, metals, alloys, ceramics, glasses and more.

Single & dual cantilever

Contrarily to the 3 points bending specimen holder, the Single & dual cantilever holder imposes to clamp the specimen.

The specimen is clamped inside the 3 jaws and the excitation is provided by the central jaws without requiring a static load to be controlled.

The span, adjustable from 30 mm up to 112 mm and the support width of 26 mm, allows accommodating with a large range of materials and specimen dimensions.

This testing solution is especially valuable for thermoplastic polymers exhibiting large change of properties due to temperature effect.

It can be used as well for thermosets polymerization tests.

Generally speaking, the cantilever support represents a practical tool for evaluating the behavior of materials, but the clamping effect limits the accuracy of the data obtained.

Specimen holders: Specific devices for rubber testing

Heat Build Up holders

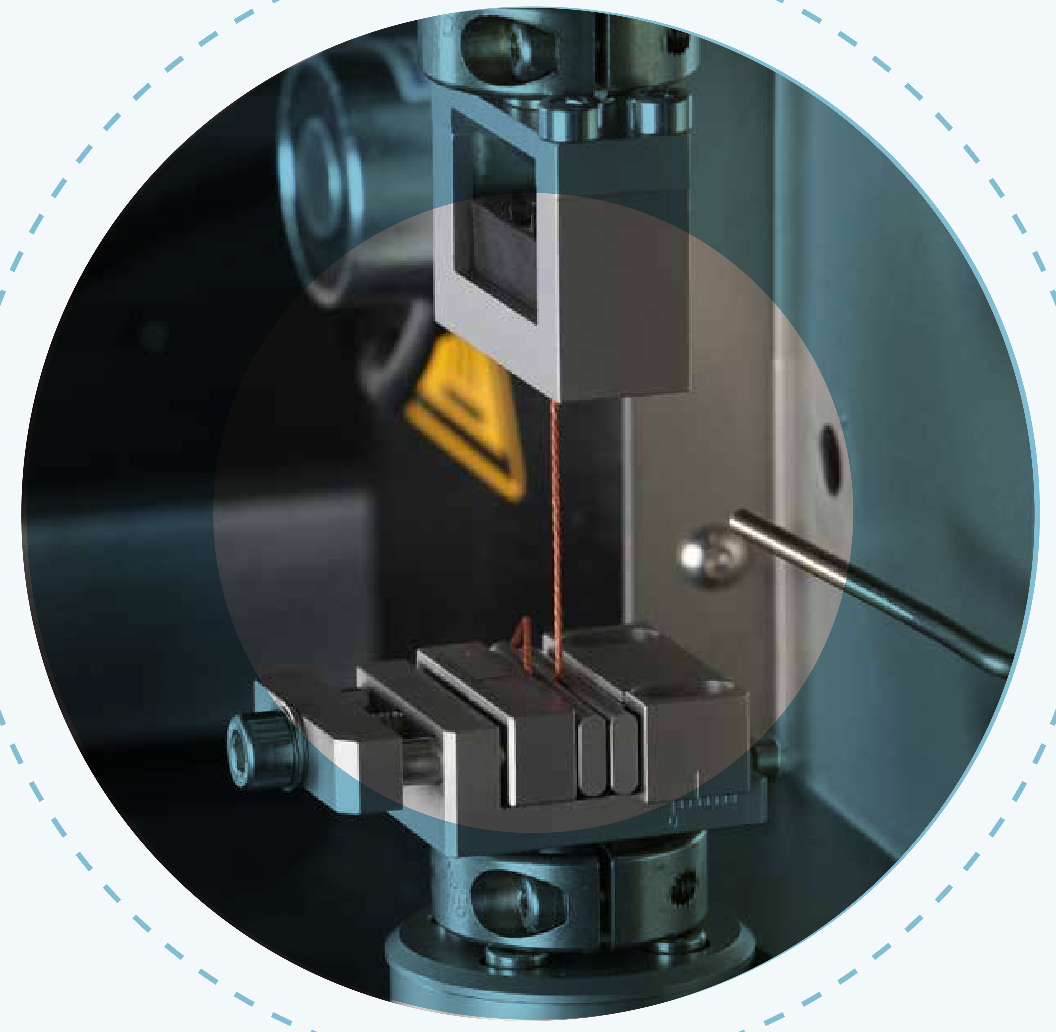
The Heat Build Up holders are devices dedicated to analyze the heat generation of a rubber specimen submitted to repeated deformation.

The holders are compression holders including thermal insulated plates.

A thermocouple is located inside the inferior plate in order to measure the temperature at the bottom surface of the specimen (Goodrich standard). The temperature change can be measured continuously over the dynamic test.

The most advanced HBU support version includes an active device for operating a needle temperature probe. The measurement can be programmed automatically at the end of the fatigue test, after stopping the excitation.

Then the needle temperature probe is pushed into the specimen to measure its core temperature.



Tension holders for pull out tests

This holder is dedicated to the analysis of the adhesion of a reinforcement textile cord with a rubber compound.

It requires a specific specimen made of a cord molded in the middle of a rectangular rubber specimen.

The rubber part is set into bottom holder jaws and the cord is fixed in the opposite jaws.

The holder can be used for tensile test, creep test or dynamic tests.

For dynamic testing, a combination of static load and dynamic stress is applied to the extremity of cord specimen.

The holder is compatible with the standard DMA+ series thermal chamber and may be used for temperature-controlled test over a large temperature range.

Fatigue crack growth tests holders

Different specimen holders allow mounting either rubber strips or specific test specimens molded with padding to optimize fixing during the fatigue test and avoid any risk of slipping in the jaws.

The use of pure shear specimen with a width up to 80 mm allows to follow up to 4 cracks in one single test and achieve reproducibility tests at once.

Specimen holders: Reservoirs for immersion tests

Liquid reservoir for bending holders

This accessory allows tests in bending modes of any rigid material featuring mechanical properties sensitive to liquid absorption.*

Liquid reservoir for tension, compression and shear holders

This accessory allows tensile, compression and shear testing of all materials with mechanical properties sensitive to liquid absorption.*

*Temperature control is provided by the standard DMA+ series thermal chamber. Accurate analysis of the impact of liquid absorption on the mechanical properties of the material can be performed even at controlled temperature up to 80°C.

The stainless steel construction of the holders, the aluminum construction of the liquid reservoirs and the seal-less design ensure compatibility with a wide range of liquids e.g. saline solutions, oils.

The precise temperature is measured by a thermocouple placed near the specimen in the liquid reservoir.

Cold and environmental sources

All sources are compatible with the DMA+ series thermal chamber.

The diversity of sources makes possible to deal with the specific configuration and needs of each test laboratory, in order to offer the solution best suited to its technical, economic or managerial criteria.

Cryogenic sources

Cryogenic sources are providing cold nitrogen gas generated by the controlled evaporation of liquid nitrogen.

The tank capacity and the corresponding investment can be chosen accordingly to the estimated laboratory needs and can be replaced easily if testing at low temperatures becomes more frequent or longer.

Chillers

An air chiller system replaces advantageously a classical cryogenic source in avoiding completely the use of liquid nitrogen, and removing all associated constraints of periodic supply and safety management; it offers additionally a fast return on investment.

Providing in the chamber temperature down to -70˚C, a chiller represents a cost-effective cold source for the most frequently used temperature range.

The use of chiller is especially recommended for automated DMA+ systems coupled to an Xpander robot for a 24/7 use.

Nitrogen Control Unit (NCU)

A nitrogen control unit can be used for connecting the DMA+ to a liquid nitrogen network or to a self-pressurized cryogenic source.

Humidity generator and controller (RH200)

For tests of material sensitive to humidity, the RH200 is an environmental system allowing a precise temperature control of the specimen and relative humidity over a wide range of operating conditions.

Specific atmospheres

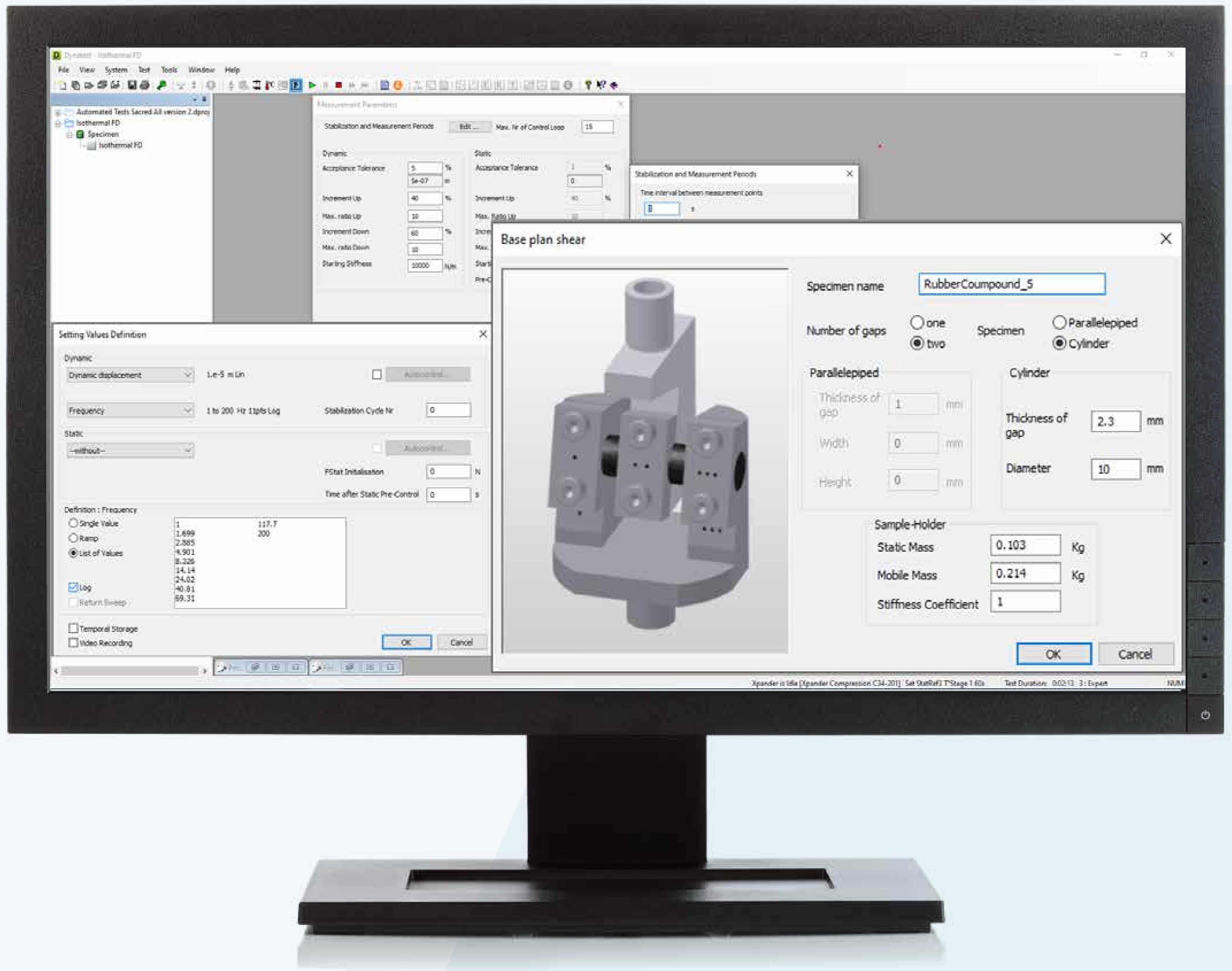
Specific systems allow to analyze the sensitivity of the material to specific conditions, such as the oxygen level or an inert atmosphere.

Dyna+ software: An extended test platform

Dyna+ is a powerful Software Test Platform dedicated to the DMA+ series, including all dynamic testing capabilities: all current and advanced DMA tests, fatigue testing for all materials, and advanced specific fatigue testing for rubber-based materials such as heat build up testing, and fatigue crack growth testing.

Associated with the capabilities of the DMA+ series mechanical design, Dyna+ makes possible to characterize most of polymer-based materials over unrivaled range of force, displacement, frequency and temperature.

Dyna+ provides unimaginable testing possibilities and unrivalled performance to satisfy the material testing need of advanced industry and research.



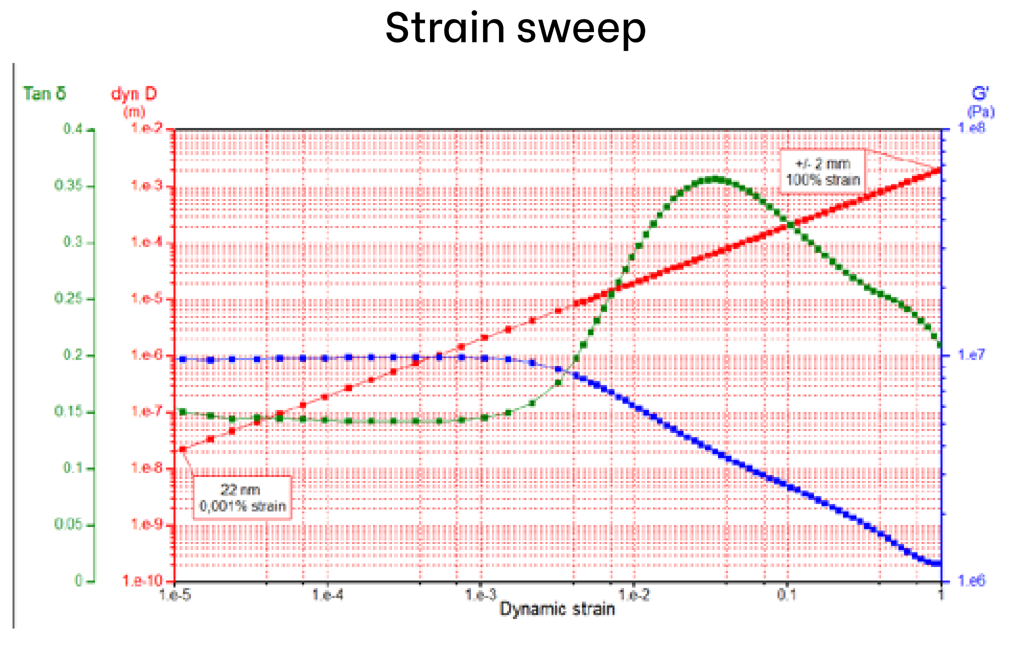
Advanced test control

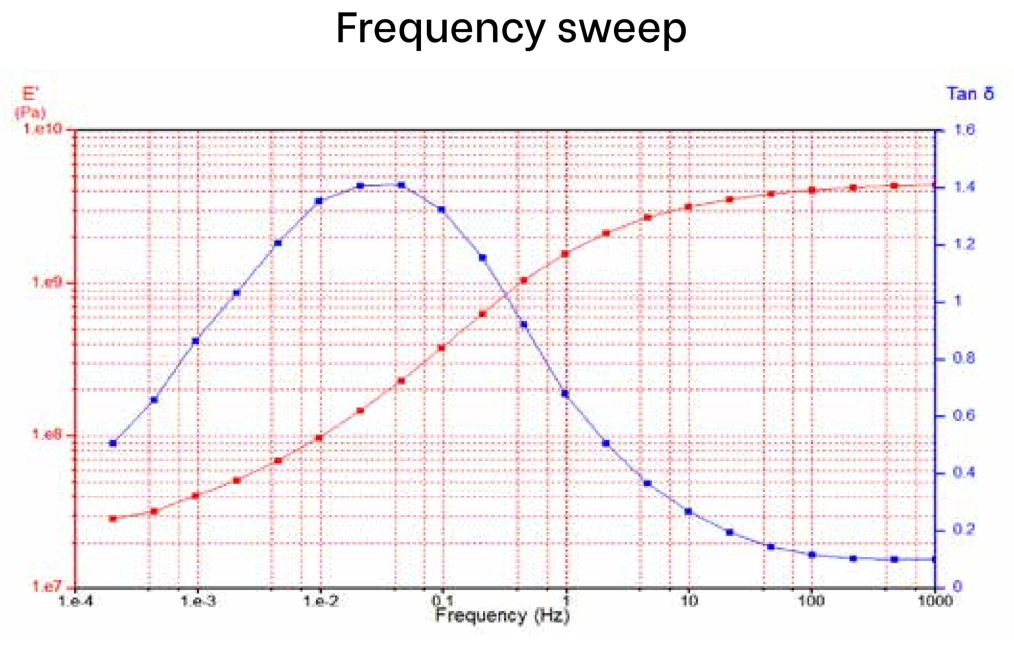
- Imposed strain /stress
- Free setting values and data points number
- Ergonomic user interface
- Full traceability of tests conditions
- Advanced control algorithms for linear and non linear behavior
- Identification of data points out of tolerance
- Auto-control mode
- Auto-tension mode
- Chaining DMA & Fatigue tests
- Temporal signal recording
- Advanced Thermal Calibration (ATC)

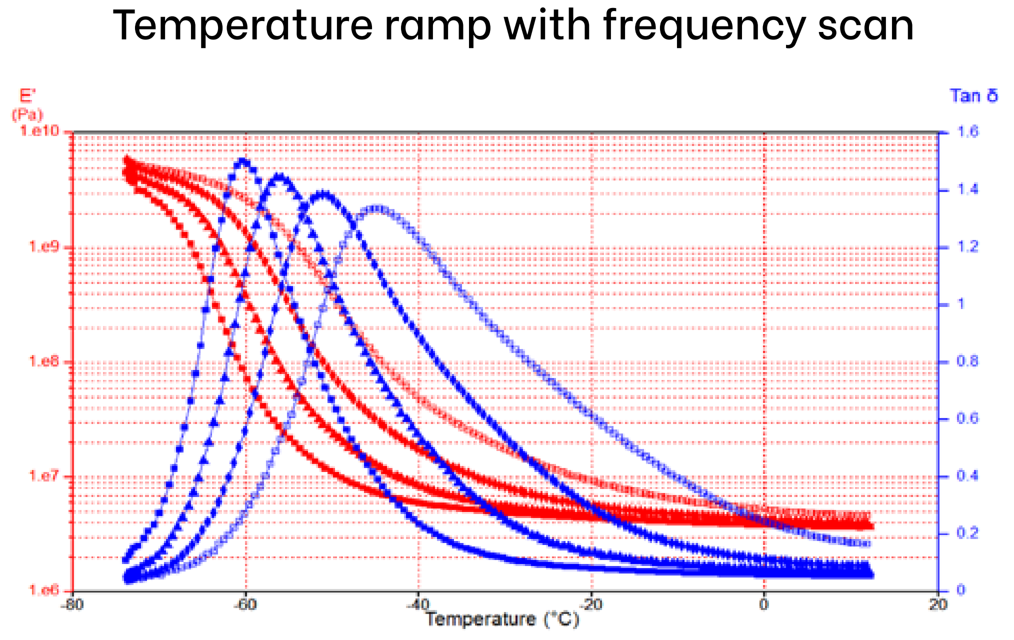
Dyna+ software: An extended test platform

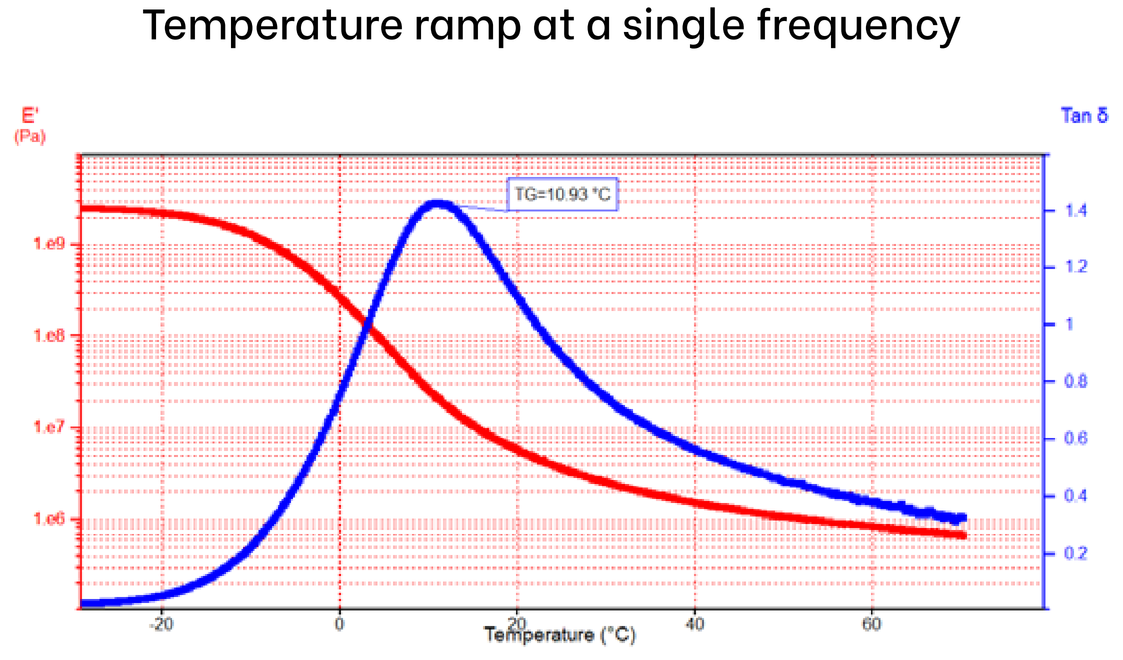
DMA testing

- Temperature ramp test at single frequency
- Temperature ramp test at multiple frequencies
- SLR mode to compensate for the specimen thermal expansion
- Frequency sweep on temperature stages
- Strain sweep (up & downwards) on temperature stages
- Stress sweep (up & downwards) on temperature stages
- Double sweep Frequency/ strain & stress on temperature stages
- Curing follow up at single or multiple frequencies
- Master curves computation
- Creep & relaxation test on temperature stages
- Long term creep prediction by Time Temperature Superposition (TTS)









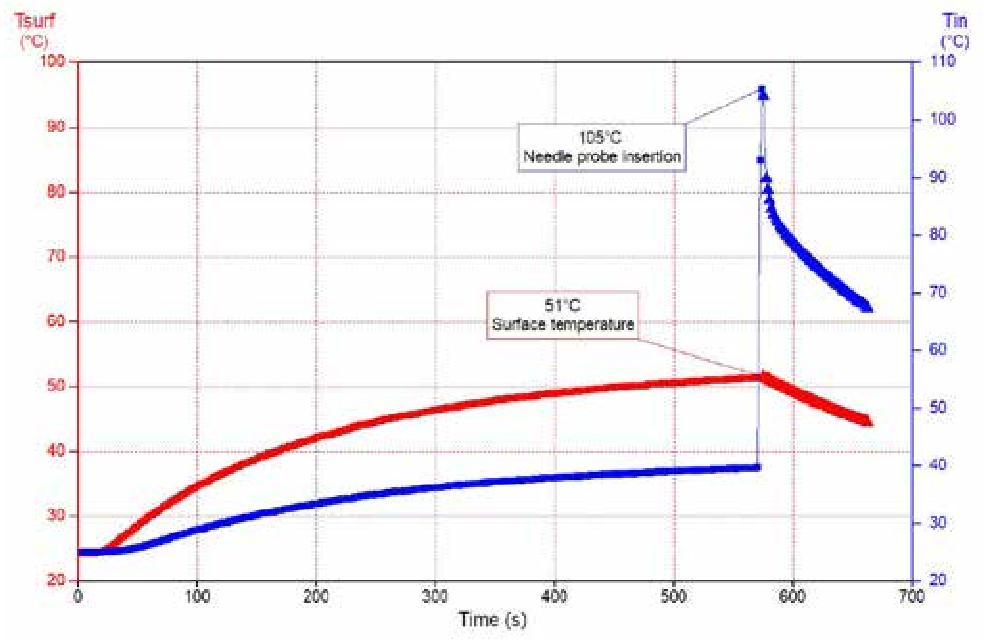
Dyna+ software:
An extended test platform

Fatigue testing

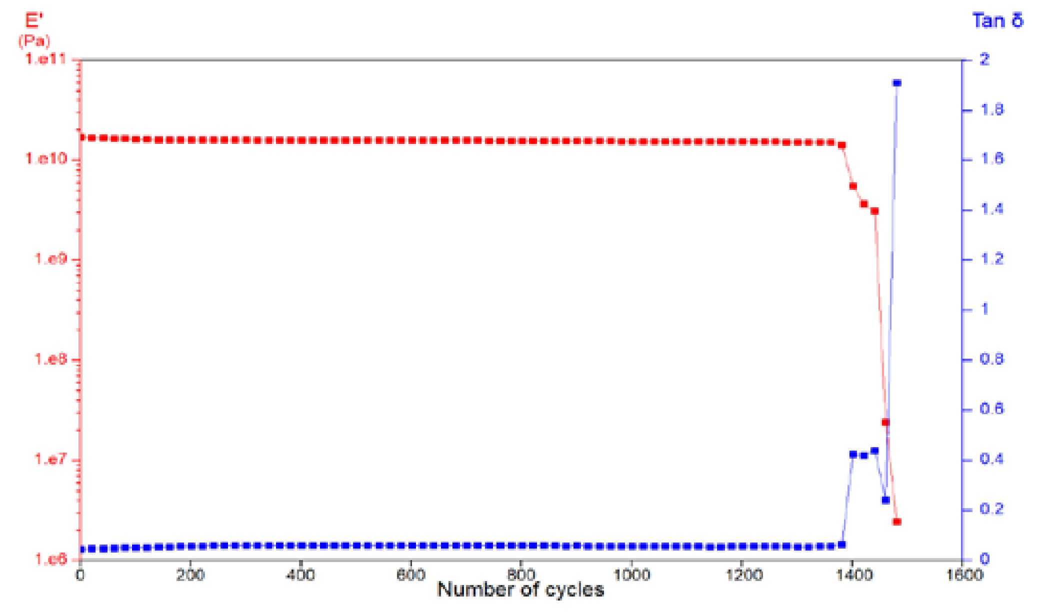
- Waveform control: Sine, Haversine, Pulse, Triangle, Square
- Customized wave forms importation
- Multiple harmonics control
- Fatigue - Heat Build Up module *
- Fatigue - Crack growth module*

(* option)

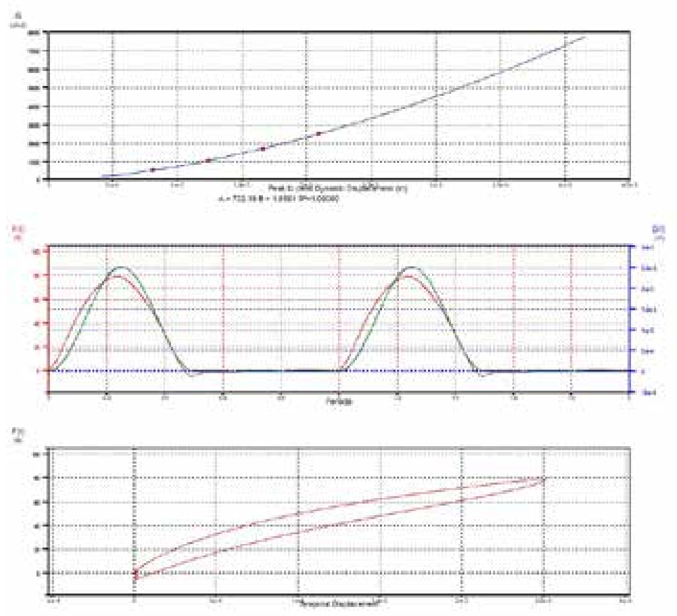
Heat build up test



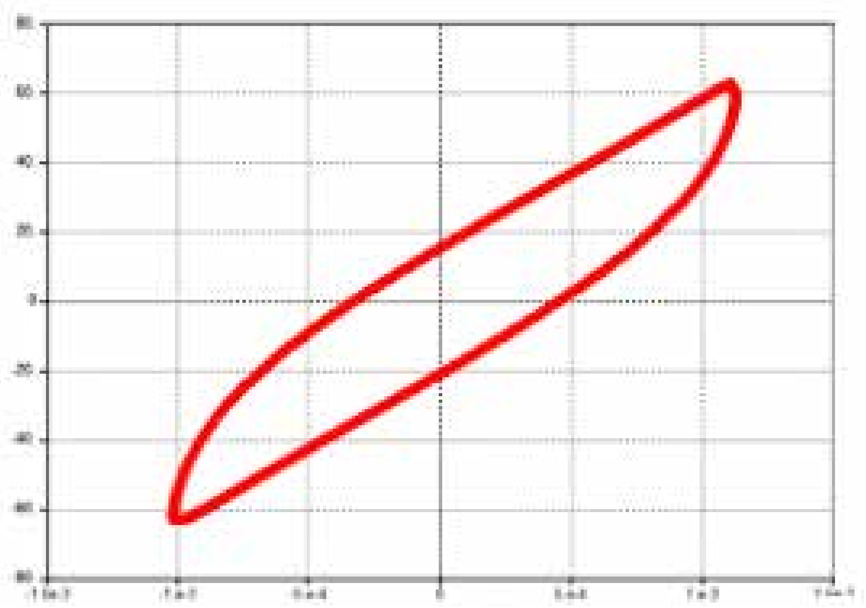
Fatigue to rupture test



Waveform control



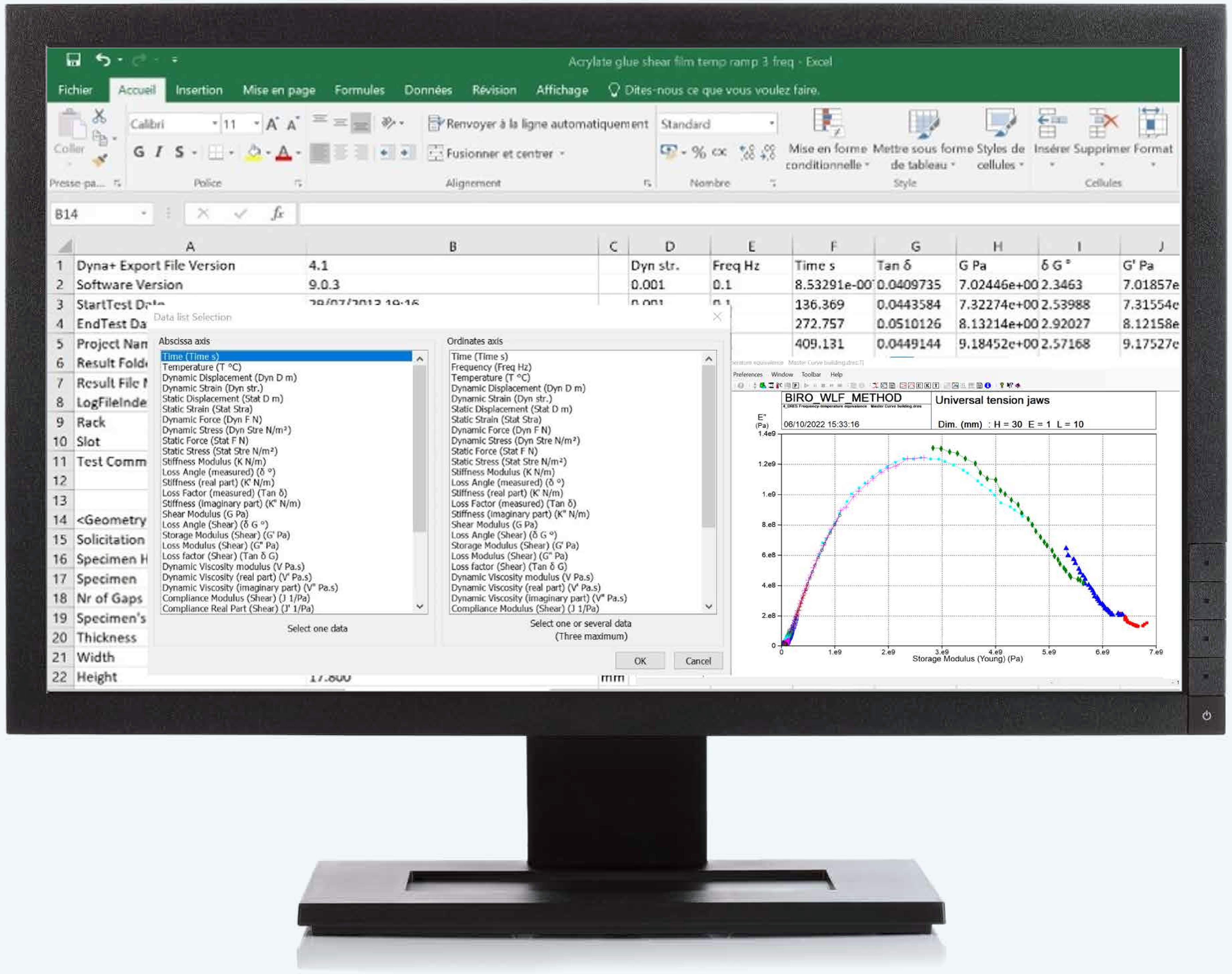
Lissajous curve



Dyna+ software: An extended test platform

Data exploitation

- Real time monitoring views
- Automatic selection of data out of tolerance control
- Automatic data storage (Ex: power line shut down)
- Automatic current views
- Automatic characteristic points determination (TG, Tan δ peaks)
- Customized views
- Customized report
- Automated report
- Csv file export (Excel compatibility)
- Compatible with LIMS (Laboratory Information Management System)



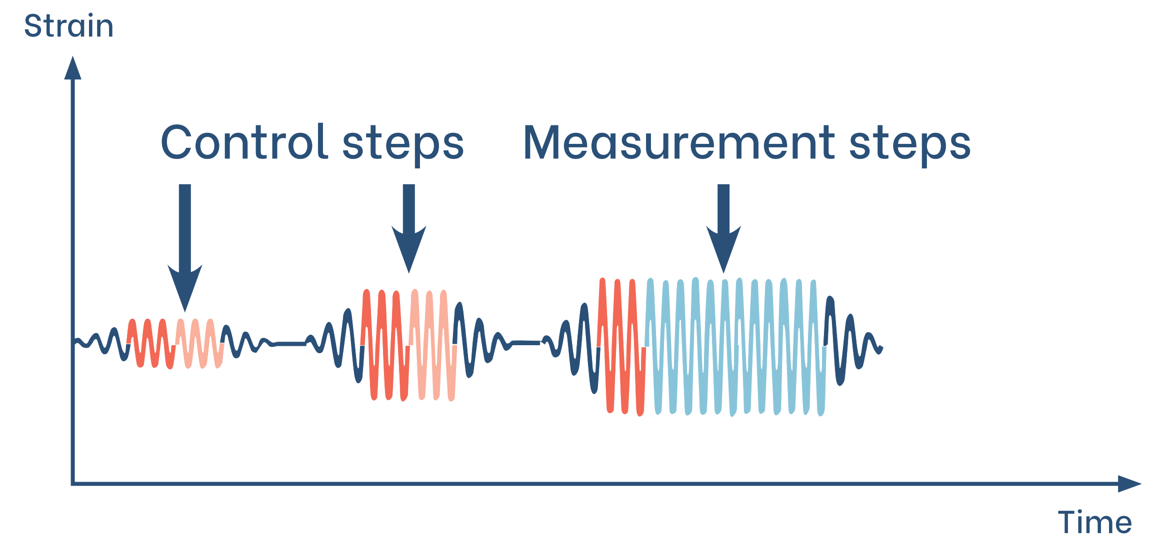
Dyna+ software: Smart functions for a perfect test control

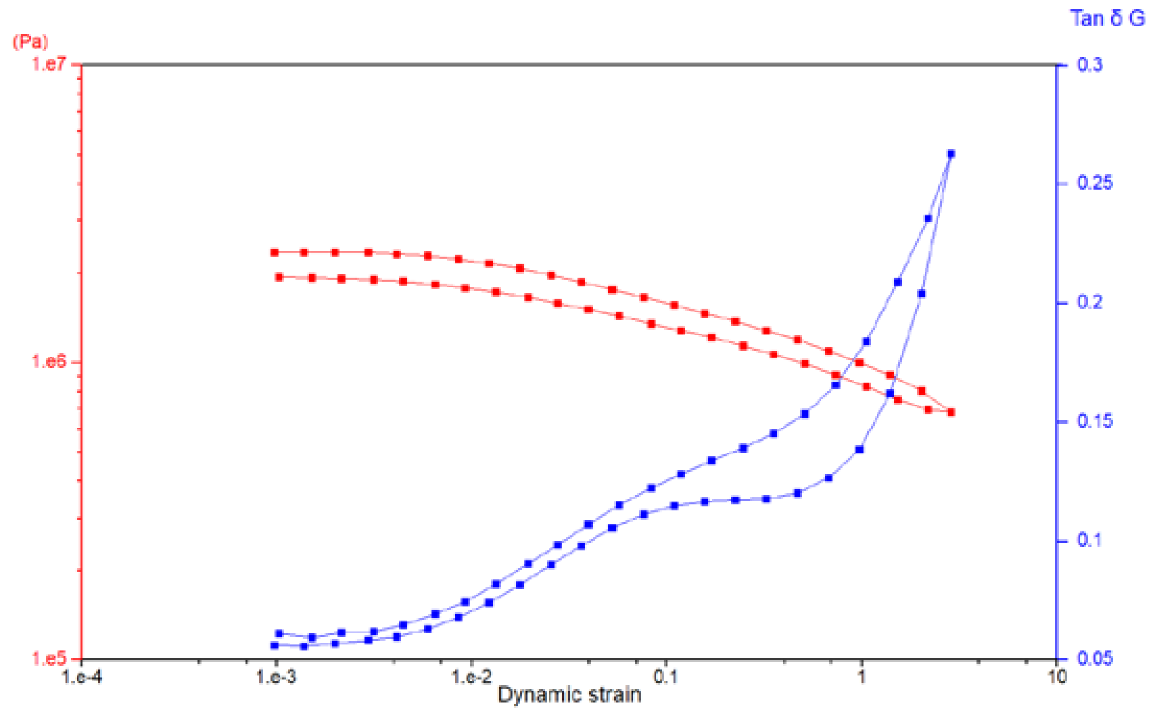
Advanced test control

Analyzing the viscoelastic properties of a polymer is a complex process.

Starting from zero, the DMA cannot predict the necessary amplitude of the signal input sent to the electrodynamic shaker providing the mechanical vibration.

Managing a specific test condition in terms of specimen's deformation or applied stress requires to measure the corresponding magnitude and proceed to a repetitive loop control process in order to reach a target value within an acceptable tolerance range.





A single data point is then the result of lot of stabilization, control and measurement cycles!

Things become even more complicated when the material exhibits a non linear behavior. This is the case for a majority of rubber based materials which exhibit complex response to dynamic excitation.

The change in strain amplitude, the number of excitation cycles may have a strong influence on the material's response. Moreover an excitation level above a critical strain value may generate irreversible damage in the material molecular structure. Therefore applying an excitation overshoot is totally prohibited.

Dyna+ offers a smart and powerful excitation control based on advanced algorithms making possible to adjust excitation control parameters to accommodate with the material nature, responding to specific constraints the expected accuracy, ensuring the perfect knowledge and traceability of the really applied testing conditions.

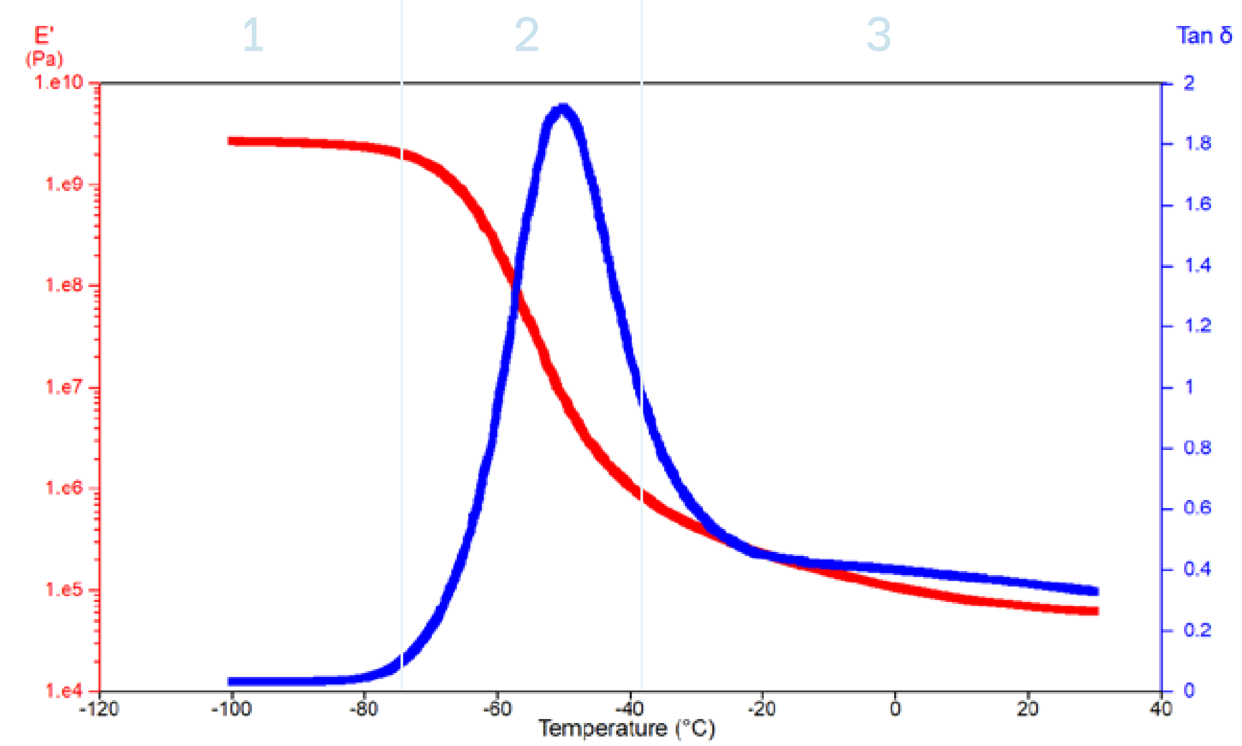
For the most complex materials, the ELS (Excitation Level Storage) function allows from a pre-test on a first specimen to learn and store the required excitation signal to replay it on a blank specimen.

Dyna+ software: Smart functions for a perfect test control

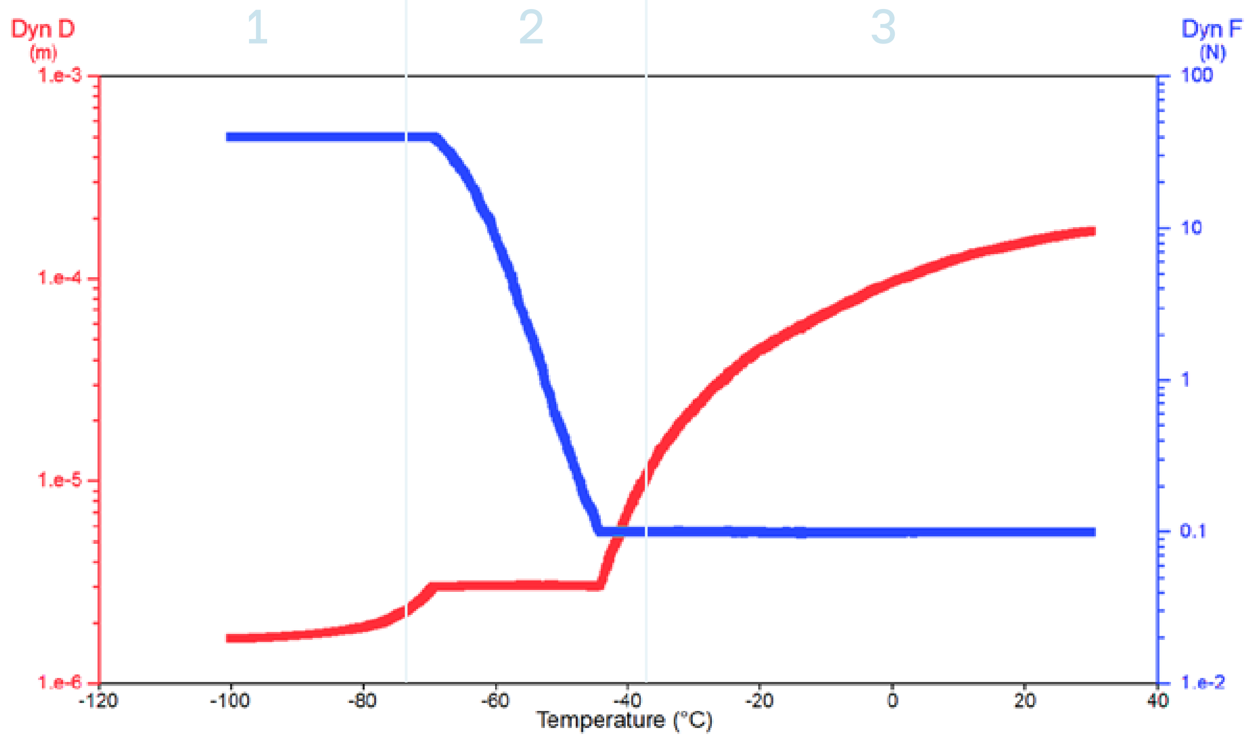
Auto-control mode

Controlling precisely the excitation amplitude all over the test is highly necessary for several reasons:

- The amplitude has to be adequate with the specimen geometry
- The Elastic modulus of the material is subject to change versus temperature
- The Elastic modulus of the material is subject to change versus strain (non linear behavior of elastomers especially)
- The specimen may be fragile and may not support excitation beyond a critical value (deformation or stress)
- The test mode may require specific dynamic control in regards to static load (compression, tension, bending)
- Signal to noise ratio must be controlled to guarantee the measurement quality



Polyurethane specimen : Modulus and Tan δ



Displacement & Force signals vs temperature at for the 3 steps of the Auto-control function

When performing a typical temperature ramp test, one uses to set preferably a constant strain to be applied during entire test. Such control is applied with a set of parameters defining how many cycles will be used for the control process and with which control tolerance.

Nevertheless it can happen, depending on the materials mechanical properties that change with temperature in that case the DMA cannot respect the nominal excitation.

In such a case, an alternative excitation control is often necessary in order to guarantee that the test continues in conditions acceptable in regards to what can accept the specimen.

The beside figures show a temperature ramp test applied to a polyurethane specimen. A setting value of 3 µm has been programmed. Additionally the auto-control function is used and set to limit the force between a maximum of 40 N and a minimum of 0.1 N. In the step 1, below the glass transition when the specimen becomes stiffer, the force is limited to 40 N in order to avoid breaking the specimen.

As the modulus decreases in the glass transition area (step 2), the DMA applies the setting value of 3 µm. At higher temperature, the auto-control limits the force to a minimum of 0.1N (step 3) in order to keep a good signal-to-noise ratio to optimize the resolution of the measurement.

Dyna+ software: Enhanced testing productivity

Operator alert

The Operator alert function allows the operator to be informed of the status of the current test or test campaign.

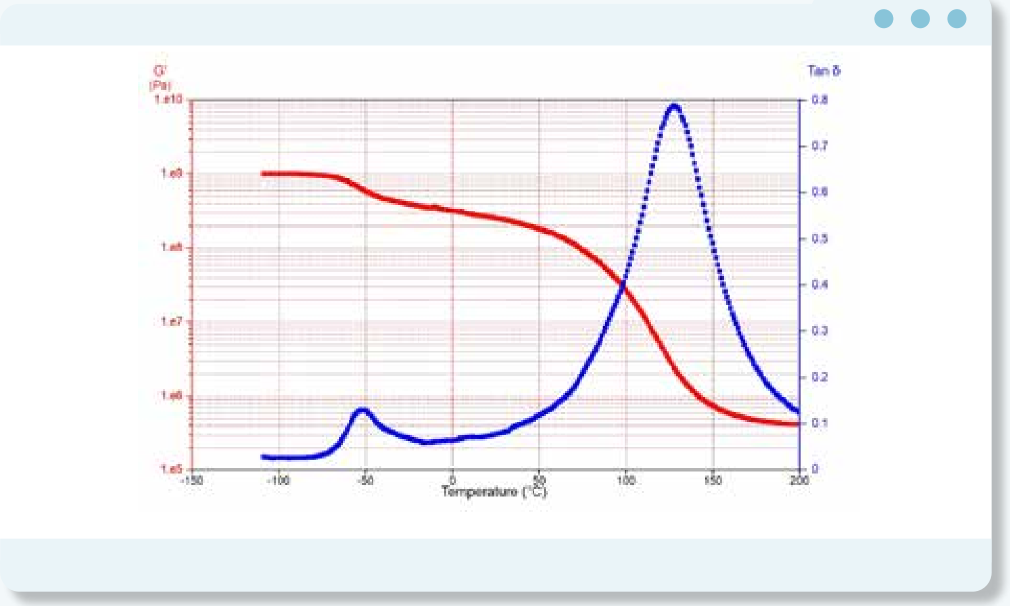
The operator can choose to be notified by text message, E-mail or phone call. This allows to avoid keeping the DMA+ unused after the end of a test and then to improve the laboratory productivity.

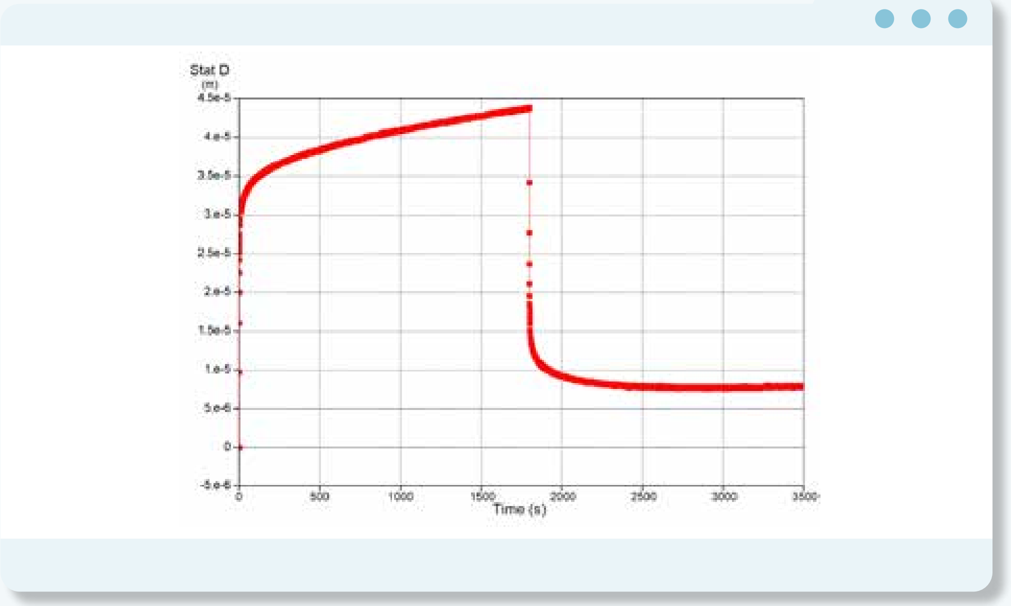
For long tests campaign carried out with Xpander in fully automated mode, it allows to ensure the proper execution of the tests in progress.

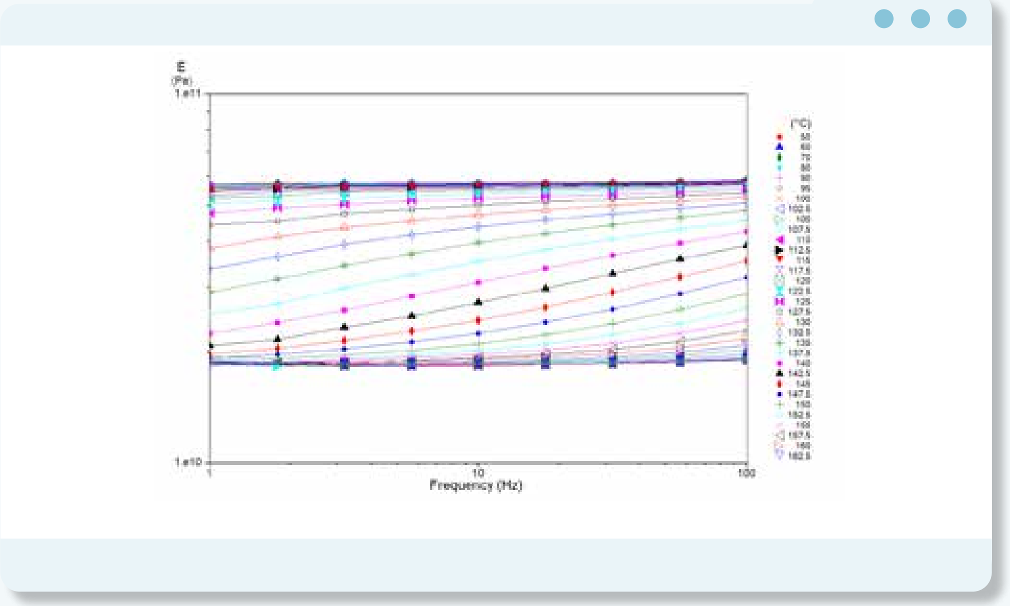
Tests chaining

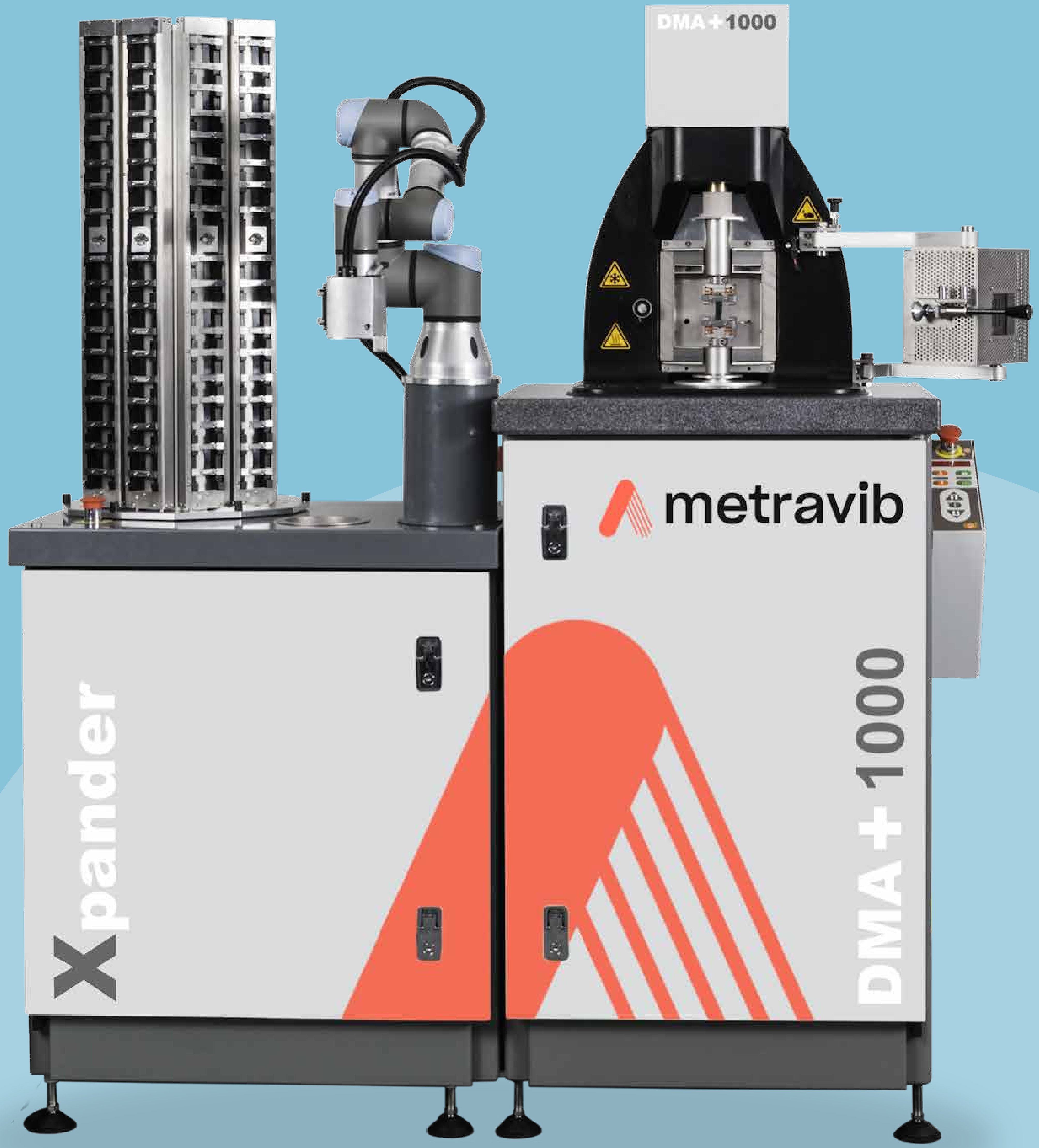
Any DMA test or fatigue test may be automatically chained to a previous test on one single specimen.

Combined with operator alert, both functions are very helpful to optimize the laboratory productivity and reduce the constraints on the mobilization of operators.







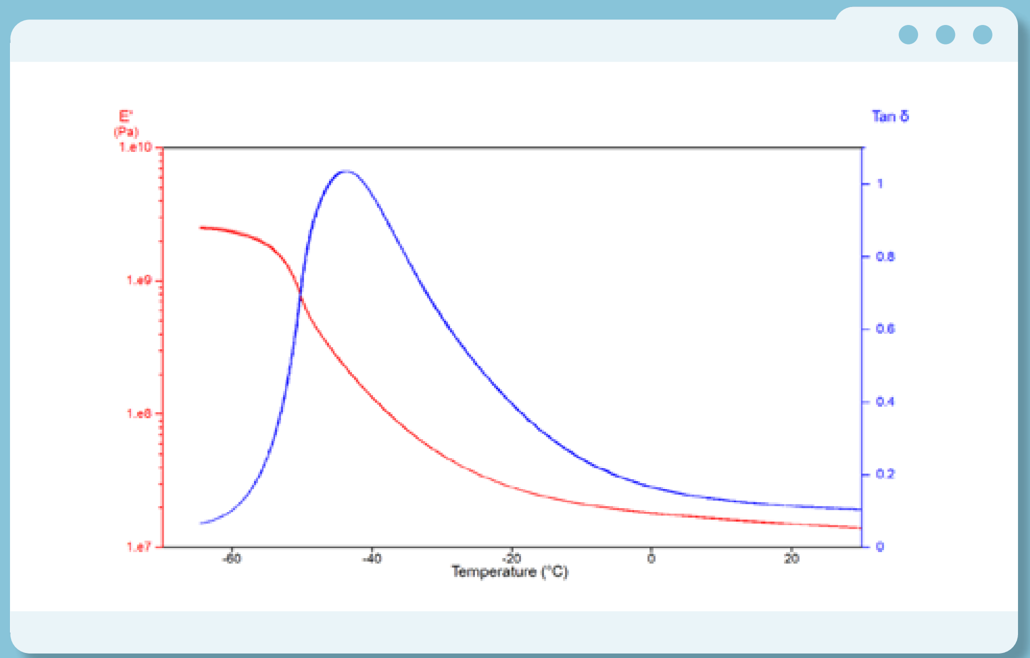


Full automation ready with Xpander

The original design and ergonomics of the DMA+ has been cleverly elaborated to be easily automated with Xpander.

Xpander is an automated specimen handling system dedicated to the DMA+ series.

Xpander is composed of a 6-axis robot arm, a rotative magazine-carousel, with up to 12 removable racks to contain up to 444 specimens.



Tension tests data with Xpander:
Data superimposed from 10 consecutive tests of the same rubber compound specimen – a perfect repeatability!

Xpander allows DMA and fatigue test campaigns to run continuously around the clock and autonomously without any operator intervention, in tension, compression and shear modes.

Xpander can be coupled to any DMA+ by a simple 2h installation without any modification of the original DMA+.

The operator can switch from the classical DMA+ use to the fully automated mode with Xpander in less than 15 minutes.

Xpander's flexibility makes possible to accommodate with material specimens of various shapes and sizes.

Xpander is a unique cost-effective solution to address the increasing test throughput requirement of testing laboratories.

Crack growth testing module

Resistance to fatigue, ageing & crack growth are major issues for rubber based materials. Current commercial methods for crack growth testing have been restricted to time to failure test.

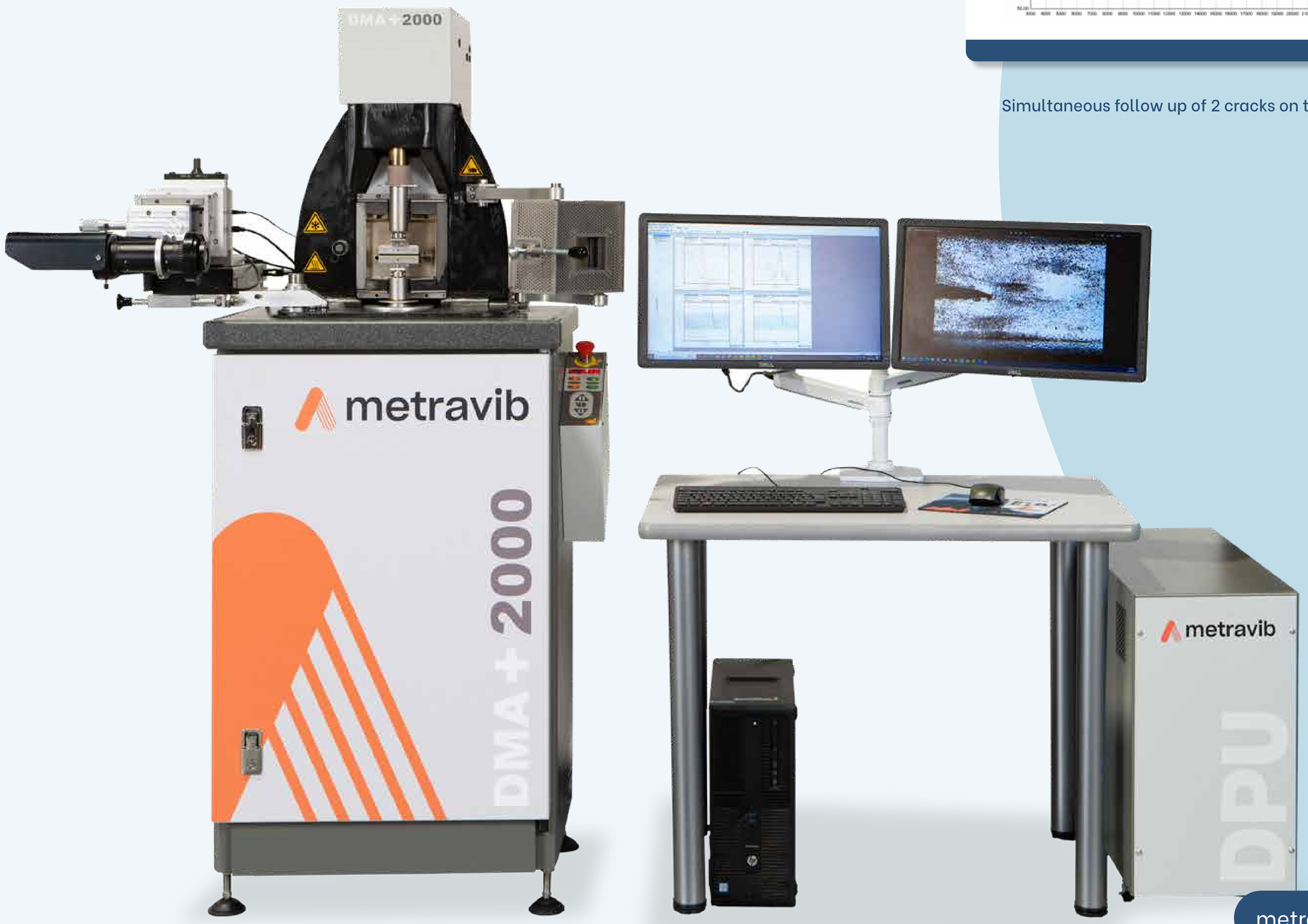
The crack growth testing module turns the DMA+ into a fatigue crack growth testing machine for a cost effective investment.

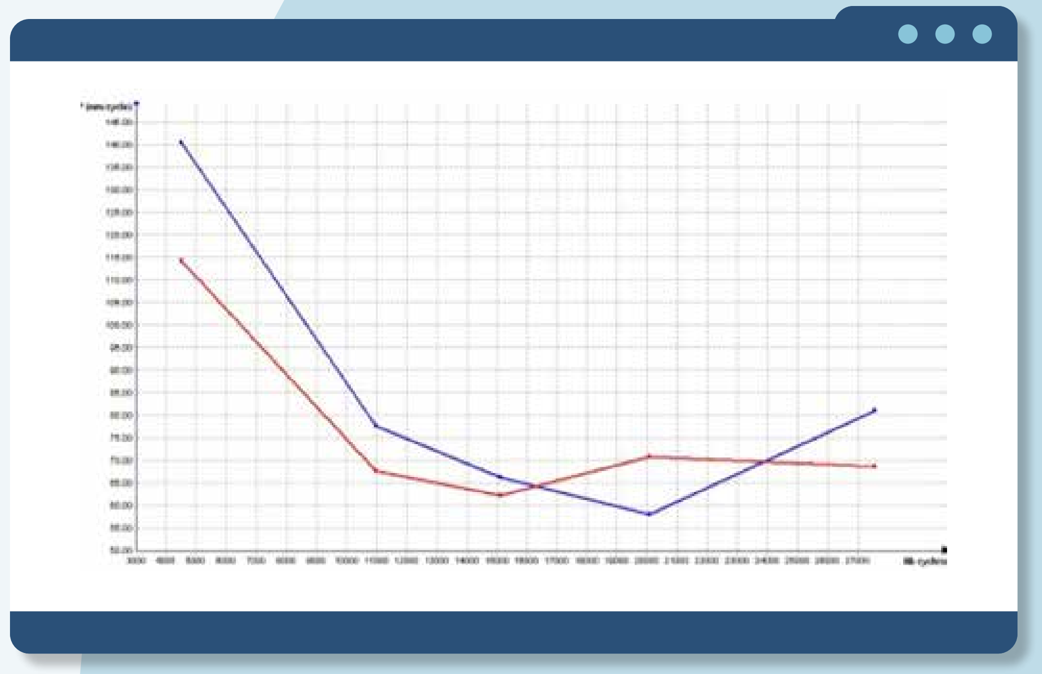
The rubber compound specimen is initially cut. After a prior characterization test, the crack growth test is applied at a specific controlled energy. This makes easy comparison between various compounds.

The crack tip position is periodically determined by a video camera and a real time image processing with an accuracy of 5 µm.

One single test allows up to 4 simultaneous crack follow up on the same specimen .

The retractable optical system can be pivoted to the left of the DMA+ and frees up the classic use for all common applications.



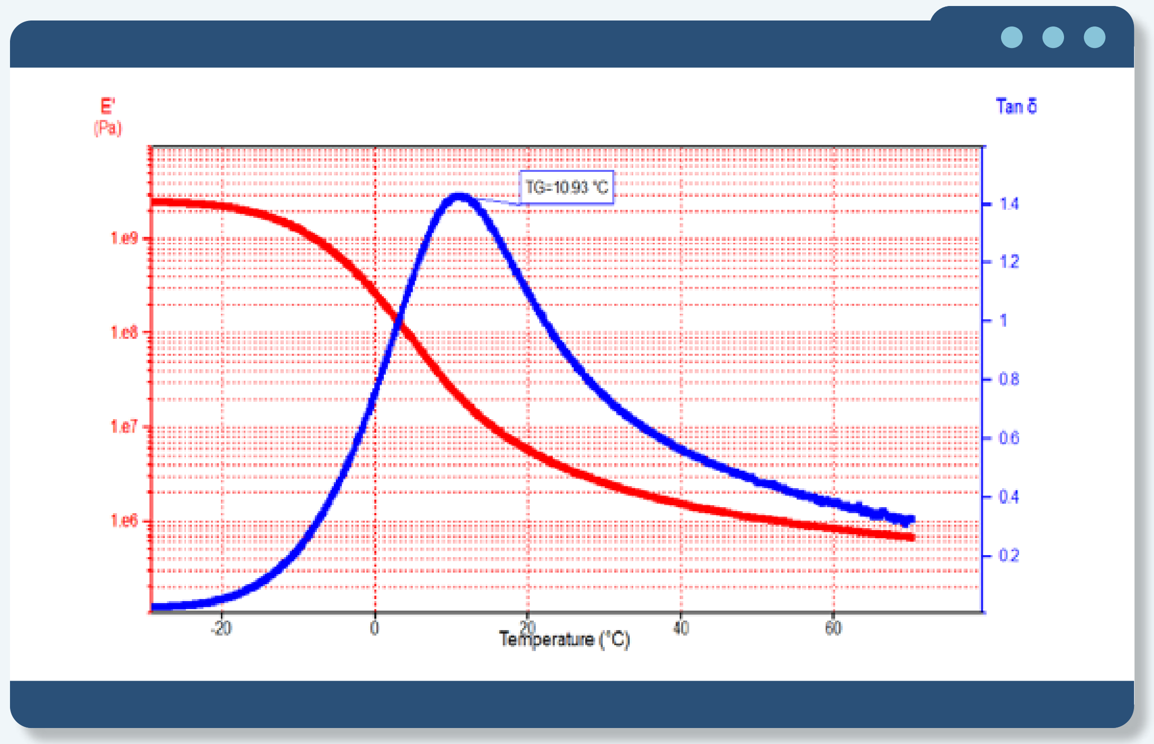


Simultaneous follow up of 2 cracks on the same specimen

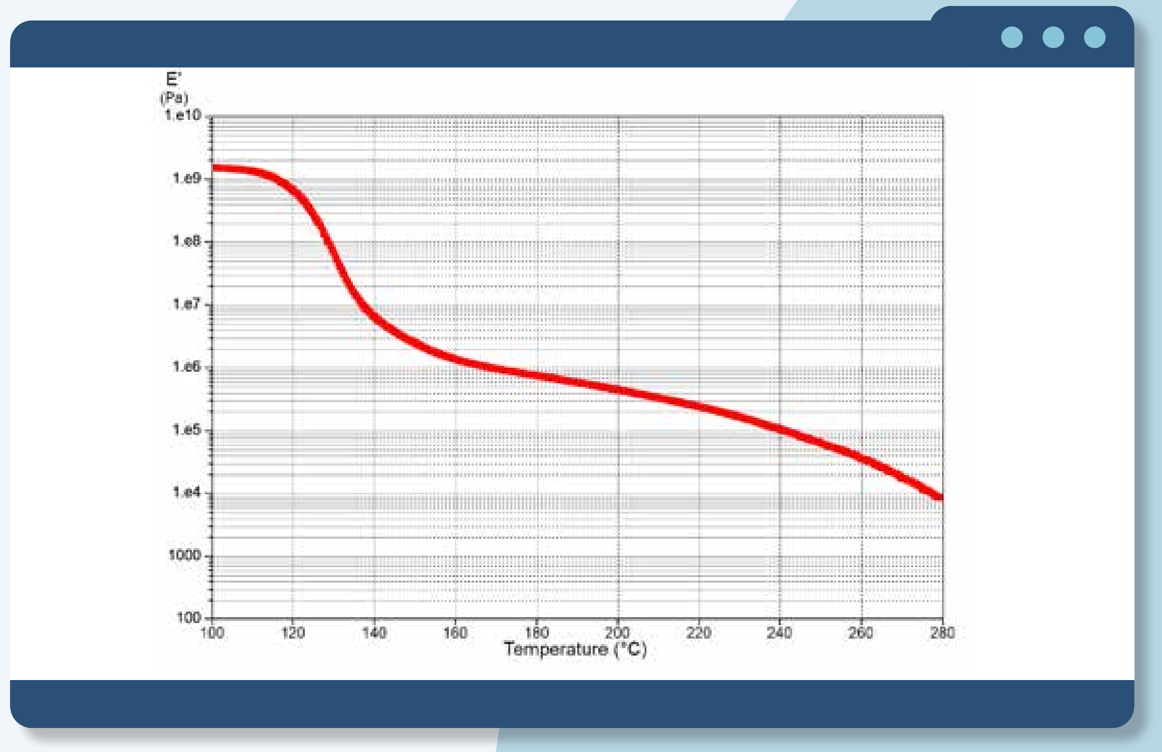
Focus on the main applications of the DMA+ series

Nowadays the DMA technique is identified as one of the most powerful and relevant analysis technique to obtain the thermomechanical characteristics of materials.

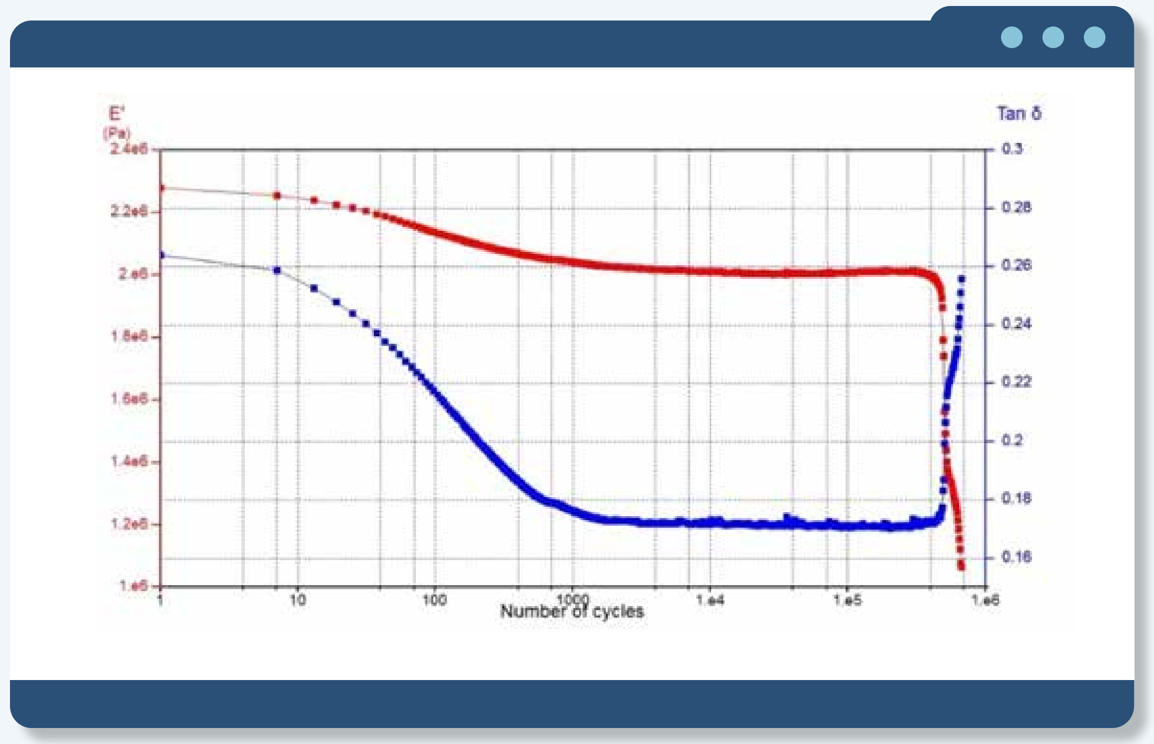
Beside classical DMA tests, Metravib has drastically extended the testing capabilities of the DMA+ series, taking advantage of excitation, electronics, transducers, software technologies to give testing solutions for a large variety of materials and applications.



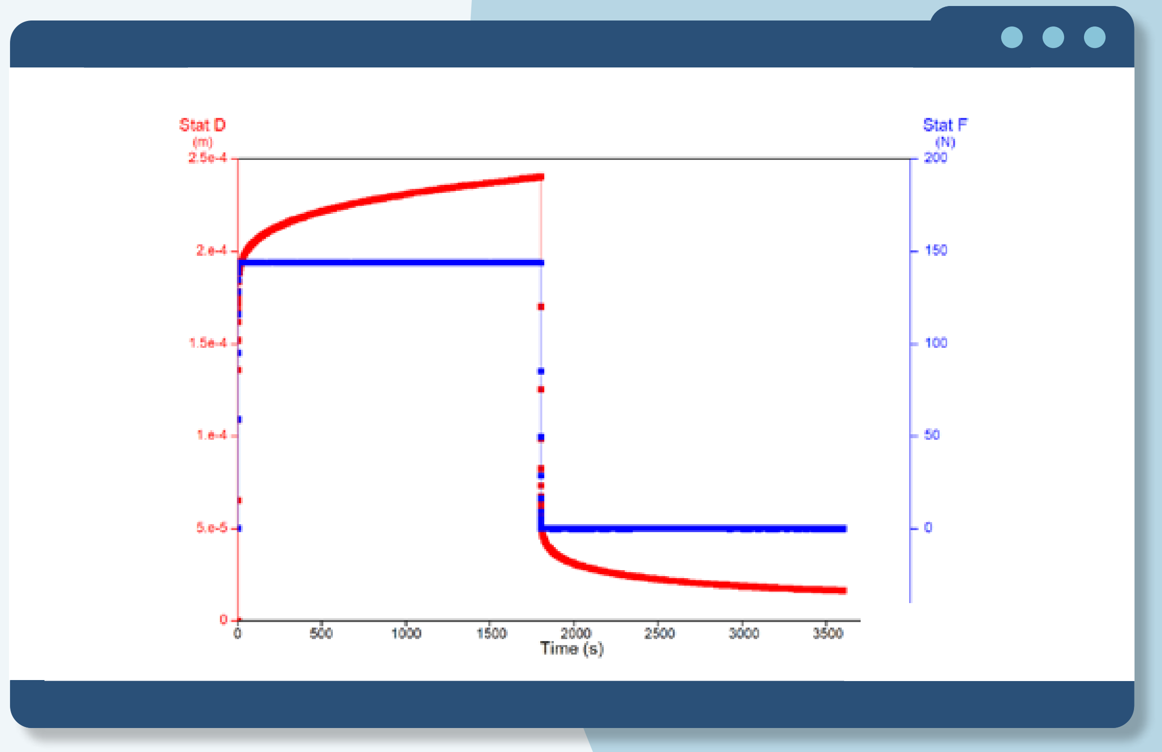
Glass transition determination



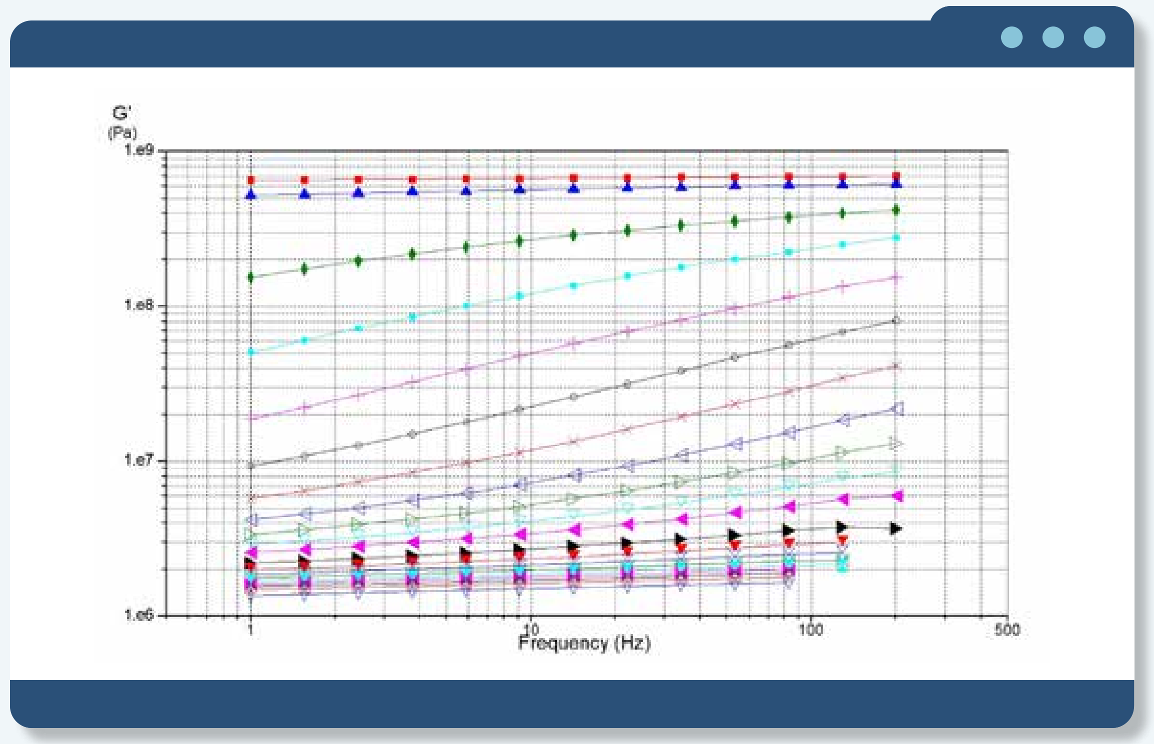
Thermomechanical properties



Fatigue testing

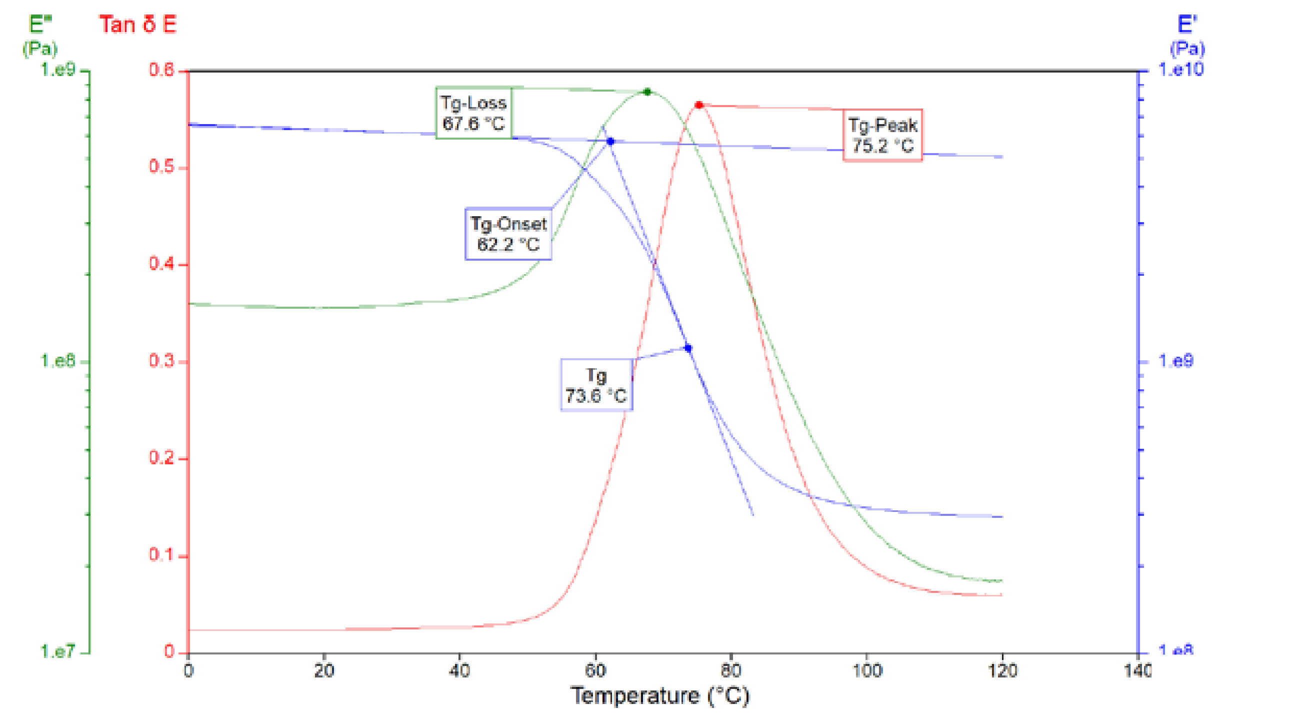


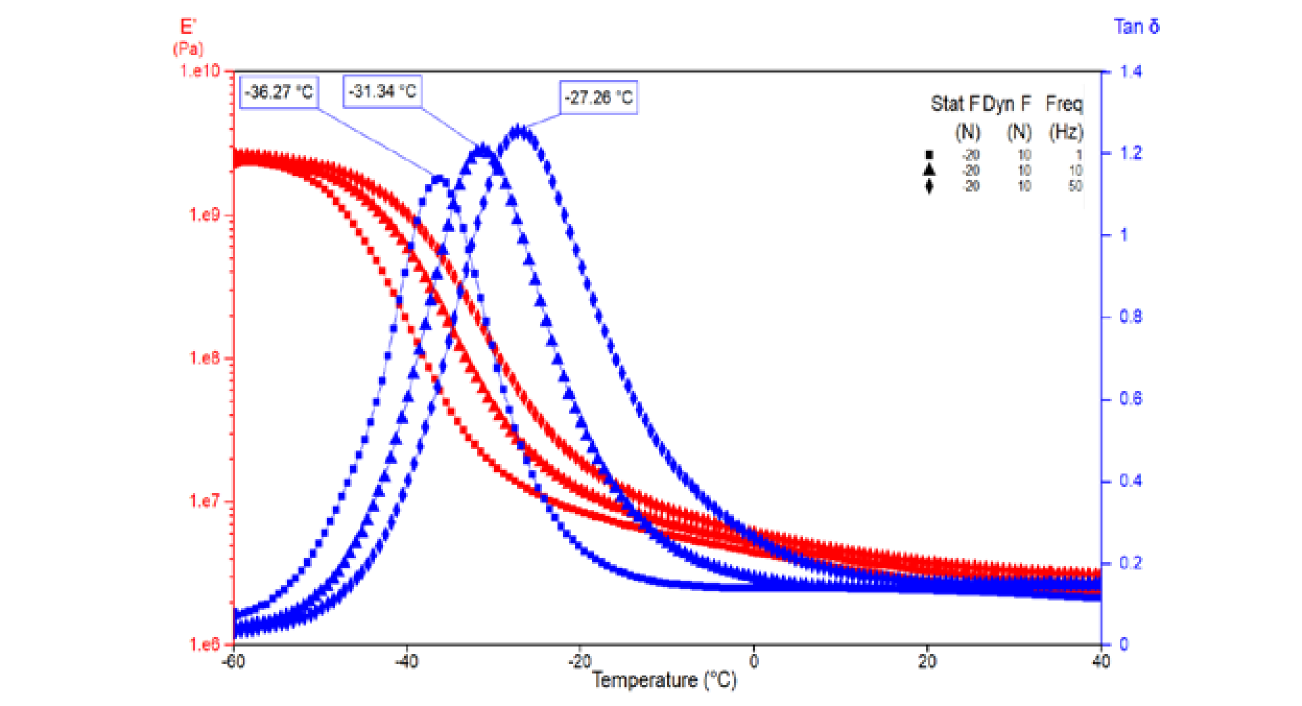
Creep and stress relaxation



Frequency dependence

Applications





Glass transition determination

DMA is known as the most accurate technique for precisely determining the glass transition (Tg) of polymer materials.

The test is carried out on a continuous temperature ramp at a controlled variation rate.

This test, one of the most commonly used in DMA, also makes possible to know precisely the viscoelastic properties and their dependence on temperature; these data are crucial to determine the temperature range of use of a given material for an industrial application.

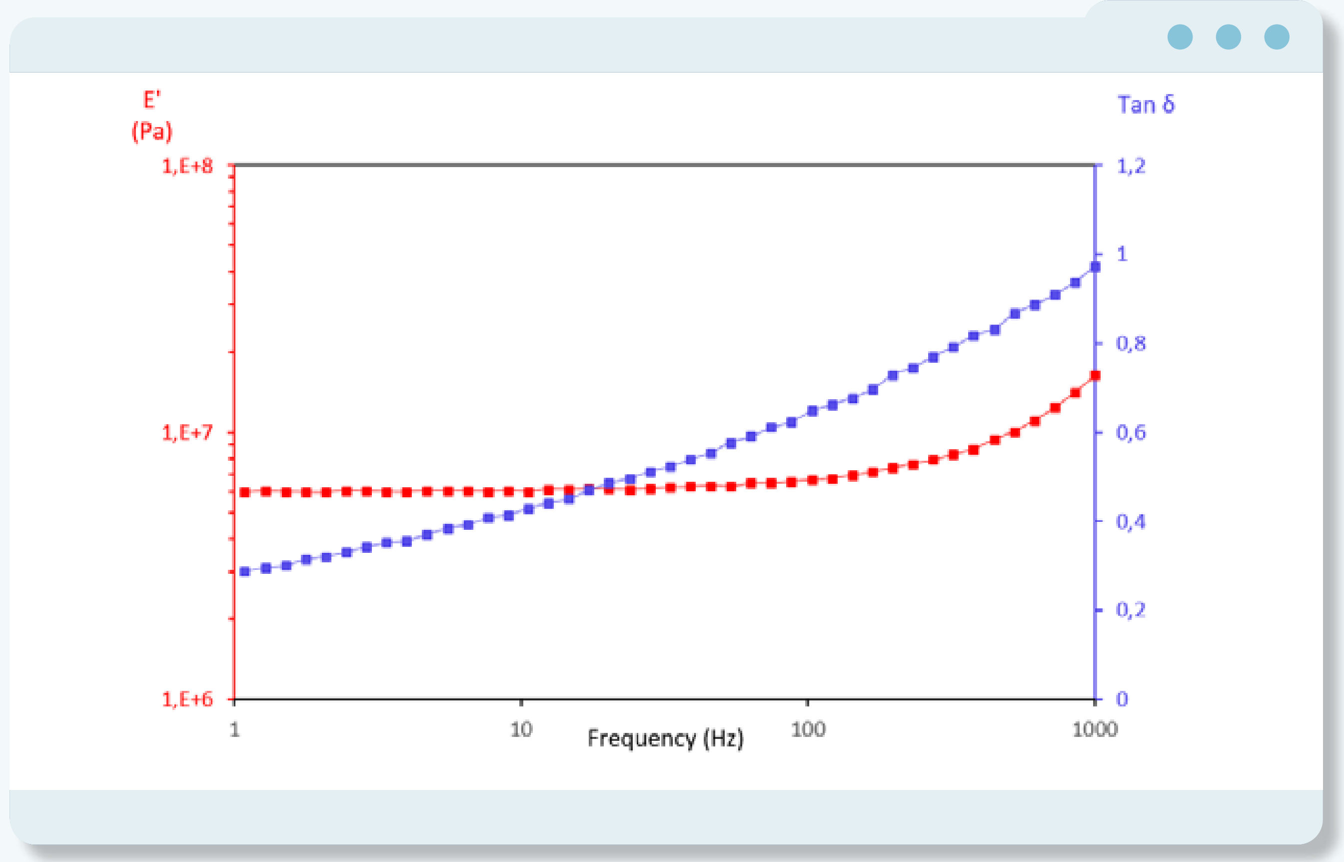
The example shows a specimen of PMMA tested in tension at 1Hz. The Modulus curve typically describes the material state change from the glassy plateau to the rubbery plateau, while Tg may be determined from the peak of Tan Delta or E”, or the inflection point of E’.

With a particularly wide range of excitation force, the DMA + series makes possible to work equally on small specimens but also on specimens of larger dimensions; this allows to obtain data better representative of the structure of the material and to gain in precision by reducing the impact of inaccuracy of dimensional measurements on the Modulus measurement.

Frequency dependence

The viscoelastic properties of materials are very depend on the excitation frequency. If the glass transition is in a conventional way determined at a frequency of 1Hz, the materials damping and modulus may vary in a drastic and different way with temperature and frequency. DMA analysis give the keys to a perfect selection of the right materials for a specific end use application that require specific performance. The example beside shows the frequency dependence of an epoxy resin’s mechanical properties.

Applications

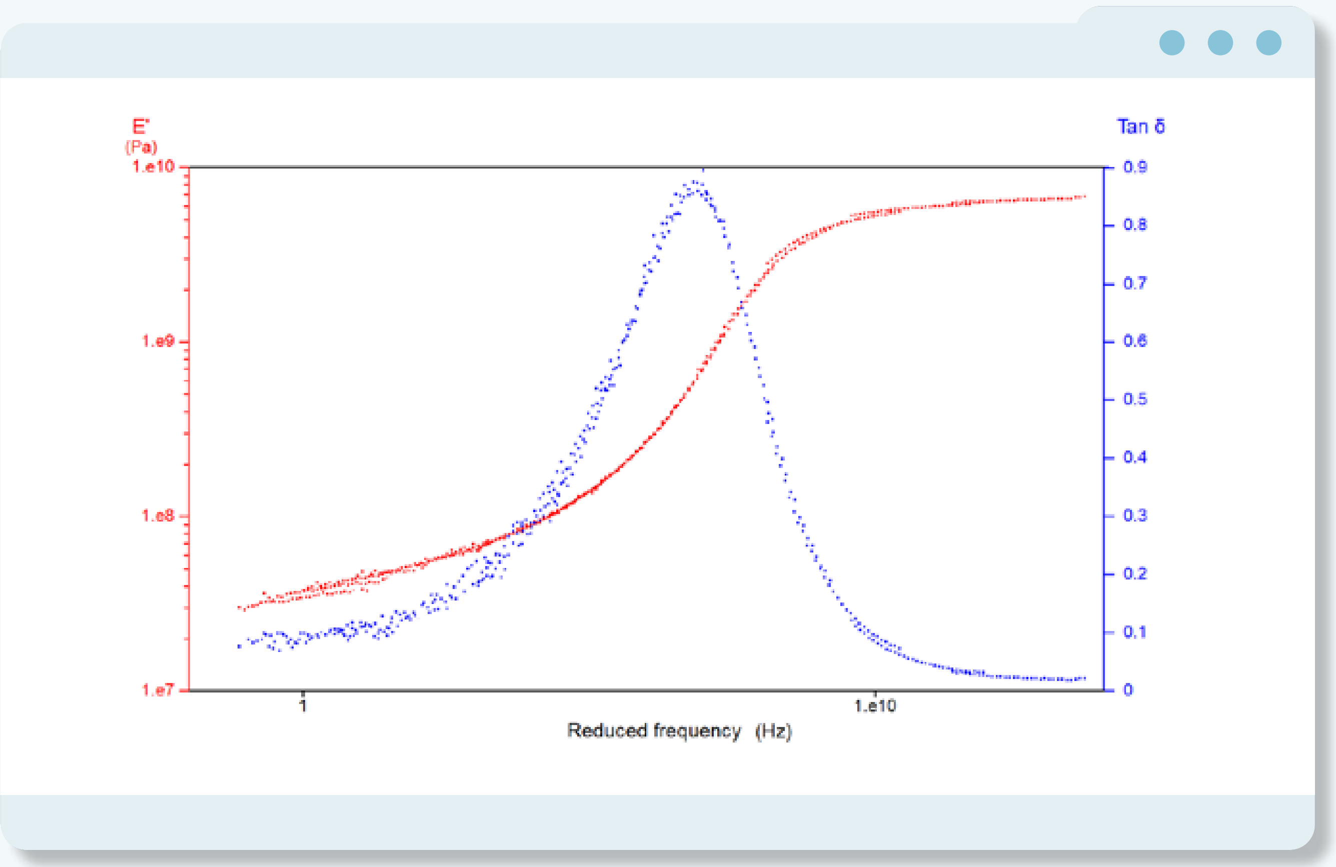


High frequency testing

One of the great advantages of DMA testing technique is the possibility to study viscoelastic properties at various excitation frequencies.

The figure below shows both modulus and Tan Delta up to 1000 Hz for a cylindrical specimen of rubber extracted from a running shoes sole. These typical results show a slight increase of both E' and Tan Delta with frequency due to a different response of the polymers chain under a quick vibrational mechanical excitation. Frequency sweep testing has proven to be useful to understand the behavior of many different polymers during their use, such as the grip ability of the running shoes.

The unique design of the DMA+ mechanical test frame offer a unique frequency range of analysis from quasi static up to 1 KHz.



Master curves calculation

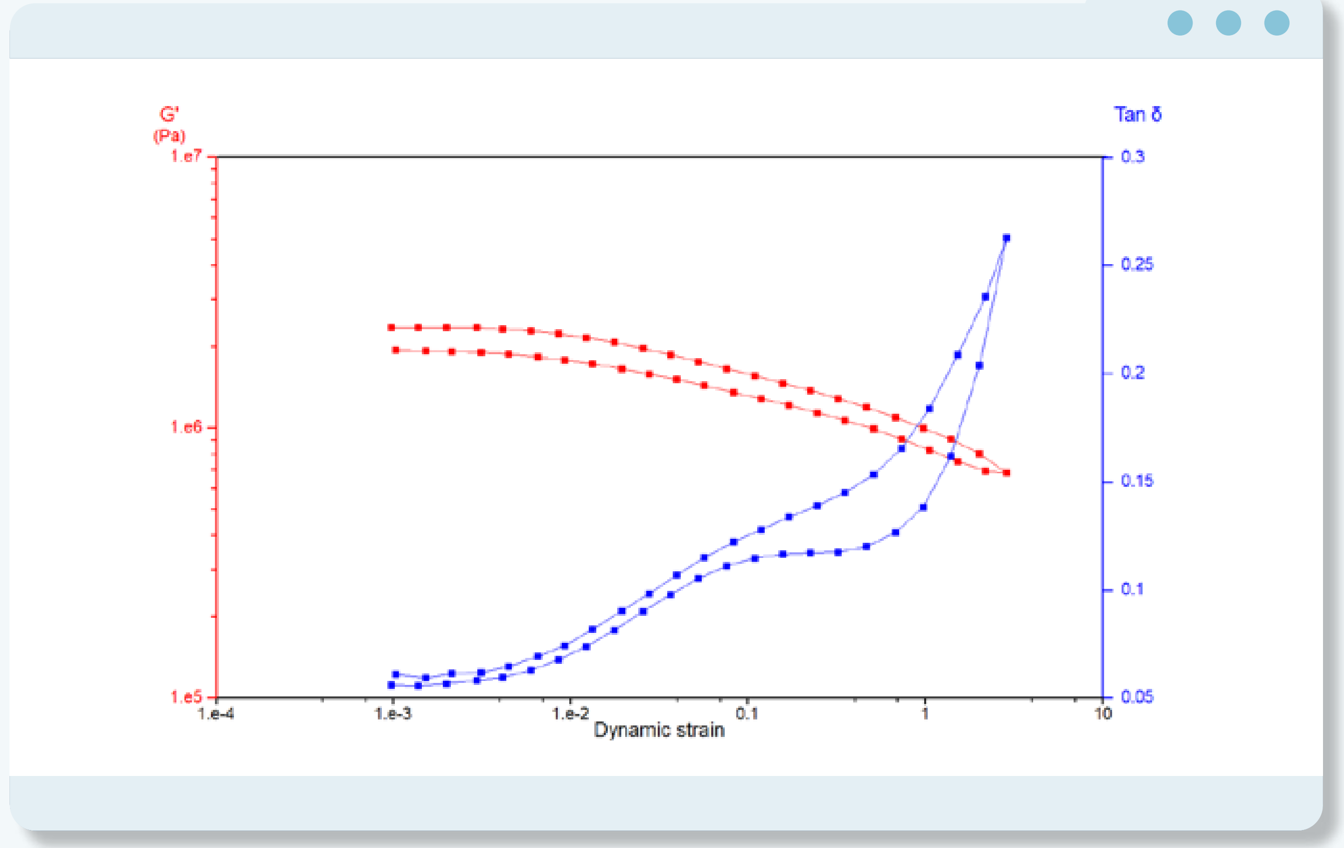
The determination of viscoelastic properties on a large frequency domain is a crucial point in the development of rubber based materials (anti-vibration damping, noise reduction, tire grip...).

Although Metravib DMA+ are able to measure directly the materials viscoelastic properties for excitation frequencies up to 1kHz at various temperatures, the frequency/temperature equivalence law principle allows extrapolating the modulus and Tan Delta for a much higher frequencies domain through master curves calculation.

The example shows master curves of a rubber material, fitted with a combination of WLF and Arrhenius models.

A smart parameter selection allows ensuring a perfect repeatability of master curves calculation avoiding any operator effect.

Applications

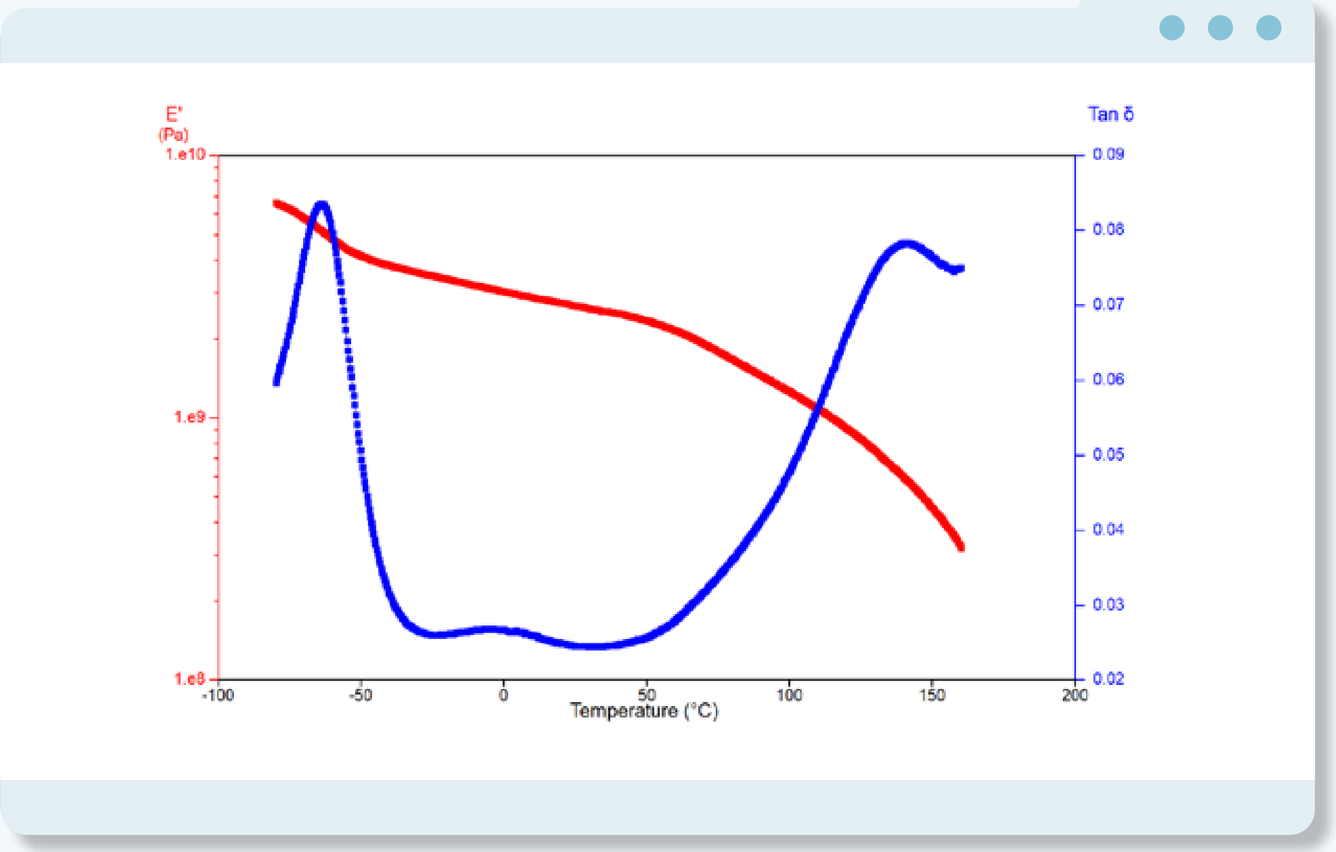


Payne and Mullins effects

Rubber based materials exhibit currently non linear mechanical response. A single test including successively a strain sweep upwards and a strain sweep downwards allows analyzing both Payne and Mullins effects.

The test of a rubber compound specimen in double shear at 10Hz shows a decrease of Shear modulus with increasing strain (Payne effect); the second strain sweep back shows a softening of the compound which characterize the compound's sensitivity to the strain previously applied (Mullins effect). Reversible or irreversible effects on the matrix/fillers links may be analyzed precisely and give key information to the rubber products manufacturers for the compounding optimization in regards to the expected product performances.

Rubber materials may be extremely sensitive to the excitation history (duration, amplitudes, cycles, relaxation times...) ; Dyna+ software advanced excitation parameters allow handling precisely all the excitation process of the test in order to observe very fine differences of response of various compounds.

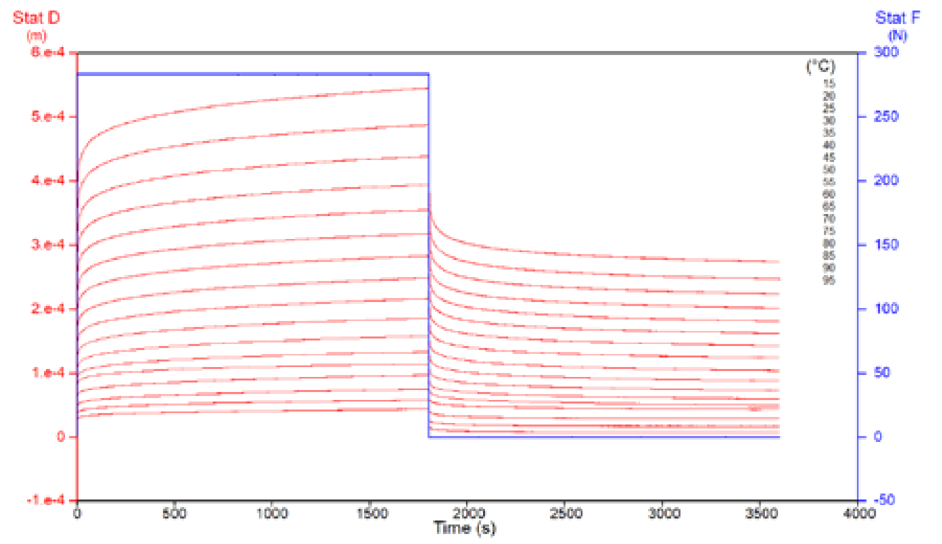


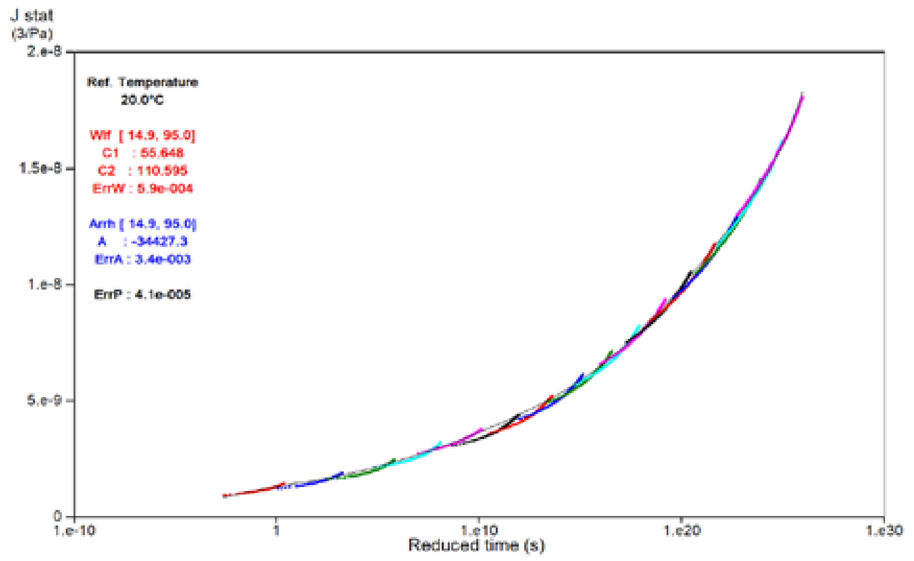
Secondary transitions

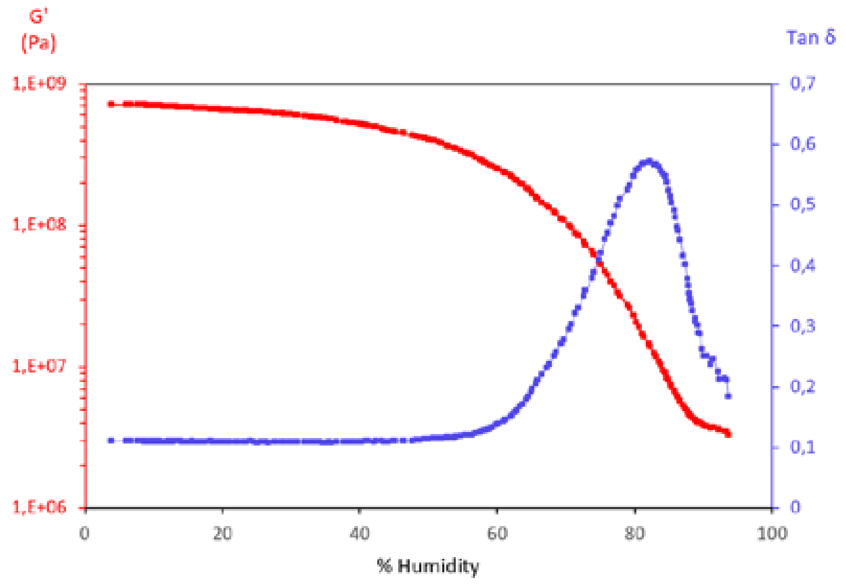
In DMA tests, Tan Delta is a key indicator especially informative; Tan Delta variations expresses material's state change: the glass transition of course, but as well polymerization, crystallization, fusion, relaxation.

Secondary transitions correlate with the motion of a side group of atoms on a polymer chain or at the end of the chain. Beside, a temperature ramp test of a Polyoxymethylene (POM) is a good example of the ability of the DMA+ to clearly reveals multiple transitions of very different amplitudes.

Applications







Creep test and long term creep prediction

Creep test consists in applying a static stress over a fixed time, and to measure the resulting strain. The recovery test consists in releasing the stress and follow up how the material's specimen relaxes.

Such creep experiment can be performed at consecutive various temperature stages in one single test.

Relaxation time, permanent deformation can precisely describe the materials stability within temperature under specific stress or strain.

With the dedicated TTS function of Dyna+, based on time/temperature equivalence principle, such data can be used to predict the material's creep at long term. Relatively short experiments allows ranking precisely how end-use products performances will age within their expected lifetime.

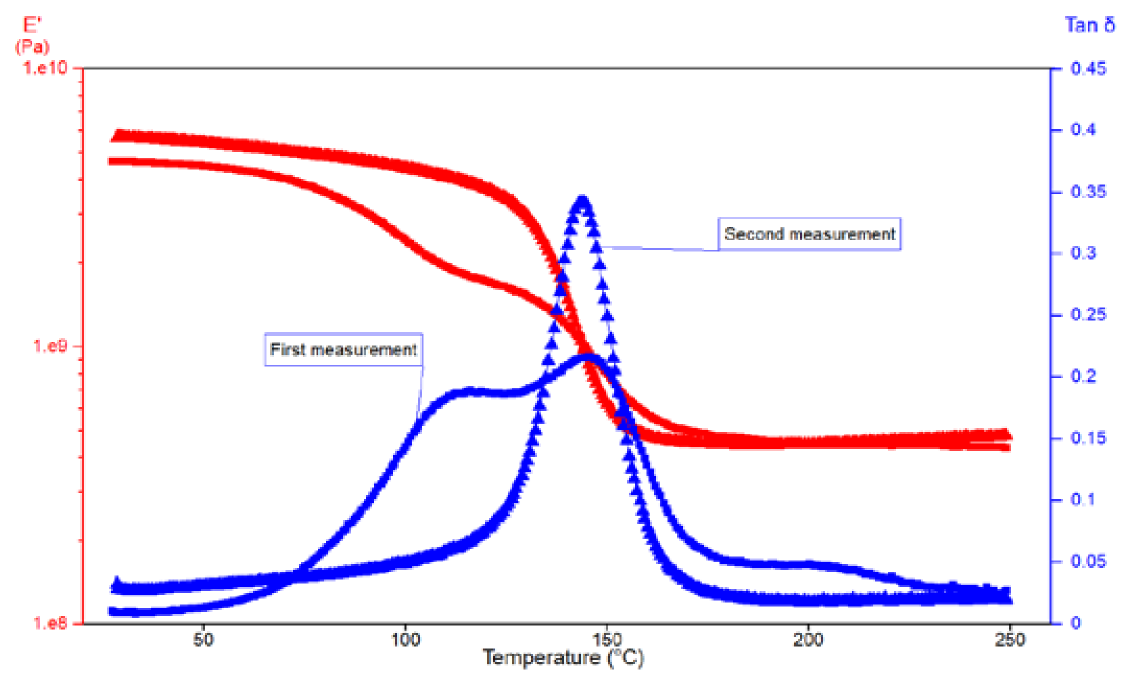
Humidity dependence

Materials sensitive to water can be tested at controlled humidity with a dedicated humidity module.

The example of Chitosane, a biodegradable polymer, exposed to increasing humidity shows the drastic impact of water on the polymer's structure and on its viscoelastic properties: The shear modulus decreases by a factor of 100 at 90% RH while the Tan Delta reaches a peak value at 80% RH.

Additional accessories allow immersing the specimen in liquids while testing.

Applications



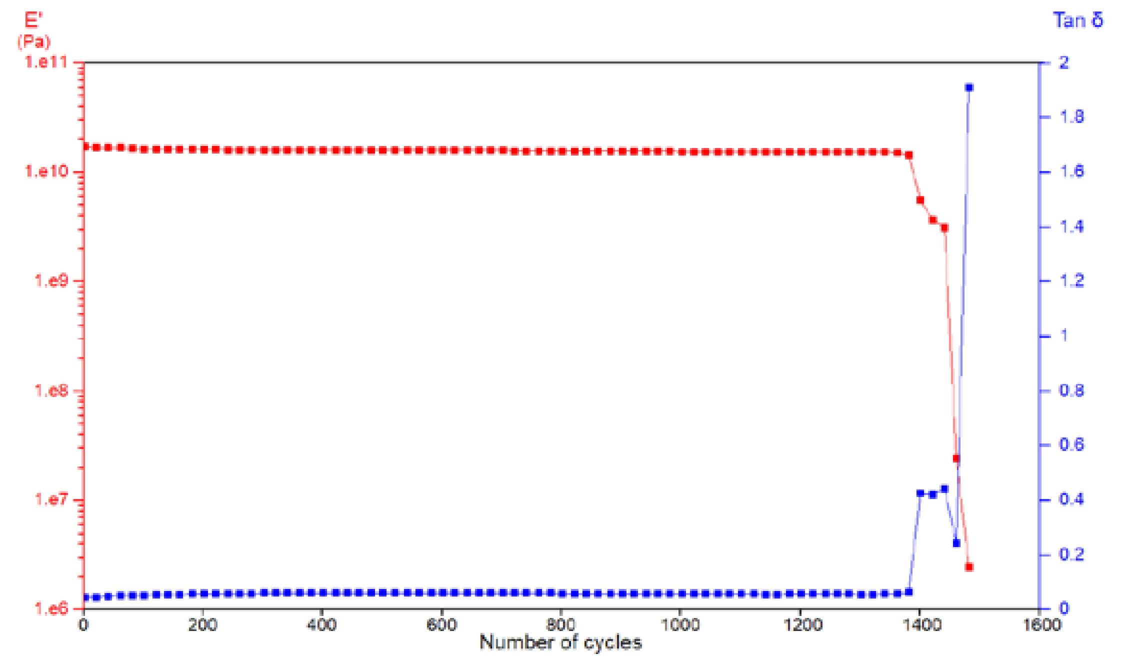
Curing effect

The knowledge of the degree of cure is significant for ensuring that the nominal mechanical properties of a polymer matrix are achieved. Under-curing can lead to under-strength adhesion between fibers layers, or skins and cores, and potential geometric distortions upon part removal from the mold.

In the example beside, in test #1, a specimen of an epoxy-based material is tested over a temperature ramp up to 250°C; it exhibits a smeared glass transition featuring two Tan Delta peaks.

A second consecutive identical test (#2) reveals a sharper glass transition with a much higher amplitude Tan Delta. Test #2 highlights that the specimen was initially poorly cured, and has been post-cured during test #1 .

A third consecutive test shows E and Tan Delta totally superimposed with tests #2 and demonstrates that the nominal mechanical properties are achieved.



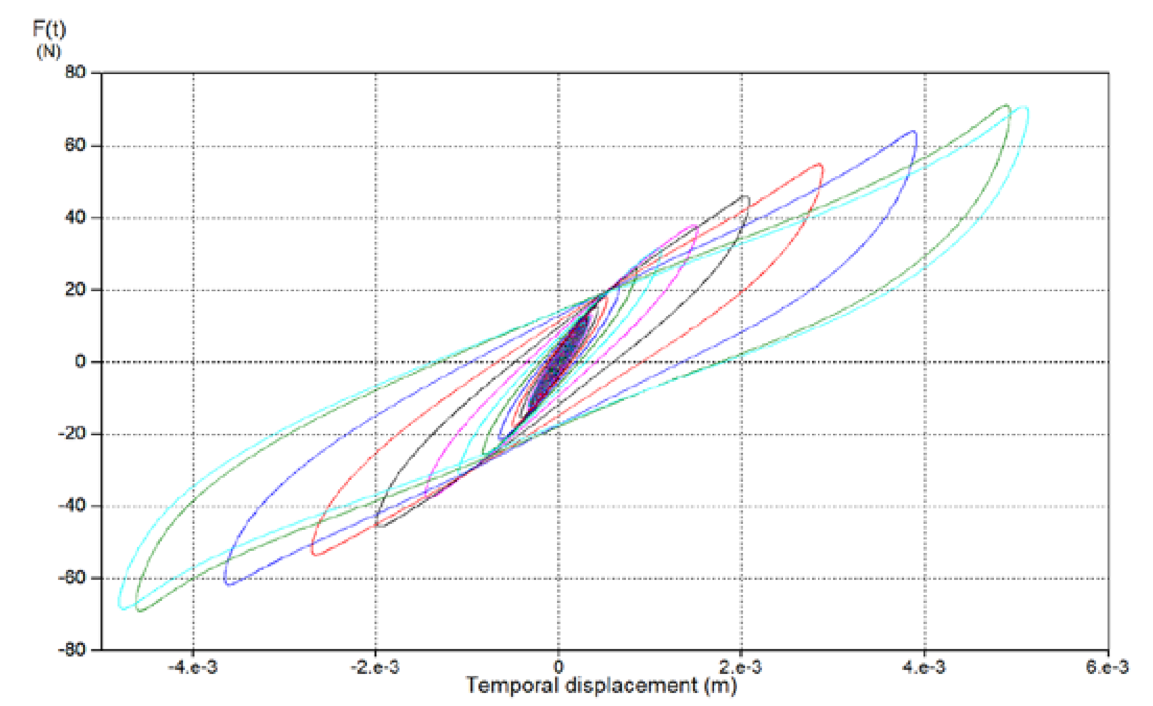
Fatigue testing

Fatigue testing can be achieved with the Dyna+ fatigue module with controlling wave form, amplitude, frequency and strain or stress.

Tests can be performed over hundreds, thousands or millions of cycles. In the example beside, a glass epoxy composite specimen exhibits constant mechanical properties until 1400 cycles where the drop of modulus and Tan Delta soaring are characteristic of a mechanical rupture inside the specimen.

Coupling Xpander with the DMA+ makes possible automated fatigue testing of hundreds specimens without operator action.

Applications



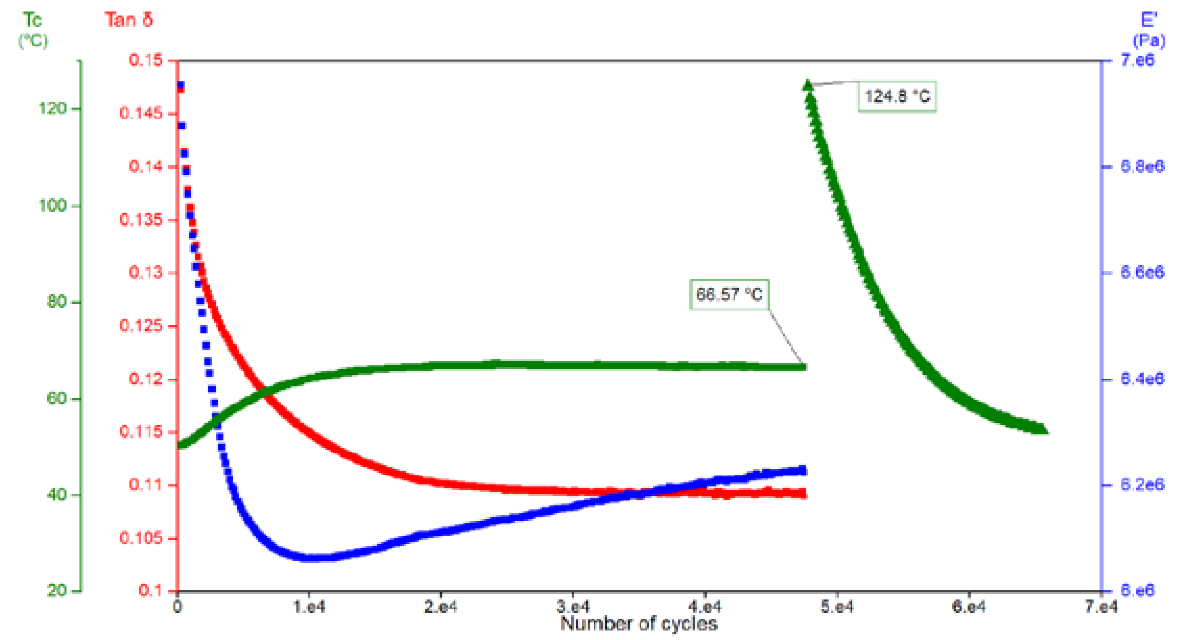
Temporal signals

The Lissajous stress-strain curve is a quick, straight-line display for identifying the material's behavior.

For rubber-based materials exhibiting complex response to dynamic excitation, Lissajous curves provide insight in the non-linear response in displaying this complex mechanical behavior in a compact manner.

It is especially helpful to make easy the comparison between various materials and to perform the calculation of energy dissipation and the energy dissipation ratio.

Dyna+ makes possible the storage of the displacement and force temporal signals. This powerful information allows to perform quickly a relevant expertise of the material's behavior and a quick and efficient response's comparison between various materials.



Heat build-up (HBU) testing

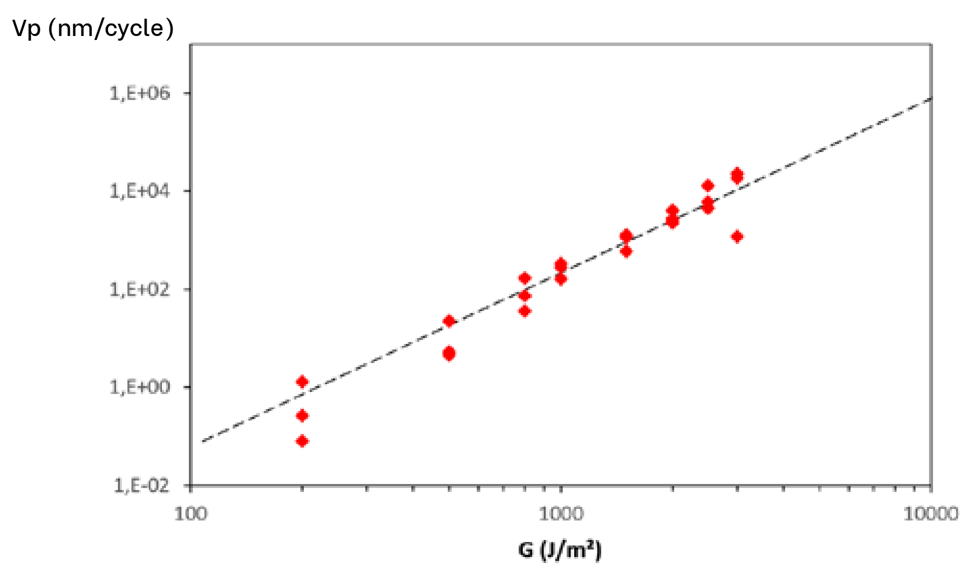
Heat build-up occurs in a rubber-based material when it is submitted to high strain dynamic amplitudes. Such property may be crucial or critical depending on end-use application.

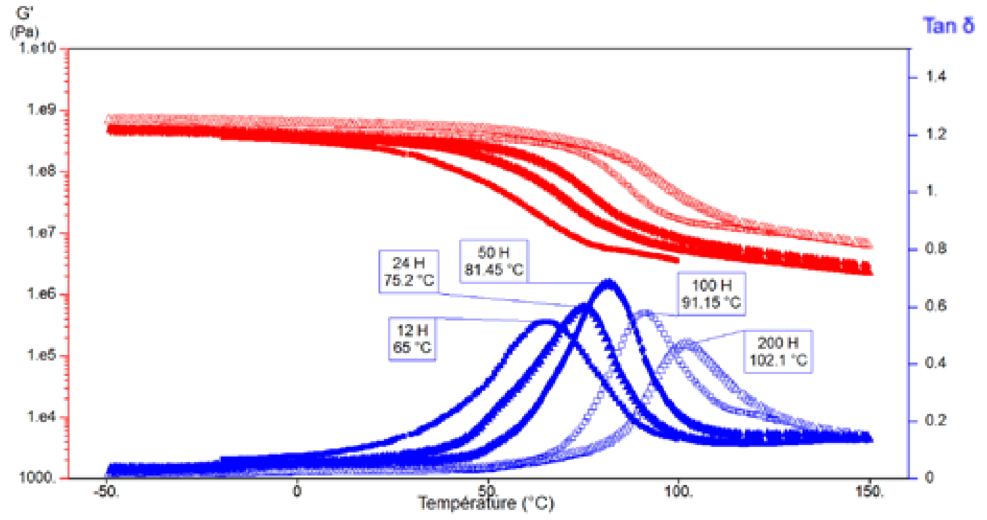
A cylindrical specimen is submitted to a static force an excited continuously at a fixed frequency and at controlled strain amplitude.

The dedicated HBU specimen holder allows to measure the surface temperature on one extremity of the specimen (66.57˚C), while a needle thermal probe can be punched inside the specimen at the end of test to measure the core temperature (124.8˚C).

Heat build-up tests provides key data to estimate the service performances and expected service life of rubber based end-use products.

Applications





Fatigue crack growth testing

Resistance to fatigue and crack growth are major issues for rubber based materials. A fatigue crack growth module allows to achieve crack growth test with a DMA+.

After having initiated a crack in the specimen, it is subjected to a fatigue test with controlled wave form, frequency and deformation or stress.

The change of position of the crack tip is precisely detected by a motorized digital camera for up to 4 cracks during the same test over the continuous fatigue test.

The data presented in the figure beside show the dependence of the crack growth rate (in nanometers per cycle) versus the energy G (in joules per m²) applied to the specimen. Those data are essential for the life time prediction of the end-use product.

DMA+ series are the unique DMA combining high DMA testing capabilities and advanced fatigue crack growth testing in one single instrument.

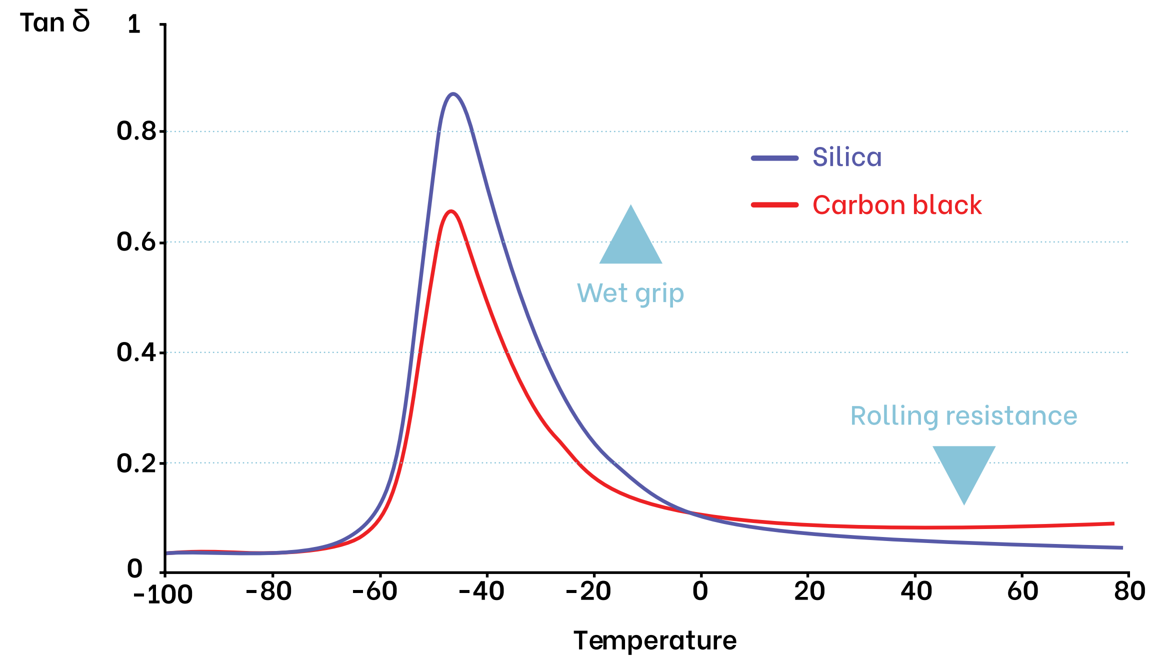
Ageing

The fine analysis performance of a DMA allows easily distinguishing mechanical behavior changes due to many factors including chemical, or physical ageing.

The example beside displays the thermal response of 6 specimens of the same PVC featuring different ageing.

One can notice the strong difference between the various curves of Shear modulus and Tan Delta : the glass transition of the material is drastically affected by ageing. Such data are key parameters to handle the materials lifespan, various mechanical performances, strong to fragile limit ...

Applications



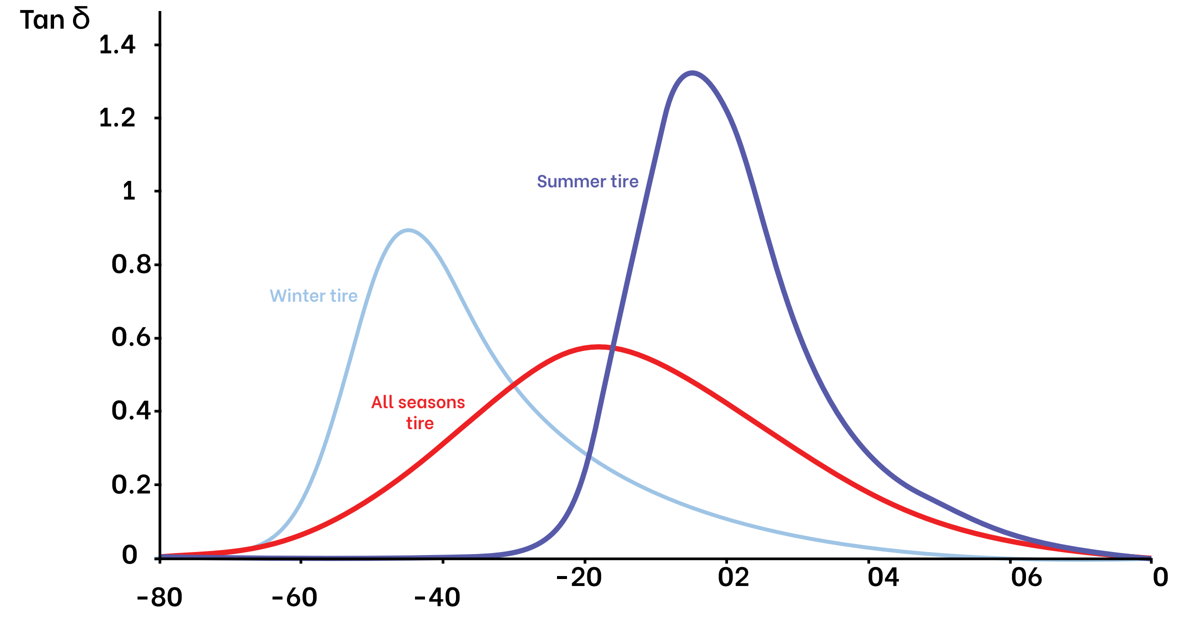
Fillers effect

The close correlation between the viscoelastic properties and the tire performances is established for more than 30 years. Therefore the tire industry uses intensively the DMA technique as a key technique to help compounders to optimize the rubber compounds formulation and meet car manufacturer expectations.

Reinforcing filler is one of the most critical ingredients in tire production. Carbon black is typically used to improve the overall performance of the tires.

A partial replacement of carbon black by silica may bring many benefits.

In the example beside the introduction of silica in the rubber compounds filled with carbon black increases the Tan Delta peak amplitude, improving wet grip performance, while at the same time, it reduces at 60˚C the Tan Delta value, improving the rolling resistance performance.



Tire performance prediction

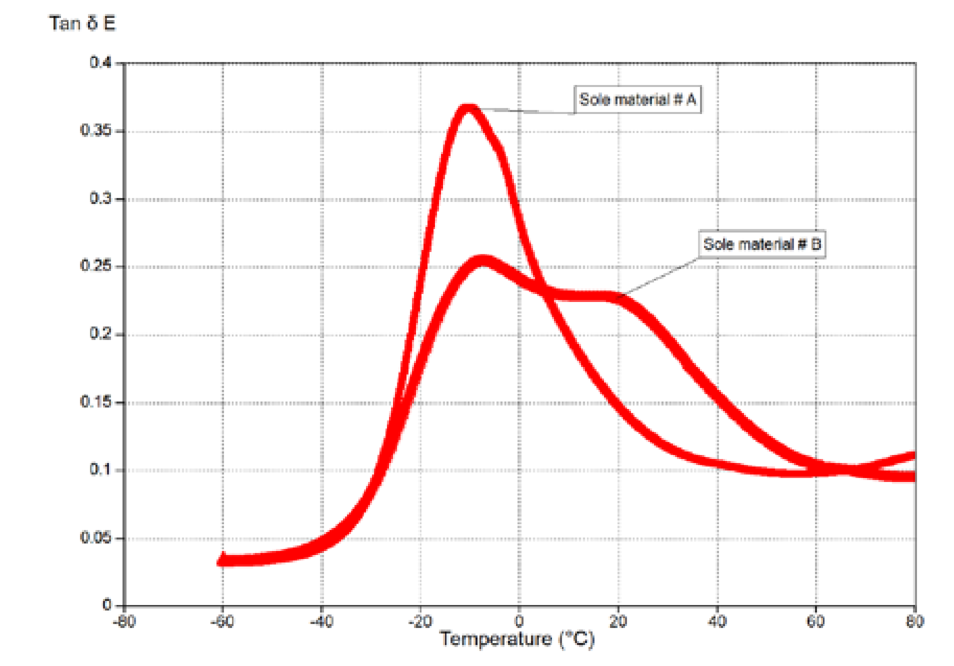
For years the close connection between the rubber compounds viscoelastic properties and the tire performance has been established and the DMA technique has become an essential testing method for the tire industry.

The contribution on tire performance of all components of the compound: rubber, reinforcing fillers, oils, and various chemicals used to crosslink or cure the compounds, can be clearly established.

Numerous predictors have been developed using the shear modulus G, G', and Tan Delta data at specific temperature and frequency to predict rolling resistance, handling, and traction on dry or slippery roads.

As an example the figure beside displays the Tan Delta thermal graph for 3 different tire tread rubber compounds typically used in winter, summer and all seasons tires. Tan Delta has become a major key indicator in the development of tire performance.

Applications

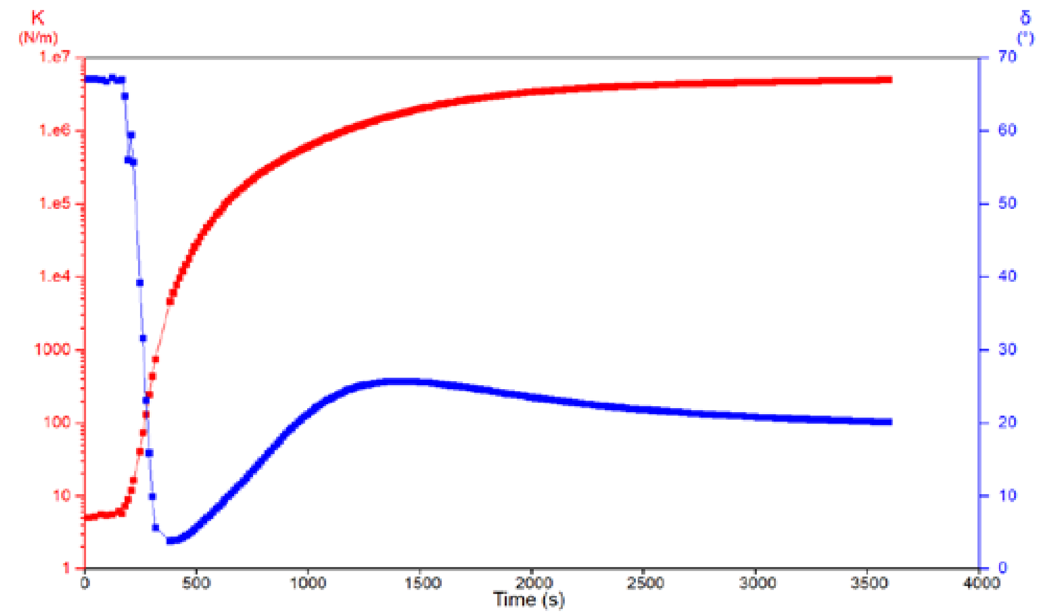


Sport shoes performance optimization

During a run, the impact of the foot with the ground produces a shock that causes vibrations to travel down the legs. Signal frequencies during running are between 5 to 20Hz. The attenuation of mechanical stress waves in this frequency range is essential to prevent injuries.

Additionally as for tires, the shoes sole's grip is also an essential performance. Both damping and grip are closely connected with the viscoelastic properties of the sole.

In the graph beside we can see the damping difference versus temperature of two different foams selected for the composition of a running shoes sole. The selection will go for the material featuring an average damping covering a larger temperature range (Material B).

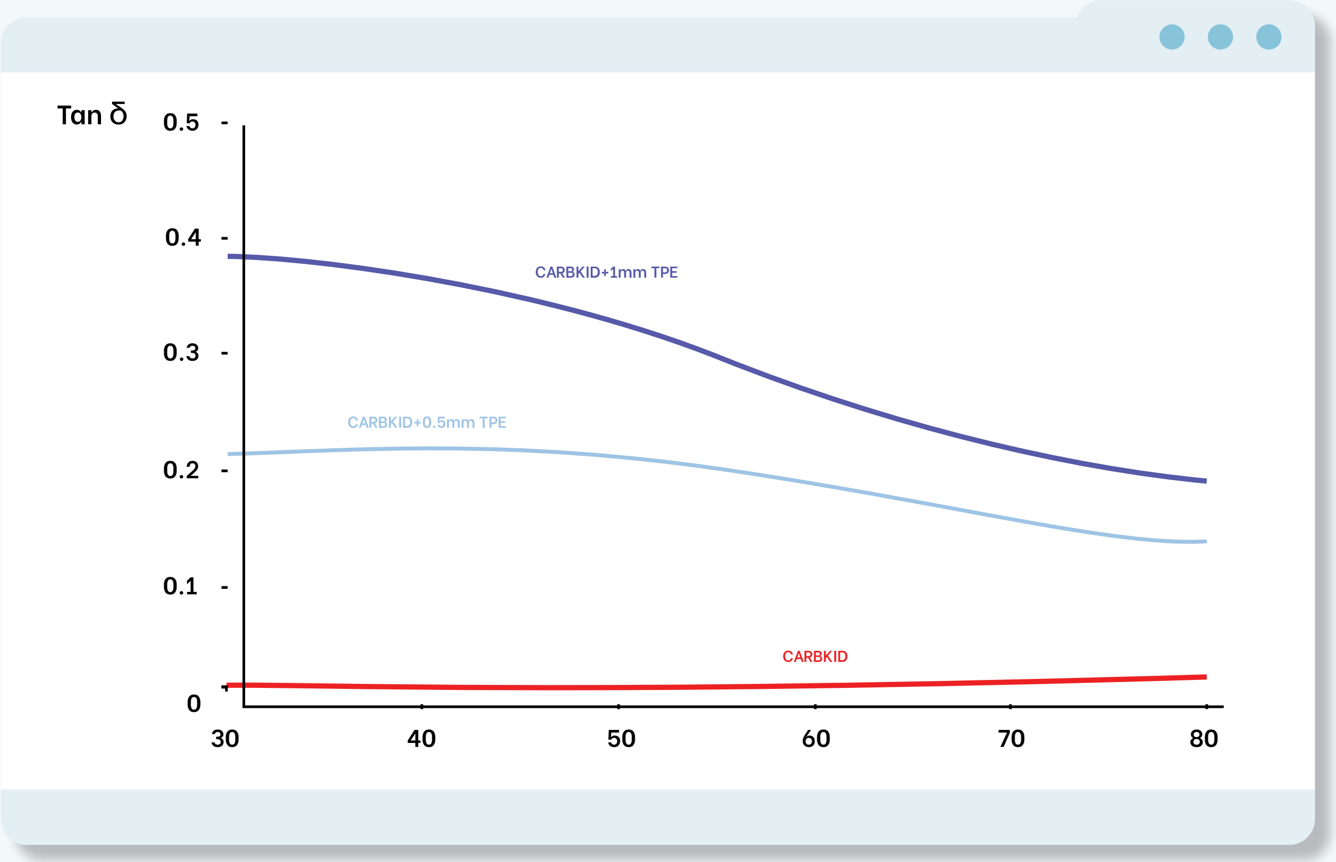


Polymerization process optimization

A shear specimen holder dedicated to the analysis of pasty materials allows following up the viscosity change on a huge scale, nearly a 10 millions ratio! This accessory is especially valuable for simulating a curing /polymerization process and optimizing the curing conditions (time and temperature cycle) necessary to reach the expected mechanical properties.

In the example beside, a resin epoxy has been exposed to a temperature ramp then a temperature stage at 130˚C; During the thermal cycle the specimen is sheared at 10Hz at controlled amplitude. The high acquisition data frequency allows getting a high data curves resolution without requiring interpolation between data points.

Applications



Electric vehicles new challenges

New Electric vehicles developments bring new challenges: by removing the thermal engine, the noise level inside the vehicle is much different than with former thermal vehicles impacting in a different way the acoustic comfort for users.

The continuous request of energy savings and new NVH brings a constant pressure on weight reduction and selection of materials.

Sheet Molding Compounds (SMC) have considerable potential as lightweight alternatives to traditional materials used in automotive components.

New researches have focused on the optimization of their dampening ability by incorporating Thermo-Plastic Elastomers (TPE) in their structure.

The graph beside presents the Tan Delta (damping) of 3 SMC specimens tested in 3 points bending mode with a DMA+300. The 3 SMC specimens are composed of TPE layers of different thicknesses. The benefit of adding TPE layer inside SMC is clearly a significant gain of damping: higher is the TPE layer, higher the damping.

Main specifications / Features

			DMA+300	DMA+1000	DMA+2000
Excitation & measurement channels					
Actuator			Electrodynamic actuator (METRAVIB technology)	Electrodynamic actuator (METRAVIB suspensions)	Electrodynamic actuator (METRAVIB suspensions)
Frequency	min		0.00001 Hz	0.00001 Hz	0.00001 Hz
Frequency	max		200 Hz	200 Hz	200 Hz
Frequency	max	option	1 000 Hz	1 000 Hz	1 000 Hz
Dynamic force	max		300 N *	1000 N *	2000 N *
Dynamic force	min		0.01 N *	0.01 N *	0.01 N *
Dynamic force	resolution		< 0.0001 N	< 0.0003 N	< 0.0003 N
Static force	range		-70 N up to +70 N	-250 N up to +250 N	-500 N up to +500 N
Static force	range	option	no	-500 N up to +500 N	no
Dynamic displacement	max		12 mm *	12 mm *	12 mm *
Dynamic displacement	min		0.05 µm *	0.1 µm *	0.1 µm *
Dynamic displacement	resolution		< 1 nm	< 1 nm	< 1 nm
Static displacement	range		12 mm	8 mm	10 mm

* peak to peak

Main specifications / Features

			DMA+300	DMA+1000	DMA+2000
Thermal chamber & environment control					
Thermal chamber internal dimensions	(HxWxD)			120 x 140 x 95 mm	
Hot mode					
Temperature (min)	min			room	
Temperature (standard thermal chamber)	max			500°C	
Temperature (thermal chamber with window)	max			300°C	
Temperature (thermal chamber with infrared window)	max			200°C	
Temperature (crack growth thermal chamber)	max			150°C	
Variation rate	min (heating)			0.1°C/min	
Variation rate	max (heating)			10°C/min	
Thermal control stability				+/-0.1°C	
Cold mode - Cryogenic sources		option			
Temperature	min			-150°C	
Variation rate	min			+/-0.1°C/min	
Variation rate	max			+/-10°C/min	
Thermal control stability				+/-0.2°C	
Cold mode - Air chiller		option			
Temperature	min			-70°C	
Variation rate	min			+/-0.1°C/min	
Variation rate	max			+/-10°C/min	
Thermal control stability				+/-0.1°C	
Controlled oxygen atmosphere system		option		10ppm to 20% O2	

Main specifications / Features

		DMA+300	DMA+1000	DMA+2000
Material Properties				
Modulus	min	250 Pa	500 Pa	500 Pa
Modulus	max	3E12 Pa	3E12 Pa	3E12 Pa
Modulus	Measurement range	> 7 decades	> 6 decades	> 6 decades
Delta	min	0°	0°	0°
Delta	max	90°	90°	90°
Tan delta	resolution	<0.000001	<0.000001	<0.000001

The features presented are not contractual and are subject to change without notice | July 2024

Metravib experts and partners assist you worldwide.

- Instruments installation
- User training
- Hot line assistance
- Calibration and maintenance
- Testing service
- Customization
- Tests procedures and standards definition

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