



High-Force Dynamic Mechanical Analysis (DMA)

Dynamic characterization of viscoelastic materials

be certain.

Introduction to DMA

In performance-critical applications, researchers and product developers depend on accurate dynamic materials characterization. Elastomers, thermoplastics and thermosets all require dynamic characterization to evaluate behavior before these materials are used to improve cars, planes, medical devices and consumer products.

Dynamic Mechanical Analysis, or DMA, is a dynamic characterization technique that measures stress as a function of strain, or force as a function of displacement. Viscoelastic materials, like polymers, behave both like an elastic solid and a viscous fluid. DMA measures the viscoelastic properties under dynamic oscillatory (often sinusoidal) test conditions.

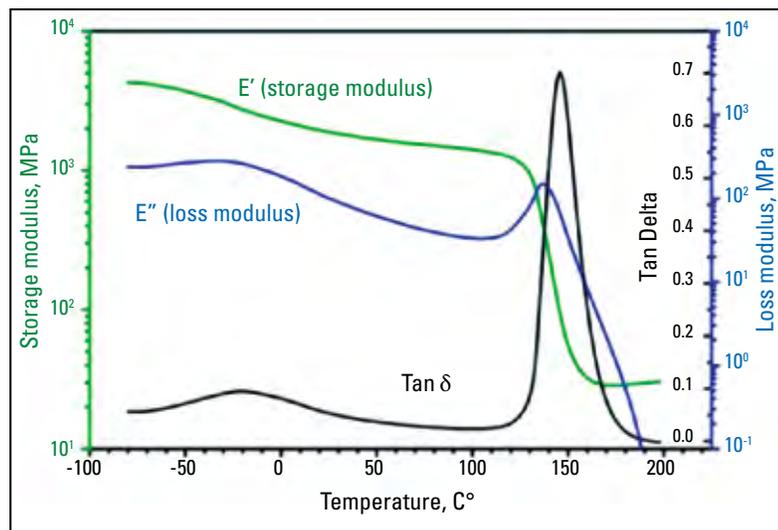
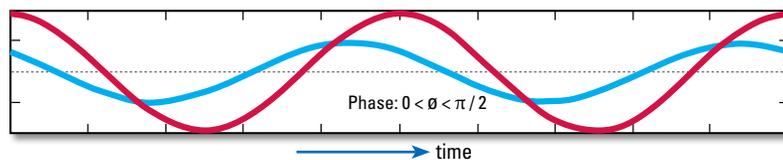
When dynamic stress is applied to the material and a resultant strain is measured:

- » The phase difference, δ , between the stress and strain waves is measured
- » The phase lag will be 0° for purely elastic materials and 90° for purely viscous materials
- » Viscoelastic materials will exhibit an intermediate phase difference

Typical viscoelastic properties include the following parameters:

- » (E') The storage modulus is the elastic component and describes the sample's stiffness
- » (E'') The loss modulus is the viscous component and is related to the sample's ability to dissipate mechanical energy through molecular motion
- » ($\tan \delta$) The tangent of phase difference provides information on the relationship between the elastic and inelastic components
- » (E^*) The complex modulus equals stress divided by strain
- » When the complex modulus (E^*) and the measurement of δ are known, the storage modulus, (E'), and loss modulus (E''), can be calculated.

Viscoelastic Stress & Strain



- » **Loss Modulus (E'')** measures the energy dissipated as heat, representing the **viscous** portion
- » **Storage Modulus (E')** measures the stored energy, representing the **elastic** portion
- » **Tan Delta ($\tan \delta$)** is simply a ratio between the two, loss/storage, or E''/E'

MTS Dynamic Characterization Experience

With core competencies in force control and motion measurement, MTS is ideally suited to developing new solutions for accurate and repeatable dynamic materials characterization. The materials testing division originated more than fifty years ago with a focus on durability testing to meet the needs of the aerospace industry. During this time, MTS pioneered closed-loop control solutions for testing applications, and soon developed into a test and measurement leader. Today, with opportunities to solve testing challenges in a wide range of industries across the globe, MTS continues to create better solutions for materials testing.

MTS has a long history of developing solutions for viscoelastic characterization, especially through working with the automotive industry to improve elastomer testing. This collaboration has led to many innovations and the discovery of best practices for testing viscoelastic materials.

As a result of these decades of dynamic characterization experience, MTS can offer an extensive product family that encompasses a wide range of forces and frequencies; comprehensive characterization expertise for materials and components; and software packages to address DMA, fatigue & fracture, durability, tension/compression, flex/bend, static deflection, and additional templates to address specific standards, including FDA regulatory tests for biomaterials and medical devices.



Critical Elements for Accurate DMA Test Systems

When choosing a test system for conducting DMA, it is critical to have a machine that does not introduce mechanical resonances, can measure across a great range of amplitudes and provides superior dynamic control.

No Mechanical Resonances

High-frequency excitation can inadvertently produce mechanical resonant modes within the load frame structure which will often corrupt the measurement accuracy of displacement and force transducers.

Displacement transducer measurements (either LVDT or Encoder) can be corrupted by erroneous motion at the transducer's mounting base due to resonant vibrations.

Force transducer measurements can be corrupted by resonant vibrations that induce high acceleration on the transducer. This acceleration can result in significant inertial errors in the force measurement. A good DMA system will not exhibit any detrimental mechanical resonant modes in the axis of measurement.

Measuring Across Extreme Dynamic Range Amplitudes

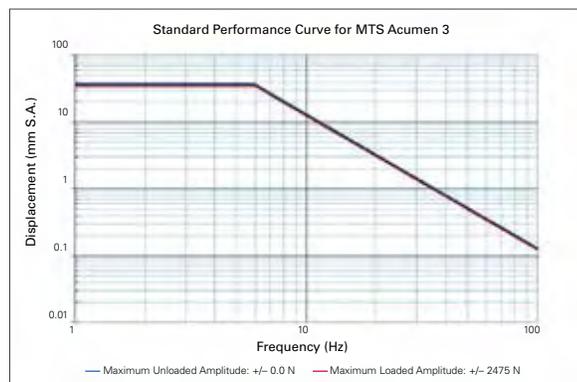
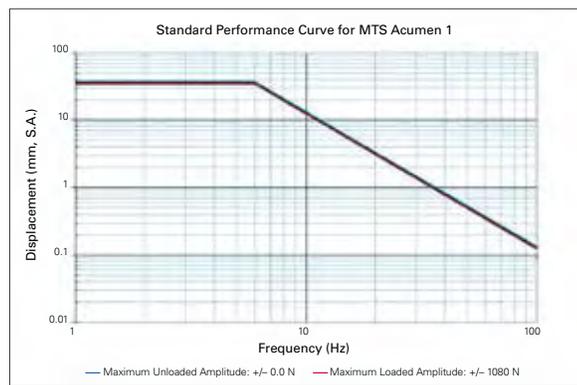
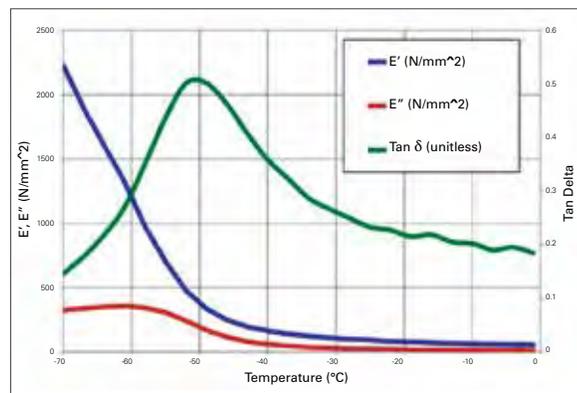
For elastomer and thermoplastic materials, measurements are often required both above and below the glass transition temperature within the same test set-up. The material elastic modulus (and stiffness) can change by a factor of 1,000 when transitioning from temperatures below the glass transition in which the material is hard like glass, to temperatures above the glass transition temperature at which the material is rubbery and relatively soft.

This implies that if a constant sinusoidal force were applied throughout the test, the material displacements in the glassy region could be relatively small. But that same sinusoidal force amplitude applied above the glass transition could result in displacements changing by a factor of 1000. Conversely if a constant sinusoidal displacement were applied throughout the test, the material forces in the rubbery region could be relatively small. But that constant displacement amplitude applied above the glass transition could result in forces changing by a factor of 1000. Both scenarios require a test system with the ability to measure across extreme dynamic range amplitudes.

Dynamic Amplitude Control

Most elastomers and thermoplastics have a significant amplitude non-linearity, meaning that the measured elastic modulus (or stiffness) of the material is dependent on the amplitude of the applied excitation. Since the material modulus and response are so amplitude dependent, it is extremely critical that the test input excitation amplitude is known and controlled accurately. This amplitude control becomes even more difficult at higher test frequencies.

Low bandwidth control loops or sluggish outer loop compensators can impose additional cycles on the specimen and cause internal specimen heating, resulting in misleading material characteristic data. Lack of extremely stable control laws can also result in control loop instability which can damage the specimen. Therefore accurate dynamic control is a critical DMA test system attribute.



MTS Acumen® Solution for High-Force DMA

DMA testing requires control of force and displacement, and accurate measurement in order to calculate dynamic stiffness. In a typical DMA profile, one controls both a mean load or displacement and a dynamic load or displacement. If the signals include extraneous resonances, the dynamic stiffness will not be accurate.

Superior Accuracy

The MTS Acumen Electrodynamic Test System provides a singular solution for DMA and fatigue testing in a quiet, energy efficient package. The electrodynamic load frame design also eliminates detrimental mechanical resonances, providing additional accuracy specifically for DMA testing.

Acceleration Compensation

Exceptional compensation of accelerating load train components is especially important in DMA due to high test frequencies and their impact on calculated material properties. MTS uses a patent-pending acceleration compensation technique that results in more accurate correction. High-fidelity sensors provide compensation for very small inertial errors, which results in the ability to collect accurate DMA data at significantly lower force amplitudes.

Higher Resolution Displacement

The MTS Acumen digital encoder provides higher resolution than an analog sensor, and can be used for testing peak to peak displacement as low as eight microns (0.008 mm) for higher repeatability and performance control. This capability is crucial for DMA measurement on stiff samples with small displacements.

Low-Force Testing Option

This high-performance sensor package provides force measurement for dynamic loads as low as 0.2 N peak to peak (standard system sensors test as low as 5 or 10 N peak to peak). The low-force option includes an Advanced Dynamic Response (ADR) sensor that is mounted on the system load cell and measures the dynamic loading while the system load cell measures the mean load. It offers the ability to measure force across more than two orders of magnitude (a factor of 200

or more) which can be important for DMA testing through the glass transition where the material modulus can change by a factor of 100 or more. Additionally, the ADR sensor takes advantage of acceleration compensation to allow for accurate and high-fidelity correction of inertial errors.

Secondary load cells are sometimes used for low-force testing, but MTS' experience suggests that tandem load cells do not provide adequate accuracy in DMA.

Creep and Stress Relaxation

The Acumen System can also perform static tests such as creep and stress relaxation. In creep, a stress is applied to the sample and held constant while deformation vs. time is measured. Stress is then removed and the recovery is measured. In stress relaxation, a deformation is applied and held constant and degradation of the stress vs. time is measured.

MTS ACUMEN DMA DIFFERENCE

- » No mechanical resonances
- » Patent-pending acceleration compensation for increased accuracy of dynamic properties
- » High-resolution digital encoder ideal for stiff samples with small displacements
- » ADR sensor option provides superior dynamic resolution for low-force testing
- » Test space that accommodates larger specimens to ensure representative material evaluation
- » Graphical representations of test setup, tuning and limits



A Full Complement of DMA Test Accessories

Grips, Fixtures & Environmental Chambers

Every testing application is unique, from the nature of the component or material to the design of the test. That's why MTS provides grips, fixtures and environmental chambers that have been tested and approved for use in DMA applications.



Environmental Chambers



Double Lap Shear Fixtures



Bend Fixtures



Shear Fixtures



Tension/Compression Grips



Thumb Screw Grips



Compression Platens



Wedge Action Tensile Grips



Cam-action Tensile Grips



Fully Reversed Loading Double Cantilever Bend Fixtures

DMA Application Software

With MTS Model 793.31 DMA/Dynamic Characterization software, you can measure the dynamic properties of viscoelastic materials or components under a wide range of variables. The shape of a viscoelastic specimen often adds complexity to modulus calculations. To help ensure accurate calculation of modulus, this software allows you to specify the specimen shape used in your test.

Key Features

- » Nested sweeps—define nested sweeps so that all of the variables can be changed together or separately
- » Mixed mode—specify the mode of control for the dynamic amplitude and the mean level as follows:
 - Mean control parameter—Load, displacement, strain, or stress
 - Amplitude control parameter—Load, displacement, strain, acceleration, or stress
- » QC limit definition—define calculated value data ranges and limits, which is useful in pass/fail QA/QC applications
- » Automatic correction—when specimen feedback does not align with the desired waveform, the software compensates for errors with a patented Amplitude/Phase Control (APC) algorithm. It also measures and corrects for errors due to unwanted accelerations, load path compliance, mechanical and electrical phase errors, and transducer roll-off.

GLASS TRANSITION MEASUREMENT

Four decades of strain amplitudes can be accomplished with frequencies that can range from 0.01 Hz to 100 Hz. Over four decades of modulus can be measured to allow for complete glass transition temperature measurements.

FEA TOOL COMPATIBILITY

The ASCII file format allows data transfer to standard finite element analysis (FEA) packages for efficient modeling of the material's behavior.

COMPONENT CHARACTERIZATION

For component performance analysis, MTS Model 793.31 software measures a wide array of parameters, including spring rate (storage stiffness), damping (C), transmissibility, and hysteretic energy.

ELASTOMEREXPRESS™ SOFTWARE

This simplified version of DMA application software includes standard test templates designed to automate decision-making processes. Perfect for high-throughput QA/QC environments, ElastomerExpress software minimizes operator interaction and maximizes efficiency.

Measure these parameters and many other calculated properties:

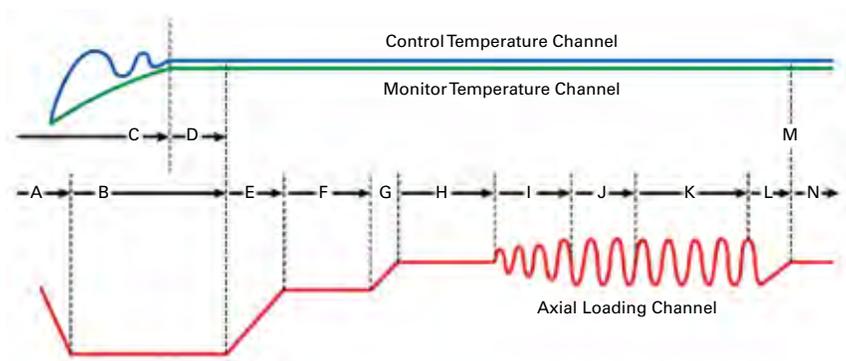
- » Stiffness (K)
- » Damping (C)
- » Modulus (E or G)
- » Tan delta (tan δ)

Specify the following as fixed or sweep in a linear, logarithmic, or arbitrary manner:

- » Frequency
- » Mean
- » Amplitude
- » Temperature
- » Phase (if multiple channels)

DMA Software Options

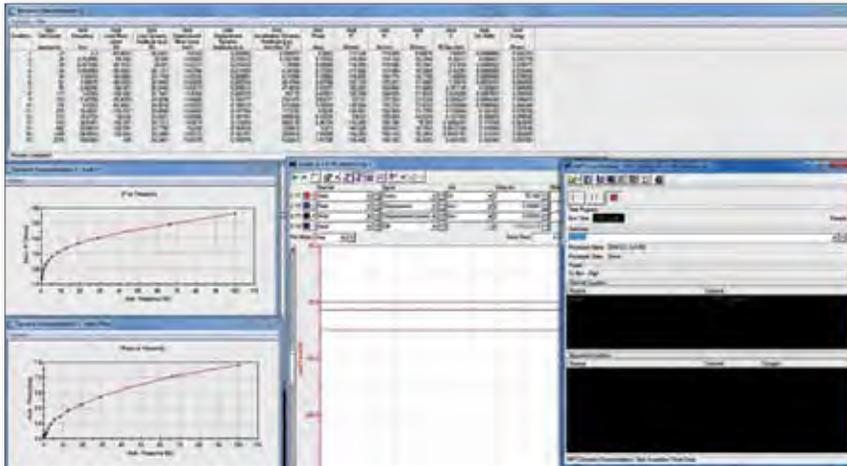
- » 793.31 DMA/Dynamic Characterization
- » 793.30 Elastomer Express (for QA/QC testing)
- » 793.33 Static Deflection
- » Master Curve Software



Format of a typical DMA test condition

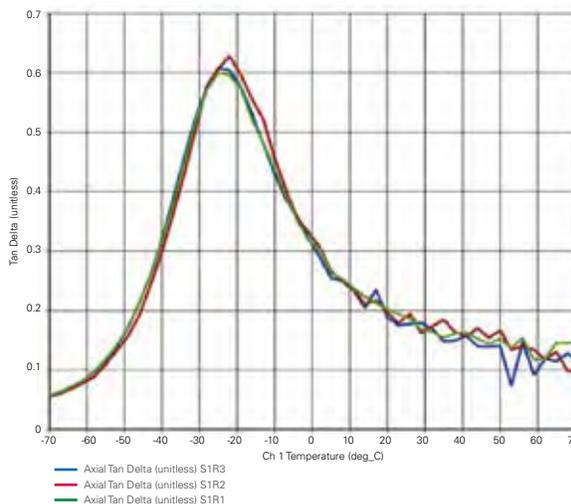
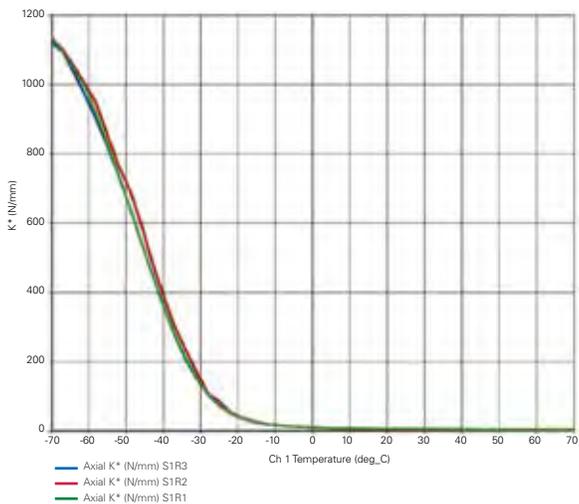
- A. Ramp to hold value
- B. Hold during temperature dwell
- C. Stabilize temperature
- D. Dwell at temperature
- E. Ramp to zero
- F. Relax at zero
- G. Ramp to mean or set point
- H. Dwell at mean
- I. Converge dynamic amplitude
- J. Pre cycle
- K. Acquire data cycles
- L. Return to base or mean level
- M. Record temperature
- N. Process data

Example of a Frequency Sweep Test



Test Parameters
 Frequency: 0.2 Hz to 100 Hz, 15 Log. steps per sweep.
 Mean Level: -100 N.
 Dynamic amplitude (p-p): 25 N.

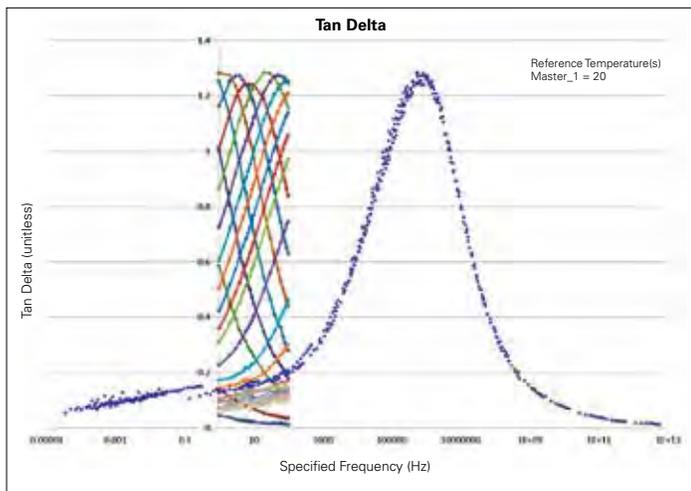
Typical Plots for K^* and $\tan \delta$



Time-Temperature Superposition & Master Curves

The Time-Temperature Superposition (TTS) principle is based on the concept in polymer physics that time and temperature have similar effects on the properties of linear viscoelastic materials. It is used to determine temperature-dependent properties of viscoelastic materials at temperatures above and below laboratory test (reference) temperatures.

MTS Master Curve software leverages the TTS principle by using Williams-Landel-Ferry (WLF) model calculations to generate master curves for a given material. These easy-to-use, Excel-based graphs can show temperature-dependent mechanical property behavior out to MHz or even GHz, in other words far beyond a lab's physical testing capabilities. For this reason, master curves are insightful for comprehensive material characterization.



Create Master Curves via Time Temperature Superposition (TTS) – Williams-Landel-Ferry (WLF) Model

Comprehensive Viscoelastic Characterization Capabilities

As a viscoelastic test solutions leader, MTS has helped the automotive industry characterize their elastomer products for several decades. Today, many industries leverage MTS' in-depth understanding of dynamic characterization to create testing solutions for materials and for components, some of which may need to be tested at higher frequencies.

Broad Test System Portfolio

With one of the widest ranges of force capacity (0.0002 kN – 250 kN) and frequency capability (0.01 Hz – 1000 Hz), MTS solutions accommodate diverse applications and appeal across industries. And with options available in servohydraulic and electrodynamic technologies, MTS can deliver the most appropriate solution for specific applications.

Characterization Expertise

MTS provides a single, comprehensive suite of software for dynamic characterization of viscoelastic materials across all product lines. In addition to its industry-leading DMA package, this software can also perform Fatigue & Fracture, Durability, Tension/Compression, Flex/Bend, Static Deflection, and more.

Applications Support

The MTS global service and support team can help optimize your testing with training classes or consulting services. Systems Integration Engineers and Applications Engineers bring knowledge of a wide array of test applications and testing techniques.



Examples of MTS Test Systems for Viscoelastic Materials Testing

Unparalleled Service and Support

MTS test systems are backed by the global MTS Service and Support organization. This highly experienced team offers lifecycle management services and is committed to maximizing the uptime and operational efficiency of each system. With the expertise to support test equipment from pre-installation to decommission and at every point in between, MTS has the service solutions to meet your needs for test schedule predictability, data integrity, system performance optimization and budget management.

Consulting Services

MTS offers a complete set of professional engineering services, including systems engineering, test consulting and facilities design services.

Onsite Services

MTS field service engineers have a worldwide reputation for applications expertise and will respond to requests for support or repair quickly and efficiently.

Training

MTS training programs are designed to improve operator efficiency and optimize system performance. Expertly led and completely customizable, these courses provide engaging hands-on learning experiences.

Calibration and Alignment

All test labs must calibrate their testing equipment to help ensure data accuracy. MTS provides top-quality, accredited calibration services and load frame alignment services to minimize data variance.

Maintenance and Monitoring

Based on decades of service experience, MTS has developed a set of well-defined routine maintenance offerings tailored for specific systems and components, to help extend equipment life.

Upgrade Solutions

As technology improves, an upgrade is often the most economical way of expanding lab capabilities and extending the life of existing test equipment. MTS offers upgrades and replacements for mechanical components, controllers and software.

ASK MTS ABOUT:

- » Comprehensive DMA and Fatigue Solutions
- » DMA Test Methods (Standards)
- » DMA Market Segment Packages for Elastomers, Biomaterials, Polymers, Composites, Contract Test Labs, QA/QC Labs
- » TTS Master Curves and Analysis
- » DMA Consulting Services



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