Rubber Testing Solutions

...Innovative testing solutions made in Germany!
MonTech Rubber Testing Instruments

Supplying testing systems that answer every diverse business need covering a full range of instruments, software systems and service dedicated to the rubber industry. Whether for static or dynamic rheology, viscosity or processability testing, measurement of mechanical and physical properties, sample preparation, integrated laboratory software along with global on-site calibration and emergency services - we have solutions to meet all your laboratory and rubber testing needs!

Get in touch with us today to find your rubber testing solution!
Huge changes in rubber industry affecting quality control and laboratories

- Rising quality demands and more demanding applications
- Increasing pressure on production costs and integration
- Changing National and international standards
**Moving Die Rheometer and Rubber Process Analyzer Application Examples**

**Isothermal Cure**

Isothermal cure experiments are the most common type of test for quality control in rubber and elastomer processing.

MonTech Moving Die Rheometers provide high precision data as well as a simple operation of the instruments.

All the important characteristics, such as minimum / maximum elastic torque, scorch times, cure times and reaction rates are precisely calculated, with over 3500 different datapoints. All data is available in numerical as well as graphical form; limits, control gates and tolerance graphs can easily be set, and Pass / Fail status is automatically evaluated after each test.
In general, the mechanical properties of materials depend on frequency. A good understanding of the influence of frequency on a material is therefore very important for its practical use. For example, a material appears stiff under the action of a force at high frequency, but soft when the force is applied slowly. Isothermal frequency sweeps provide information about the weight distribution MWD (crossover modulus) as well as average molecular weight AWM (crossover frequency). But as the behavior of viscoelastic materials like polymers not only depends on frequency, it also depends on temperature.

MonTech has incorporated further advanced testing capabilities such as the Time-Temperature Superposition principle (TTS), which is based on the equivalence between frequency and temperature behavior during transition processes, forming the basis of WLF master-curve modelling available on MonTech dynamic Rheometers, even for predicting material performance at frequencies outside the range that can be measured with a dynamic mechanical analyzer.
Moving Die Rheometer and Rubber Process Analyzer Application Examples

Strain Sweep for Filler Loading "Payne-effect"

The Payne effect is a particular feature of the stress-strain behaviour of rubber, especially rubber compounds containing fillers such as carbon black and silica. Physically, the Payne effect can be attributed to deformation-induced changes in the material's microstructure, i.e. to breakage and recovery of weak physical bonds linking adjacent filler clusters. Measurement of modulus vs. strain is therefore essential to understanding and quantifying Filler loading, filler dispersion and filler-filler interaction in the low strain region, and polymer-filler interaction at higher strain.

The resulting characterisations of material structure are essential as they directly impact dynamic stiffness and damping behaviour of final products such as rubber bushings, automotive tyres and all other rubber goods. Similar to the Payne effect under small deformations is the Mullins effect, which is observed under larger deformations in the non-linear viscoelastic range.
Quality control testing
In the rubber industry

**RAW POLYMER TESTING**
- Viscosity
- MW Distribution
- Branching
- Gel Content
- Ageing

**PROCESSABILITY TESTING**
- Mill / Extrusion Behaviour
- Die Swell
- Mould Flow
- Process Viscosity
- Filler Dispersion

**ADVANCED CURE TESTING**
- Process Scorch
- Cure Rate
- Cure Time
- Reversion
- Non-isothermal Cure Testing

**DYNAMIC MECHANICAL TESTING**
- Modulus
- Elasticity
- Viscoelastic Properties
- Heat Build-up
- Damping
RPA POLYMER APPLICATION (Example 1)

EPDM Raw polymers
Tangent Delta

EPDM 1 & 3 have equal Mooney viscosity

14% stain - 125° C
RPA PROCESSABILITY APPLICATIONS (Example 4)
RPA POST CURE APPLICATIONS (Resilience)

Correlation Resilience (Amb Temp) and G’’ (RPA)  
80 °C and 2% strain after cure

Resilience (\%) vs. G’’ (MPa)

Correlation coefficient: 0.999

Relevant to:
- Rebound
- Hysteresis,
- Heat Build-up
- Rolling Resistance
International standards:
Dynamic testing replacing traditional tests

e.g. Viscosity

INTERNATIONAL STANDARD

ISO 13145

First edition
2012-09-15

Rubber — Determination of viscosity and stress relaxation using a rotorless sealed shear rheometer

Caoutchouc — Détermination de la viscosité et de la relaxation de contrainte au moyen d’un rhéomètre à visulement sans rotor étanche
International standards: Dynamic testing replacing traditional tests
e.g. Viscosity

Introduction

The rheological properties of rubbers are related to their structural characteristics and will influence the behaviour of the rubber during processing and the performance of the final product.

For these reasons, the industrial environment requires instruments that can quickly and easily evaluate the rheological properties.

As a consequence, this standard test method was formulated using a rotorless sealed shear rheometer for rheological evaluation under defined conditions.

This test could be an alternative to the Mooney viscometer, still used as standard in many parts of the rubber industry to measure Mooney viscosity (in accordance with ISO 289-1). The defined conditions have been selected to provide a shear rate range similar to that used for Mooney viscosity and a good repeatability level.

This new test procedure should be performed over a short time and preferably in the automatic mode to optimize test efficiency.
Test sequence:

9.1 Testing sequence

The test method specified in this International Standard consists of three different steps applied in sequence on the same test piece:

a) **Preheat**: 1 min at 100 °C with cavity closed and both dies stationary.

b) **Time sweep** at 100 °C: The lower die oscillates with a frequency of 0.1 Hz, applying a strain of 150 % for six oscillations in total.

c) **Conditioning and stress relaxation**: The test piece is kept for 1 min at 100 °C with the dies stationary (this conditioning is necessary for stress recovery after the time sweep). Then a step strain of 150 % is applied and kept constant for 35 s. During this time, the decreasing torque (stress relaxation) is measured. If stress relaxation is not needed, the third stage may be omitted.

Different temperatures may be considered for specific rubbers as agreed between the interested parties.
Results:

<table>
<thead>
<tr>
<th>Step 1:</th>
<th>Pre-heat</th>
</tr>
</thead>
<tbody>
<tr>
<td>Time</td>
<td>1,00 min</td>
</tr>
<tr>
<td>Temperature</td>
<td>100,0 °C</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Step 2:</th>
<th>Time Sweep</th>
</tr>
</thead>
<tbody>
<tr>
<td>Temperature</td>
<td>100,0 °C</td>
</tr>
<tr>
<td>No of Cycles</td>
<td>6</td>
</tr>
<tr>
<td>G' @ 150%</td>
<td>20,62 kPa</td>
</tr>
<tr>
<td>G'' @ 150%</td>
<td>29,17 kPa</td>
</tr>
<tr>
<td>tan delta</td>
<td>1,415</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Step 3:</th>
<th>Stress Relaxation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Temperature</td>
<td>100,0 °C</td>
</tr>
<tr>
<td>Peak torque</td>
<td>13,47 dNm</td>
</tr>
<tr>
<td>Torque after 1 sec</td>
<td>5,13 dNm</td>
</tr>
<tr>
<td>Torque reduction after 1 sec</td>
<td>61,9 %</td>
</tr>
<tr>
<td>Torque after 20 sec</td>
<td>2,57 dNm</td>
</tr>
<tr>
<td>Torque reduction after 20 sec</td>
<td>80,9 %</td>
</tr>
</tbody>
</table>

< Correlates to Mooney Viscosity
Dynamic rheological testing

- Broad penetration into R&D Laboratories, especially of major companies
  - High potential for production Quality Control
    - Great cost / performance
    - Easy handling
    - Automated data process / result analyses
- Production control based solely on MDR no longer sufficient, additional data required
Fully automated
Online testing systems
For direct mixing line integration

Transformation in
Quality Control processes and
Instrumentation in the Rubber Industry
MonTech shopfloor testing instrument automation capabilities
MonTech was the first company that integrated traditional lab testing machinery to the shopfloor production equipment.

Since 2008 MonTech has successfully automated the testing and QC On over 90 mixing lines in turnkey projects with various different automation systems.

MonTech is globally the only turnkey provider being able to provide and integrate high end testing machiners with rubber mixing line production equipment.

The following slides will provide a short overview of our automation capabilities and possible systems and their integration. These kind of systems however can be specifically tailored to your mixing line and testing needs!

MonTech automation systems stand for a perfect integration of the testing machinery with the automation and mixing line / lab host systems
MonTech testing instrument references
Continental Tires, Korbach plant, Germany
MonTech testing instrument references
Continental Tires, Port Elizabeth, South Africa
Question:

How applicable to the process and to the final product is the standard, isothermal cure test in a Moving die Rheometer?

Most Tire manufacturers nowadays only regard a combination of physical (density), static (rheology) as well as dynamic (RPA) sufficient to truly discriminate and assess properties of the mix.
How to integrate this relevant testing into a modern rubber compounding factory?

NEW APPROACH:

Automated testing cells for compounding line integration
How to integrate this relevant testing into a modern rubber compounding factory?

How much / how many samples to test?
4,5 grams sample for each batch of up to 400 kgs?
> Line integration and oversampling
>>> Regard testing as a part of the process!

Batch to Batch and in batch consistency?
> Combination of static and dynamic testing

When and what to test?
> Process and control integration
Revolution in rubber testing: Process and System Integration

Most companies and rubber factories operate at this level of technology for more than a decade. Modern integrated testing systems are a true revolution and innovate the rubber compound quality control process.
How to sample? Volumetric sample is important.

Test sampling is most critical for a proper, fully automated line integration and reproducible test results. State of the art approach:

- Volume controlled sample cut – fully automated
- Sampling frequency based on software analysis with fixed or variable testing frequency and batch separation
  > Integration between ERP, Mixer Control, MES and Batch-off controls.
Question: Where to sample?  Line integration possibilities
Hot compound testing directly after mill / twin screw before dip tank
Line integration possibilities
Warm sampling integration - Example
Line integration possibilities
Warm sampling integration - Example
Question: Where to sample? Line integration possibilities
End of line testing integration
Line integration possibilities
End of line testing integration - Example
Line integration possibilities
End of line testing integration - Example
MonTech online testing cells – production line integration
Revolution in rubber testing!

- Sampling and testing full automated – with even increased sampling frequency
- Includes MDR, RPA, Viscosity, … Eliminates QC lab/ for routine testing
- Oversampling for continuous production lines > 3 samples per batch
MonTech online testing cells – Commercial point of view
Revolution in rubber testing!

- Typical ROI time for MonTech online testing cells:

1 to 3 years at even doubled testing frequency
MonTech online testing cells – example layout configuration
Quality control testing cells – production line integration
Revolution in rubber testing!

What tests and what testing frequency has to be covered online?

- **Test and instrument types for online integration:**
  - Volumetric sample cutting
  - Sample washing and drying
  - MDR and RPA
  - Compression Density testing
  - Compression and transfer moulding
  - Hardness (Shore, IRHD) measured on cured specimen
  - Cured specimen conditioning (heating, cooling)
  - …
Quality control testing cells – production line integration

Revolution in rubber testing!

Testing Cell – example sequence of operation:

- Material dumped from mixer, LIMS will create a XML file with required tests and test specification as well as information on samples for Release test (in the cell) / no-release test (in the lab) requirement. For queuing purposes.
- Actual sample cutting on Batch-off > Notification by Batch-off to Cell of cut sample (Batch number, Material code, Order Number, Mixer, date). (First cut sample will be used for release testing, possible second sample for non-release)
- For each sample the cell confirms receipt of sample to Batch-off
  - Sample for release testing
    - Processing of sample according to test spec by LIMS.
    - Pickup
    - Die cutting
    - Washing / Drying
    - Placement of sample in
      - MDR queue (5 samples)
      - RPA queue (5 samples)
      - Weighing + Density tester (3 cuts)
    - Remaining rubber will be put in bin for scrap bin.
  - Sample for non-release testing
    - Barcode sample label with unique LIMS order number is printed by the cell and application to slab
    - Putting non-release test sample with applied label in a specific non-release lab test bin.
- Notification of test results to LIMS whenever a test is completed (Storage of results and raw data also in MonTech DB)

One cell status monitor to be placed in the lab for Cell status, Alarm and Error message
Rubber quality control testing – line integration
Bring your quality control testing to the next level – start today!

Fully automated triple MDR Rheometer test cell installed at a global tire company.
Reference online testing cell – turnkey supplied by MonTech
New mixing lines are fitted with fully automated robotic cells including Sample cutting, MDRs, RPAs, Density testing, Vision sensors, …

Frontview with tested samples
Reference online testing cell – turnkey supplied by MonTech

New mixing lines are fitted with fully automated robotic cells including Sample cutting, MDRs, RPAs, Density testing, Vision sensors, …
Quality control testing – the future
Next level of intelligent testing

- TESTING AS A CLOSED LOOP CONTROL WITH THE MIXER CONTROL
- Fully automated data analysis
- Automatically generated, multi dimensional decision model
- Self-learning system
- Variable control of testing frequency and quality feedback
- Takes side-effects such as e.g. first-batch into account
**Software integration**

*Common data platform for all system within a single and between sites*

- Data integration is critical for being able to correlate all quality relevant data
- Data warehousing with manual and automatic data evaluation
Thank you!