Ten Steps to a Better Rheological Measurement

2005 Rheology training seminar

Ten Steps to Better Rheological Measurements

1. Understand why you are doing the measurement.
2. Understand your instrument
3. Know your sample
4. Selecting the correct test fixture
5. Loading the sample
6. Oscillation testing guidelines
7. Transient testing guidelines
8. Steady shear testing guidelines
9. Some instrument limitations
10. Presenting the data

Flow of a Rheological Experiment

- Identify the purpose of the experiment
- Design, program & run test
- Evaluate data and 0ptimize (if necessary)
- Results

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Points 3 through 8 are sample specific
- •Polymers
- •Structured Fluids
- •Solids

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Understand Why Your Doing the Measurement

- A successful rheological experiment requires thought and careful planning.
- The first step in any rheological experiment is to identify the purpose of your experiment.
- Ask youself:
  - What is the goal of the testing?
  - What do you want to know about your material?
  - Do you know what kind of experiment I should do?
- More specific questions will depend on reason the reason
- for the experiment!

Understand Why Your Doing the Measurement

The first step in any rheological experiment is to identify the purpose of your experiment

The 3 main reasons for rheological testing are:

- Characterization of structure
  MW, MWD, formulation, state of flocculation, etc.
- Predicting process performance
  Extrusion, blow molding, pumping, leveling, etc.
- Understanding end-use performance
  Strength, use temperature, dimensional stability, settling stability, etc.

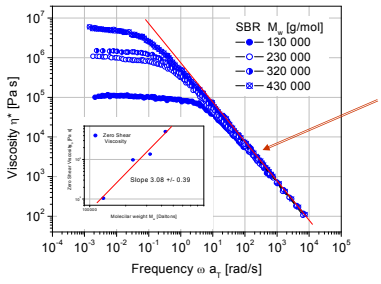
Rheological Approach

Use rheometer to characterize flow and viscoelastic properties to:

1. Understand structure
   - Requires understanding of how structure affects rheological properties
2. Predict processing performance
   - Requires understanding of what rheological properties are desired for processing
3. Predict product performance
   - Requires understanding of desired performance characteristics and how they relate to rheological properties

Influence of MW and MWD on Viscosity

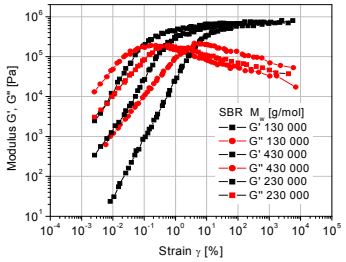
The zero shear viscosity increases with increasing molecular weight. TTS is applied to obtain the extended frequency range



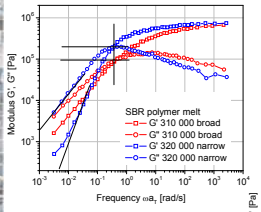
The high frequency behavior (slope -1) is independent of the molecular weight

Influence of MW on G‘ and G‘‘

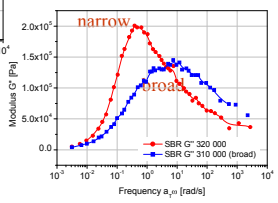
The G‘ and G“ curves are shifted to lower frequency with increasing molecular weight.



Influence of MWD on G‘ and G‘‘

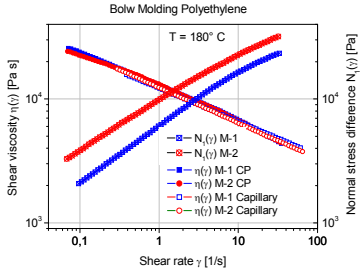


The maximum in G“ is a good indicator of the broadness of the distribution



The G‘, G“ cross-over point is another measure of the broadness of the distribution

Molecular Structure and Processibility

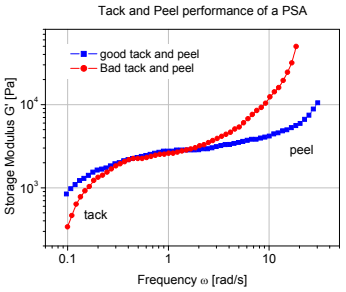


Sample M-2 has a much higher elasticity i.e normal force

Sample M-2 produces significantly heavier bottles!!!

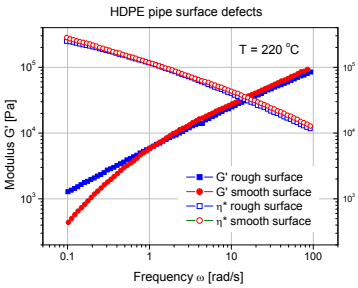
Tack and Peel of Adhesives

Bond strength is obained from peel (fast) and tack (slow) tests and can be related to the viscoelastic properties at different frequencies



Tack and Peel have to be balanced for an ideal adhesive

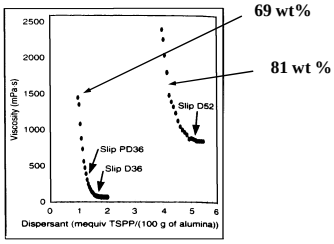
Surface Defects during Pipe Extrusion



Surface roughness correlates with G' or elasticity => broader MWD or tiny amounts of a high MW component

Sample 1 shows a rough surface after extrusion

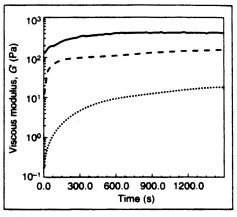
Deflocculation Curves for A16G Alumina



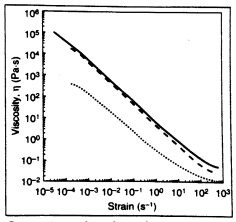
Deflocculation curve for slips D36, PD36 and D52. D36 and D52 represent the lowest viscosity states and PD36 refers to partial deflocculation.

[Reed, J.S. Introduction to the Principles of Ceramic Processing J. Wiley & Sons Inc 1988]

Rheological Characterization of Slips



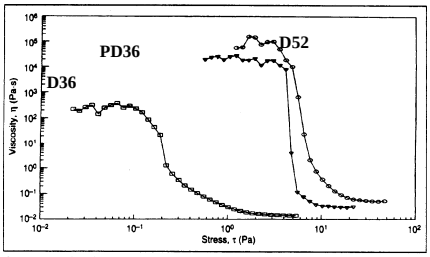
Dynamic time sweeps depicting the dependence of G' on time for three alumina slips ((—) D52, (---) PD36 and (···) D36) (stress = 0.1 Pa, frequency = 1.0 radian/s, temperature = 25°C and preshear at 100 s^{-1} for 60 s).



Comparison plot of steady-stress sweeps showing the dependence of viscosity on strain rate, demonstrating the extensive strain-rate range of the stress rheometer ((—) D52, (---) PD36 and (···) D36).

J.M. Keller, R. Ulbrich & R. Haber, American Ceramics Bulletin (76) 3 pp 87-91

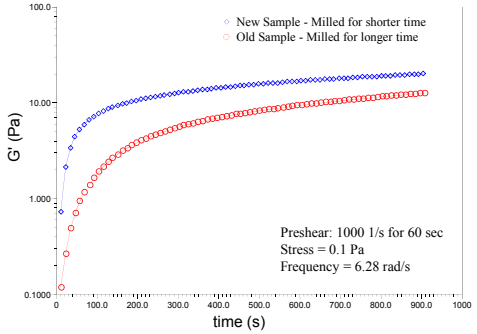
Rheological Characterization of Slips



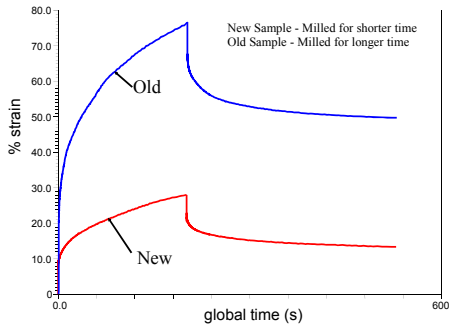
Comparison plot of steady-stress sweeps for A16SG slips: (○—○) D52, (□—□) D36 and (▽—▽) PD36. Dependence of viscosity on stress is shown depicting the critical stress.

J.M. Keller, R. Ulbrich & R. Haber, American Ceramics Bulletin (76) 3 pp 87-91

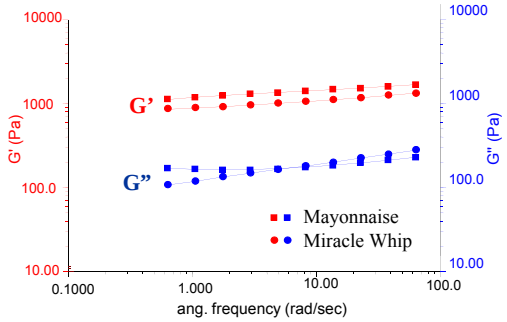
Automotive Paint: Breakdown - Rebuild



Automotive Paint : Creep Recovery Test



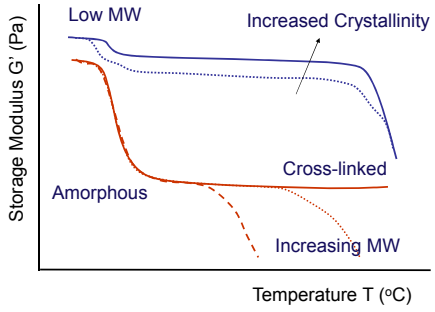
Mayonnaise and "Miracle Whip"



Why do the measurement?

- Predict Processability
- Predict End-Use Performance
- Glass Transition and secondary transitions
- Damping behavior
- Determine working temperature range, impact behavior, residual strain trapped in moldings, etc..
- Degree of curing, post curing
- Ageing
- Compatibility, Dispersion, …
- Morphology characterization, Crystallinity,
- Fingerprint of materials

Polymer Materials Change with Temperature



Alternative Approach

Use rheometer to simulate processing or end use conditions to determine suitability for application.

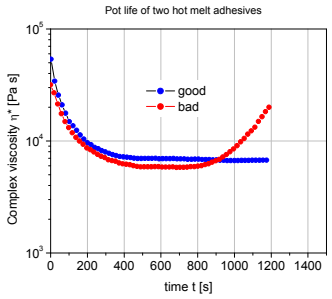
1. Requires definition of processing or end use conditions such as shear rate, strain, stress, and temperature, in order to translate test to rheometer or DMA.
2. Must understand instrument and instrument limitations in order to determine if rheometer can simulate processing or end use conditions.

Defining Shear Rate Ranges

Situation	Shear Rate Range	Examples
Sedimentation of fine powders in liquids	10^{-6} to 10^{-3}	Medicines, Paints, Salad dressing
Leveling due to surface tension	10^{-2} to 10^{-1}	Paints, Printing inks
Draining off surfaces under gravity	10^{-1} to 10^{1}	Toilet bleaches, paints, coatings
Extruders	10^{0} to 10^{2}	Polymers, foods
Chewing and Swallowing	10^{1} to 10^{2}	Foods
Dip coating	10^{1} to 10^{2}	Confectionery, paints
Mixing and stirring	10^{1} to 10^{3}	Liquids manufacturing
Pipe Flow	10^{0} to 10^{3}	Pumping liquids, blood flow
Brushing	10^{3} to 10^{4}	Painting
Rubbing	10^{4} to 10^{5}	Skin creams, lotions
High-speed coating	10^{4} to 10^{6}	Paper manufacture
Spraying	10^{5} to 10^{6}	Atomization, spray drying
Lubrication	10^{3} to 10^{7}	Bearings, engines

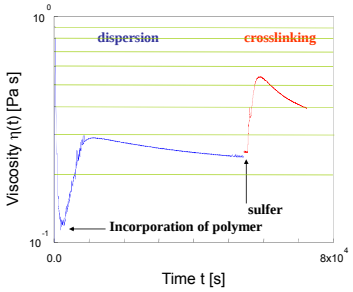
Pot Life of an Hot Melt

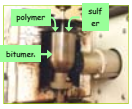
Viscosity stability at procees temperature is critical for hot melt adhesives. The viscosity builds up due to oxidative crosslinking



Open time of this polyamide resin depends on moisture

Formulation of Bitumen





- monitor the formulation process
- determine product performance

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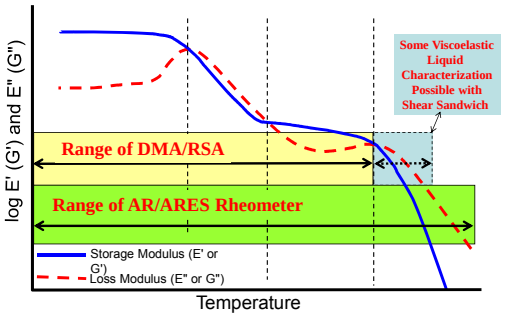
Ranges of Rheometers and DMA's



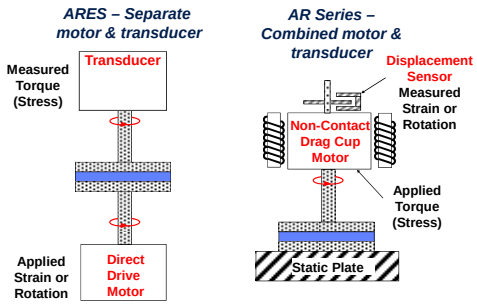
Understand Your Instrument First!

Two types of rotational rheometers and DMA's

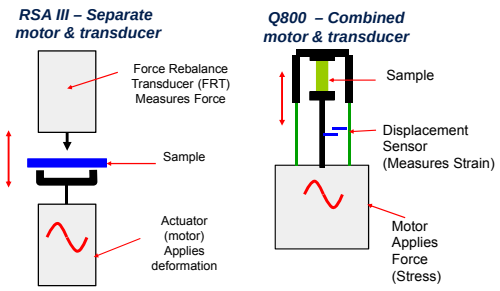
- AR-Series Rheometer and Q800/2980 DMA
  - Single/combined motor transducer
- ARES Series Rheometers and RSA III Solids Analyzer
  - Dual head/separate motor transducer

Both techniques, depending on the configuration, have different specification, different features and different performance for different applications

Rotational Rheometers Designs



DMA Designs



How Rheometers and DMA's Work

Rheology is the science of flow and deformation of matter
or
the study of stress– deformation relationships.

Fundamentally a rotational rheometer or DMA will control or measure:

1. Force or Torque
2. Displacement or Angular Displacement
3. Linear or Angular Velocity
4. Normal Force
5. Temperature

Important Rheometer Specifications

- Torque Range
- Angular Resolution
- Angular Velocity Range
- Frequency Range
- Normal Force
- Temperature System and Range

ARES or SMT Instrument Specifications

ARES Technical Specfications	ARES-LS2	ARES-LS1	ARES
Motor	High Performance LS	High Performance LS	Standard HR
Torque Range	2 µN.m - 200 mN.m	0.2 µN.m - 100 mN.m	2 µN.m - 200 mN.m
Strain Amplitude	5 µrad - 500 mrad	5 µrad - 500 mrad	5 µrad - 500 mrad
Angular Velocity Range	2 E-6 - 200 rad/s	2 E-6 - 200 rad/s	1 E-3 - 100 rad/s
Angular Frequency	1 E-5 - 500 rad/s	1 E-5 - 200 rad/s	1 E-5 - 500 rad/s
Norma/Axial Force Range	0.002 - 20 N	0.002 - 20 N	0.01 - 20 N

ARES Technical Specfications	ARES-RFS	ARES-RDA
	Standard HR	Standard HR
Motor	0.2 µN.m - 100 mN.m	20 µN.m - 200 mN.m
Torque Range	5 µrad - 500 mrad	5 µrad - 500 mrad
Strain Amplitude	1 E-3 - 100 rad/s	1 E-3 - 100 rad/s
Angular Velocity Range	1 E-5 - 200 rad/s	1 E-5 - 500 rad/s
Angular Frequency	0.01 - 20 N	Qualitative
Norma/Axial Force Range		

AR or CMT Instrument Specifications

AR Technical Specifications	AR 2000	AR 1000	AR 550/QCRII
Torque range CS	0.1 µN.m - 200 mN.m	0.1 µN.m - 100mN.m	1 µN.m - 50mN.m
Torque range CR	0.03 µN.m - 200mN.m	0.1 µN.m - 100mN.m	1 µN.m - 50mN.m
Displacement Resolution	0.04 µrad	0.62 µrad	0.62 µrad
Angular Velocity Range CS	1 E-8 - 300 rad/s	1 E-8 - 100 rad/s	1 E-8 - 100 rad/s
Angular Velocity Range CR	1E-4 - 300 rad/s	1E-3 - 100 rad/s	1E-3 - 100 rad/s
Angular Frequency Range	7.5 E-7 - 628 rad/s	6.3 E-4 - 628 rad/s	6.3 E-4 - 250 rad/s
Normal/Axial Force Range	0.005 - 50 N	0.01 - 50 N	0.01 - 50 N

RSA III and Q800 Instrument Specifications

Maximum Force	35 N	18 N
Minimum Force	0.001 N	0.0001 N
Force Resolution	0.0001 N	0.00001 N
Strain Resolution	1 nanometer	1 nanometer
Modulus Range	$10E^{3}$ to $3x10E^{12}$ Pa	$10E^{3}$ to $3x10E^{12}$ Pa
Modulus Precision	+/- 1%	+/- 1%
Tan δ Sensitivity	0.0001	0.0001
Tan δ Resolution	0.00001	0.00001
Frequency Range	$2xE^{-5}$ to 80 Hz	0.01 to 200 Hz
Dynamic Sample Deformation Range	+/- 0.5 to 1,500 µm	+/- 0.5 to 10,000 µm
Temperature Range	-150 to 600ºC	-150 to 600ºC
Heating Rate	0.1 to 60ºC/min	0.1 to 20ºC/min
Cooling Rate	0.1 to 60ºC/min	0.1 to 10ºC/min
Isothermal Stability	+/- 0.1ºC	+/- 0.1ºC
Time/Temperature Superposition	Yes	Yes

Selecting the Correct Test Fixture

- The measured quantity (angular deformation and torque) are transferred into a material quantity (stress, strain, viscosity, etc.)

Measured parameters :		Calculated parameters :	
$\theta(t)$	angular displaceme nt (rad)	$\tau(t) = K_\tau M$	stress (Pa)
$d\theta/dt = \Omega(t)$	angular ve locity (rad/s)	$\gamma(t) = K_\gamma \theta$	strain ( )
$M(t)$	torque (N m)	$\dot{\gamma}(t) = K_\gamma \, d\theta/dt$	strain rate (1/s)
		$\eta(t) = \tau(t)/\dot{\gamma}_o$	viscosity (Pa s)
		$G(t) = \tau(t)/\gamma_o$	modulus (Pa)

K_τ and K_γ relate the measured instrument data with the desired material parameter

Polymers

- Identify the purpose of the experiment
  - *1. Understand why your doing the measurement*
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Know your sample

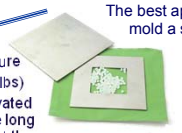
- Polymer samples come in different forms (powder, flakes, pellets) and are sensitive to environmental conditions.
- Careful sample preparation techniques are required to prepare good test specimens for reproducible testing.
  - Molding a sample...
  - Handling powders, flakes...
  - Controling the environment...

Molding Polymers Pellets

Molding temperature:
10 - 20°C > than test temperature

- apply pressure (8 – 12 000 lbs)
- keep at elevated temperature long enough to let the sample relax
- cool down slowly under pressure to avoid orientations

The best approach is to mold a sample plate (100x100 or 50x50mm).



Punch out a sample disk 8, 25 or 50mm

Molding Powders and Flakes

- Before molding at high temperature, the sample has to be compacted cold to reduce the volume.
- The compacted samples are transferred to the mold.
- Follow steps for molding pellets procedure

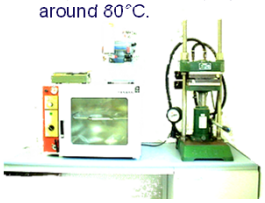
Note: sample may need to be stabilized or dryed to avoid degradation

Preparing Semi Solid Samples

- Cut rectangular sheets of prepreg or adhesive (30x30mm)
- Alternate direction of layer; approx. 5 layer on top of each other (remove release paper from PSA)
- Compress the stag of sample layers in a press (4 – 5000 lbs)
- Punch out 25 mm disks

How to Control the Environment

- PP and Polyolefines in general tend to degrade fast – need to be stabilized by adding antioxidents
- Moisture sensitive materials such as polyamides and polyester require drying in vacuum or at temperatures around 80°C.



- Materials such as PS or PMMA absorb moisture also. In the melt phase, the gas separates in bubbles and the sample foams => pre - drying in vacuum prior to testing is necessary.

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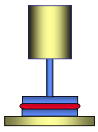
Parallel Plate

- For filled and unfilled polymer systems. This geometry is predominately used for oscillation measurements in the linear-viscoelastic range.



Strain Constant: $K_\gamma = \frac{R}{H}$

Stress Constant: $K_\sigma = \frac{2M}{\pi(R)^3}$

Caution: the **shear rate** varies with the radius!

Parallel Plates

Advantages:

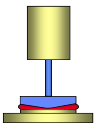
- variable sample thickness (recommended 0.5 to 2mm)
- shear rate range adjustable, changing gap H or plate diameter R
- temperature ramps / sweeps can be carried out without Gap-adjustment
- easy sample loading for high viscosity samples (pre-mold disks or pellets)
- small sample volume
- viscosities: - low to high
- wall slippage problems can be overcome by using serrated, roughened plates

Disadvantages:

- shear rate not constant (can be corrected)
- instabilities in shear field can occur at high rates (liquid shear-field breaks up and liquid is lost due to centrifugal force)

Cone and Plate

- For unfilled polymer melt systems. Primarily to be used in transient and steady test modes, outside of the linear viscoelastic range.

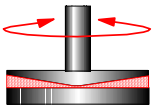


Strain Constant: $K_\gamma = \frac{1}{\beta}$

Stress Constant: $K_\sigma = \frac{3 * M}{2\pi(R)^3}$

Note: the shear rate is constant over the radius.

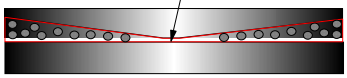
Limitations of the Cone & Plate



Cone & Plate

Truncation Heights:
1 degree ~ 20 - 30 microns
2 degrees ~ 60 microns
4 degrees ~ 120 microns

Truncation Height = Gap



Gap must be > or = 10 [particle size]!!

Cone and Plate

Advantages

- homogeneous shear, shear rate and stress in the gap
- small sample volume (ca. 1ml)
- viscosities: - low to high
- cleaning easy
- ideal for normal force measurements

Disadvantages

- highly sensitive to the relative position (gap) of cone and plate
- gap depends on temperature
- sample loading for high viscosity samples is difficult
- not suitable for dispersions with solid particles
- instabilities in shear field can occur at high rates (liquid shear-field breaks up and liquid is lost due to centrifugal force)

Choosing the Geometry Size

- Assess the approximate viscosity or consistency of your sample.
- When a variety of cones and plates are available, select appropriate diameter
  - geometry
  - Medium viscosity (polyolefines MI>5, polyester,..) - 40 or 50 mm geometry
  - High viscosity (polyolefines MI<5) – 20 or 25 mm
- Examine data in terms of absolute instrument variables [torque/speed/displacement] and modify geometry choice to move test range into the optimum instrument range if necessary.

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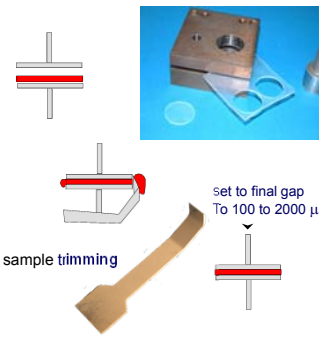
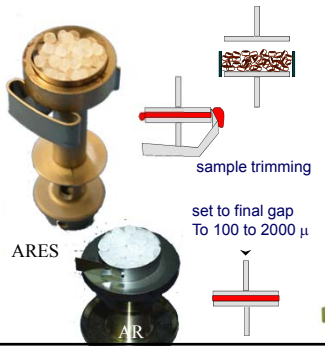
Loading a Molded Disk

- Environmental system to test temperature.
- Load disk onto plate
- Close oven.
- Bring upper plate to gap position.
- Watch Normal force
- Open oven
- Trim sample.
- Close oven.
- Adjust gap

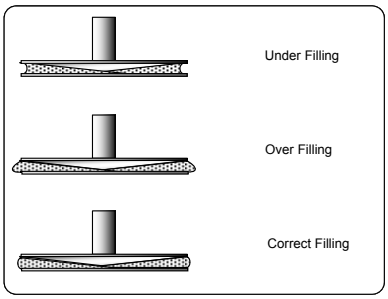


Loading Pellets



- Mount Guard ring and shim. Load pellets.
- Close oven.
- Heat to temperature.
- Compress sample.
- Open oven.
- Remove guard ring.
- Set gap. Watch normal force
- Trim sample.
- Set final gap

Correct filling



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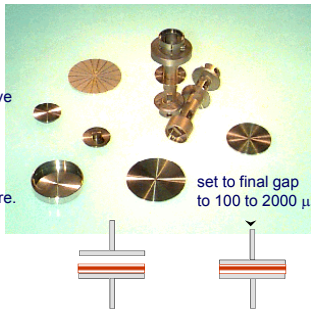
Loading Prepreg and Adhesives

- Mount disposable plate.
- Insert 3-5 layer prepreg or adhesive sample (minimum 0.1mm) between plates.
- Close oven.
- Heat to temperature.
- Set gap. Watch normal force.
- Use normal force control if required.



Normal Force Gap Closing

- Most sensitive as the sample itself determines how fast the gap is closed
- Gap Closure is halted when a normal force > the registered maximum value, and the sample is allowed to relax before closure continues. This avoids overload of the transducer when loading high viscosity melts
- The greater the maximum registered value the faster the close
- Closure may take some time for highly viscous polymers. Loading at higher temperature will speed the process up

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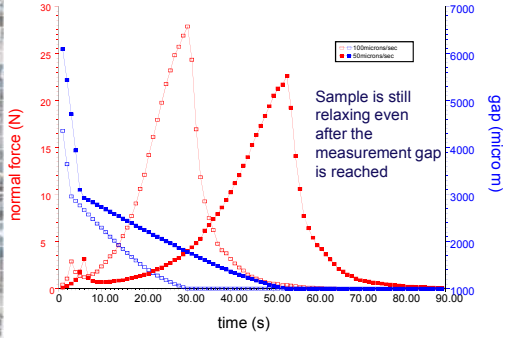
Normal Force During Loading PDMS



Hints for Loading the Sample

- Set trim gap 5% greater then final gap to insure proper filling.
- When using 25 mm cones, pressed disks 0.5 mm thick makes loading faster and reduces excess sample.
- Use force controlled gap setting to avoid overloading the normal force transducers.
- Make sure that torque and normal force have decayed to zero before starting the test.

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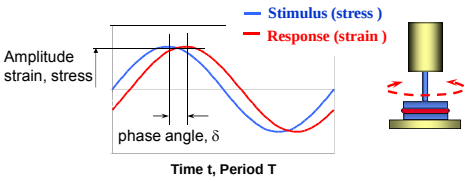
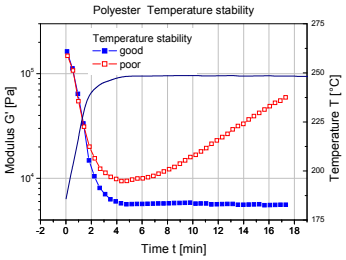
Oscillation Testing Review



Applied: Frequency, strain or stress amplitude
Measured: Phase angle and ratio of strain and stress amplitude
Calculated:

Elastic Modulus: stored energy or elasticity
Viscous Modulus: dissipated energy
Tan δ: damping
Complex Viscosity: resistance to flow

Step #1: Testing Material Stability Time Sweep



Determines if properties are changing over the time of testing
- Degradation
- Molecular weight building
- Crosslinking

Important, but often overlooked!

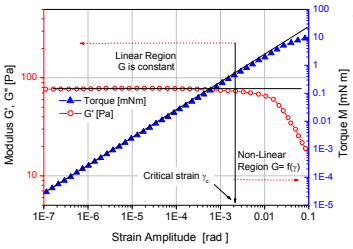
Hints for Time Sweep on Polymers

- Visually observe the material => Use Camera on new ovens
  - Is material degraded?
    - Did color change
      - Use N_2 purge

Testing parameters

- Time: Recommended starting point: 10 minutes
- Temperature: Application Temperature
- Angular Frequency 6.28 to 10 rad/s
- Strain amplitude: 1 – 10%

Testing in the Linear Region

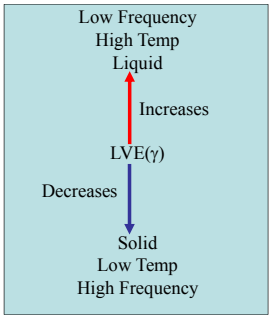


- In the LVR, the dynamic parameters are indepen-dent of strain.
- Test results must be compared in the LVR.

Outside of the LVR the material response is no longer sinusoidal!

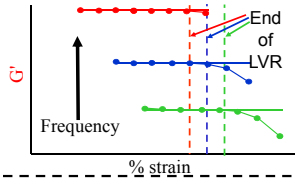
Linear Region Considerations

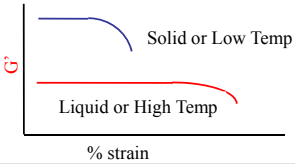
- Need to define linear region accurately.
- The linear region depends on:
  - Frequency
  - Temperature
  - State of sample – solid or liquid



Linear Region Considerations

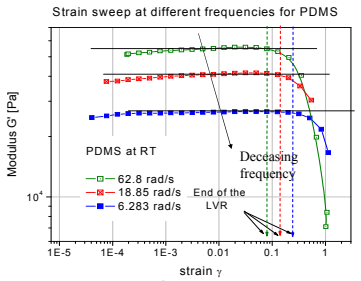
Consider when selecting the strain for the frequency scan





Consider when selecting the strain for temperature scans

Frequency Dependence of LVR



The LVR region decreases with increasing frequency

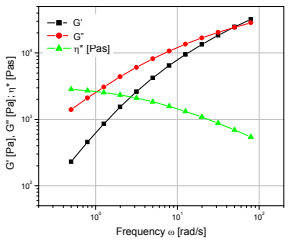
=> Consider when selecting the strain for the frequency scan

Linear region varies from 7 to 25 % strain.
10% strain would be too high for frequency sweep to 100 rad/s

Hints for Testing Linear Region

- Determine linear region only after accessing stability
- Suggested tests and parameters
  - ARES – Run strain sweep: 0.1 to 100%
  - AR – Run stress sweep: 100x's min torque to 2/3 max torque
  - Sweep mode: log 5 points/decade
  - Frequency: 6.28 to 10 rad/s
  - Temperature/s: Depends on subsequent tests
- Run test, evaluate data in spreadsheet, remove any of data outside specifications
- Plot G' vs. Strain
- Use 5% rule to determine linear region

Testing the Frequency Dependence



- Represents the viscoelastic nature of a material in time
- Provides information about the material at different processing or application rates (ω~γ)

The upper frequency is limited by the instrument, the lower frequency is typically 10^{-5} rad/s, a practical limit is 0.1 or 0.01 rad/s

Importance of Frequency Sweeps

- Tells about the material's reaction to short or long excitations (elastic or viscous or both)
- Provides a quantitative measurement of the elasticity (reversible deformation) in a material
- Measurement of Viscosity
  - Zero Shear viscosity a low frequency,
  - Steady viscosity at larger frequencies (Cox-Merz)
  - power law index,…
- The frequency dependence of viscosity and modulus correlates with MW & MWD.

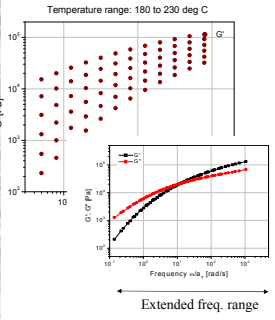
$$\eta_0 \approx M_w^{3.4} \text{ and } J_e = \frac{G'}{(G'')^2} \approx \left(\frac{M_w}{M_z}\right)^{3.4}$$

Hints for Frequency Sweeps on Polymers

Testing parameters

- Determine the LVR first
- Run 100 - 0.1rad/s in log sweep; 5 pts/decade
- Choose Strain has to be in the LVR (determined in a strain sweep)
- Can be run low to high or high to low
  - Low to high
    - No problem for stable sample Problem for sample with limited stability
  - High to low
    - Provides a sizable number of data points in short period of time - test can be aborted when the terminal region is reached – thus reduces test time.
    - Collects more data with samples of limited stability

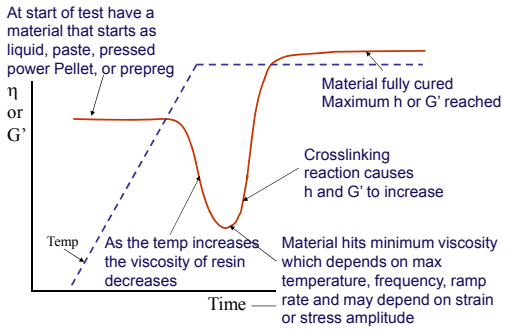
TTS to Extend the Frequency Range



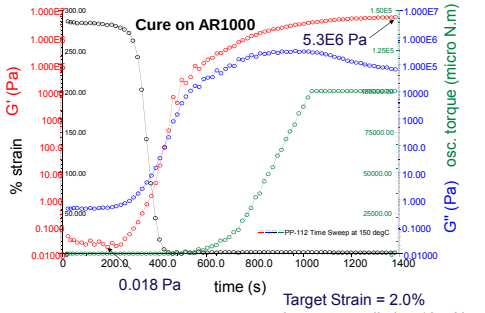
Frequency Sweeps over a range of Temperatures

TTS is an empirical relationship and works only when the material is "thermo-rheologically simple"

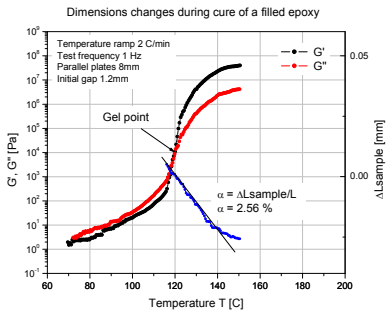
Thermosetting Polymers – Temperature Ramp



Thermosetting Polymers - Isotherm



Dimension Change during Cure



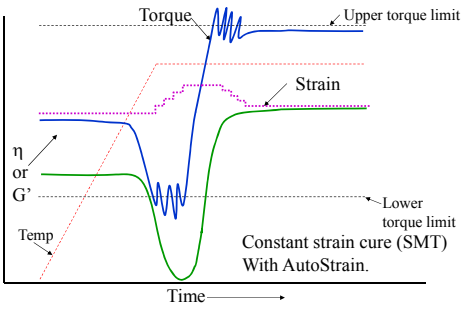
Hints for Temp. Ramp for Thermosets

- Use Normal force control to adjust for shrinkage (AutoTension) and/or thermal expansion in parallel plates
- Use AutoStrain to keep the torque in specified limits for ARES

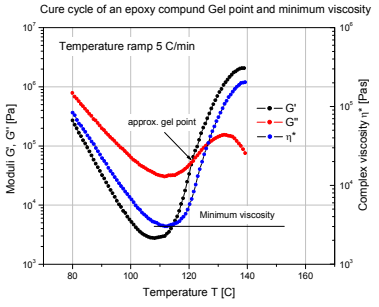
Testing parameters

- Strain
  - Depends on sample consistancy at the start: powder, paste, low viscosity liquid
- Frequency – Typically 6.28 to 10 rad/s or higher with ARES (faster data acquisition)
- Ramp Rate – 1 to 5°C/min.

Thermosetting Polymers - AutoStrain



Thermoset Polymer - Temperature Ramp



AutoStrain increases the strain to keep the torque within the instrument range in order to accurately measure the viscosity minimum

Thermosetting Polymers: Consideration

- Frequency considerations
  - ARES can run at higher frequencies to generate higher torques without risk of inertia issues at near minimum viscosity
  - Higher Frequencies also means faster data acquisition between points for capturing rapid material changes.
  - AR can run into system inertia limitations at minimum viscosity. Monitor raw phase
- AutoStrain considerations
  - AutoStrain only adjusts when torque is above or below limits. Need to get adjustment factor right. 20% adjustment factor works for almost all samples.
  - The AR2000 uses a lower torque limit as opposed to Auto Strain. Strains can get large. Keep an eye on Strain.

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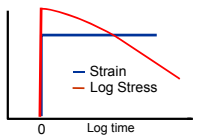
Types of Transient Tests

- Relaxation
  - A step strain is applied and the stress recorded
- Creep and Creep Recovery
  - A step stress is applied and the strain recorded
  - The recoverable strain is recorded after removing the stress

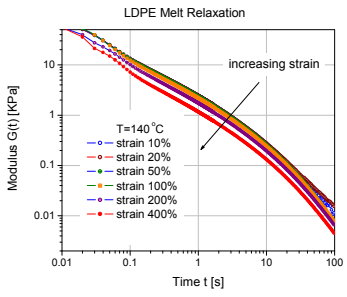
Stress Relaxation Experiment

- AR series: Response time dependant on feedback loop (Strain is applied to sample instantaneously
- Stress is monitored as a function of time $\sigma(t)$.



For small deformations (LVR) the ratio of stress to strain is a function of time only.
The material property is the STRESS RELAXATION MODULUS, G(t)
$G(t) = \sigma(t)/\gamma$

Stress Relaxation and Linear Region



- Fast visco-elastic characteriza-tion of a polymer
- Results less accurate for short and for long times

Stress Relaxation Parameters

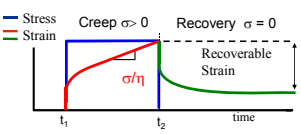
- Strain
  - Small strains (LVR)
    - G(t) is independent of strain
  - Large strains
    - G(t) depends on the strain and decreases with increasing strain.
    - The ratio $G(t)/G_o(t)$ is a measure of non linearity (to calculate the damping function)
- Temperature
  - As application or sample comparison dictates.
  - Temperature steps are seldom used to extend the time range (see freq. sweep)

Hints for Relaxation Tests on Polymers

- Never start with too high strain
  - ARES – transducer overload; motor stopped
  - AR –target strain at full torque is not hit
- Useful to run a single oscillation test at 1 rad/s to estimate the materials' modulus G(t). Estimate the maximum strain for the maximum instrument torque.
- Use logarithmic sampling
- For materials with short relaxation times, oscillation is preferred => torque relaxes too fast below the instrument resolution
- Stress relaxation is used often for stiff samples, which relax much slower.
- Very little used for t-T superposition studies today

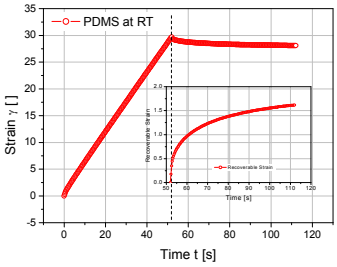
Creep / Recovery Experiment

- ARES Response time dependant on feedback loop and transducer zero
- Stress is applied to sample instantaneously at t_1. The strain is monitored as a function of time □(t).
- The stress is set to zero at t_2. The strain is monitored as a function of time□ γ□(t□).



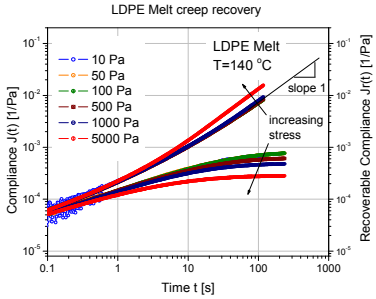
In the creep zone:
COMPLIANCE
$J(t) = \gamma(t)/\sigma$
In the recovery zone:
RECOVERABLE COMPLIANCE
$J_r(t) = \{ \gamma_u - \gamma(t) \} / \sigma$

Creep on PDMS



- The best test approach to measure long relaxation (retardation times)
- Recovery is the most sensitive parameter to measure elasticity

Creep and Recovery with Increasing Stress



Non linear effects can be detected in recovery before they are seen in the creep (viscosity dominates)

Creep Parameters

- Stress in creep zone
  - In linear viscoelastic region J(t) will be independent of the applied stress in LVR
  - Select the highest stress in the LVR.
- Recovery Zone (Stress zero by definition)
  - Start recovery only, after steady state creep has been obtained.
  - Select a recovery time, twice the creep time
  - The recoverable compliance J_e is a very sensitive measure of the melt elasticity
- Temperature(s)
  - As application or sample comparison dictates

Creep – ARES and AR series

- AR series
  - Drag cup motor: Steps torque to programmed stress and holds constant with time.
  - Optical Encoder: Records displacement with time, during creep and recovery.
  - Limitation: torque resolution during creep and residual torque during recovery.
- ARES series (LS Motor Only)
  - Motor/Transducer Feedback control: Motor drives angular velocity to control feedback from transducer to constant torque (stress).
  - Encoder measures motor strain and strain rate Limitation:
- Limitation
  - Requires initial viscosity value to back calculate a starting shear rate to initiate feedback loop.
  - Offset between sample torque and transducer zero.

Hints for Creep Tests on Polymers

- Use only AR series rheometer for recovery measurements on polymer melts
- Operate always at the highest stress in the LVR
- Recovery time zone must at least be twice the creep time zone.
- Do not exceed one strain unit during creep (End of LVR).

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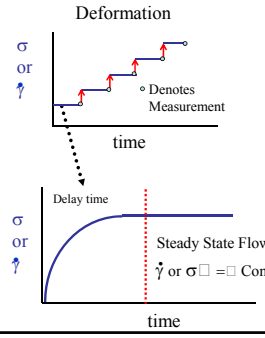
Steady State Flow on Polymer Melts

- In polymer rheology, the steady state viscosity as a function of shear rate is the most used material function – referred to as viscosity function
- The steady state stress as a function of shear rate is referred to as flow curve
- Rotational rheometers can only measure the steady state viscosity and 1st normal stress coefficient at low rates (<10 1/s) due to edge failures. Higher shear rates may be obtained from oscillation measurements using Cox Merz.

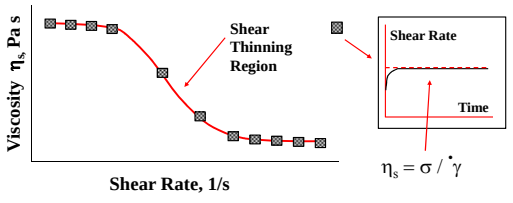
Steady State Flow Experiment



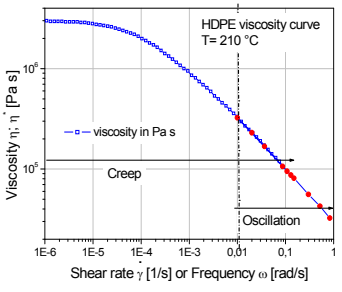
Stress or rate is applied to the sample. Viscosity measurement is taken when the material has reached steady state flow. The stress or rate is increased (logarithmically) and the process is repeated yielding a viscosity or flow curve .

Steady State Flow Experiment

Whether steady state has been reached, by applying stress or rate, has no influence on the final result. In the polymer industry, the viscosity function is always shown as a function of the shear rate; each steady viscosity value giving a single viscosity data point:



Steady State Flow

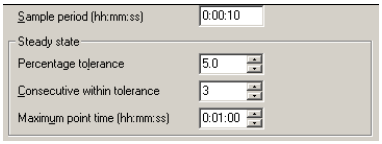


- With stress controlled AR, the viscosity can easily be measured down and below 10^{-6} 1/s
- ARES with LS motor can **control** rates down to 10^{-6} 1/s

Rate Controlled Steady-State Flow Parameters

- Controlled Rate
  - Initial and final shear rate
    - Start at lowest settable rate
    - Suggest final value – 100 to 1000 1/s
      - Use your judgment
  - Step time
    - Stepped Flow
      - 10 seconds
    - Steady State Flow
      - AR: use algorithm (see slide on topic)
      - ARES: use guideline (see slide on topic)
  - Temperature
    - As defined by application

AR: Steady State Algorithm Flow

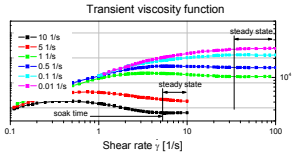


Default values shown

- During the test, the dependant variable (speed in controlled stress mode or torque in controlled shear rate mode) is monitored with time to determine when stability has been reached.
- An average value for the dependant variable is recorded over the **Sample period**.
- When consecutive average values **(Consecutive within tolerance)** are within the **tolerance** specified here, the data is accepted.
- The software will also accept the point at the end of the **Maximum point time**, should the data still not be at a steady state value.

Hints for Steady State Flow on ARES

- Defining time required to reach equilibrium:
  - Run 'Single Point Test' at lowest shear rate to determine 'delay before measure' for steady rate sweep. Use this equilibrium time as 'delay before measurement'.
  - Higher shear rates will require less time to reach equilibrium, ensuring all data acquired was collected under steady state conditions
- May sequence tests to shorten experiment time



Stress Controlled Steady-State Flow Parameters

- Controlled stress
  - Initial and final stress
    - Suggest: Control Torque- use 10 times lowest settable torque
    - Suggest final value – use maximum
      - Likely to get instrument over-speed message
        - Can throw sample out; Use covers
  - Step time
    - Stepped Flow
      - 10 seconds
    - Steady State Flow
      - Use algorithm (see slide on topic)
  - Temperature
    - As defined by application

Stress Controlled Steady State Flow Hints

- No guarantee that the sample reaches steady state at maximum point time
- Can run in stress control enabling stress and flow information in one test
- Easier to controlling shear stress can measure much lower shear rates than controlling rate in CMT or SMT with HR motor

Structured Fluids

- Identify the purpose of the experiment
  - *1. Understand why your doing the measurement*
- Design, program & run test
  - *2. Understand your instrument*
  - *3. Know your sample*
  - *4. Selecting correct test fixtures*
  - *5. Oscillation testing guidelines*
  - *6. Transient testing guidelines*
  - *7. Steady shear testing guidelines*
  - *8. Loading the sample*
- Evaluate data and 0ptimize if necessary
  - *9. Instrument limitations*
- Results
  - *10. Presenting the data*

Ten Steps to Better Rheological Measurements

1. Understand why you are doing the measurement.
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3. **Know your sample.**
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Know Your Sample

- Structured fluids can range in consistency from low viscosity such as milk to pasty like tooth paste. Structured fluids are very sensitive to mechanical and environmental conditions. Correct pre-conditioning and loading into the rheometer is important.
- Structured fluids are time-dependent – how you treat the sample may affect the test results!

Handling Low Viscosity Fluids

- Fluid samples which pour freely are relatively easy to handle prior to loading:
  - Keep the flask closed to avoid evaporation of solvent or the continuous phase
  - Shake or stir may be necessary to concentration gradients in suspensions
  - Adequate shelf temperature may be required to avoid phase separation in emulsions
- Never return used sample into original flask to avoid contamination

Dealing with Pastes and Slurries

- High viscosity pastes and slurries may suffer structure changes with time.
- Food samples, for example Dough, changes continuously. The test samples need to be prepared very carefully and consistently for each experiment in order obtain reproducibility
- Slurries that may settle build a cake. These samples have to be tested before sedimentation.

Dealing with Gels

- Gels, especially chemical gels, may change irreversibly when applying large deformations.
  - Prepare (formulate) the sample and bring it into the final shape required for the measurement without deforming the sample (cut, punch,..)
  - Prepare (formulate) the sample in situ (test fixture, rheometer,.=> systemic rheology)

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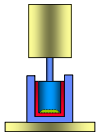
Selecting the Correct Geometry

- Low viscosity fluids are best contained in a cup and bob geometry, pastes are better loaded into parallel plate or cone plate geometry, you may say – Is this always correct?
  - A typical bob (30x30 mm) generates the same torque as a 50mm parallel plate – no advantage!
  - Low viscosity fluid stays in a 500 micron gap due to capillary forces and requires much less fluid – this is an advantage!
  - Evaporation is easier to eliminate in a concentric cylinder system with a solvent layer.
  - In general high viscosity systems are difficult to load in a cup and bob.

Concentric Cylinder

- Use with low viscosity fluids and systems with volatile components. Use low viscosity oil or the volatile component itself on top of the sample to avoid evaporation. Requires a large sample volume.



$$\text{Strain Constant: } K\gamma = \frac{2}{1-(R1/R2)2}$$

$$\text{Stress Constant: } K_\sigma = \frac{1}{2\pi L(R_1)^2}$$

Note: the average shear rate is represented by the value in the middle of the gap

Concentric Cylinders

Advantages:

- good for low viscosity fluids easy loading
- allows high shear rates (>10^3 s-1) without losing liquid due to centrifugal force
- Sample evaporation is easy to eliminate with a solvent layer

Disadvantages:

- shear rate constant for small gaps only
- instabilities in shear field can occur at high rate (homogeneous liquid shear-field breaks up, wall-slip)
- large sample volume (>20ml)

Special Cylinder Geometries

- Vane is ideal for avoiding slippage, sample breaking during loading, etc..
- Starch rotor and Helical screw are ideal for mixing and avoiding sedimentation, etc..

Avoiding Slip with Plate Geometries

- Different surface treatments are used to avoid slipping of gels, suspensions, etc..
  - Crosshatched surface
  - Serrated surface
  - Sand blasted or roughened surface
  - Porous surface

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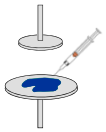
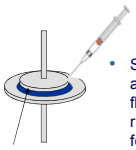
Loading Low Viscosity Fluids

Check volume requirements for testing geometry

- Deposit fluid in the middle of the plate, rotate plate while setting the gap to insure even filling

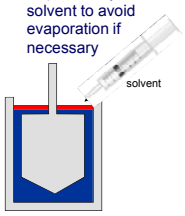


- Set gap first and deposit fluid at the rim. Capillary forces suck in the fluid

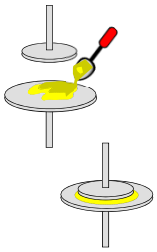


Meniscus due to surface tension

- Fill cup, lower the bob to the mark. Deposit a layer of solvent to avoid evaporation if necessary



Loading Pastes and Slurries



Pastes and slurries

- Deposit the paste with a spoon (syringe with cut-off tip for less viscous systems) in the middle of the plate.
- Load 10-20% excess material to insure complete filling
- Close the gap automatically using logarithmic speed or force gap setting to minimize shear in the sample
- Trim sample and set final gap

Closing the Gap

- Linear speed profile after reaching the user defined 'Compression Distance'
- Exponential speed profile after reaching the user defined 'Compression Distance'
- Normal Force set not to exceed a certain value of after reaching the user defined 'Compression Distance'

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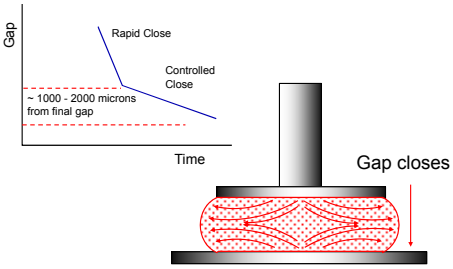
Linear Gap Closing



Effect of squeeze is to shear sample

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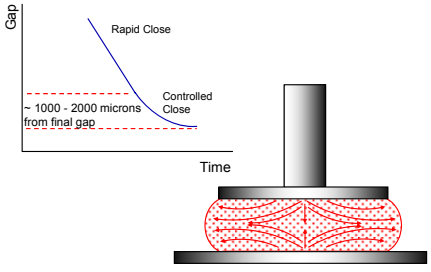
Exponential Gap Closing



Squeeze Flow effect is less pronounced

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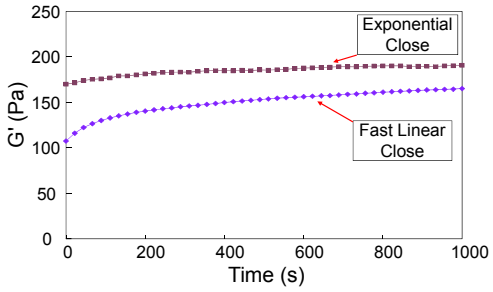
Comparison of Linear and Exponential Gap Closing



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Loading Gels

- Prepare gel sample with the desired shape
- Use cross-hatched, roughened plates
- Form the gel between the plates

- Insert vane without breaking the sample (yoghourt, sour cream)

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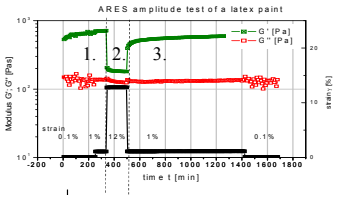
Structured Fluid: Pre-Testing



- Select low strain high enough to generate a good signal, typical > 0.1%. The high strain should be 10 to 100 times higher than the low strain
- Switch strain manually (ARES) or go to next step (AR2000) when equilibrium has been reached.

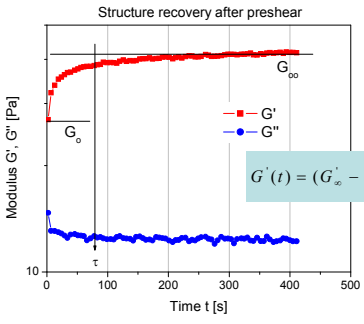
Structured Fluids: Results of the Pre-Testing

- **The pre-test may indicate the following:**
  - Effect loading observed by the change of G' in section 1
  - Possible yield in the material observed by a significant drop in G' in the low and high strain section.
  - Possible thixotropy observed by the time require to obtain equilibrium in section 3
  - Reversibility of material structure observed by the difference between equilibrium in section 1 and 3

Need for Pre-Shearing

- Important, but often overlooked
- If pre-testing indicates irreversible structure, pre-shear and delay will be required to establish a known history to insure reproducible test results
- Simulation shear processes in applications

Testing Time Dependence -Time Sweep



$$G'(t) = (G'_{\infty} - G'_{0})(1 - \exp\left(\frac{t}{\tau}\right))$$

τ is a characteristic restructuring time

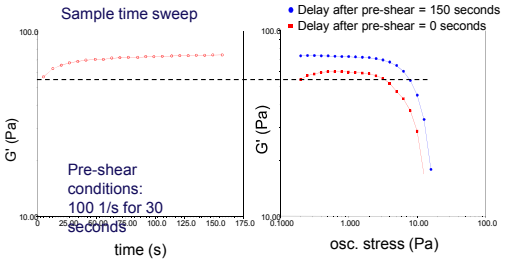
Hints for Time Sweep on AR & ARES Series

- Load new sample, pre-shear to breaking structure (time from section 2 during the pre-test) and follow structure building at low amplitude (section 3of the pre-test) at 1 Hz i.e. 1rad/s
  - Typically, the restructuring curve can be approximated by an exponential function. In this case a characteristic restructuring time can be calculated
  - Sometimes the structure recovery is more complex f.example a fast initial recovery to 90% of G_{oo} and very slow recovery of the rest 10%

Suggestions for Strain or Stress Sweep Testing

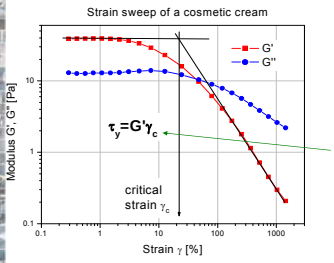
- Dispersions
  - Is structure broken during loading ?
    - May need to pre-shear and wait for structure to build.
  - If the above condition exists:
    - Pre-shear at 10 1/sec for 30 sec
    - Run time sweep to see when sample is stable, then run strain or stress sweep
  - When comparing samples, condition all with the same pre-shear history.

Importance of Waiting for Structure Rebuild



- End of LVR is indicative of "Yield" or "Strength of Structure"
- Useful for Stability predictions (stability as defined by yield)

Testing in the Linear Region - Strain Sweep

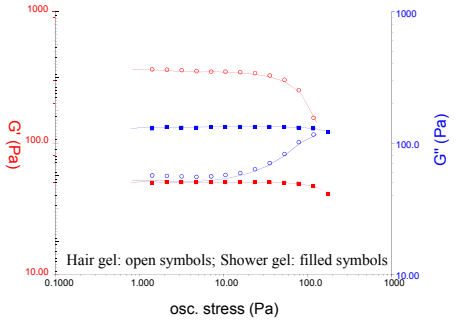


Structured sample

Estimate the yield stress from the on-set of linear behavior

If the material has shown significant thixotropy, the next test should be a "dynamic time sweep" after pre-shearing at the typical application shear rate

Testing in the Linear Region - Stress Sweep



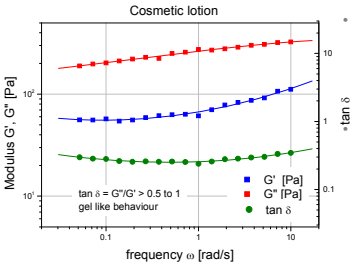
Hints for Strain Sweep: ARES & AR

- Pre-shear and delay determined from time sweep
- Strain Range
  - Initial strain: Lowest settable value
  - Final Strain: Dispersion: 10% strain
- Frequency: 6.28 rad/s
- Sweep Mode
  - Logarithmic for samples with linear regions with wide linear region;10 pts/decade
  - Linear mode for highly structured materials with short linear regions, i.e. toothpaste

Hints for Stress Sweep : AR

- Pre-shear and delay determined from time sweep
- Oscillation Stress Range:
  - Recommend to program Oscillation Torque
  - Initial Torque: 10 times minimum torque
  - Final Torque: 1/2 maximum torque
- Frequency: 6.28 rad/s
  - Sweep Mode
    - Logarithmic for samples with wide linear regions; 10 pts/decade
    - Linear mode for highly structured materials with short linear regions, i.e. toothpaste

Testing the Frequency Dependence



- G' and G" are virtually independent of frequency, as well as tan d.
- Also the material behaves predominately elastic (G'>G") => which stands for structure in the material, capable of storing energy

Hints for Frequency Sweeps

- Determine the LVR first
- Pre-shear and delay determined from time sweep
- Run 100 - 0.1rad/s in log sweep; 5 pts/decade
- Strain has to be below the critical strain τ_c, determined in the strain sweep

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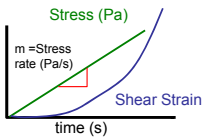
Transient Tests for Structured Fluids

- Transient test measures the instantaneous response of a material as a function of time
- Stress ramp
  - – this is a very common method to determine the yield stress
- Rate ramp
  - The rate on the sample is continuously increased and the stress recorded. The rate ramp is a fast method to measure a flow curve
- Thixotropic loop
  - A rate ramp up is followed by a down ramp. The difference between the flow curve during the up and down segment relates to thixotropy

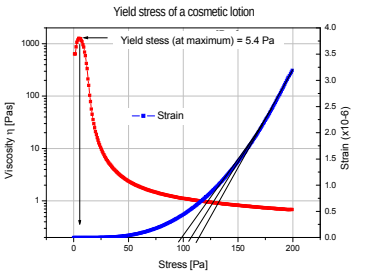
Continuous stress ramp

- A Stress changing at a constant rate is applied to material
- Resultant strain is monitored with time.



The stress ramp is predominately used to measure the yield stress. The parameters evaluated are either the measured strain or the instantaneous viscosity

Yield Stress in a Stress Ramp



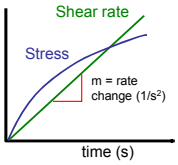
The maximum viscosity method is more representative and reproducible then the strain tangent method

Continuous Ramp Testing Parameters

- Controlled stress (AR series)
  - Initial and final stress
    - Can start at zero
    - Suggest final value –Use your judgment
  - Duration or Time of experiment
    - Typically 1 to 3 min.
    - Shorter duration can affect results (inertia effect or thixotropy)
  - Temperature
    - As defined by application
- Controlled Stress (ARES series)
  - Requires guess of viscosity
  - Test parameter similar AR

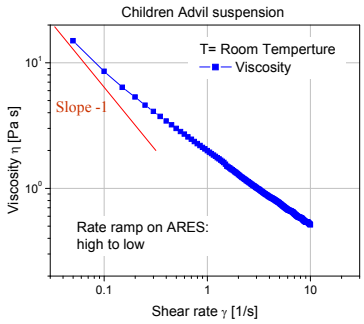
Continuous Rate Ramp

- A constant changing shear rate is applied to the sample
- The resultant stress is recorded



Rate ramps are used mostly as a fast method to measure the flow curve or the shear rate dependent viscosity of non thixotropic fluids

Viscosity Function From a Rate Ramp

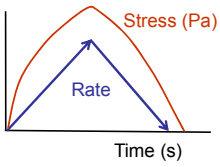


Continuous Ramp Testing Parameters

- Controlled rate (ARES series)
  - Initial and final rate
    - Can start at zero
    - Suggest final value –Use your judgment
  - Duration or Time of experiment
    - Typically 1 to 3 min.
    - Shorter duration can affect results (thixotropy)
  - Temperature
    - As defined by application
- Controlled Rate (AR series)
  - Uses feedback control
  - Test parameter similar ARES

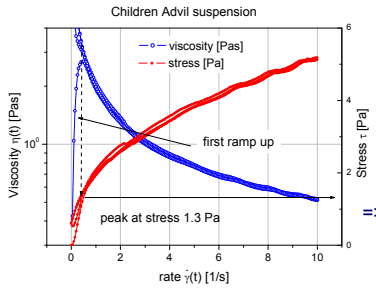
Thixotropy Loop Test

- Constant increasing rate is applied to material.
- Resultant stress is monitored with time.
- (Combination of up and down stress ramps is a pseudo-thixotropic loop)



The thixotropic loop was the traditional method frequently used with viscometers to measure thixotropy.

Thixotropic Loop

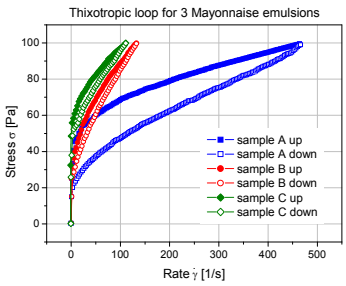


Non-thixotropic material

Up and down ramps superpose – except the first one

=> Start up from zero

Thixotropic Loop



Thixotropic material

Up and down ramps do not superpose

Area under the curve is a measure of thixotropy

Stress and Rate Ramps - Comments

- Why is a stress ramp preferred for yield measurements? What is the difference?

=> small change in stress causes a large change in strain rate
=> it is easier to control small stresses than small rates

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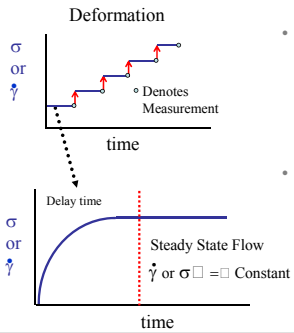
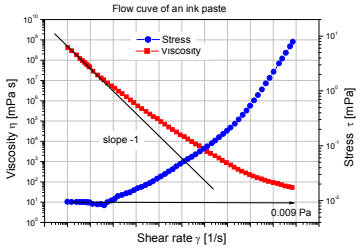
Steady State Experiment



- Whether steady state has been reached, by applying stress or rate, has no influence on the final result.
- In order to obtain a continuous curve on structured materials, however it matters how the viscosity function is obtained.

Structured Fluid: Flow Curve in Rate Mode



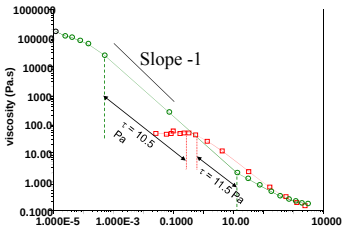
Stepped rate test on ARES

Shear rates are stepped at equal intervals

=> smooth curve

The viscosity decreases with a slope of -1 versus strain rate and the stress becomes rate independent => material with yield stress

Structured Fluid: Flow Curve in Stress Mode



Stepped stress test on AR2000

Stress is stepped at equal intervals, the data are represented as a function of rate

=> discontinuous curve curve

Flow Curve in Stress Mode - Comments

- At the yield point, the rate can vary over a few decades for a small increment in stress.
  - If the stress is stepped up or down, at equal intervals, the rate dependence will show a discontinuity in the viscosity curve

=> in this case you have to control on strain rate in order to obtain a continuous flow curve or viscosity curve.

Solids

- Identify the purpose of the experiment
  - *1. Understand why your doing the measurement*
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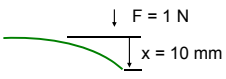
Sample Considerations

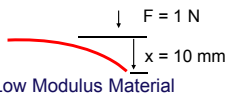
- **Deformation Mode:**
  - E : Tension, compression and bending modes - DMA
  - G: Torsion rectangular shear mode - Rheometer
- **Stiffness:**
  Stiffness of sample related to its dimensions [l,w,t].
  Stiffness may limit sample size.
- **Shape:**
  Accurate/reproducible modulus depends on good sample shape [consistent cross-section throughout length.
  Sample size cannot exceed clamp restrictions [Stiffness takes precedence].

Sample Stiffness and Material Modulus

Thick and Thin Samples Can Have The ***Same Stiffness***

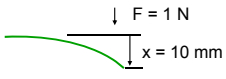


High Modulus Material

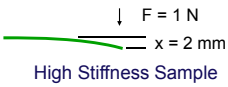


Low Modulus Material

Thick and Thin Samples That Have The ***Same Modulus***



Low Stiffness Sample



High Stiffness Sample

Changing Sample Stiffness

Clamp Type	To Increase Stiffness……	To Decrease Stiffness…….
Tension Film	Decrease length or increase width. If possible increase thickness.	Increase length or decrease width. If possible decrease thickness.
Tension Fiber	Decrease length or increase diameter if possible.	Increase length or decrease diameter if possible.
Dual/Single Cantilever	Decrease length or increase width. If possible increase thickness. Note: L/T≥ 10	Increase length or decrease width,, If possible decrease thickness. Note: L/T≥ 10
Three Point Bending	Decrease length or increase width. If possible increase thickness.	Increase length or decrease width. If possible decrease thickness.
Compression– circular sample	Decrease thickness or Increase diameter.	Increase thickness ø decrease diameter.
Shear Sandwich	Decrease thickness or Increase length and width.	Increase thickness or Decrease length and width.
Torsion	Increase thickness Decrease length	Decrease thickness Increase length

Preparation of Solid Test Specimen

- Uniform and well-defined dimensions
- Shape could be fiber, film or rod/rectangular bar
- If sample is received as pellets, mold sheet first and cut specimen of the desired size for torsion or bending experiments

- Rubber can be tested in bending or torsion (rectangular sample) or in shear (disc)
- Compression molded samples are preferred over injection molded samples because of the highly oriented nature of injection molding. Orientation does not affect Tg but tan delta before Tg.

Preparation of Solid Thin Films

- Curling of films
  - Electrostatic effects on thin films can cause static attractions that make manipulation frustrating
  - Small residual stresses in the film from processing can cause curling
- Thin brittle films are subject to cracking
  - Cutting with razor blade, not with scissor
  - Be careful while trying to flatten for loading

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DMA Clamping Guide

Sample	Clamp	Sample Dimensions
High modulus metals or composites	3-point Bend Dual/single Cant. Torsion	L/T> 10 if possible
Unreinforced thermoplastics or thermosets	Single Cantilever, Torsion	L/T >10 if possible
Brittle solid (ceramics)	3-point Bend, Dual Cant. Torsion	L/T>10 if possible
Elastomers	Dual Cantilever Single Cantilever Shear Sandwich Tension	L/T>20 for T<Tg L/T>10 for T<Tg (only for T> Tg) T<2 mm W<5 mm
Films/Fibers	Tension	L 10-20 mm T<2 mm
Supported Systems	8 mm Dual Cantilever	minimize sample, put foil on clamps

Solids Clamping Selection – Rigid Samples

- Rigid (Glassy, Crystalline) Samples in Bending
  - Rectangular or cylindrical sample desired: L/T > 10 necessary for quantitative Young's modulus, otherwise mixed mode of deformation and modulus will be low.
  - Use Dual/Single Cantilever or Three Point Bend Clamps
  - ASTM Forms - smooth edges for better gripping of samples
  - Unusual shapes - cut into rectangular bars or run as formed for qualitative information
- Rigid Samples in Torsion
  - Rectangular or cylindrical samples desired.
  - Samples up to 5 mm can be tested in torsion.
- Films and Fibers
  - Use Tension Film or Tension Fiber clamps
  - Short span cantilever may work well for stiff films

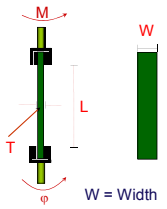
Solids Clamp Selection - Elastomers

- For sub -T_g information use dual/single cantilever clamp or Torsion
- To characterize through T_g and above, use single cantilever or Torsion. If sample is sufficiently thin [low stiffness] then tension can be used.
- Shear sandwich clamp or parallel plate
  - Can be used to characterize material above T_g
  - Can be used for curing (Vulcanizing) a rubber

Clamp Selection – Coatings and Resins

- Coatings
  - Only qualitative data from supported samples
  - Transitions of coatings on substrates are best observed in bending
- Resins
  - Use fiberglass braid, metal mesh or metal shims as support for cure characterization in dual/single cantilever
  - Use 8 mm dual cantilever clamps for low stiffness
  - Use parallel plates for rotational rheometers

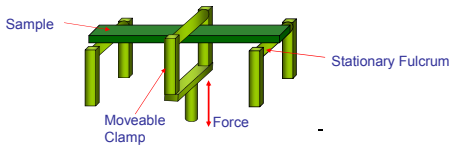
Torsion Rectangular



W = Width
L = Length
T = Thickness

- high modulus samples
- small temperature gradient
- simple to prepare
- Not suitable for films and fibers!

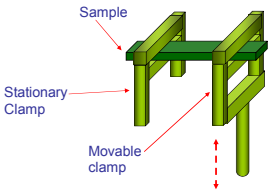
3-Point Bending



- Best mode for measuring medium to high modulus materials
- Conforms with ASTM standard test method for bending
- Purest deformation mode since clamping effects are eliminated

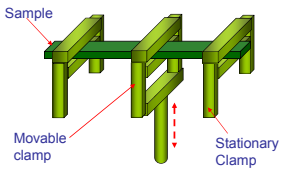
Single Cantilever

- Best general purpose mode (thermoplastics)



- Preferred mode over dual cantilever for most neat thermoplastics (un-reinforced) except elastomers
- Clamping torque is important

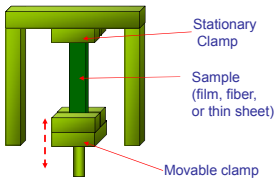
Dual Cantilever



- Highly damped materials can be measured
- Best mode for evaluating the cure of supported materials

Tensile

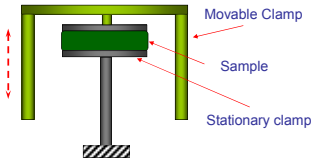
- Best mode for evaluation of thin films and fibers (bundle or single filaments)
- Small samples of high modulus materials can be measured



- TMA constant force & force ramp measurements (mini-tensile tester)
- Force Track & constant force control

Compression

- Good mode for low to medium modulus materials (gels, elasto-mers)



- Materials must provide restoring force (support necessary static load)
- Options for expansion & penetration measurements

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Before Loading the Sample

- Prepare homogenous sample of constant thickness and width
- Make sure the test specimen has no air bubbles enclosed or cracks
- Make precise measurement of the sample dimensions

Sample Dimension Errors

- When measurements on samples are inexact, the moduli values are affected.
- Specimen with uneven surfaces or geometries will not give consistent moduli.
- Bear in mind that moduli are usually plotted in log form, a slight change visually on the graph is a large change in data.
- Transition temperatures and tan δ will not be affected by dimension errors .

Avoid Clamping Effects

- Always use the same **clamping torque** for a series of samples –
  - even if stress patterns affect absolute results, the run to run variations will be minimal
  - clamp the sample **symmetrical**
- Do not apply **too much torque** on softer samples, no squeezing
  - **re-clamping** may be necessary in the glassy state (thermal contraction)

Loading Films and Fibers - Comments

- Films and fibers are often more fragile requiring a gentle treatment in loading and testing
- Production often involves tremendous stresses on the material: drawing, orienting, annealing, extruding. Internal stress causes rapid changes during and after T_g, measuring system must compensate rapidly
- Uneven thickness are more problematic since aspect ratio is extreme
- The Deformation for films and fibers is in tensile.
  - Sample length is more variable than bending modes: clamp geometry doesn't interfere with size. Normal force control keeps sample stretched.

Alignment in Tension Mode

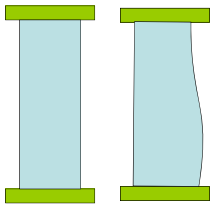
ideal inclined sagging variable thickness

Using Multiple Fibers

- In many cases, the testing needs to use bundled fibers to represent real life
- Evenly distribute or wind the bundles
- Avoid kinking that would make some fibers longer than others
- Approximate sample dimensions and do not use absolute modulus values: run to run variations may be significant

Sample Buckling

- Buckling during loading causes serious errors as buckled areas do not "feel" the force or deformation
- Buckling can be the result of non-uniform stretching, or crooked loading of a film.
- Observe film from edge while oscillating to verify goodness of load.
- If sample is buckling, reload a new sample.



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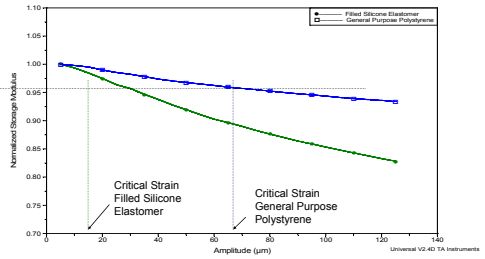
Procedure to Run a Solid Sample

1. Load sample at room temperature
2. Change temperature to the desired starting point with Normal force control active (automatically adjust gap to compensate sample and fixture's thermal expansion or contraction)
3. Run Strain Sweep or generate a stress-strain curve to confirm the linear region
4. Run desired test modes like frequency sweep or temperature step/ramp

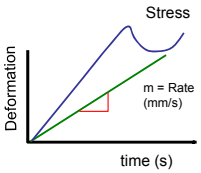
Strain Sweep and Linear Region

In bending using dual cantilever



Constant Deformation

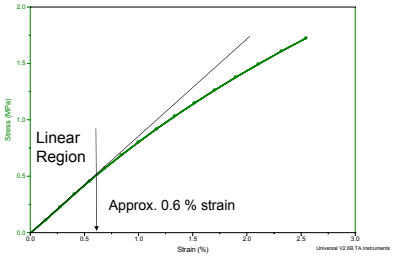
- A deformation changing at a constant rate is applied to material
- Resultant stress is monitored with time.



The constant deformation is predominately used to measure the linear deformation range of materials. The parameters evaluated are the measured stress (force) vs. strain (displacement).

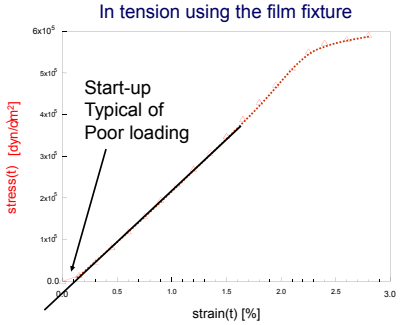
Stress-Strain Curve - for Linear Region

In tension using the film fixture



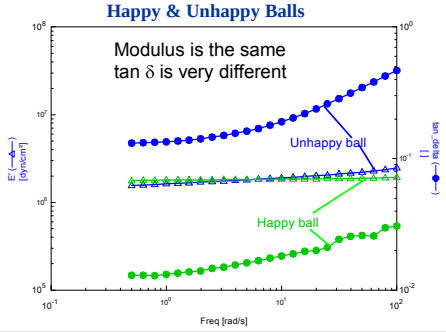
Stress/Strain Curves - for Linear Regions

In tension using the film fixture



Frequency Sweeps on Solids

Happy & Unhappy Balls

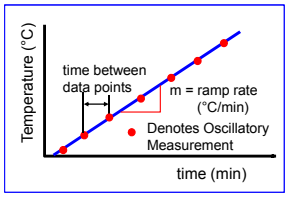


Hints for Frequency Sweep on Solids

Testing parameters

- Determine the LVR first
- Run 100 - 0.1rad/s in log sweep; 5 pts/decade
- Choose Strain has to be in the LVR (determined in a strain sweep)
- Use appropriate Force Track/AutoTension to avoid sample buckling if appropriate

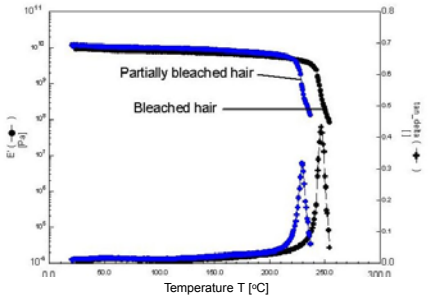
Dynamic Temperature Ramp



A linear heating rate is applied. The material response is monitored at a constant frequency and constant amplitude of deformation.

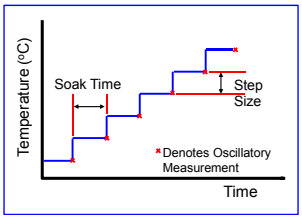
Data is taken at user defined time intervals.

Bundle of Hair in a Temperature Ramp



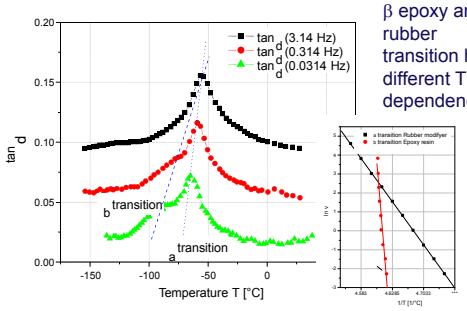
Temperature Step & Multi Frequency

A step and hold temperature profile is applied. The material response is monitored at one, or over a range of frequencies, at constant amplitude of deformation.

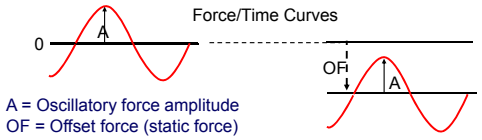


Frequency Dependence of Modified Epoxy

β epoxy and rubber transition have different T dependence



Hints for Normal Force Control



A = Oscillatory force amplitude
OF = Offset force (static force)

Clamps without offset force:	Clamps with offset force:
Single Cantilever	Tension Film
Dual Cantilever	Tension: Fiber
Shear Sandwich	3-Point Bend
	Compression
	Penetration

Force Offset Modes

Static force [General]

Used in tensioning clamps to prevent sample buckling [tension], or loss of contact with the probe [compression] during the test. Static force must exceed dynamic force at all times during experiment

- Constant Force
  - Applies same static force throughout experiment
  - Can be used with highly crystalline or crosslinked materials to measure displacement for quantitative expansion
- Force Track
  - Applies Static Force in Proportion to Sample Modulus. Used to reduce stretching as specimen weakens
  - Values from 125-150% work well for most samples

Normal Force Control - Parameters

Clamp Type	Static Force	Force Track (autostrain)
Tension Film	0.01 N	120 to 150%
Tension Fiber	0.001 N	120%
Compression	0.001 to 0.01 N	125%
Three Point Bending Thermoplastic Sample	1 N	125 to 150%
Three Point Bending Stiff Thermoset Sample	1 N	150 to 200% Can use constant static force

Normal Force Control - Parameters

RSA III Auxiliary Control Program Parameters

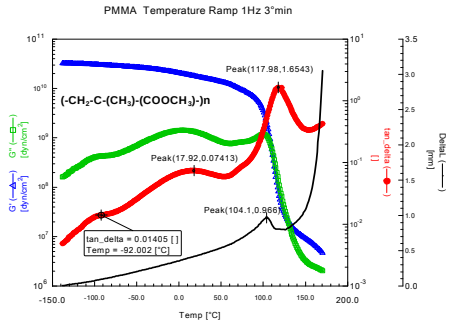
AutoTension
1.Initial Static Force:_____
2.Static F >Dyn. Force: _____
3.Min. Static Force:_____
4.AutoTension Sensitivity:_____

AutoStrain
5.Inc. strain if force drops below:_____
6.Dec. strain if force goes above:_____
7.Adjustment Factor:_____

Q800 Program Parameters

1. Initial Static Force:_____
2. ForceTrack: _____
3. Minimum dynamic Force:_____

Dimension Changes During Temperature Ramps



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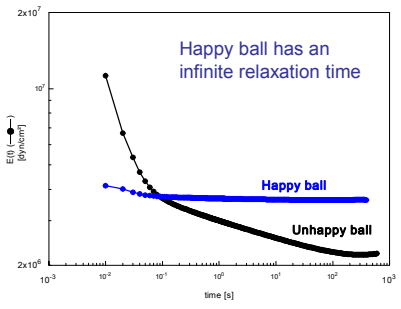
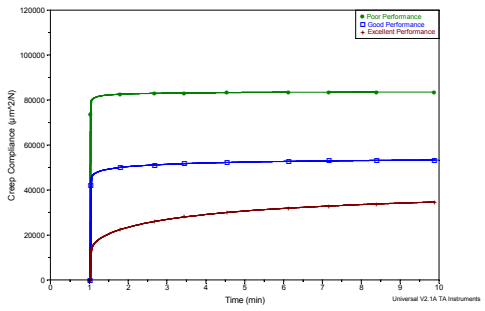
Transient and Steady Testing on a DMA

- More than 90% of all solids testing on a DMA is in oscillation
- Solids analyzer also perform time dependent or steady measurements on solid materials; for example:
  - Creep on solid fibers
  - Long term relaxation studies on solid specimen
  - Tack test

Stress Relaxation Happy & Unhappy Balls



Creep on Packaging Films



Flow of a Rheological Experiment

- Identify the purpose of the experiment
  - *1. Understand why your doing the measurement*
- Design, program & run test
  - *2. Understand your instrument*
  - *3. Know your sample*
  - *4. Selecting correct test fixtures*
  - *5. Oscillation testing guidelines*
  - *6. Transient testing guidelines*
  - *7. Steady shear testing guidelines*
  - *8. Loading the sample*
- Evaluate data and 0ptimize if necessary
  - *9. Instrument limitations*
- Results
  - *10. Presenting the data*

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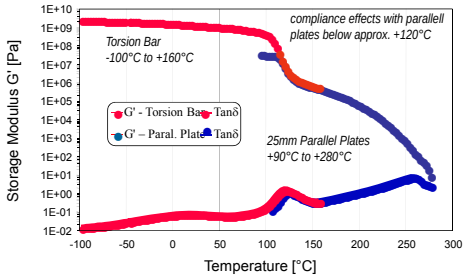
Instrument Limitations

The following are important key issues that may limit the range of operation. It is critical that these items are understood:

- Instrument Compliance which can affect data when the sample becomes much stiffer than the instrument.
  - More of a concern in dual head design
- Secondary flows or edge effects can limit the high strain and strain rates.
- Instrument Inertia may dominate measurements of low stiffness/low viscosity materials. This limits the maximum frequency in single head rheometers and DMA's.

Radial Compliance

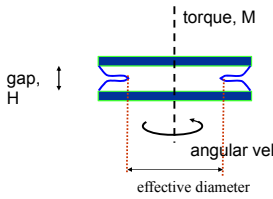


- All TA Instruments include compliance calibrations/corrections.

Instrument Compliance

- How do you see and compensate for compliance in dual head instrument
  - Look at actual and commanded strain in the spreadsheet.
  - If actual strain is < 80% of the commanded strain, then sample geometry should be changed to reduce stiffness of sample
- Refer to stiffness chart above.

Edge Failure – Secondary Flows



Large normal stress difference leads to crack formation at free surface

- Results in apparent drop in viscosity
- Remedy: decrease gap to increase stabilizing influence of surface tension

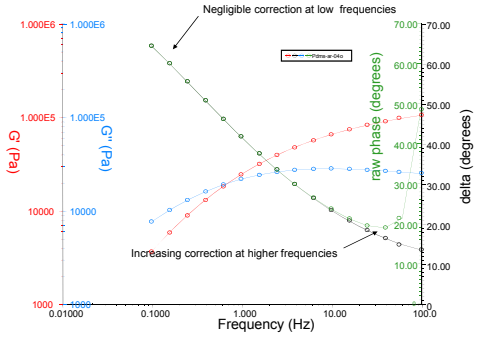
Inertial Effects

- What is Inertia?
  - Definition: That property of matter which manifests itself as a resistance to any change in momentum of a body
- Angular motion: $M = I \, d\Omega / dt$
  - M is the torque (Nm)
  - Ω is the angular velocity (rad / s)
  - I is the moment of inertia ($Nm\ s^2 \equiv kg\ m^2$)
    - $I = \Sigma\ mr^2$
    - m is the mass at distance r from the axis of rotation

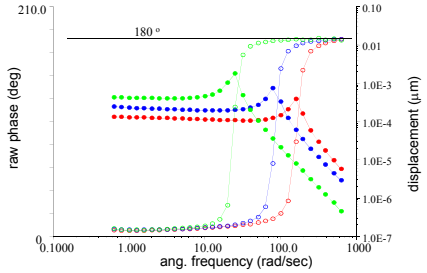
Inertial Effects in Oscillation for the AR Series

- During one cycle in oscillation, the material is accelerated to maximum speed and then decelerated to zero speed => inertia effects are always present
- Concern in oscillation experiments is particularly at high frequency with low viscosity fluids
  - Minimize correction by lowering the inertia of the moving parts (test geometries)
  - Inertia effects the measured phase. Plot raw phase. If raw phase is above 150° then inertia is dominating your test results

Correction for Inertia in Oscillation for AR

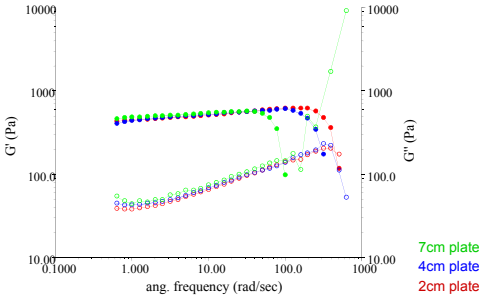


Effect of Inertia on Amplitude and Raw Phase



7cm plate, inertia: geometry = 42.69, total = 57.01 µNm s²
4cm plate, inertia: geometry = 7.90, total = 22.22 µNm s²
2cm plate, inertia: geometry = 1.109, total = 15.43 µNm s²

Effect of Inertia on G'and G"



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Presenting the Data

- Accurate data interpretation, model fitting, and comparisons of rheological data require selection of appropriate parameters and plot scaling.
- Meaningful information can be masked if the data are not presented the right way.
- After running experiment, always check and make sure the data you are presenting are within instrument specifications, and unaffected by artifacts.
- It is a good practice to include notes on plots that display information pertinent to how the data was run and important to the interpretation, such as pre-shear rate and time, temperature in a frequency sweep, frequency in a temperature sweep, clamp type or geometry, etc.

Presenting the Data

- Don't overload plots with unnecessary parameters. Better to make two plots rather then confuse the interpretation.
- Legends
- Colors/symbols/lines
- Stamp critical points and fits such as end of linear region, cross-over frequency, transition peaks, onsets, etc.

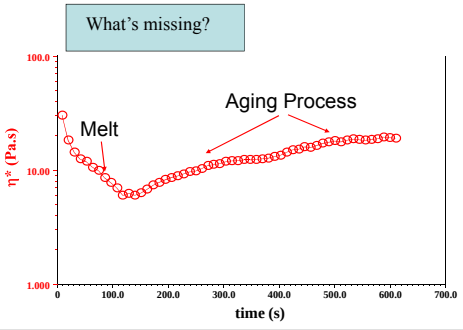
Oscillation Tests

- Time Sweeps
  - X-axis: Plot time on linear scale
  - Y-axis: Plot G' or η^* on log scale
- Strain Sweeps
  - X-axis: Plot γ same as sweep mode – log or lin
  - Y-axis: Plot G'& G" on log scale
- Stress Sweeps
  - X-axis: Plot γ or τ same as sweep mode – log or lin
  - Y-axis: Plot G' & G" on log scale

Oscillation Tests

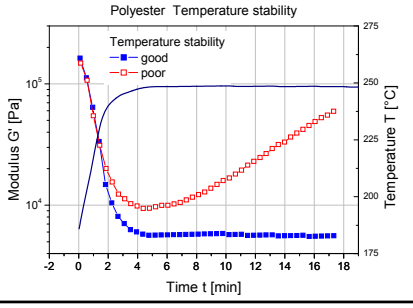
- Frequency Sweeps
  - X-axis: Plot frequency ω on log scale
  - Y-axis: Plot G', G", tan δ & η^* on log scale
- Temperature Sweeps and Ramps
  - X-axis: Plot temperature on linear scale
  - Y-axis: Plot G'& G" on log scale
  - Y-axis: tan δ can be plotted on log or linear scale
    - A single transition over a temperature range is often best observed on a linear scale.
    - When multiple transitions are present, weaker secondary transition peaks may not be obvious when tan δ is plotted on linear scale.

Data Presentation: Time Sweep on Polymer

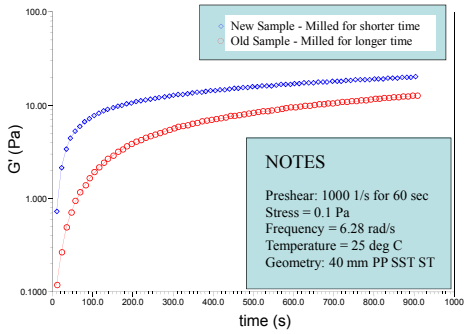


Data Presentation: Time Sweep on Polymer

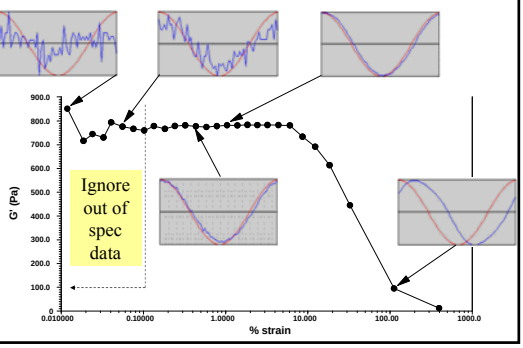
That's Better!



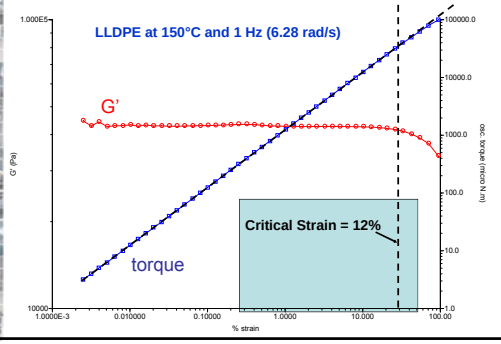
Data Presentation: Time Sweep on Structured Fluid



Remember to Check Specifications

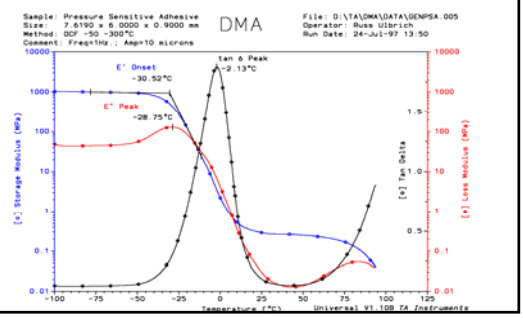


Label Important Information

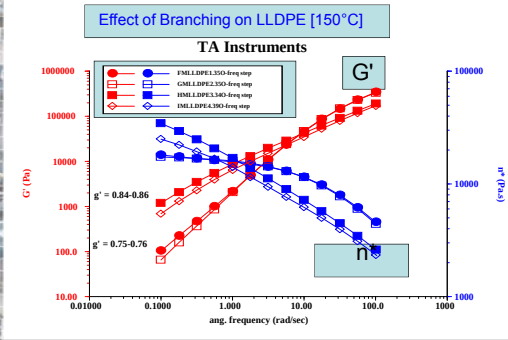


Label Important Information

PSA: Glass Transition Measurement

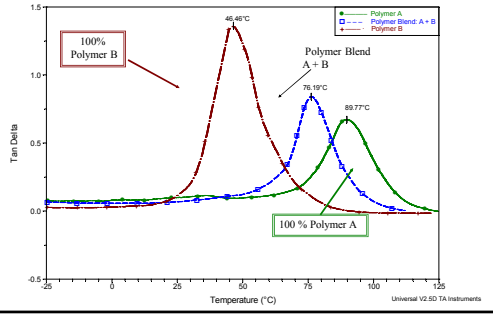


Good Use of Colors, Symbols, & Legend



Good Use of Colors, Symbols, & Legend

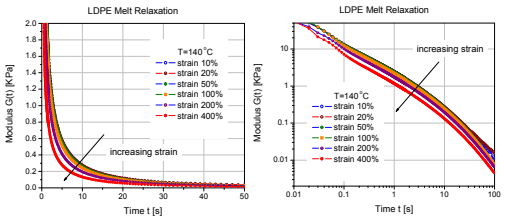
Polymer Blend - Aerospace Coating



Presenting Stress Relaxation & Creep Data

- Stress relaxation results are typically always presented on a log-log scale.
- In Creep, Strain, Creep compliance, and recoverable compliance can be plotted
  - Linear – linear
  - Linear – log
  - Log – log
- The selection of scaling will depend on the time scale of interest.
- In general, linear scales work better for looking at the longer time response of a material and log scales help to expand differences on shorter time scales.
- Some examples are as follows.

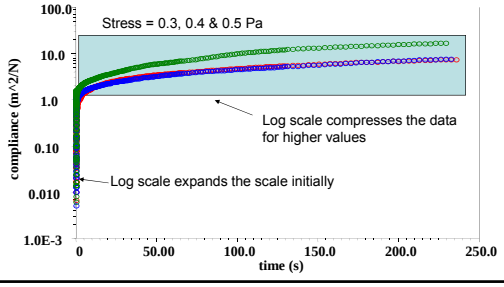
Linear and logarithmic Plots



Data curves may look very different in the logarithmic representation!!

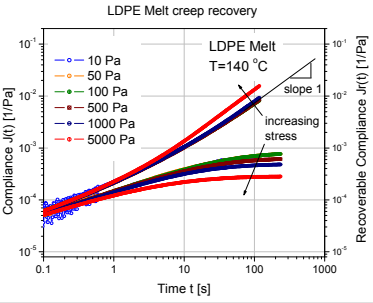
Creep Compliance with Increasing Stress

Log-linear scale provides perspective of LVE for complex fluid



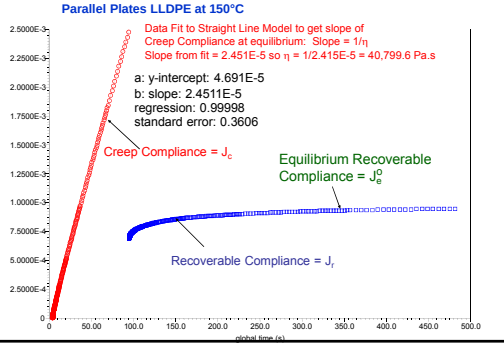
Creep and Recovery with Increasing Stress

Log-log scale provides different LVE perspective for polymer



Initial effects are expanded in time

Creep Recovery Curve on Polymer Melt: Linear Scales for Long time Equilibrium

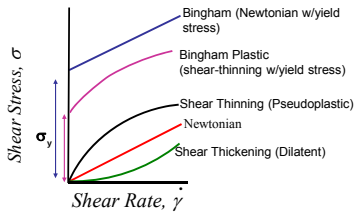


Presenting Steady State and Transient Flow Data

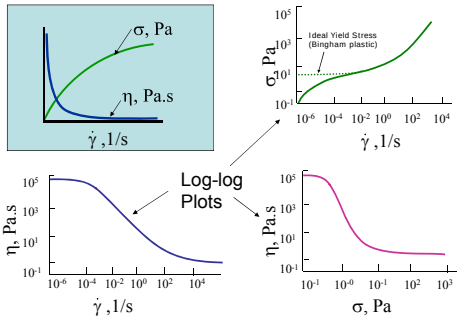
- Multiple ways of plotting steady shear data.
  - Shear stress vs. shear rate
  - Viscosity vs. shear rate
  - Viscosity vs. shear stress
- Make use of log and linear scales
- Use viscosity models

Characteristic Flow Diagrams on Linear-Linear Plot

Flow on linear-linear Plot

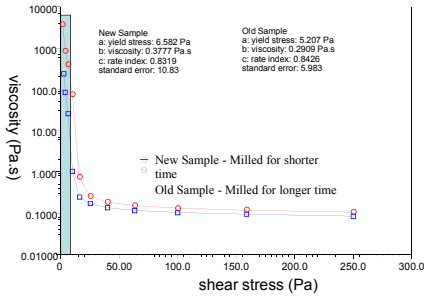


Characteristic Diagrams for Shear Thinning Fluids



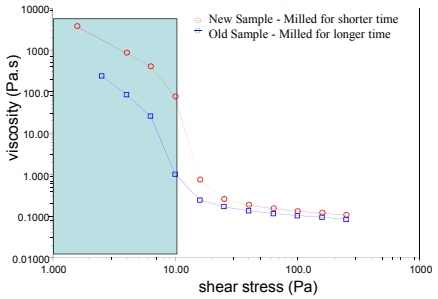
Scaling Makes a Difference

Automotive Paint Samples: Data fit to Herschel-Bulkley Model

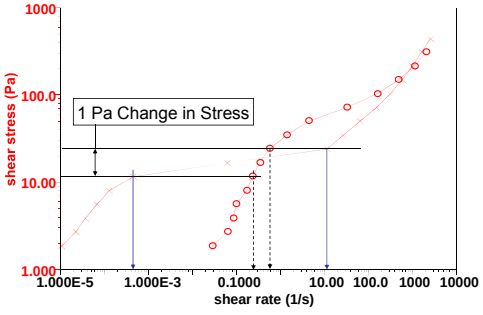


Scaling Makes a Difference

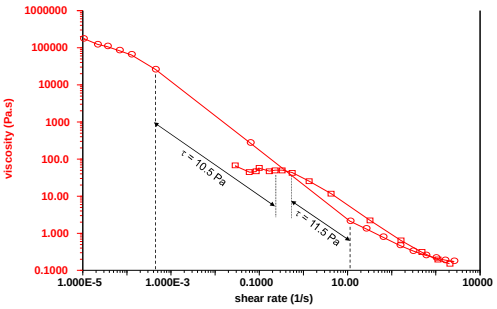
Automotive Paint Samples: Viscosity vs. Shear Stress



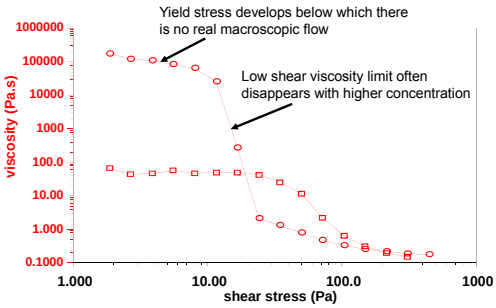
Ink Flow Curves: Shear Stress vs. Shear Rate



Ink Flow Curves: h vs. Shear Rate



Ink Flow Curves: h vs. Shear Stress



Fitting the Right Model

Predicts the shape of the complete Flow Curve

Cross $\frac{\eta_0 - \eta_\infty}{\eta - \eta_\infty} = (K\dot{\gamma})^m$

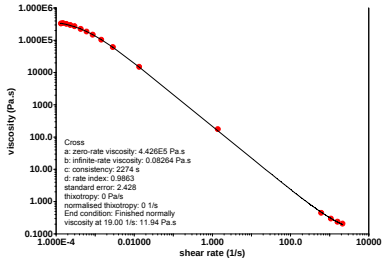
Sub-sets of the Cross Equation which predict portions of the complete Flow Curve

Power Law $\eta = K_1 \dot{\gamma}^{n-1}$

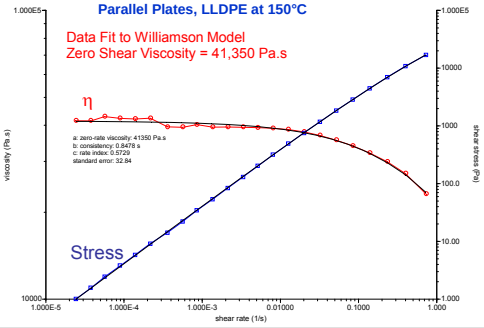
Sisko $\eta = \eta_\infty + K_1 \dot{\gamma}^{n-1}$

Williamson $\eta = \eta_o - K_1 \dot{\gamma}^{n-1}$

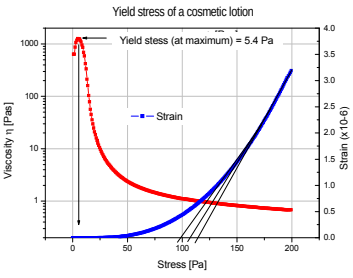
Margarine Data: Cross Model



Flow Curve on Polymer Melt

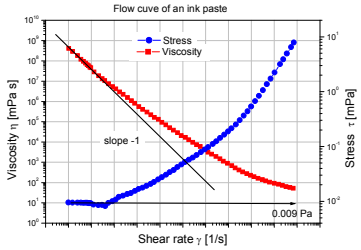


Yield stress in a stress ramp



The maximum viscosity method is more representative and reproducible then the strain tangent method

Structured fluid: Flow Curve in Rate Mode



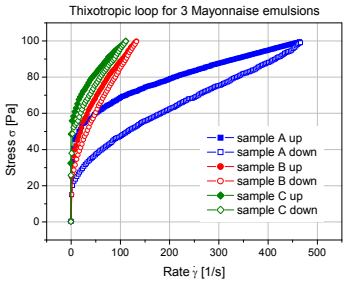
Stepped rate test on ARES

Shear rates are stepped at equal intervals

=> smooth curve

The viscosity decreases with a slope of -1 versus strain rate and the stress becomes rate independent => material with yield stress

Thixotropic loop

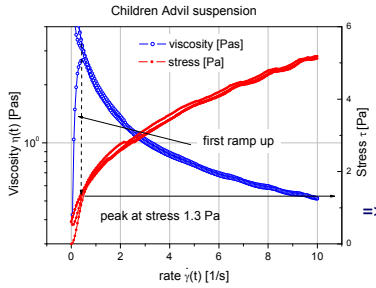


Thixotropic material

Up and down ramps do not superpose

Area under the curve is a measure of thixotropy

Thixotropic loop

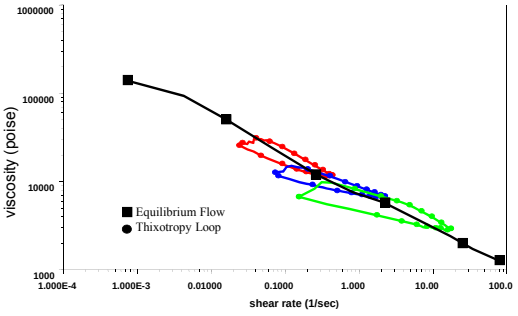


Non-thixotropic material

Up and down ramps superpose – except the first one

=> Start up from zero

Equilibrium Flow & Thixotropy Loops



Tip #11: *Take advantage of all of the training opportunities available to you!*

Tip #11: *Take advantage of all of the training opportunities available to you!*

- It is extremely important to us for our customers to get the best out of their instruments
- While we've always offered multiple training opportunities, we've dramatically increased this offering over the past several years
- We will use this last presentation to make sure you are aware of what is available
- Our web site is a good place to start to see what training is available
  - www.tainstruments.com

TA Instruments Corporate Website

Training Web page

Training Opportunities from TA

- Initial Training at Installation
- e-Training Courses
- Theory & Applications Training Courses
- Hands-On Training Courses
- Custom Onsite Training Courses
- Special Courses

Initial Training at Installation

- Preformed by Service Engineer during installation
- They will show you how to:
  - Calibrate your instrument
  - Use software to set up experimental run
  - Prepare a sample for analysis
  - Create & run a method
  - Analyze the data

e-Training Courses

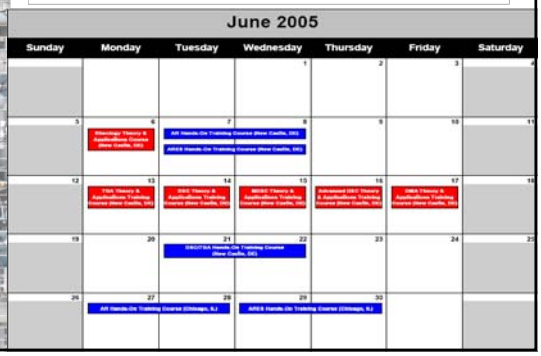
- Variety of courses that can be taken off our website
- These course include:
  - QuickStart Courses that tell you how to get started running your instrument – **These are Free!!**
  - Advanced Courses build on the basics and talk about advanced features and options
- These are available as recorded courses that you can take 24/7
- Also available “Live” (see website for details)

e-Training Courses

Theory & Applications Training Courses

- One day lecture based courses for each technique
- Covers; theory, instrumentation, calibration, method development, operating conditions, and applications
- Given at our corporate headquarters in New Castle, DE (also in CA –see special courses)

Example of Training Calendar



Hands-On Training Courses

- A two day course that consists of running the instruments, sample preparation, method development, and data interpretation
- Attendance is limited per course to maximize the benefit
- Courses are scheduled in DE, and our Chicago area office

Custom Onsite Training Courses

- These courses are taught at your facility, with your instruments, and your samples
- Can be Hands-On, Lecture, or a combination of both
- Typical agenda might be:
  - Theory & Instrumentation (Lecture)
  - Applications (Lecture)
  - Calibration (Hands-on)
  - Running your samples (Hands-on)
  - Analyzing your data (Hands-on)

Special Courses

- This section will list new specialized courses that we will be giving from time to time
- Current offerings:
- Theory & Applications Course in CA – ***Back by popular demand***
  - Scheduled the week of September 12, 2005 at Caltech in Pasadena (the week before NATAS in nearby Universal City, CA)
  - Rheology, DMA, TGA, DSC, MDSC®
  - These are the same lecture based courses that we give in DE

Special Courses

- DSC Certified User Training Course - ***Back by popular demand***
  - October 19, 2005 as part of a "DSC Week", which includes Theory & Applications courses for DSC & MDSC®, and Hands-on DSC & TGA training
  - Course is designed to take users to the next level. Covers theory, calibration & maintenance, method development, and data interpretation

Special Courses

- Rheology Certified User Training Courses – ***First time offered***
  - A separate course for both AR & ARES users. Scheduled on October 25 (AR) & 26 (ARES), 2005 as part of a "Rheology Week", which also includes the Rheology Theory & Applications course,and Hands-on training courses for the AR & the ARES
  - Course is designed to take users to the next level. Covers theory, calibration & maintenance, method development, and data interpretation

Lastly

- Always let us know if you desire training that is not listed. We are always open to suggestions, and feedback.
- For more information on any and all of these options, go to our website, or email training@tainstruments.com
- Thank you for coming and for your interest in TA Instruments