Viscoelasticity, Creep and Fiber-filled Materials

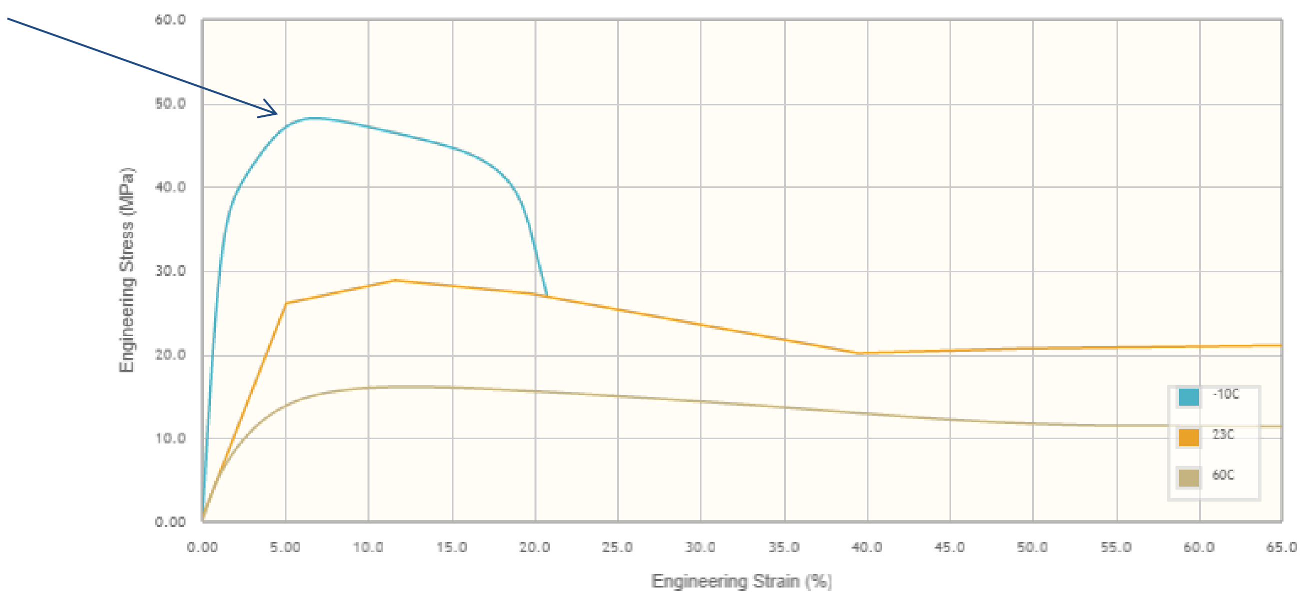
Hubert Lobo

Definitions of creep and viscoelasticity

- Creep is the change that occurs to a material due constant load over time
  - Depends on applied load
  - Depends on temperature/environment
- Stress relaxation is the change that occurs to a material due to constant deformation over time
  - Depends on applied strain
  - Depends on temperature/environment
- Viscoelasticity is the property consisting of both elastic behavior and flow (plastic) behavior, in which time varies the behavior of each component
  - Depends on time/frequency
  - Depends on load/strain
  - Depends on temperature/environment

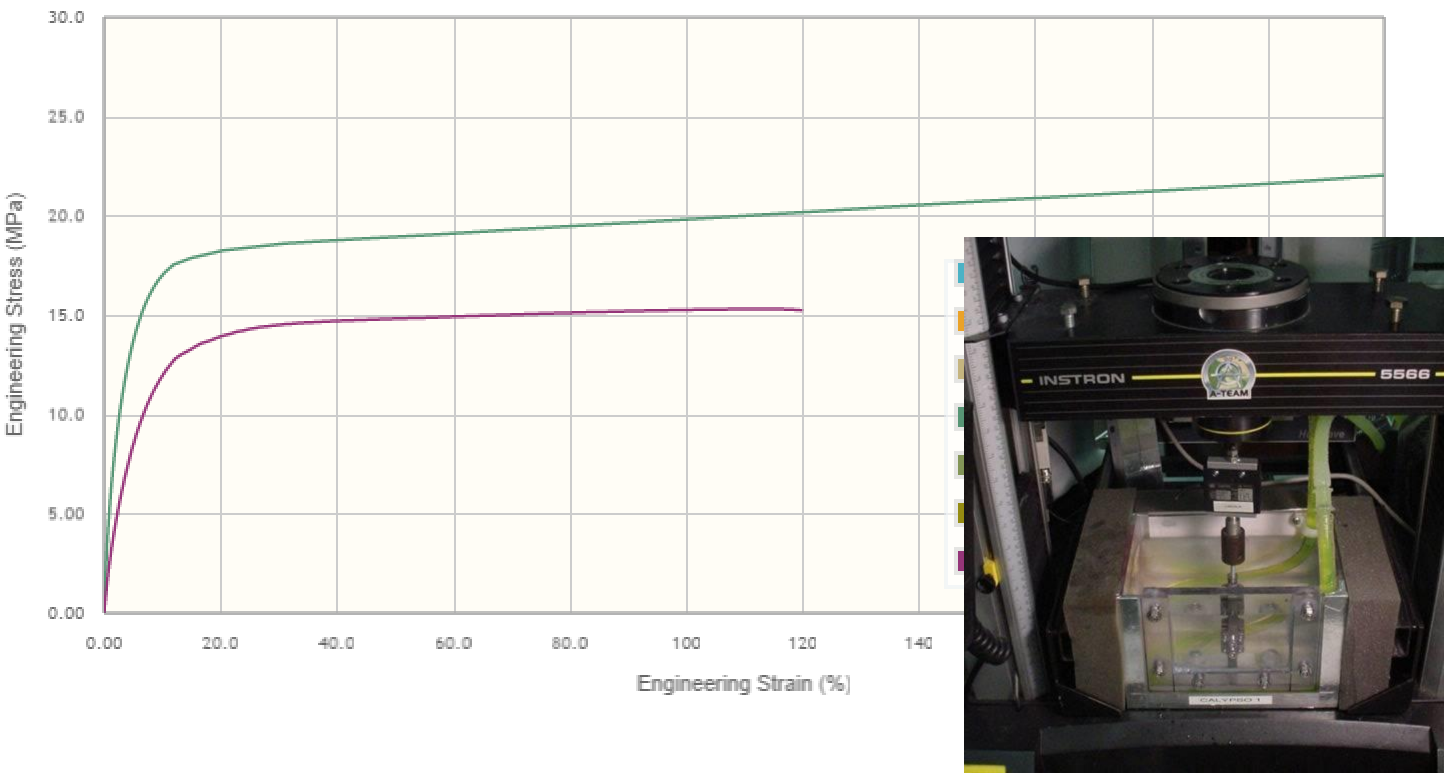
Effect of temperature

- Properties and dependencies change with temperature
  - Modulus
  - Ductile-brittle transitions
  - Rate dependency



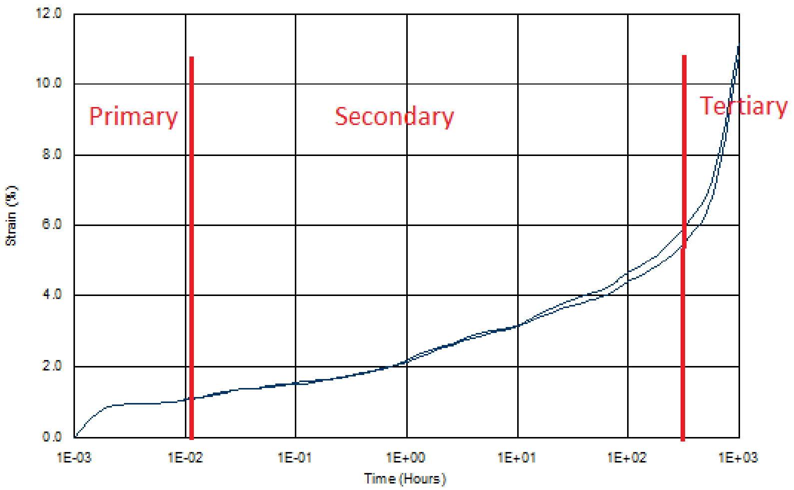
Effect of environment: *in-vivo*

- Saline solution at body temperature



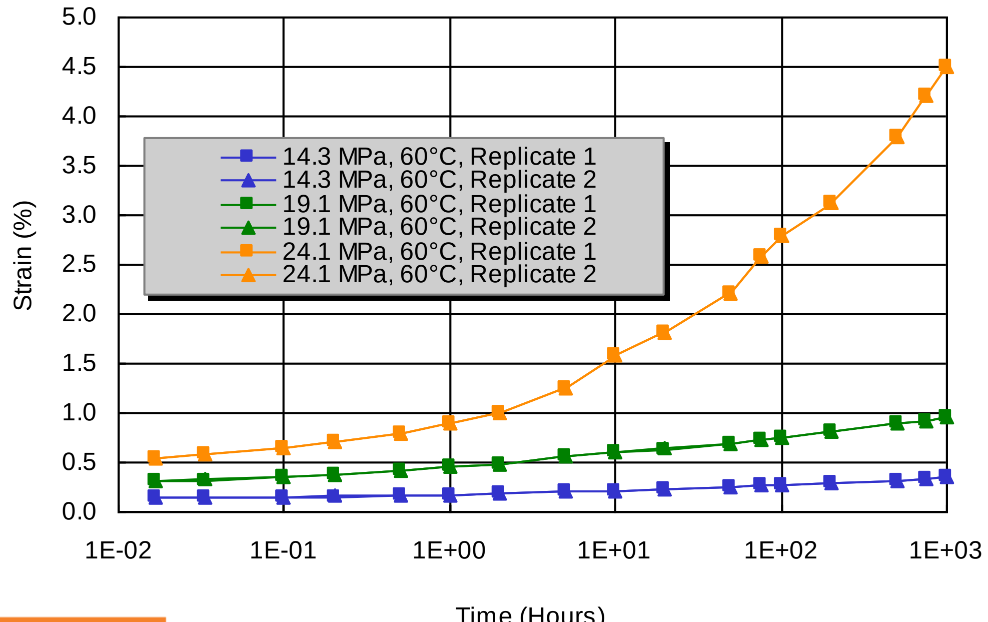
Creep behavior

- Primary
  - Short time
  - Loading phase transient
- Secondary
  - Sustained uniform strain
  - Handled by creep models
- Tertiary
  - Localization and failure
  - Creep rupture



Creep modeling

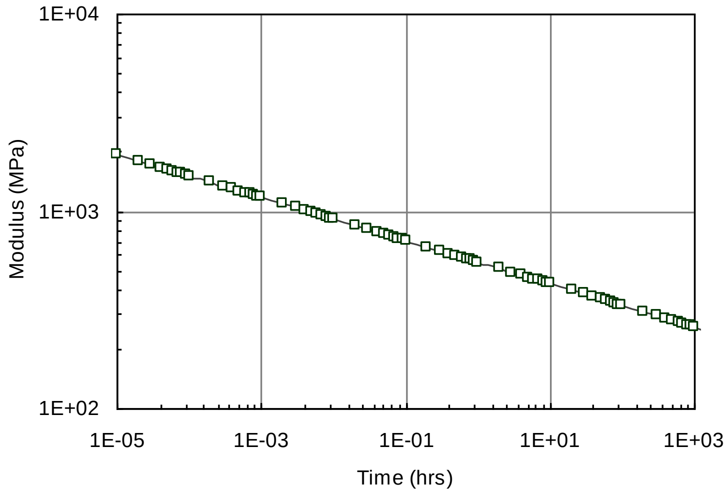
- Creep strain vs. time, fit to time hardening model
  - Minimum 3 stress levels
  - Typically 1000 hours
  - Can incorporate temperate but best handled as individual models for each temperature (more stable/accurate)
  - Plastic deformation
  - ASTM D2990 / ISO 899



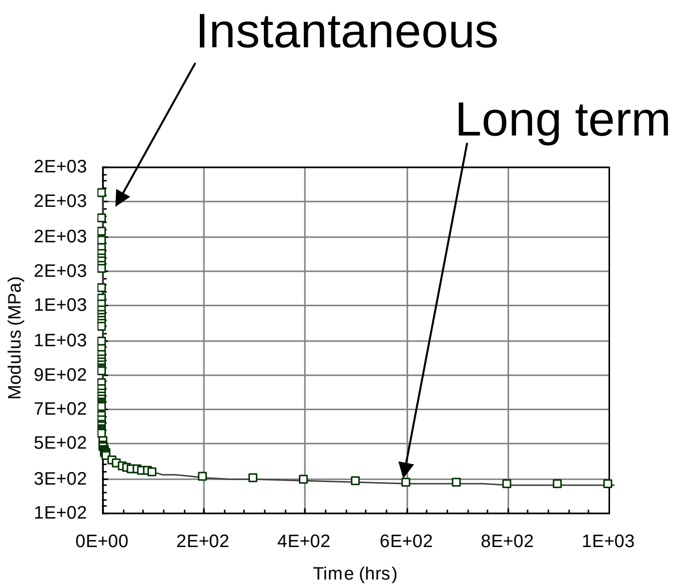
$$\dot{\varepsilon}_{cr} = C1\,\sigma^{c2}\,t^{c3}$$

Stress relaxation

- Stress relaxation
  - Stress/Modulus will relax over time
  - Logarithmic behavior
  - Typically 1000 hours
  - Plastic deformation
  - ASTM D6147



Log-Log Scaling



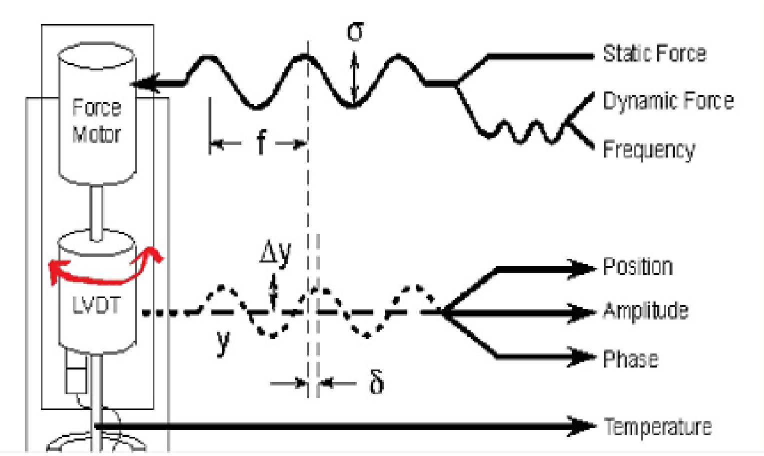
Linear-Linear Scaling

Viscoelasticity

- In small strain (linear viscoelastic) situations
  - Applied strain (stress) is recoverable
  - Frequency or time based measurements
- A time-temperature correlation exists
  - Higher temperature = longer time
  - Lowering temperature = short time

DMA (dynamic mechanical analysis) operation

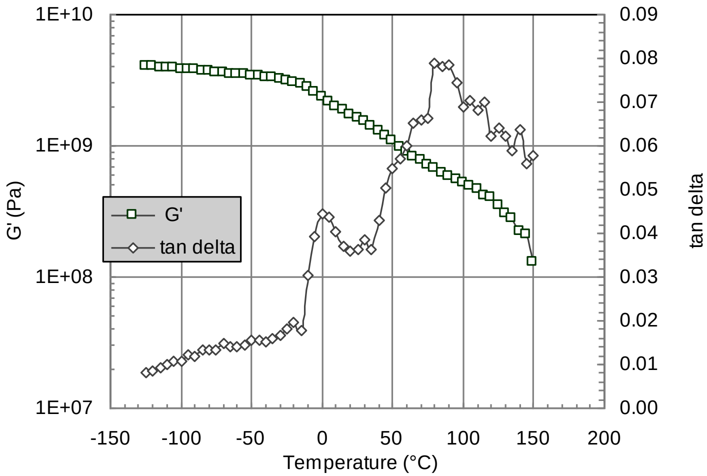
- Force or displacement control
- Dynamic or constant application



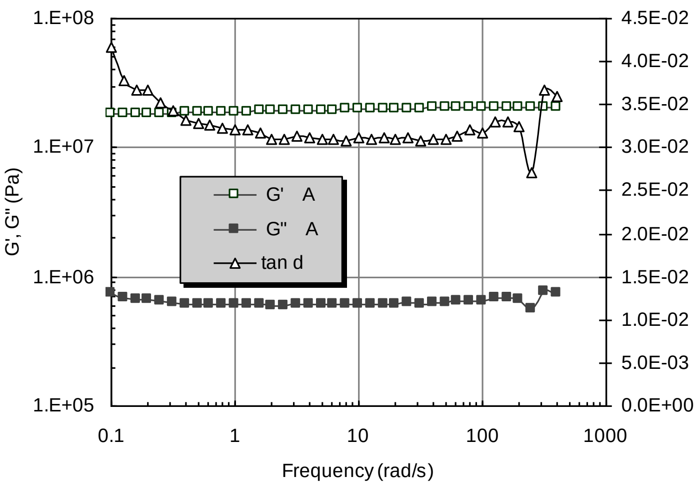
Example of force based DMA

$$G' = \frac{\sigma_0}{\varepsilon_0}\cos\delta$$
$$G'' = \frac{\sigma_0}{\varepsilon_0}\sin\delta$$

Derivation of dynamic modulii



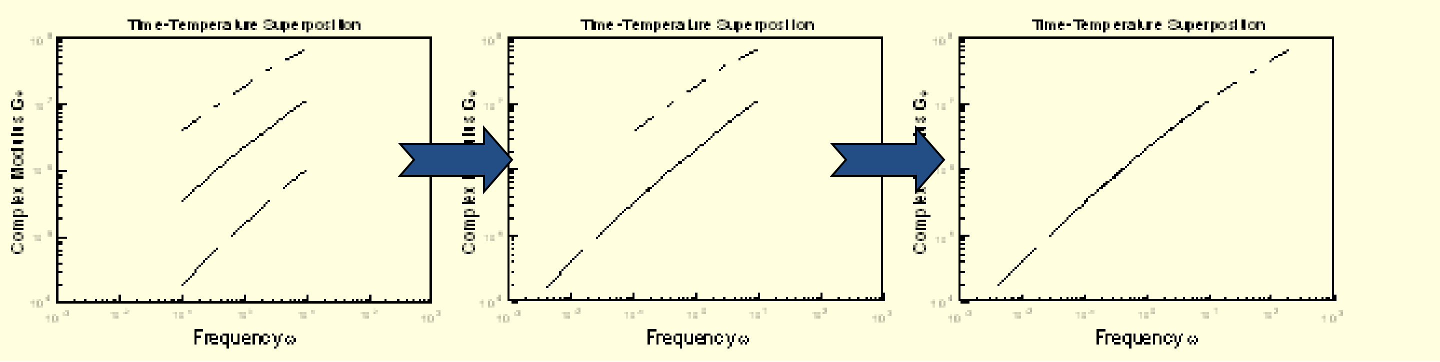
Temperature sweep



Frequency sweep

TTS (time-temperature superposition)

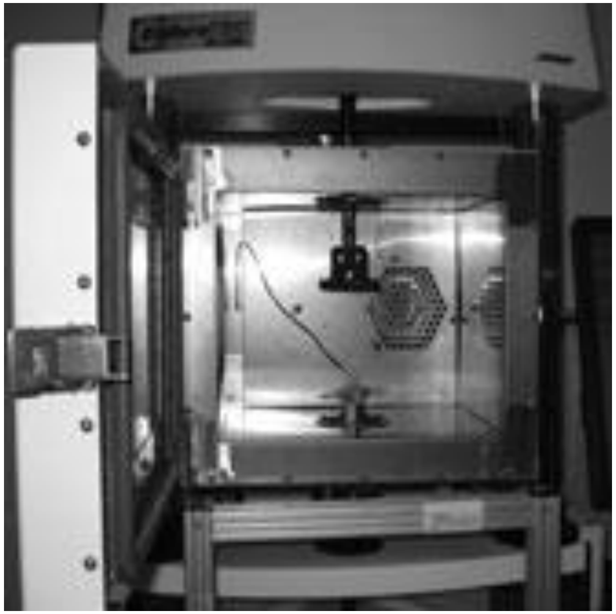
- Takes advantage of both time and temperature sensitivity of material
  - Measure frequency or time sweep at multiple temperautes
  - Shift curves in time/freq domain
  - Generate a mastercurve at times/freq higher than possible to test
  - Also generate temperature sensitivity



Torsional mode

- Torsional DMA
  - Temperature: -125 to 600°C
  - Frequency: 0.01 to 500 rad/s
  - Steady or dynamic modes
  - Torque range: 2 to 2000 gm-cm
- Specimens
  - 25 mm diam. Disc
  - Torsional (flex) bar
- Data
  - G’-G” data tan delta
  - Shear stress relaxation

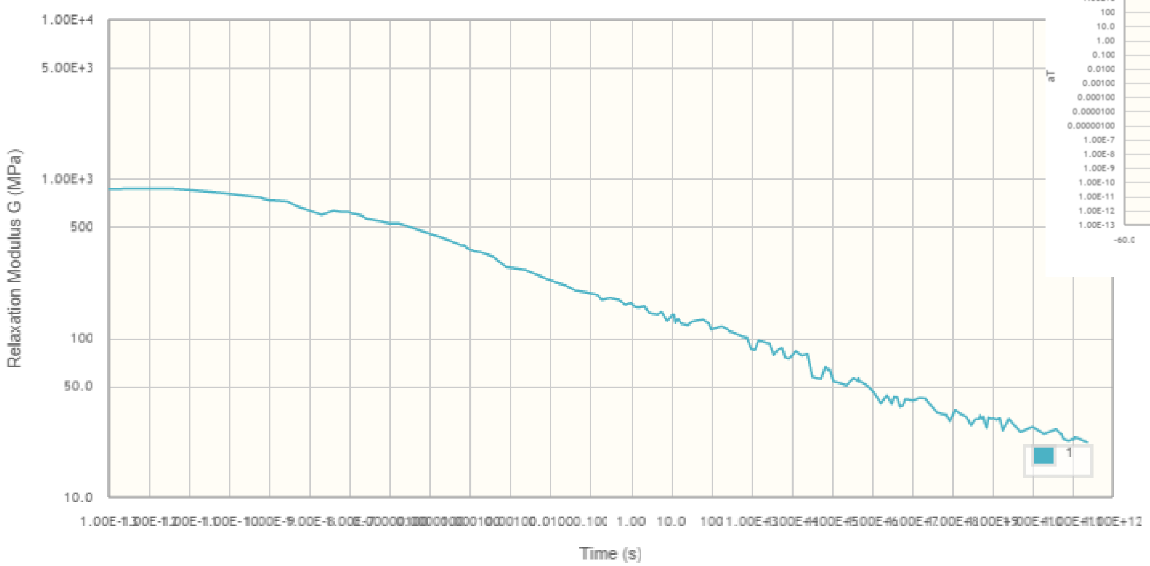
Tension/compression mode

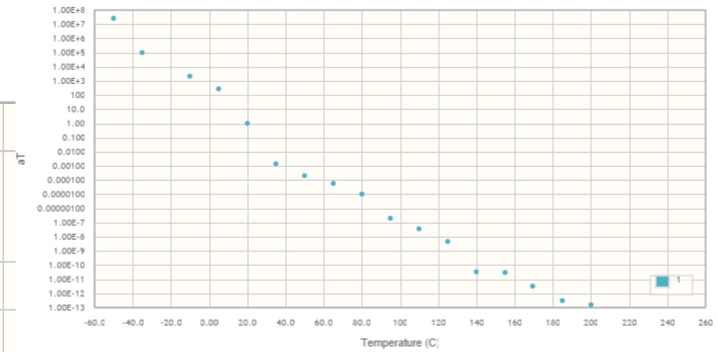


- Dynamic tensile-compression
  - Frequency: 0.01-200 Hz
  - Load: 0-250 N
  - Crosshead speed 0-3 m/s
  - Displacement: ±12.7 mm
  - Temperature: -80 to 250C
- Specimens
  - ASTM type V tensile bar
  - Compression cube (25mm depending on stiffness)
- Data
  - E’ E” (tensile loss/storage modulus) tan delta
  - Tensile relaxation modulus E f(t)

PTFE mastercurve (stress relaxation)

- Caution should be exercised in using very large time scale data
  - Environment reaction of material (deterioration of material, chemical exposure, etc.)
  - Assumes all things are constant
  - Beware of transition temperatures (TTS does not work)

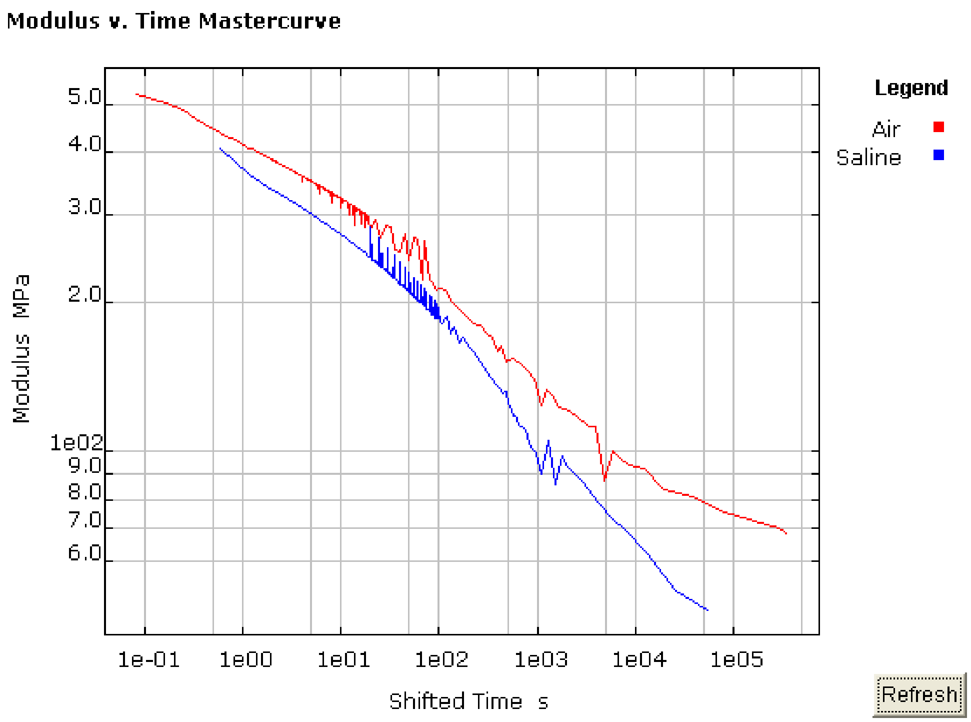




Shift factors

Viscoelasticity & environmental effects

- *In-vivo* stress relaxation for hip replacement material



Viscoelastic models

- Time-based Prony series

$$G(t) = G_0 - \Sigma_{i=1}^{N} G_i [1 - \exp(-t/\tau_i)]$$

- Frequency-based

$$G' = G_0 \left(\alpha_\infty + \sum \left(\frac{\alpha_i^G \left(\tau_i^G\right)^2 \omega^2}{1 + \left(\tau_i^G\right)^2 \omega^2} \right) \right)$$

$$G'' = G_0 \left(\sum \left(\frac{\alpha_i^G \tau_i^G \omega}{1 + \left(\tau_i^G\right)^2 \omega^2} \right) \right)$$

Viscoelastic models

- Normalized modulus data fit to 6-parameter Prony series
- Used for simulating short or long term events
- Can apply WLF shift factors for different temperatures

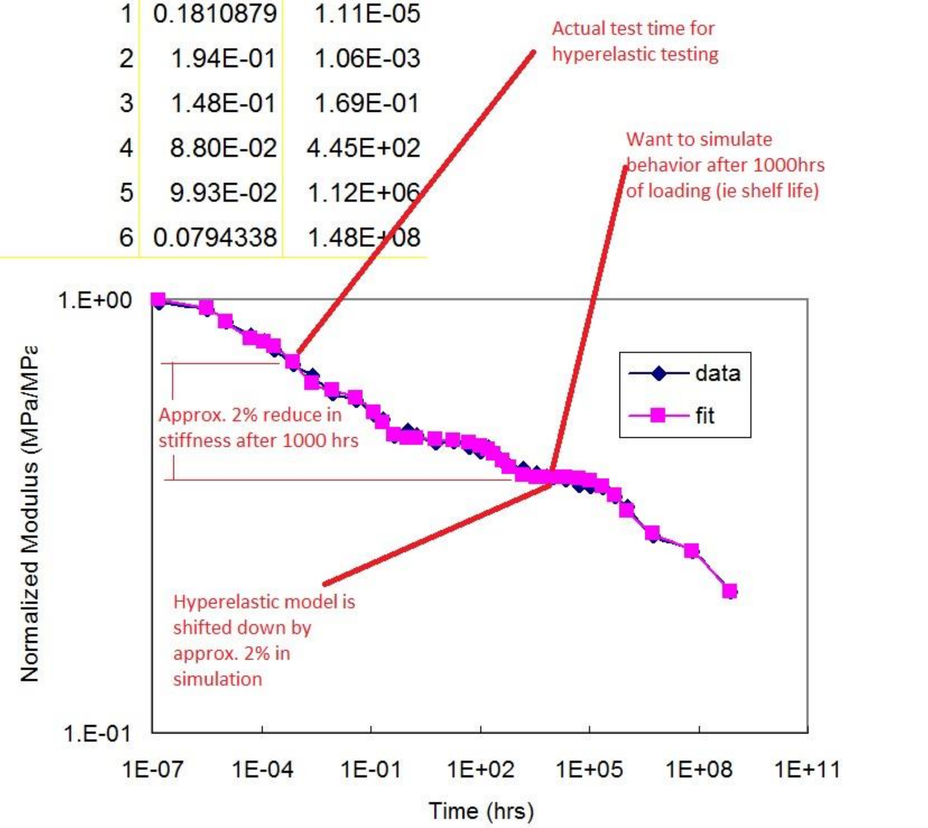
$$G(t) = G_0 - \Sigma_{i=1}^{N} G_i [1 - \exp(-t/\tau_i)]$$

Prony series

$$\log(a_T) = \frac{-C_1 (T - T_r)}{C_2 + (T - T_r)}$$

WLF equation

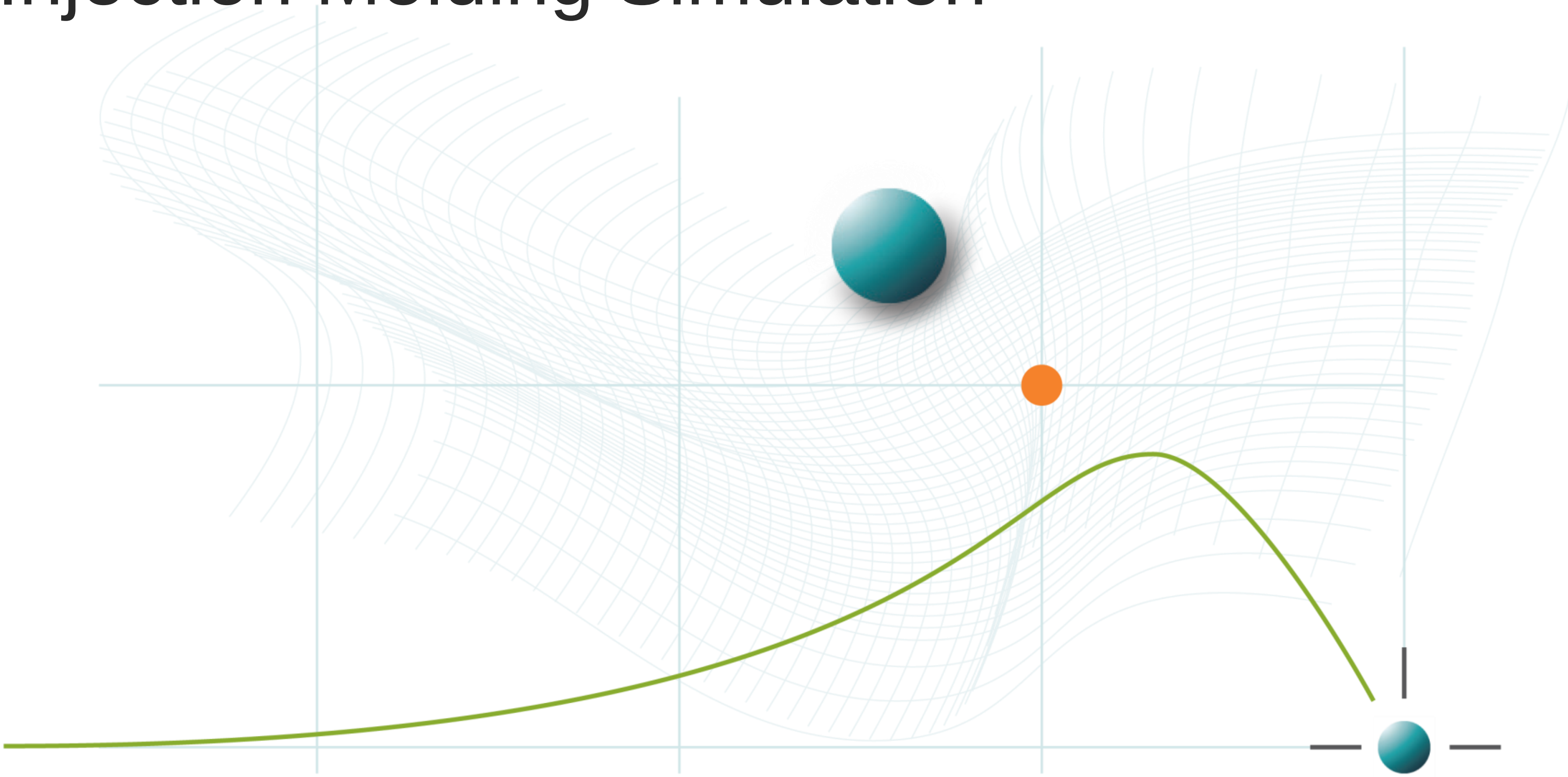
	G(I)	Tau(I)
1	0.1810879	1.11E-05
2	1.94E-01	1.06E-03
3	1.48E-01	1.69E-01
4	8.80E-02	4.45E+02
5	9.93E-02	1.12E+06
6	0.0794338	1.48E+08



Limitations

- Depends on linear viscoelastic theory
- Can predict non-linear effects
- Cannot predict large strain deformation
- Cannot predict failure
- Great caution when used with hyperelasticity or other large deformation model

Fiber Filled Plastics and the Need for Injection Molding Simulation

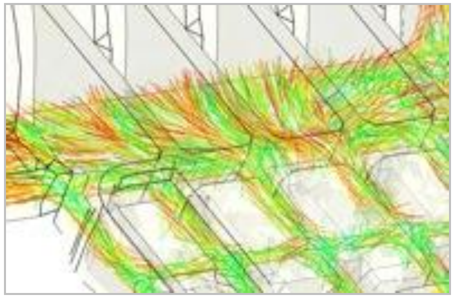


DatapointLabs TestPaks matereality knowmats picsci

expert material testing | CAE material parameters | CAE Validation | software & infrastructure for materials | materials knowledge | electronic lab notebooks

Fiber-filled plastics

- Spatial orientation of fibers
  - Properties vary spatially
  - Significant property differences with orientation
- Can approximate:
  - Worst case: use cross-flow data
- Use third party software to map then implement orientation effects to FEA
  - Injection molding simulation of part to capture fiber alignment
  - Digimat or Helius to impart material properties
  - Send to FEA



Source:e-Xstream

Injection molding simulation

- Simulate injection molding process
  - Mold design
  - Fiber orientation
  - Final part geometry (warp/shrink)
  - Frozen stresses
- Third party software
  - Autodesk Moldflow
  - Moldex 3D
  - Sigmasoft

Required testing

- Viscosity
- Specific heat
- DSC transition temperatures
- Thermal conductivity
- PVT
- Linear shrinkage
- Viscoelastic properties

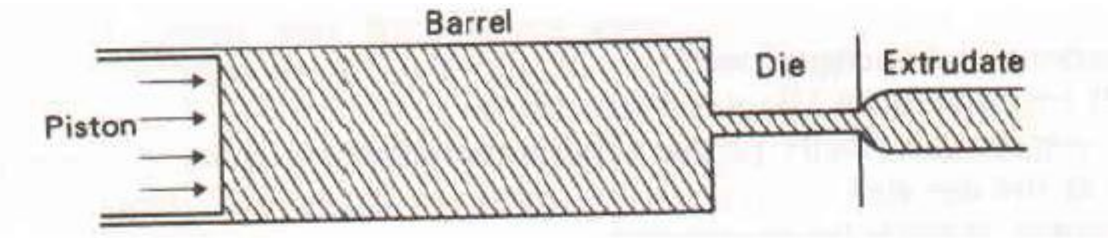
Mold filling

Packing

Post-mold
Shrinkage
Warpage

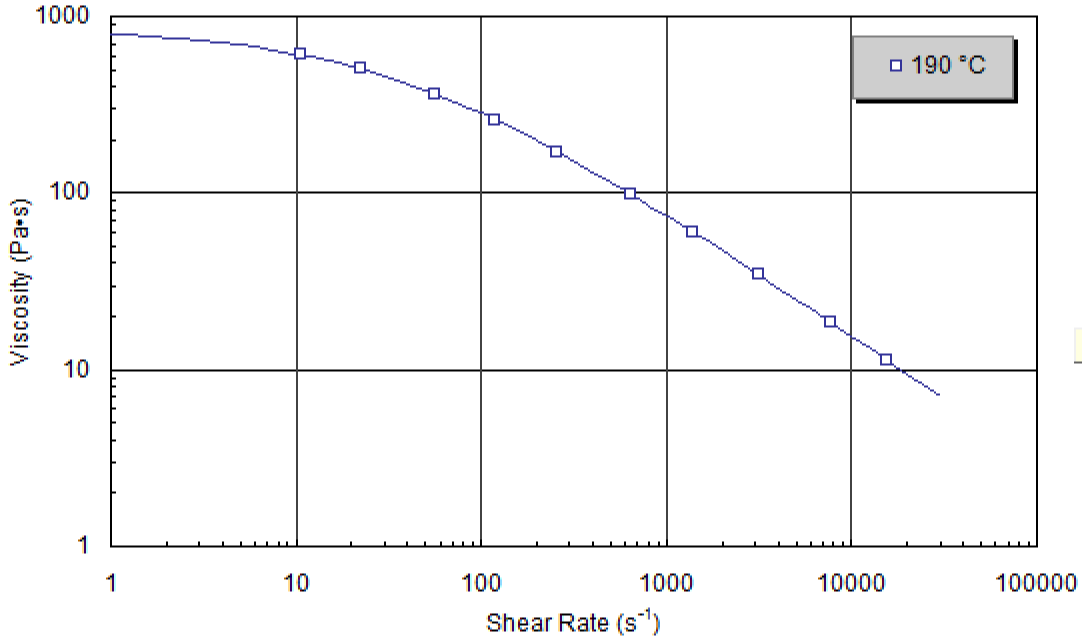
Rheometer measurement of viscosity

- Capillary rheometer is used
- Material is extruded through a restriction of known geometry (extremely high tolerance dies)
- Temperature and flow rate are controlled
- Pressure drop across the restriction is used to determine viscosity as a function of shear rate and temperature



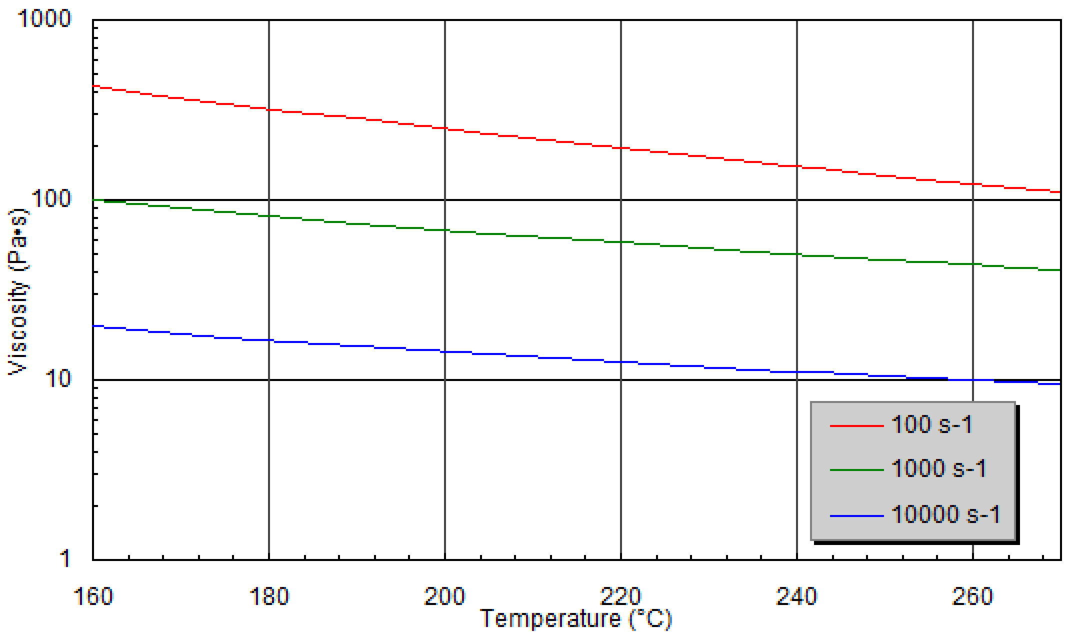
Viscosity properties

- As shear rate increases, viscosity decreases



Viscosity properties

- As temperature increases, viscosity decreases



Viscosity measurements

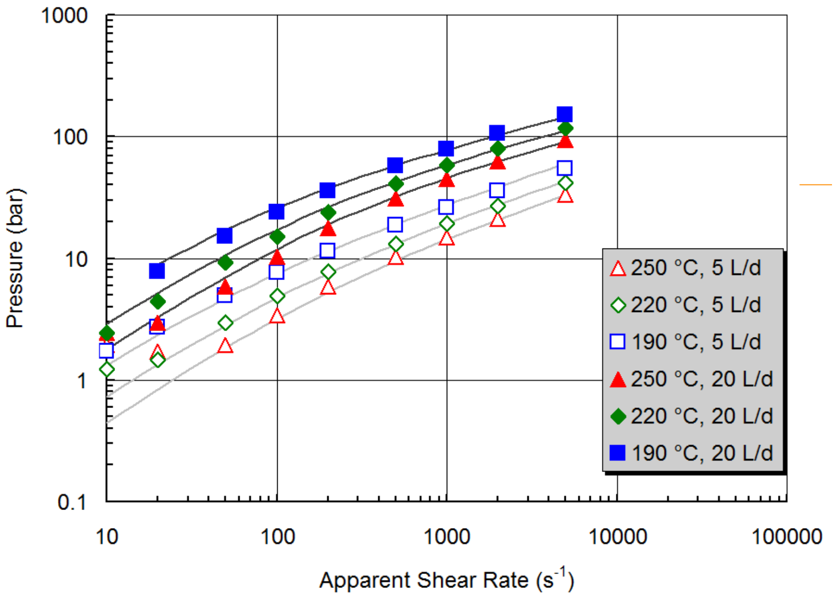
- Apparent Viscosity
- Shear rate: $\dot{\gamma}_a = \frac{32Q}{\pi d^3}$
  - Shear stress: $\tau_w = \frac{\Delta p d}{4L}$
  - Shear viscosity: $\eta_a = \frac{\tau_w}{\dot{\gamma}_a}$
- Corrections to viscosity
  - Reservoir and friction losses (transducer located at die)
  - End pressure drop (Bagley)
  - Non-parabolic velocity (Rabinowitsch correction)

where:

Q = Volume flow rate
Δp = Pressure drop
d = Capillary diameter
L = Capillary length

Bagley correction testing

- Bagley correction
  - Perform viscosity measure on two different die ratios at equal shear rates
  - Evaluate pressure differences between die geometries (capillary diameter remains the same)
    - $\tau = R/2(dP/dL)$

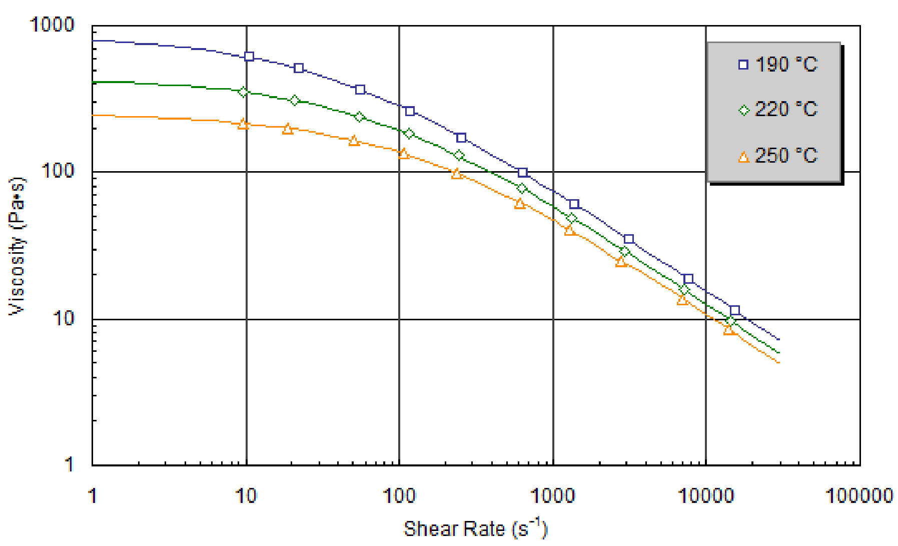


Viscosity modeling

- Very strong rheological models
  - Cross WLF, Cross Arrhenius
  - Combines a model of shear rate dependency with temperature dependency
  - Allows us to predict beyond testing range

$$\eta(T,\dot{\gamma}) = \frac{\eta_0(T)}{1+\left(\frac{\eta_0\dot{\gamma}}{\tau^*}\right)^{1-n}}, where$$

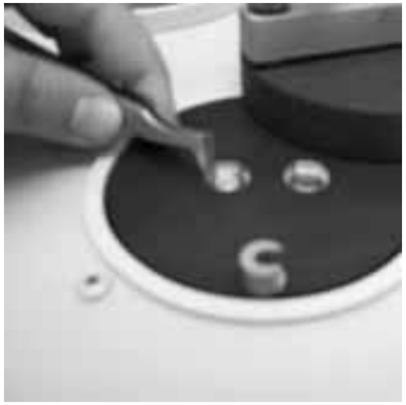
$$\eta_0(T) = D_1 \exp\left[-\frac{A_1(T-D_2)}{A_2+(T-D_2)}\right]$$



n	0.28400
τ*	32096.1
D1	3.86E+13
D2	263.15
A1	30.87
A2	51.6

Specific heat

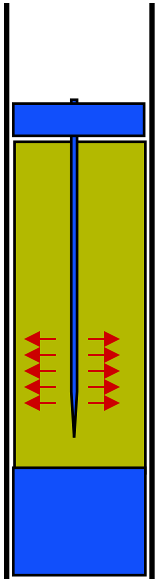
- DSC (differential scanning calorimeter)
  - Small samples sizes (7-15 mg)
  - Differential heat required to raise the temperature of the sample as compared to a reference
  - Performed in cooling to replicate molten material cooling to solidification
- Used in the simulation to determine how much energy must be dissipated to promote solidification



Thermal conductivity

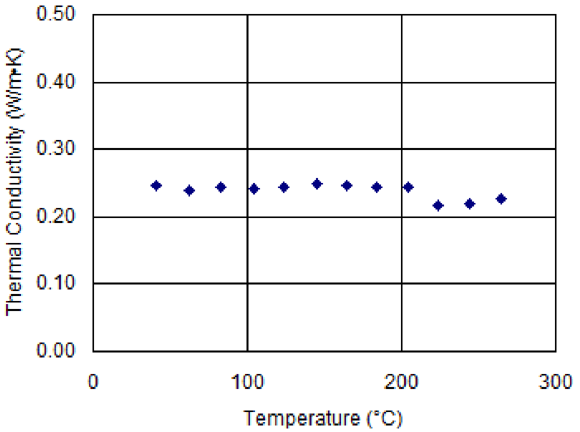
- A measure of how well a material transfers heat
  - Measured using transient line source
  - Measured in melt and solid state
  - Different behaviors for semi-crystalline and amorphous

Measure time to dissipate the heat pulse away from probe



Thermal conductivity

- Semi-crystalline materials show an increase in thermal conductivity in solid state
- Amorphous materials show a decrease in thermal conductivity in solid state
- The addition of fillers increase thermal conductivity
- Thermal conductivity of polymers is much lower than metals
  - Copper: 400 W/mK
  - ABS: 0.176 W/mK

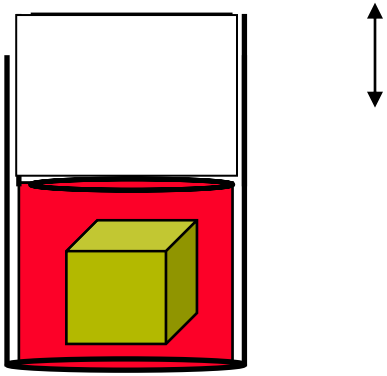


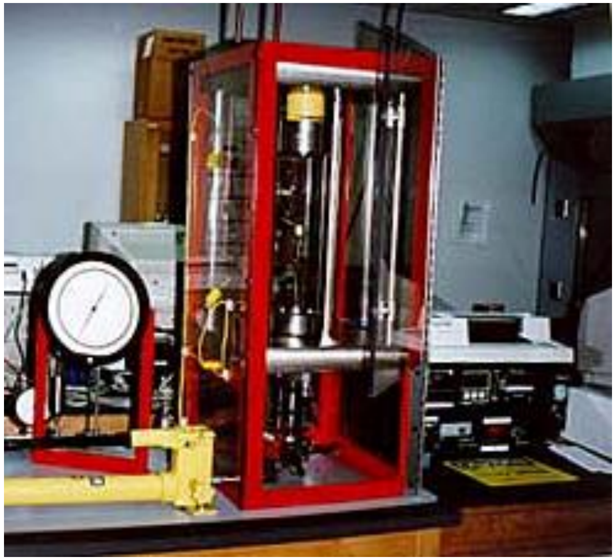
Density as a function of PVT (pressure, volume and temperature)

- Isobaric cooling scan (for semi-crystalline materials)
  - Need to accurately capture the onset of crystallization
  - Much longer run times
- Isothermal heating scan (for amorphous materials)
  - No crystallization so transition is independent of mode
  - Much faster (relatively)
- Pressures of 10 – 200 Mpa
- Measure both solid and melt domains

PVT testing

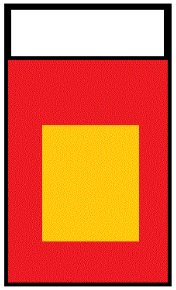
- Difficult and time consuming test
  - Initial density at ambient conditions
  - Mercury used as confining fluid
  - High temperatures and pressures
  - Complex datasets
  - True hydrostatic state

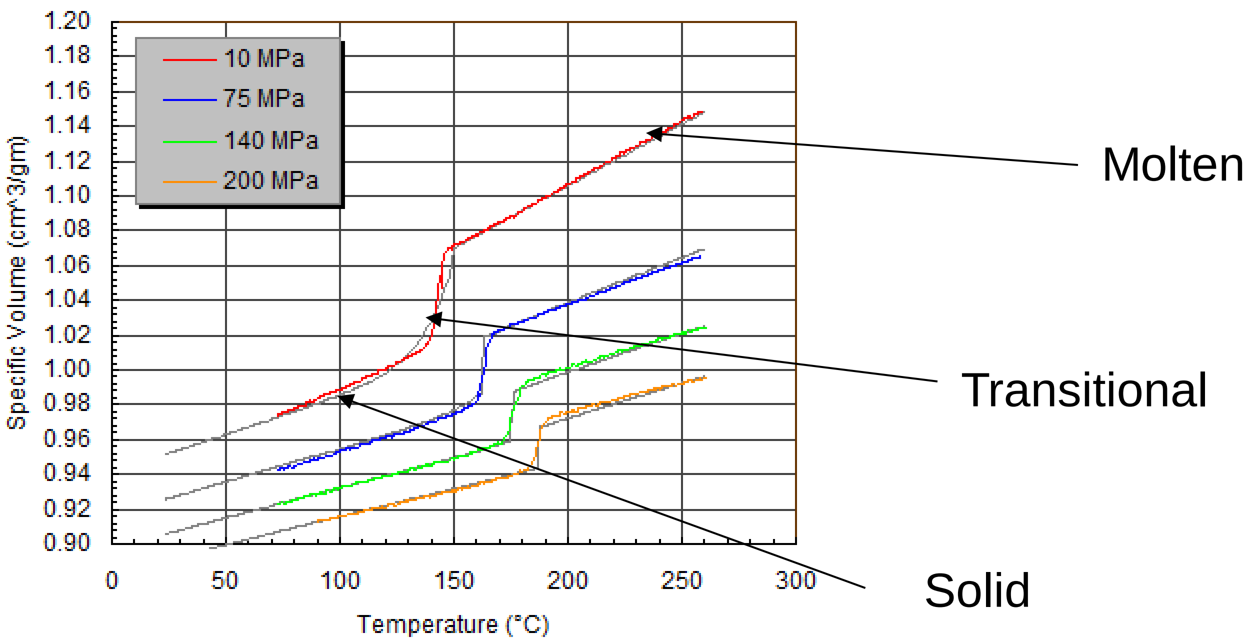


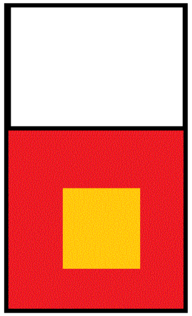


PVT test data

- Semi-crystalline material
  - Transition region is critical
  - Rise in temp. = rise in spec. vol.
  - Rise in press. = drop in spec. vol.

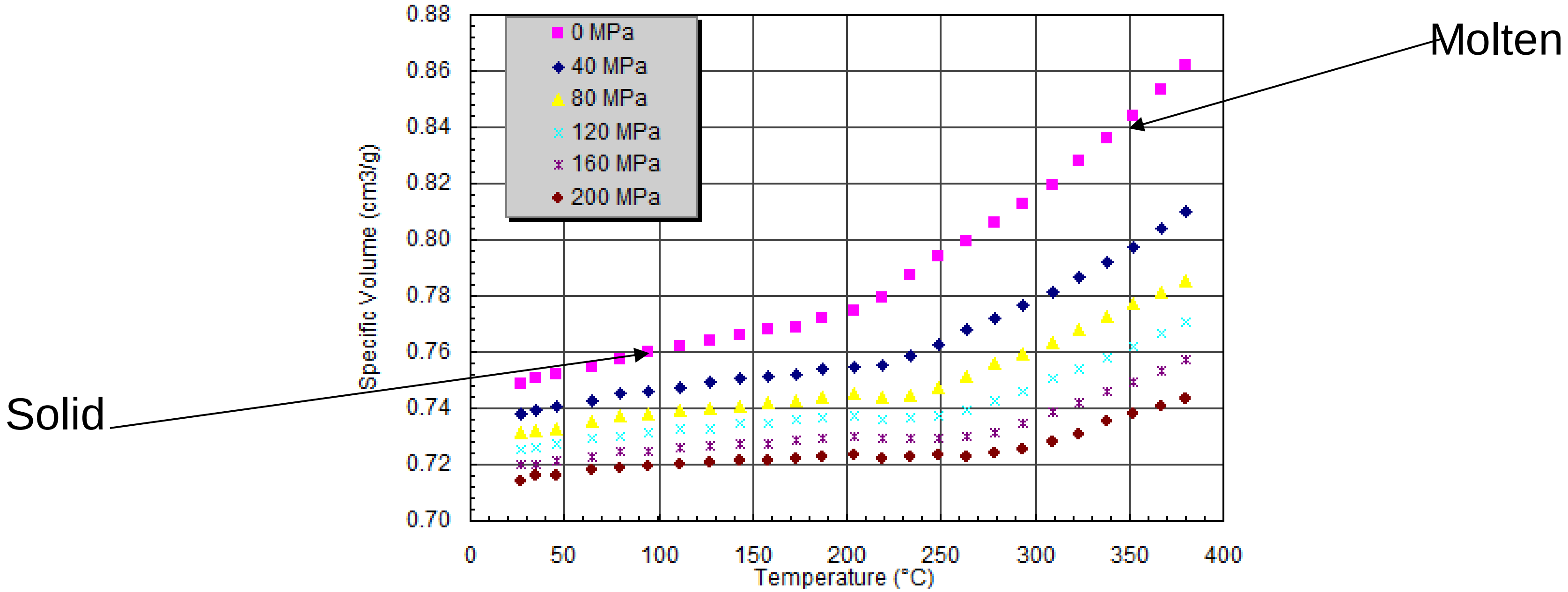






PVT test data

- Amorphous material
  - Transition is not dependent on mode

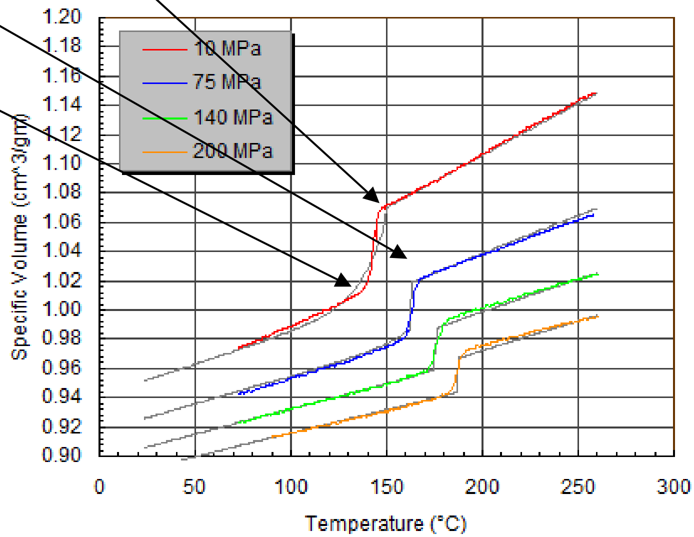


PVT modeling

- Two-domain Tait model
  - 13 parameters
  - Three groups of paramters
    - b5 is the transition of the low pressure
    - b6 is the slope of the transition
    - b7, b8, and b9 describe the shape of the crystalline transition

Two-Domain Tait PVT Model:

b5	4.202E+02	K
b6	2.000E-07	K/Pa
b1m	1.081E-03	m^3/kg
b2m	7.707E-07	$m^3/kg•K$
b3m	6.864E+07	Pa
b4m	3.209E-03	1/K
b1s	1.011E-03	m^3/kg
b2s	4.442E-07	$m^3/kg•K$
b3s	1.397E+08	Pa
b4s	1.752E-03	1/K
b7	7.064E-05	m^3/kg
b8	8.027E-02	1/K
b9	4.311E-08	1/Pa

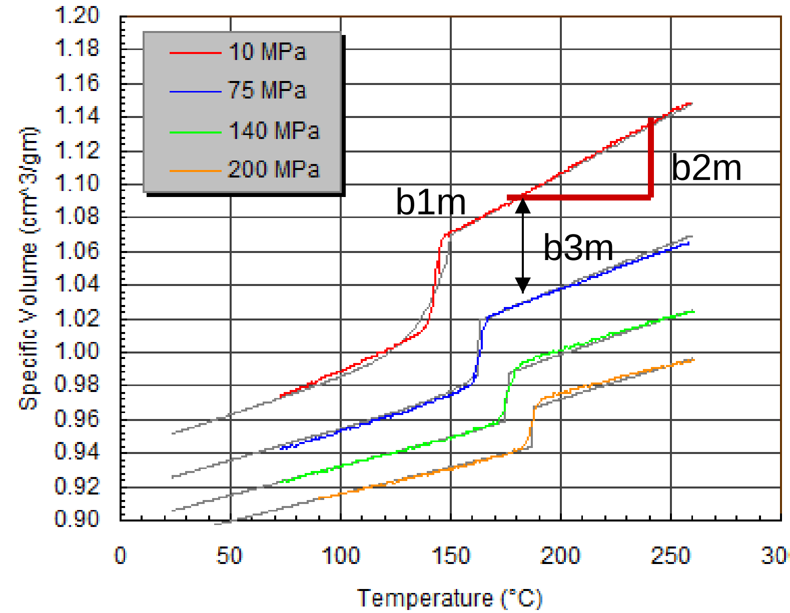


PVT modeling, cont.

- b1m is the specific volume at b5
- b2m is the slope of the melt region
- b3m is the pressure sensitivity or spread of the melt fit
- b4m is the pressure sensitivity of the melt state slope
- b1s through b4s are the same but for the solid state

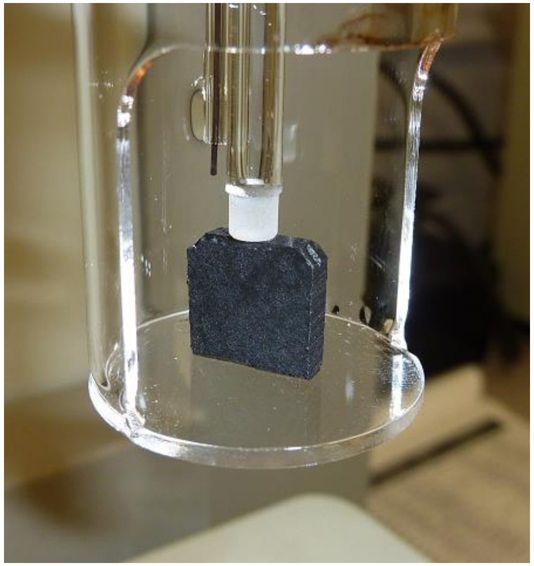
Two-Domain Tait PVT Model:

b5	4.202E+02	K
b6	2.000E-07	K/Pa
b1m	1.081E-03	m^3/kg
b2m	7.707E-07	$m^3/kg \cdot K$
b3m	6.864E+07	Pa
b4m	3.209E-03	1/K
b1s	1.011E-03	m^3/kg
b2s	4.442E-07	$m^3/kg \cdot K$
b3s	1.397E+08	Pa
b4s	1.752E-03	1/K
b7	7.064E-05	m^3/kg
b8	8.027E-02	1/K



Thermal expansion

- TMA (thermo-mechanical analyzer)
  - 10 x 10 mm x thickness plaques
  - Low expansion quartz probe and station
  - Constant heating rate
  - Slope of δL over temperature
- Orientation
  - One direction for no fiber
  - Two directions for fiber-filled



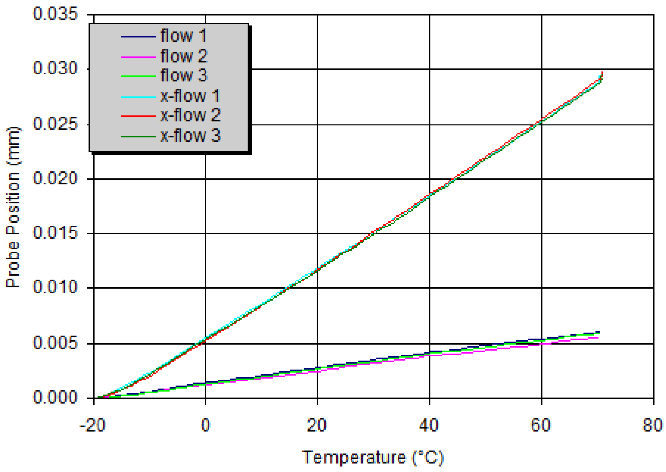
Thermal expansion test data

- Data presented as calculated slopes that are constant over the test range
  - Plot of probe position vs. temperature ensures linear relationship
- Anisotropic materials
  - Measurements across the flow always higher
  - Fibers have less thermal expansion than polymer

CLTE

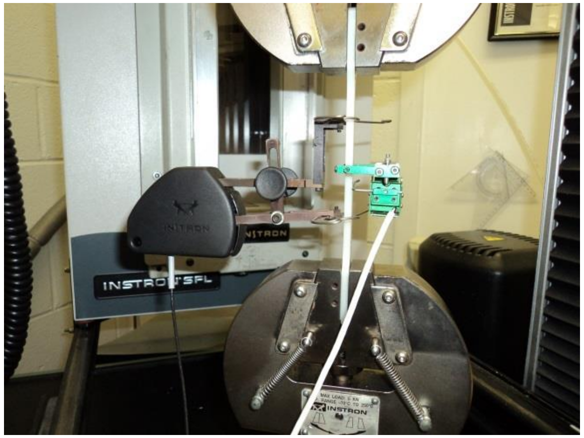
flow direction (α1)	
	0° to 60°C
replicate 1	6 x 10-6 / °C
replicate 2	6 x 10-6 / °C
replicate 3	6 x 10-6 / °C
average	6 x 10-6 / °C

cross-flow direction (α2)	
	0° to 60°C
replicate 1	34 x 10-6 / °C
replicate 2	33 x 10-6 / °C
replicate 3	31 x 10-6 / °C
average	33 x 10-6 / °C



Mechanical properties

- Tensile tests performed on a UTM
  - Temperature chamber
  - Axial and transverse strains
- Measure only the unfilled polymer
  - Fibers added in with the micro-mechanical model
- Stress strain curves at multiple temperatures
  - Modulus (σ/ε)
  - Poisson's ratio ($\varepsilon 2/\varepsilon 1$)
  - Viscoelastic properties

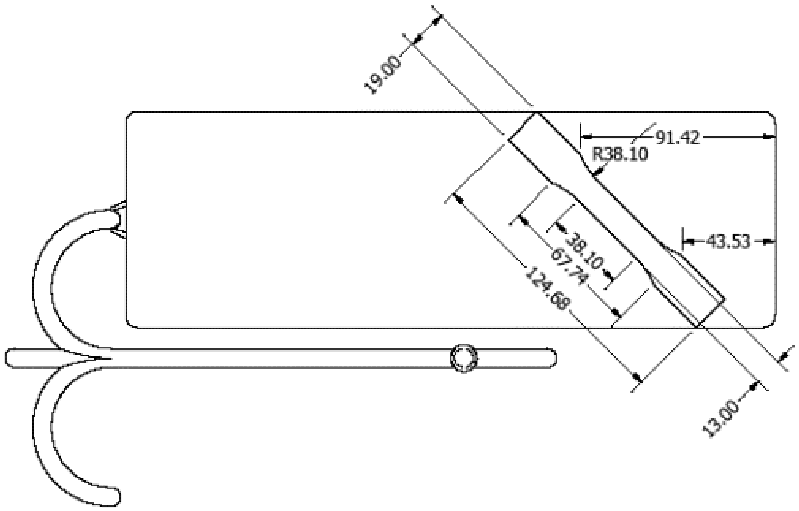


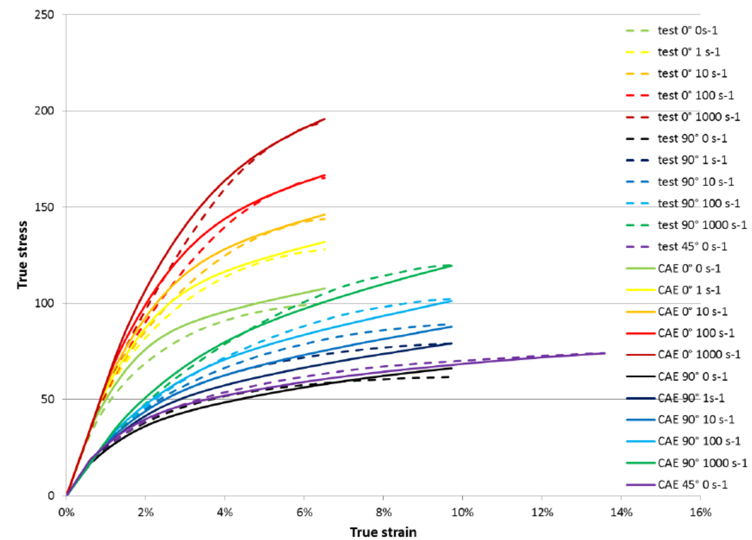
Problematic Materials

- Moisture sensitive materials
  - Improperly dried materials cause reduction in viscosity
  - Over-dried materials cause a rise in viscosity
  - PET, PA, PC, PBT etc.
- Highly filled materials
  - Can “log jam” the die entrance
  - Special dies must be used
  - Higher scatter in test data requires engineering judgment on behavior
- Thermally unstable materials
  - Require very careful attention to residence times
  - PVC

Basic DIGIMAT MX *TestPak* protocol

- Mold 100 X 300 X 3.16mm plaques
  - Edge gated on 100 mm end
  - Long flow length
  - Fully developed flow
  - Highly fiber orientation
- Cut test specimens by CNC
- 5 specimens each (0°, 90°, other orientations…)
- Obtain true stress-strain data
- Calibrate material model





DIGIMAT MX *TestPak* outputs

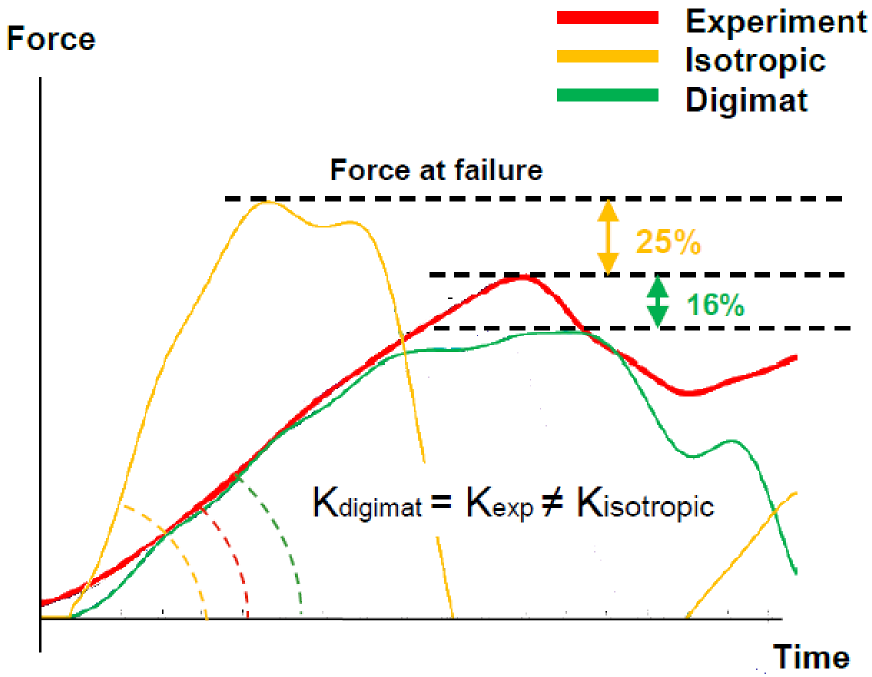
- CAD drawings of plaque and specimens
- Plaque molding conditions
- Injection molding material file
- True stress strain data (M-204) at 23°C
  - 0°, 90° orientation from plaque

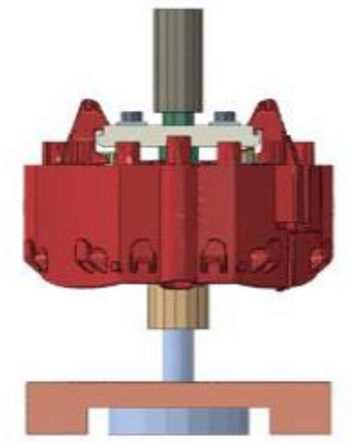
- DIGIMAT MX reverse engineering
  - Data is ready for FEA

DIGIMAT *TestPak* options

- Additional directions (10°, 20°, 45°)
- Thermomechanical (from –40° to 150°C)
- Strain-rate dependent (0.01 to 100/s)
- 3-point bend data (quasi-isotropy)
- Tensile bar data (coarse fit)
- Viscoelasticity
- Low cycle fatigue (Lemaitre-Chaboche)
- High cycle fatigue (under development)

Example: airbag housing





Source:e-Xstream

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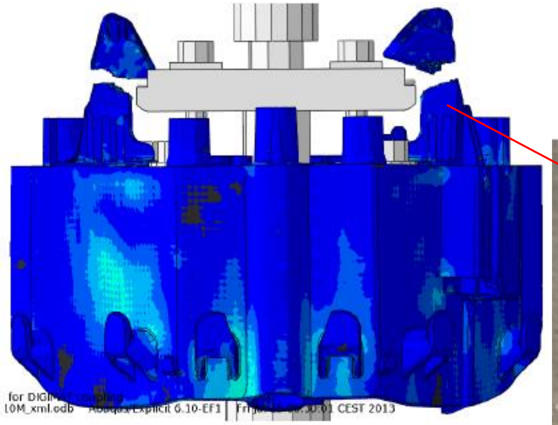
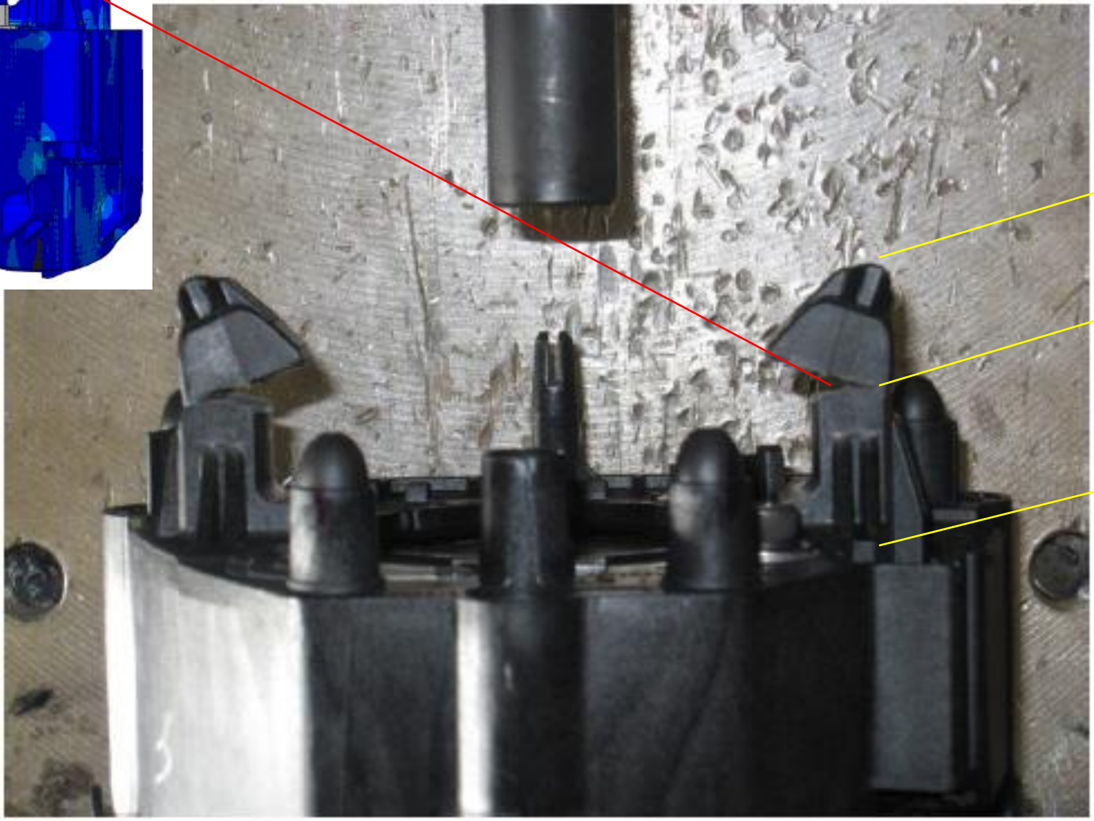
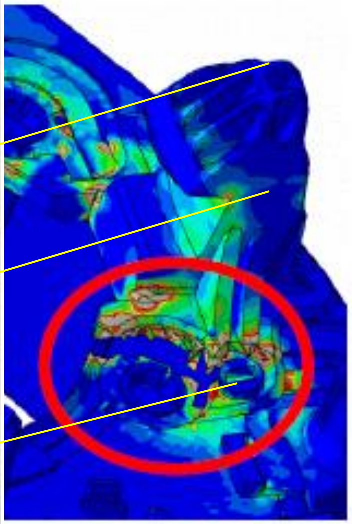
Impact on failure



With Fiber Orientation





Isotropic

Source:e-Xstream

Autoliv

Choosing the right properties

- Properties change over product operational temperature
- Properties change with environmental exposure
- Orientation (fiber-filled plastics)
- Viscoelastic (time-based behavior)
  - Creep
  - Stress relaxation
- Effect of strain rate