Applus+ DatapointLabs

Material Testing for SIGMASOFT

Douglas McMullen, Internal Sales Manager, Applus DatapointLabs

TestCart

TestPaks

CAETestBench

EST 1995

ISO 17025 Quality

Introduction to Applus

- **€1.778 billion total** revenue (2019)
- **23,000 staff** (2019)
- Present in more than **70 countries** across all continents
- Applus+ was on the Madrid Stock Exchange in May 2014

LABORATORIES DIVISION

- Testing
- Engineering
- Products & Systems Certification
- Multidisciplinary Laboratories

IDIADA DIVISION

- Design & Engineering
- Testing
- Homologation services
- Proving ground

Product Development & Validation

AUTOMOTIVE DIVISION

- Statutory vehicle inspection services for safety and emissions

Vehicle In-Service

ENERGY & INDUSTRY DIVISION

- Industrial and environmental inspection
- Technical assistance
- Non-destructive testing (NDT)
- Technical staffing

Asset design, construction and operation

Laboratories Division- Scope of Services

Testing Services

Structural Testing

- Highest load Test accredited for Aero sector as independent Lab
- Fully accredited & mainly focus in Aeronautical sector

Materials Characterization & Quality Assurance

- 6 Material Testing Laboratories in Europe, USA & China
- Fully accredited Labs for Aeronautical Sector

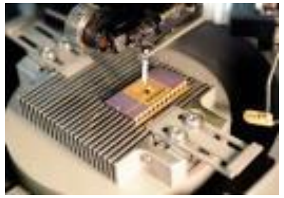
EMC & Wireless

- 3 State of the art laboratories in Europe

Fire Testing

- Full Scale Fire Laboratory in Barcelona

IT Security & Interoperability



- 3 Laboratories for product security evaluation and cybersecurity assessment in Spain and China

Metrology & Calibration

- Main focus in Spanish Local Market

Laboratories Division- Scope of Products

Applus DatapointLabs

TEST RIGS AND TEST BENCHES FOR DEVELOPMENT

Test Rigs & Tools

HILs/SILs Virtual Testing

Ground Test Equipment & Test Benches

HILs/SILs Virtual Testing

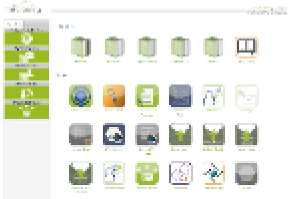
PRODUCTION EQUIPMENT

Robotic NDT Equipment

In-Line Measurement Machines

SOFTWARE SOLUTIONS

matereality
software for materials



e-Testing

Applus+ Laboratories Division Sites

Applus+ DatapointLabs

Spain | Head Quarters

Multi-technological Testing Laboratories
Engineering & Manufacturing
Several Sites in Spain
Aerospace, Automotive, IT & Industry

NDT & NDT Equipment

USA | Punta Gorda (FL)
Automated NDT Equipment Manufacturing
Aerospace

USA | Thallassee (AL)
NDT inspection for composites
Aerospace

EMC Laboratories

UK | Silverstone
Electrical & Electronics
Automotive & Industry

Italy | Amaro (Udine)
Electrical & Electronics
Automotive & Industry

Fire Test Labs

Spain | Asturias
Fire Safety in Tunnels
Infrastructures

Spain | Madrid
Product Fire Safety
Construction & Industry

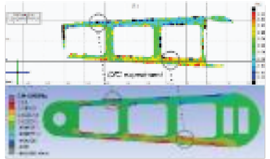
Spain | Barcelona
Product Fire Safety
Chemicals, Oil & Gas

Metrology & Calibration Labs

Spanish Network
Barcelona
Madrid
Navarra
A Coruña
Albacete
Sevilla
…

Mechanical Test Laboratories

France | St. Etiènne
Mechanical & Materials
Aerospace & industry



USA | Ithaca (NY)
Materials Testing for Simulation & CAE
Aerospace & Industry

Spain | Illescas
Mechanical & Materials
Aerospace

Germany | Bremen
Mechanical & Materials
Aerospace & Industry

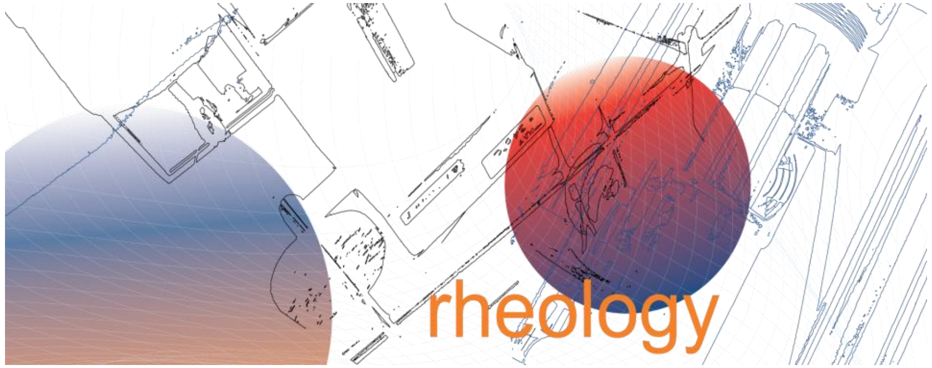
China | Shanghai
Mechanical & Materials
IT payment systems
Aerospace & IT

Norway | Bryne (Stavenger)
Mechanical & Materials
Oil & Gas

Thermoplastics

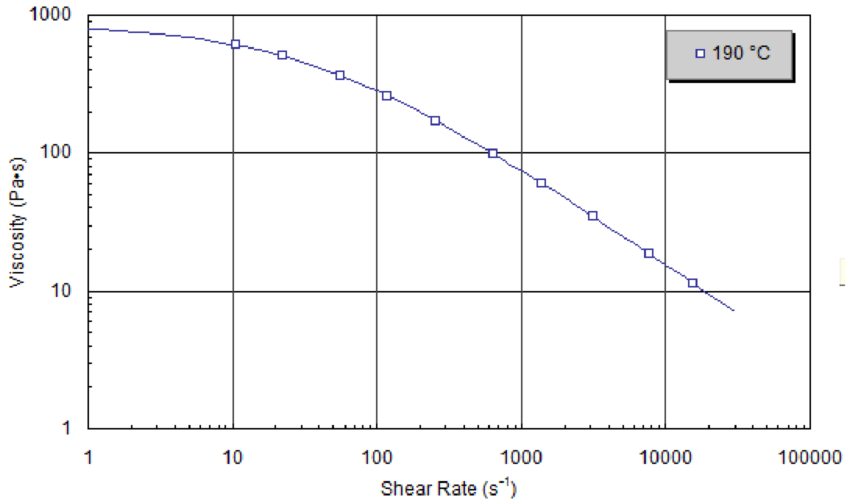
- Viscosity
- Specific Heat
- DSC Transition Temperature
- Thermal Conductivity
- PVT
- Linear Shrinkage
- Viscoelastic Properties

Rheology

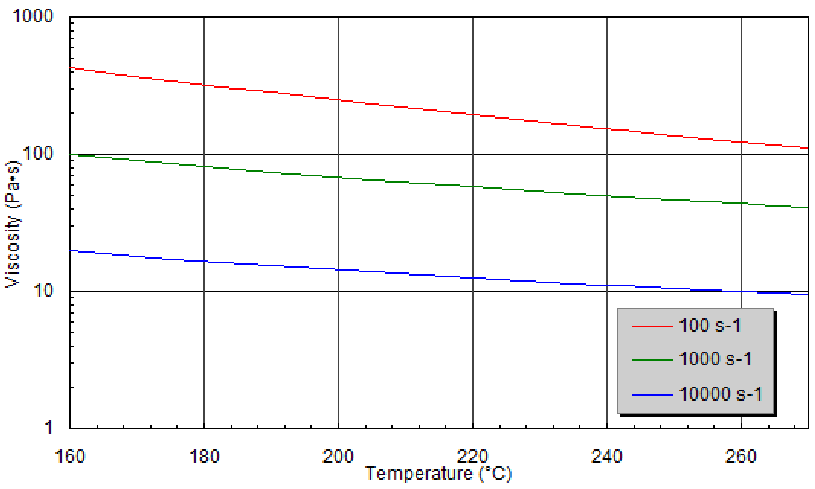


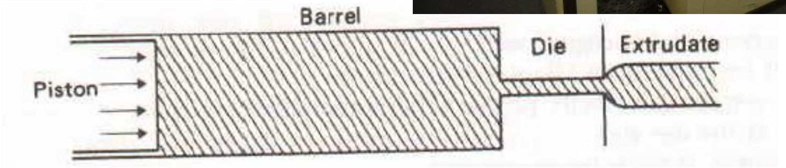
- Capillary viscosity
- Material is extruded through a restriction of known geometry (extremely high tolerance dies)
- Temperature and flow rate are controlled
- Pressure drop across the restriction is used to determine viscosity as a function of shear rate and temperature

- As shear rate increases, viscosity decreases

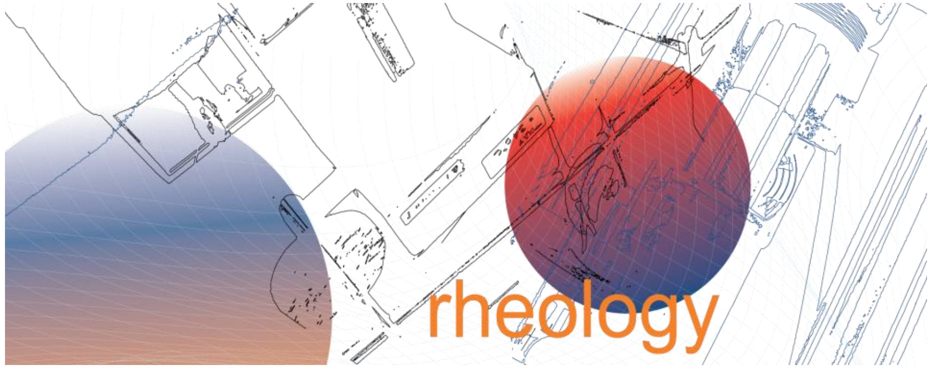


- As temperature increases, viscosity decreases





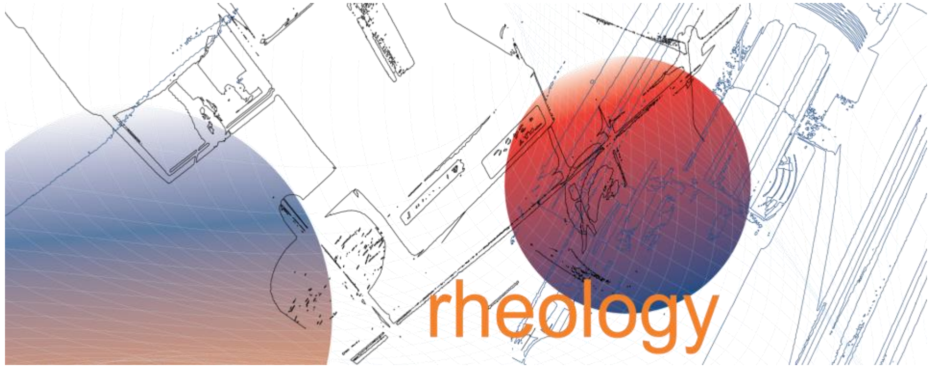
Rheology



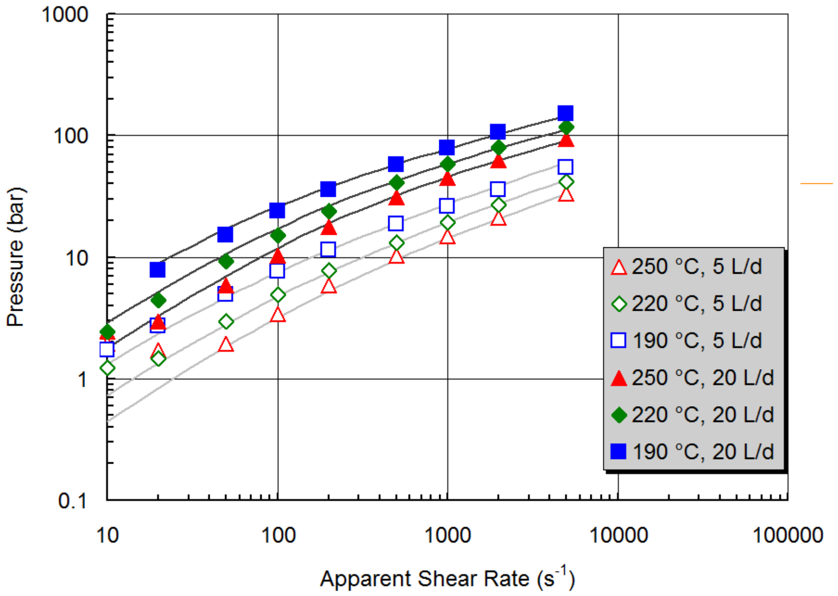
- Apparent Viscosity
- Shear rate: $\dot{\gamma}_a = \frac{32Q}{\pi d^3}$
  - Shear stress: $\tau_w = \frac{\Delta p d}{4L}$
  - Shear viscosity: $\eta_a = \frac{\tau_w}{\dot{\gamma}_a}$
- Corrections to viscosity
  - Reservoir and friction losses (transducer located at die)
  - End pressure drop (Bagley)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

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

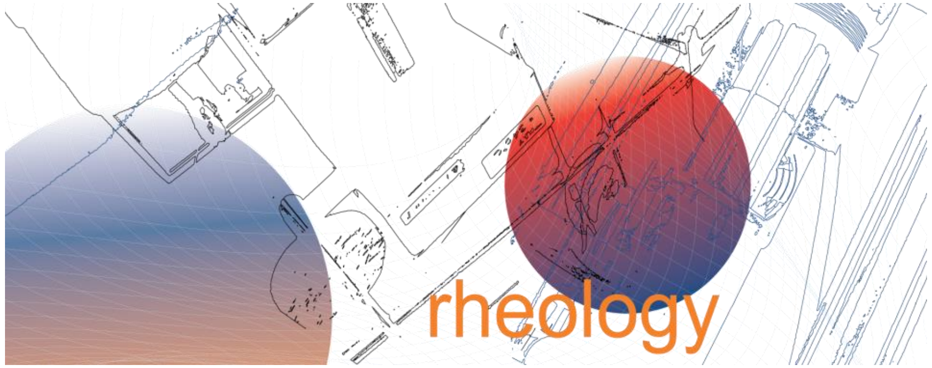
Rheology



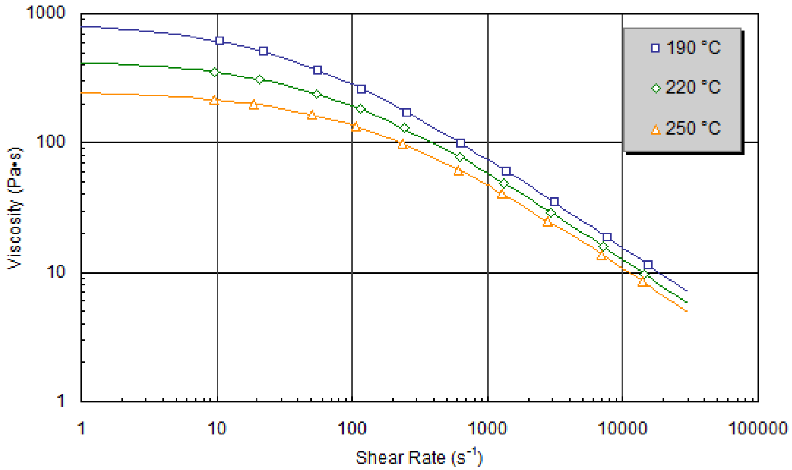
- Bagley Correction Testing
  - Perform viscosity measure on two different die ratios at equal shear rates
  - Evaluate pressure differences between die geometries (capillary diameter remains the same)
  - $\tau = R/2(dP/dL)$



Modeling



- 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



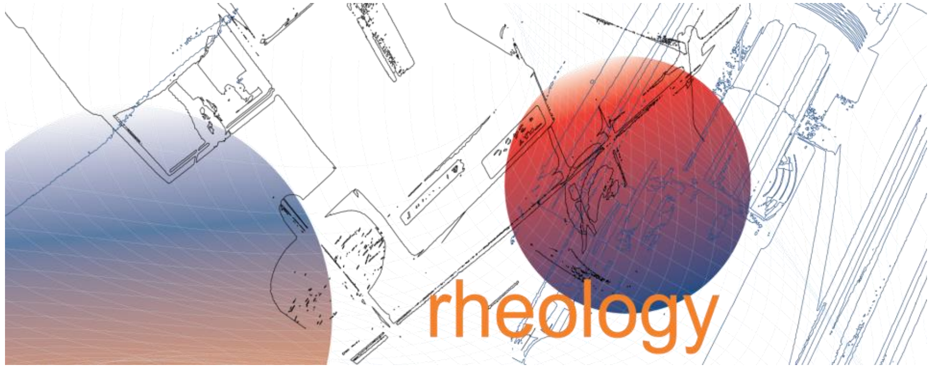
n	0.28400
τ*	32096.1
D1	3.86E+13
D2	263.15
A1	30.87
A2	51.6

- Evaluating Cross WLF Parameters
  - The parameters are tied to real physical behavior
  - N - measures shear thinning behavior
    - inverse of the power-law index
  - rules for N
    - 0 < N < 1
    - small N = shear sensitive
  - τ* is the critical transition stress for shear-thinning behavior
    - if τ* is large, wide Newtonian region
    - if τ* is small, narrow Newtonian region
    - τ* is small for simple linear polymers
      - eg HDPE, LDPE, PP
    - τ* is large for polymers with large side chains
      - eg. PC

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

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

Modeling



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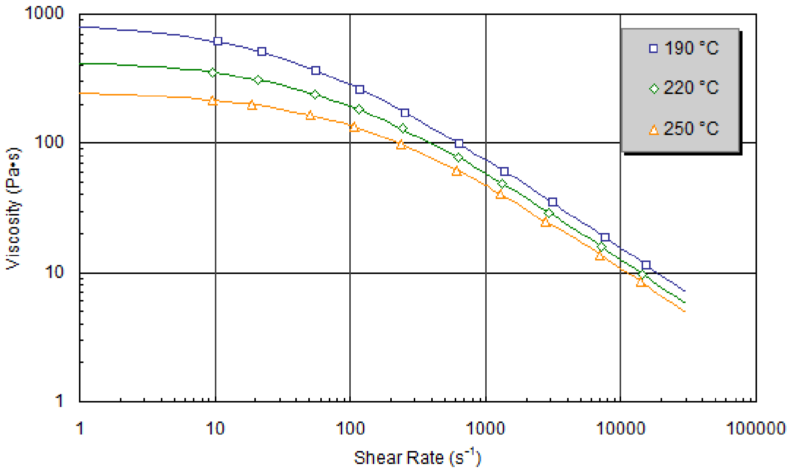
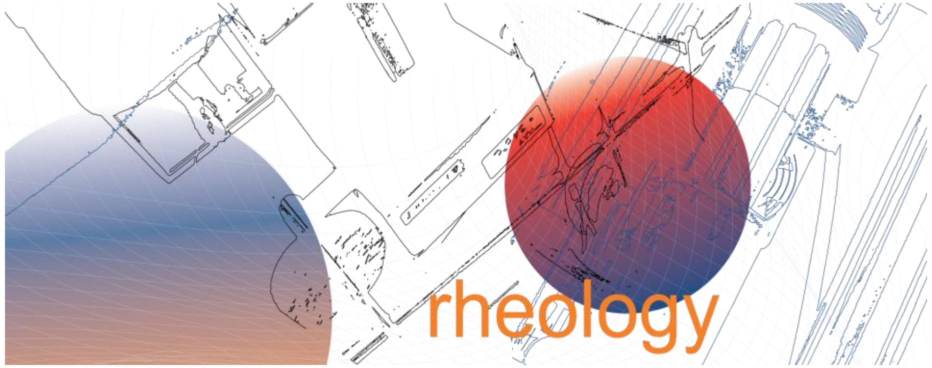


- Evaluating Cross WLF Parameters
  - D1 is coupled to the WLF temperature dependency equation
  - No direct relevance
  - D2 is the reference temperature
  - Theoretically where h goes to infinity
  - A1 & A2 - WLF parameters
  - A1 defines the temperature sensitivity of viscosity
  - A2 defines change in temperature sensitivity with temperature

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

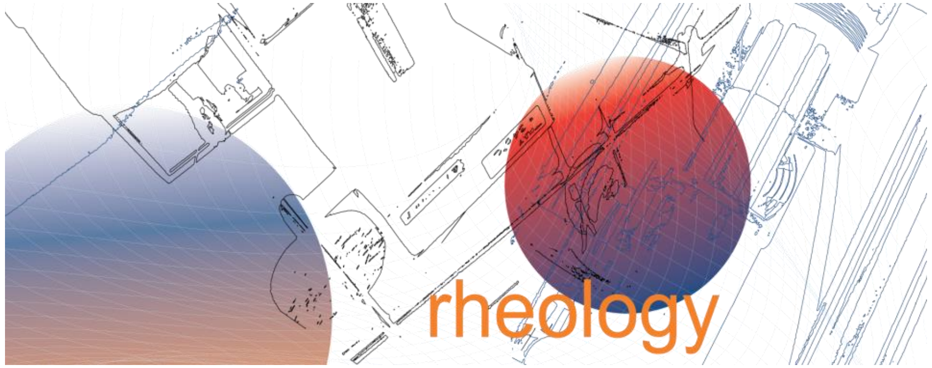
$$\eta_0(T) = D_1 \exp\left[-\frac{A_1(T-D_2)}{A_2+(T-D_2)}\right]$$

Considerations for Testing



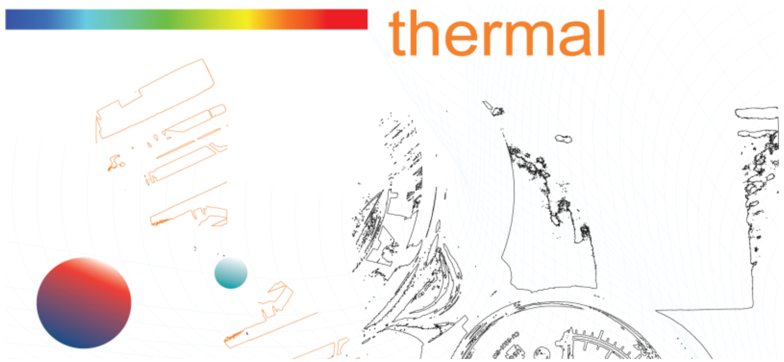
- Limited shear rates
  - Typically 10-10000 /s
  - Optional to go up to 100000 /s (uses smaller die)
- Residence times are longer in testing
  - Testing takes several minutes (approx. 6-10 min.)
  - Need to worry about thermal stability
- Processing temperatures are typically higher than test temperatures
- Typically testing is performed at two temperatures within the processing range and one below

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
  - Requires very careful attention to residence times
  - PVC

Thermal Testing



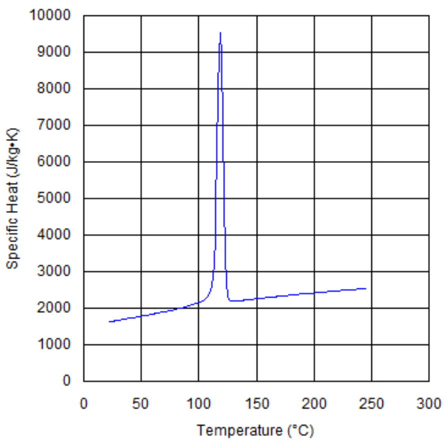
•Specific Heat

- 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

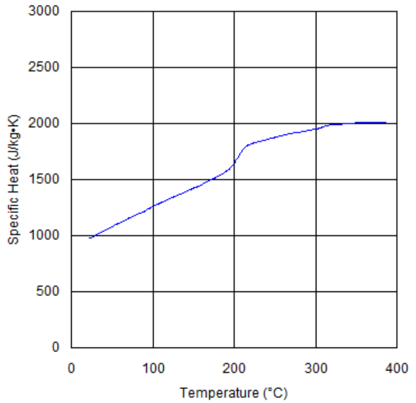
•Transition Analysis

- Semi-Crystalline materials show a peak in the specific heat curve
  - The peak is due to the addition heat needed to initiate crystallization
  - Due to thermal lag, transition temperatures measured in cooling mode will be lower than those measured in heating
  - The onset of the transition is set as the melting point to ensure complete melt of the polymer
  - The point at which the peak ends is set as the eject temperature
  - Beyond the eject temperature, no flow can take place
- Amorphous materials show a "knee" in the specific heat curve
  - The knee is the glass transition of the material, no crystallization takes place
  - The onset of the transition is set as the melting point to ensure complete melt of the polymer
  - In this case the inflection point of the knee is taken as the eject temperature
  - Beyond the eject temperature, no flow can take place

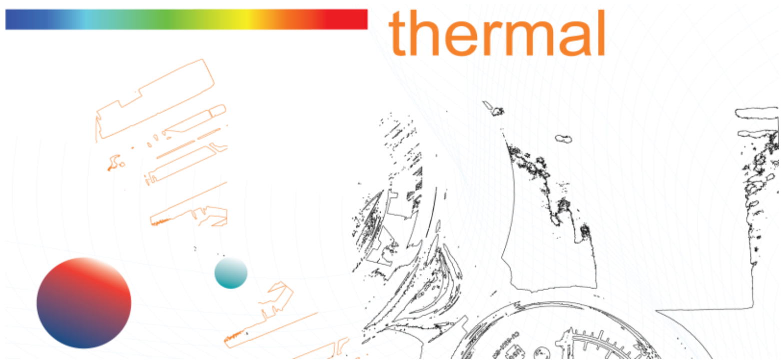
- Semi-Crystalline



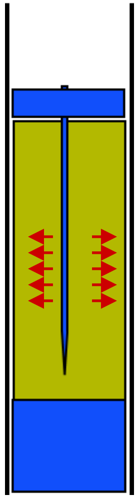
- Amorphous



Thermal Testing

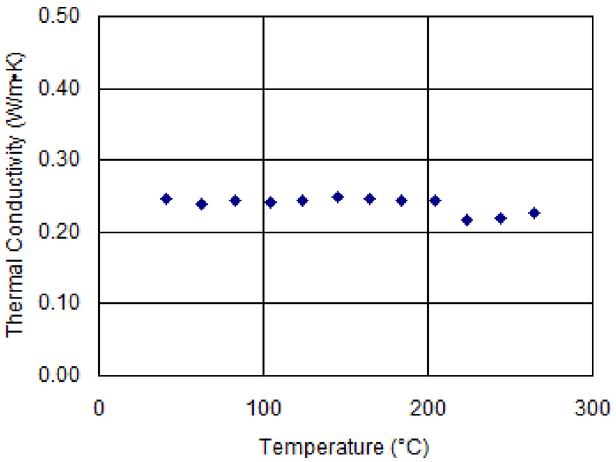


- Measure time to dissipate the heat pulse away from probe

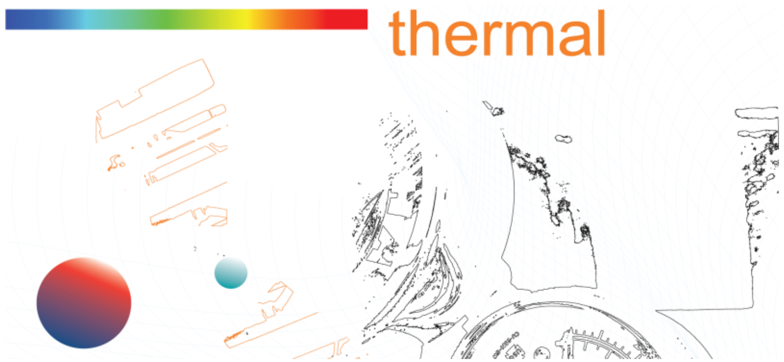


•Thermal Conductivity

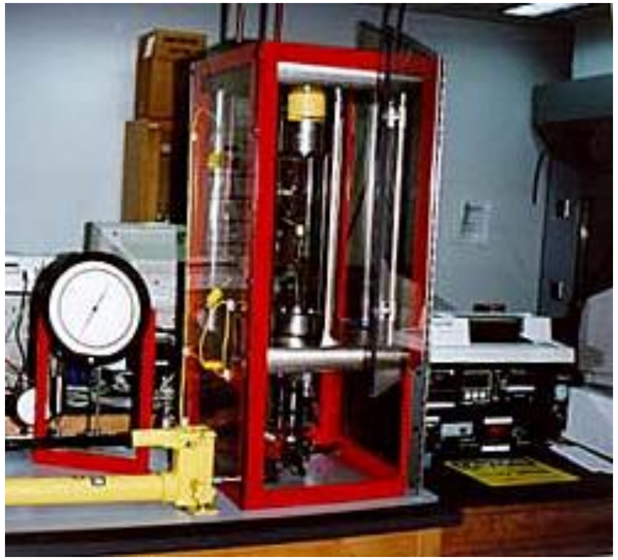
- A measure of how well a material transfers heat
  - Measured using transient line source
  - Measured in melt and solid state
  - Different behaviors for semi-crystalline and amorphous
  - Semi-crystalline materials show an increase in thermal conductivity in solid state
  - Amorphous materials show 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

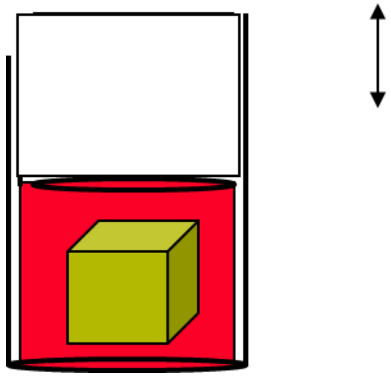


PVT (pressure, volume and temperature)



- Isobaric cooling (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
- 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 (pressure, volume and temperature)

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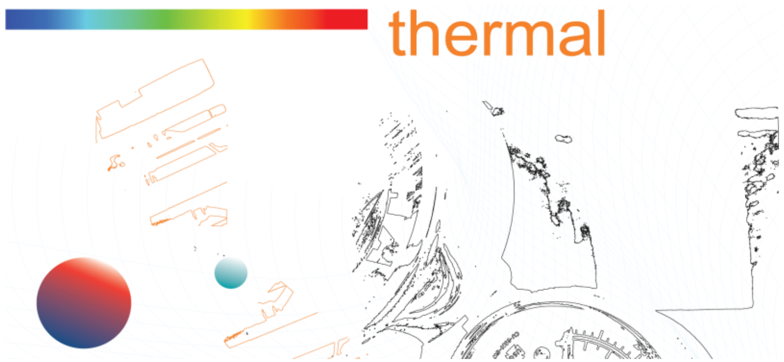
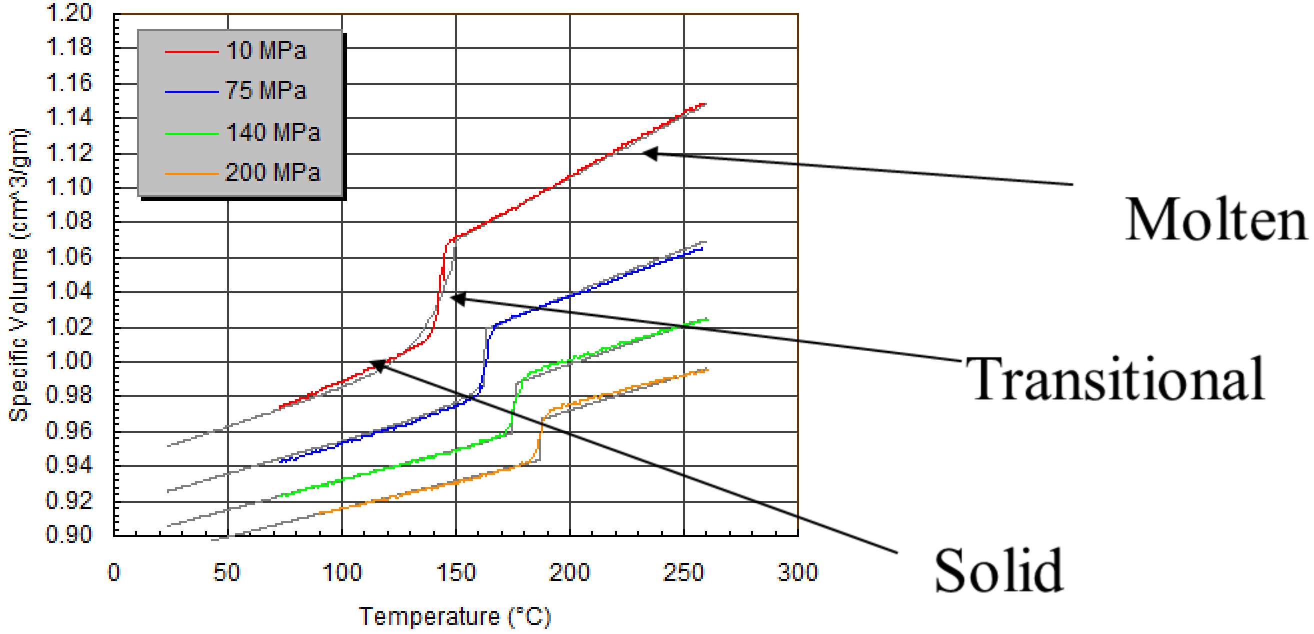
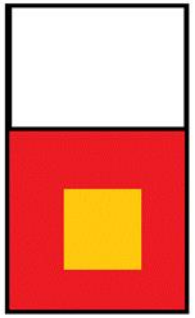
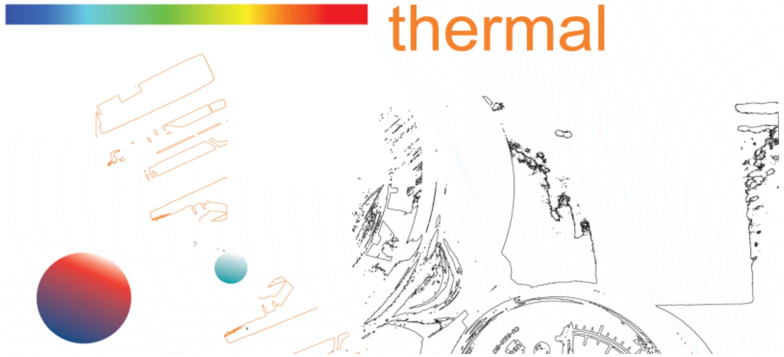


- Semi-Crystalline material
  - Transition region is critical
  - Rise in temp. = rise in spec. vol.
  - Rise in press. = drop in spec. vol.

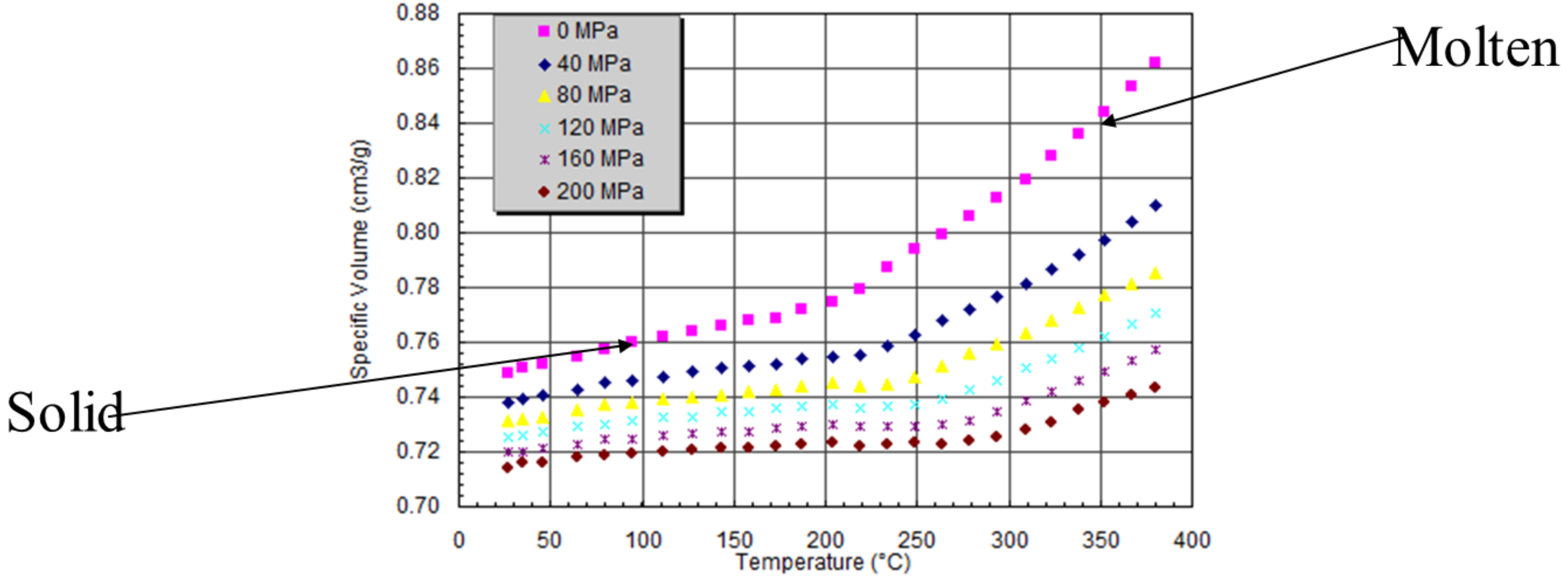




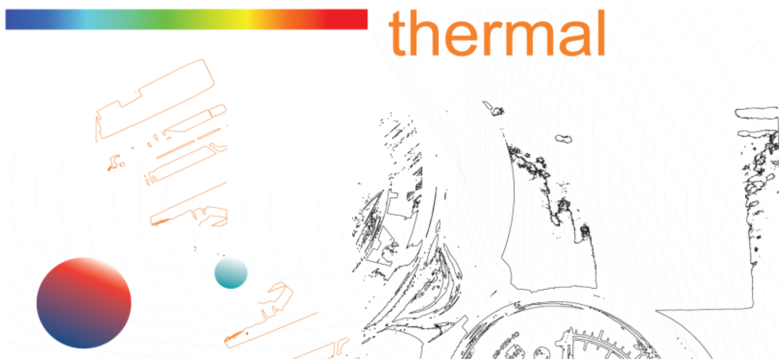
PVT (pressure, volume and temperature)



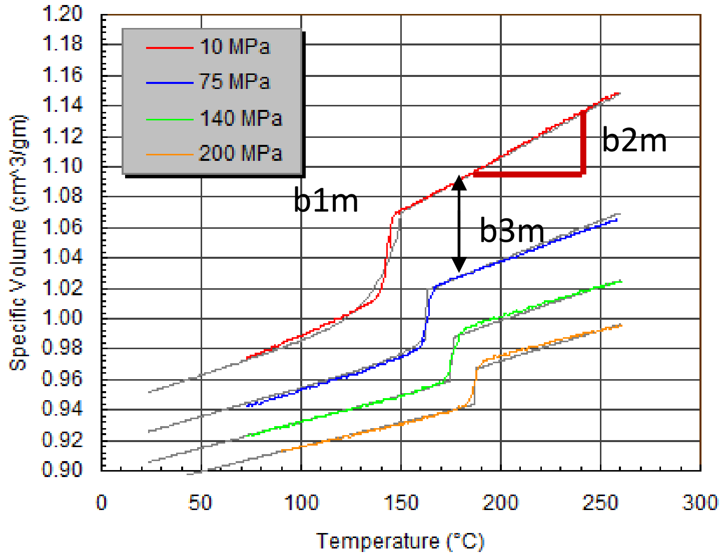
- Amorphous material
- Transition is not dependent on mode



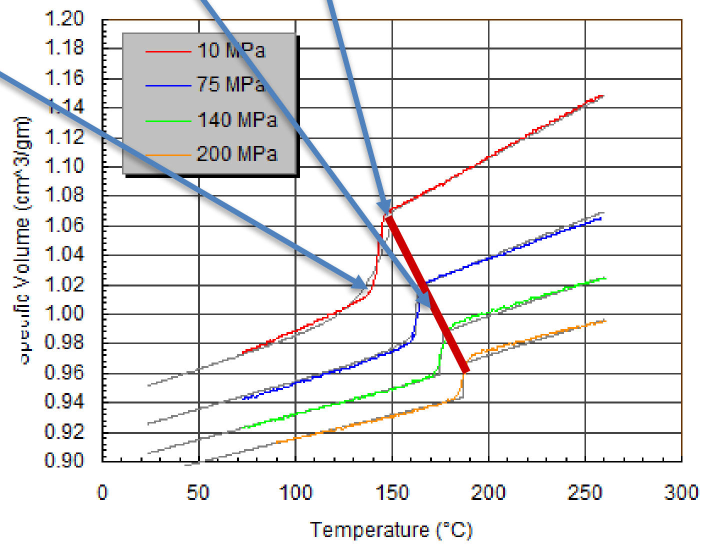
Modeling



- Two domain Tait model
  - 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



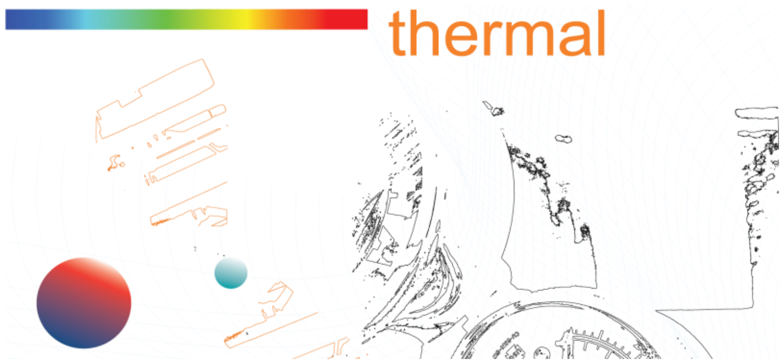
- Two domain Tait model (transition region)
  - 13 parameters
  - Three groups of parameters
    - b5 is the transition of the low pressure
    - b6 is the slope of the transition
    - b7, b8, and b9 describe the shape of the crystalline transition



Two-Domain Tait PVT Model:

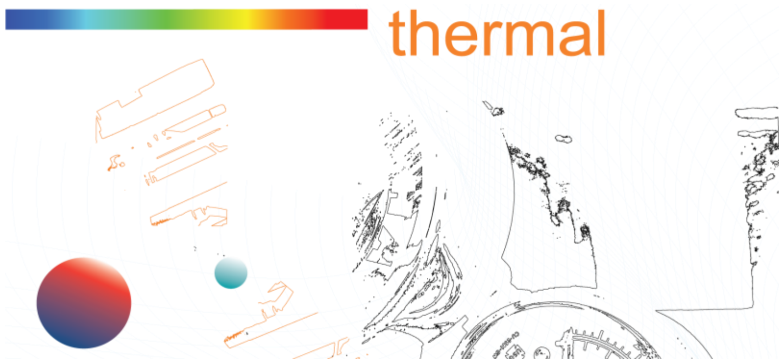
b5	4.202E+02	K
b6	2.000E-07	K/Pa
b1m	1.081E-03	m^3/kg
b2m	7.707E-07	m^3/kg•K
b3m	6.864E+07	Pa
b4m	3.209E-03	1/K
b1s	1.011E-03	m^3/kg
b2s	4.442E-07	m^3/kg•K
b3s	1.397E+08	Pa
b4s	1.752E-03	1/K
b7	7.064E-05	m^3/kg
b8	8.027E-02	1/K
b9	4.311E-08	1/Pa

Problematic Materials

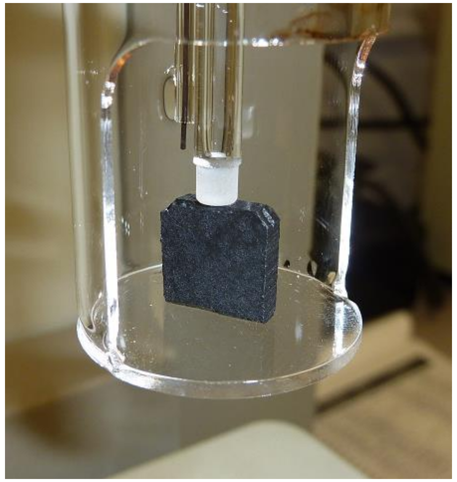


- Thermally unstable materials
- Materials that have voids
- Very high melting point materials
  - Limitation of machine is 400°C
  - Mercury boils at 356°C under atmospheric conditions (test at minimum of 10 MPa)
  - PEI, PAEK

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

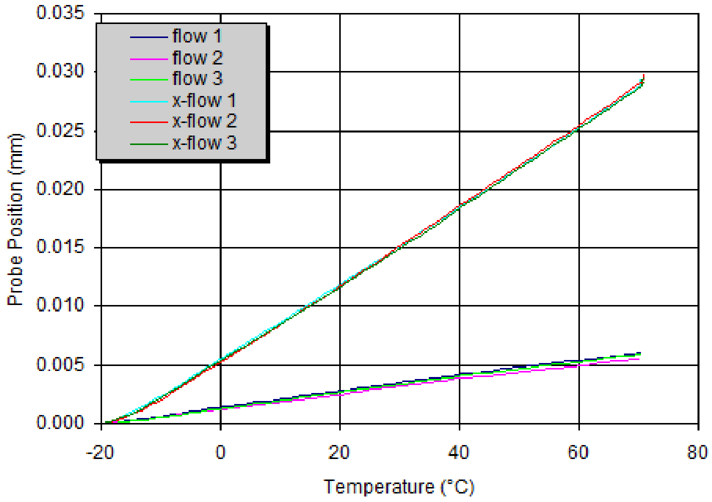


- Data presented as calculated slopes that are constant over the test range
- Plot of probe position vs. temperature ensures linear relationship
- Anisotropic materials
- Measurements across the flow always higher
- Fibers have less thermal expansion than polymer

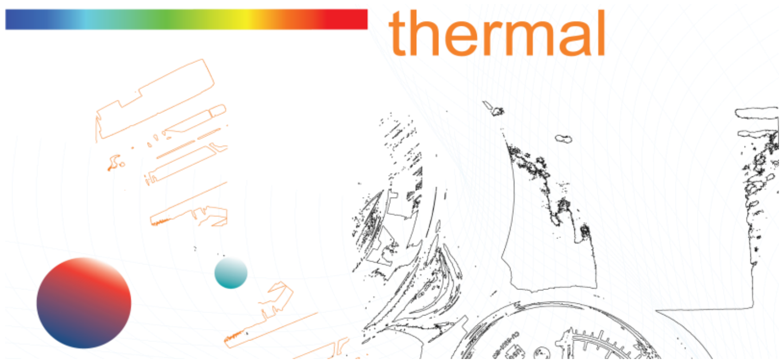
CLTE

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

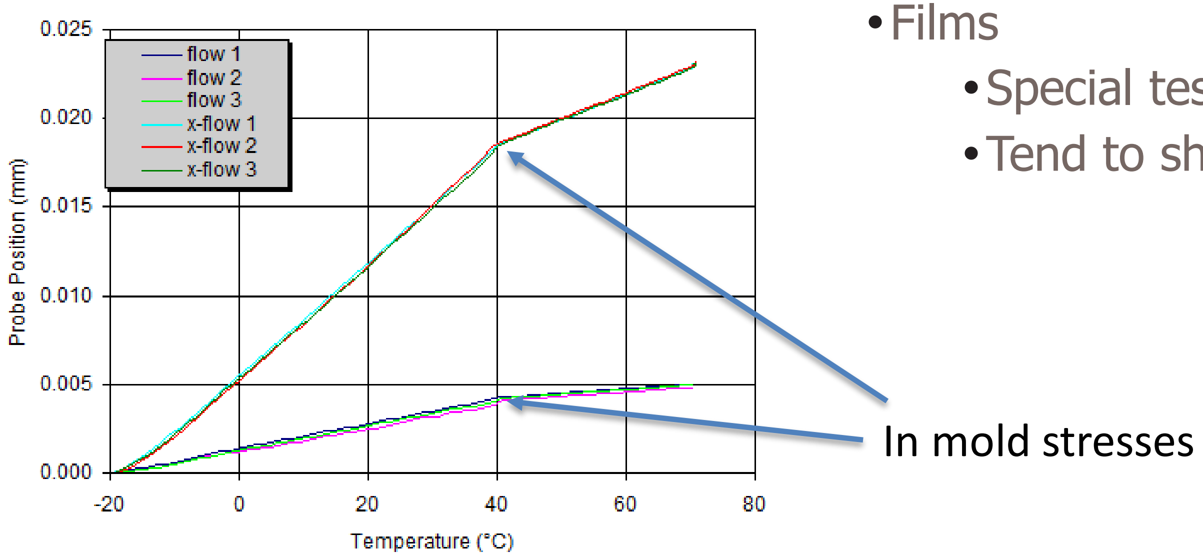
cross-flow direction (α2)	
	0° to 60°C
replicate 1	34 x 10-6 / °C
replicate 2	33 x 10-6 / °C
replicate 3	31 x 10-6 / °C
average	33 x 10-6 / °C



Problematic Materials

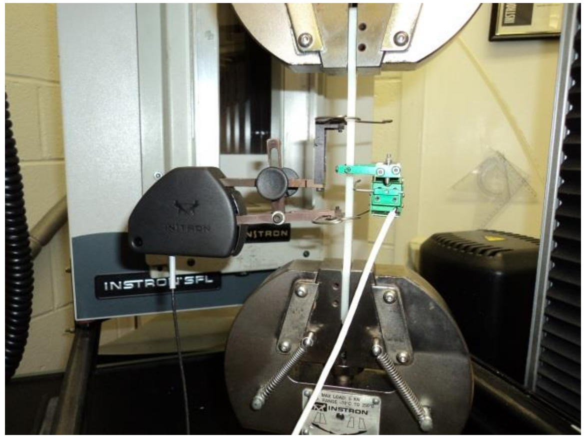


- Continuous fiber materials
  - Test probe sits directly on the fibers that have similar CLTE to probe
- Residual stress after molding
  - Require additional annealing operation to alleviate stresses
- Very soft materials
  - Probe penetrates sample
- Films
  - Special test methods are required
  - Tend to show shrinkage due to processing method

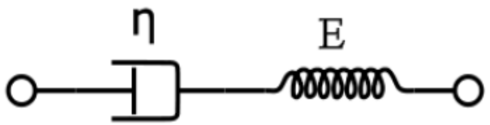


Mechanical Testing

- Tensile tests performed on a UTM
  - Temperature chamber
  - Axial and transverse strains
- Stress strain curves at multiple temperatures
  - Modulus (σ/ε)
  - Poisson's ratio (ε2/ε1)
  - Viscoelastic properties

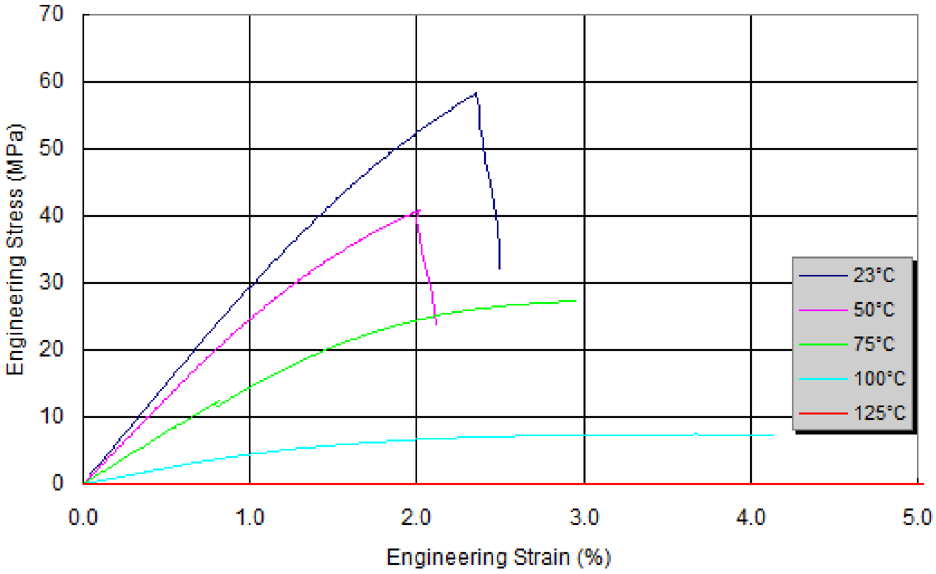


- Only valid for unfilled materials
- Performed at constant strain rate
  - Converted to relaxation times
  - Multi-temperature allows for temperature dependent relaxation



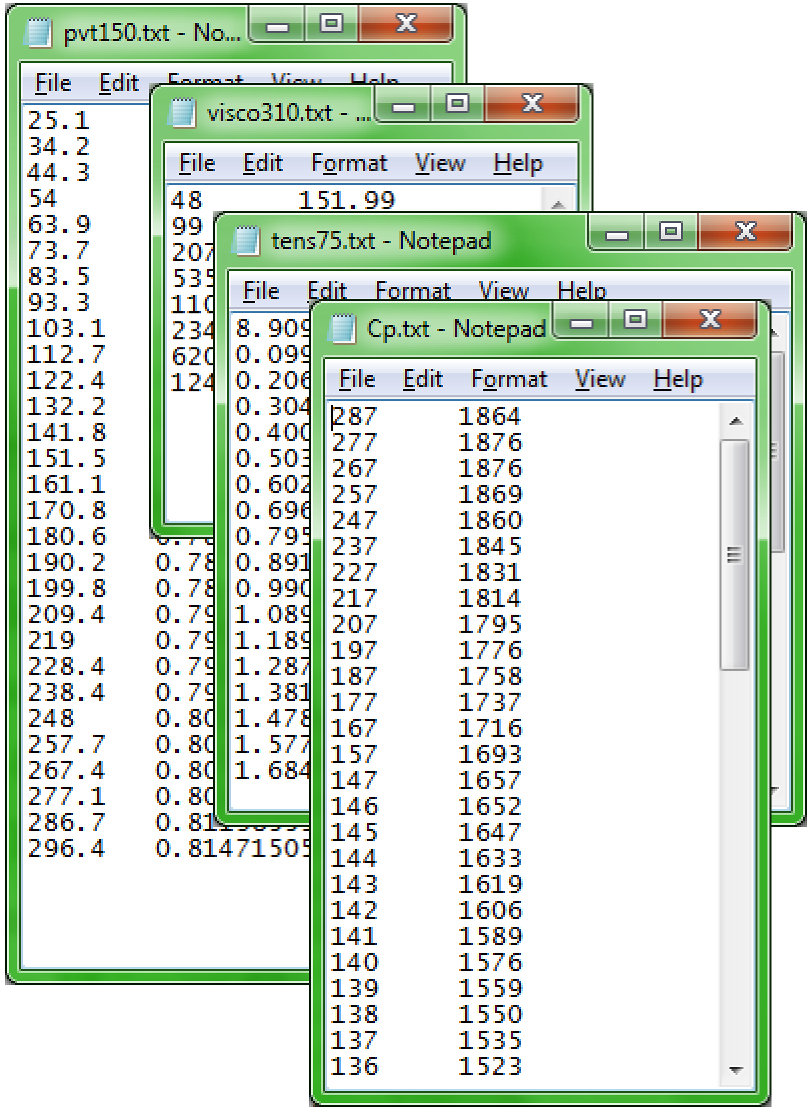
Properties

Temp. °C	Modulus E MPa	Poisson's Ratio
23	3010	0.372
50	2564	0.390
75	1555	0.426
100	480	0.465
125	2	0.480



Implementation into SIGMASOFT

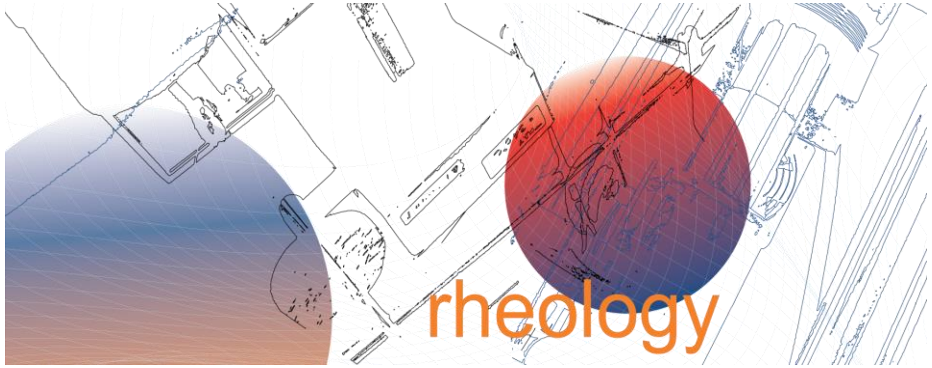
- X-Y pairs in text files
- Viscosity (3)
- Specific heat (1)
- Thermal conductivity (1)
- PVT (4-6)
- CTE (1)
- Mechanicals (5)
- At least 17 input files



Thermoset/Rubber

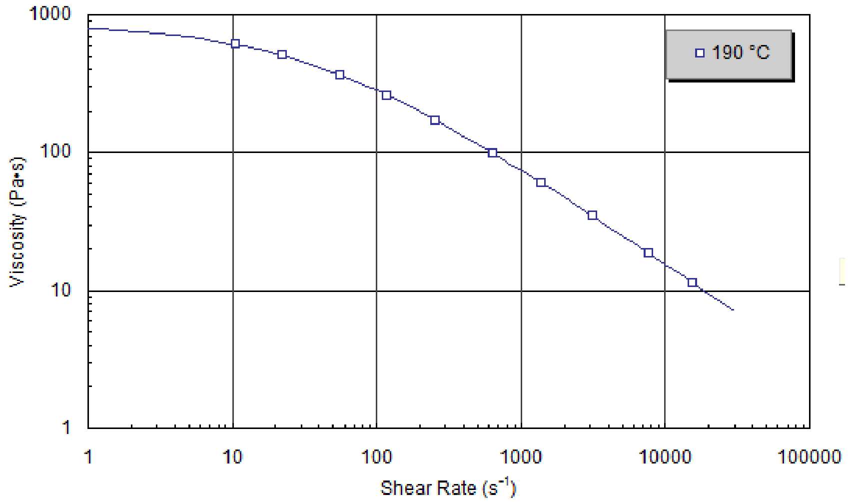
- Viscosity
- Curing Viscosity
- Specific Heat
- Thermal Conductivity
- PVT

Rheology

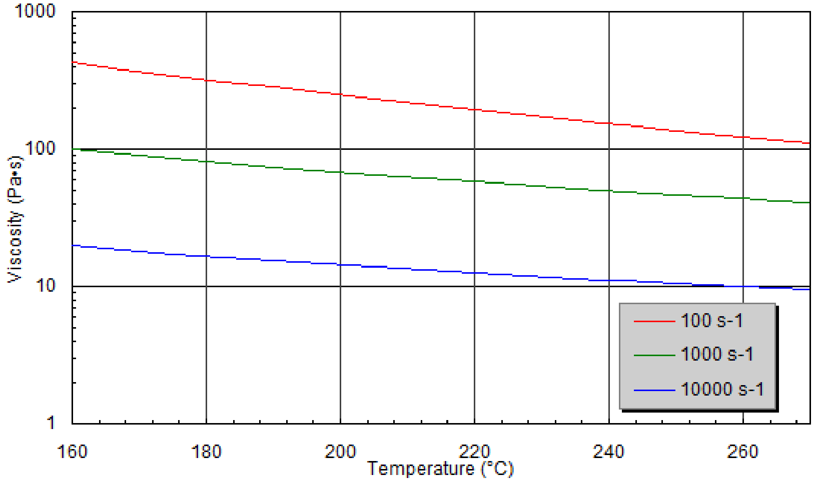


- Capillary viscosity for rubber
- Parallel plate rheometer for liquids
- Material is extruded through a restriction of known geometry (extremely high tolerance dies)
- Lower plate oscillates while top records torque
- Must be done on non-curable material or well below cure temperature

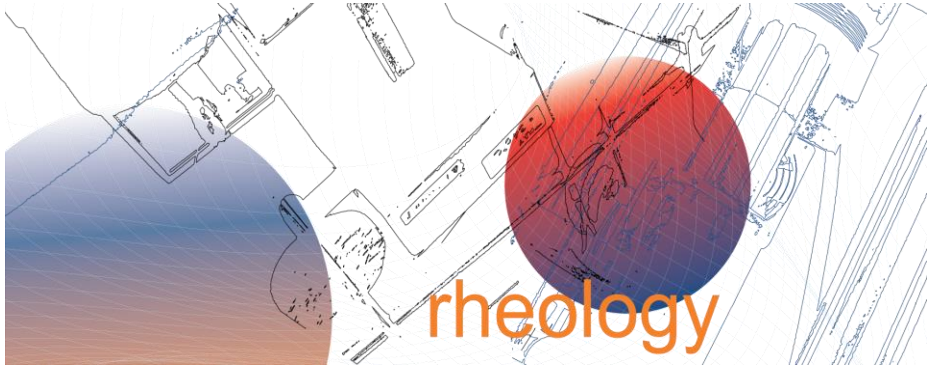
- As shear rate increases, viscosity decreases



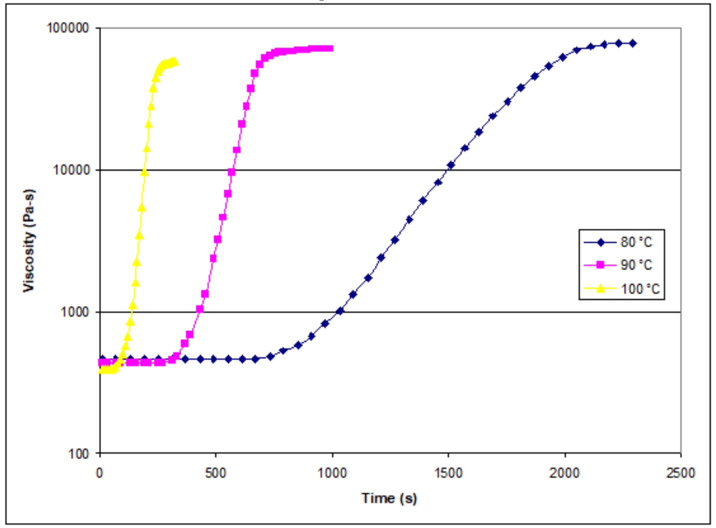
- As temperature increases, viscosity decreases



Rheology (Curing)

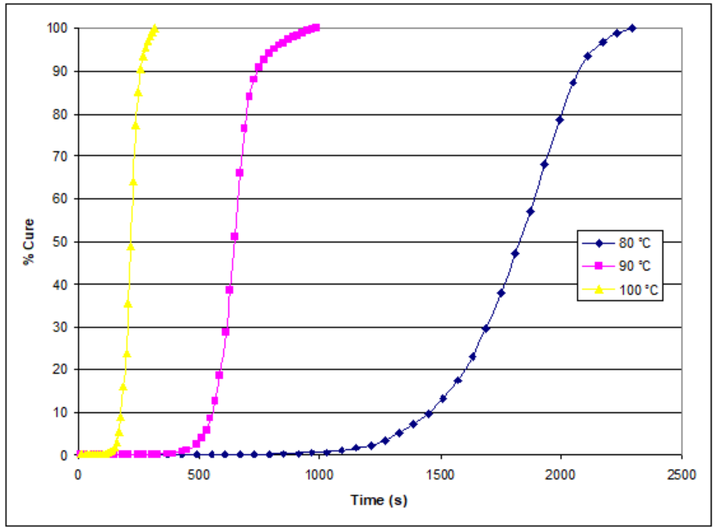


• Viscosity vs. Time

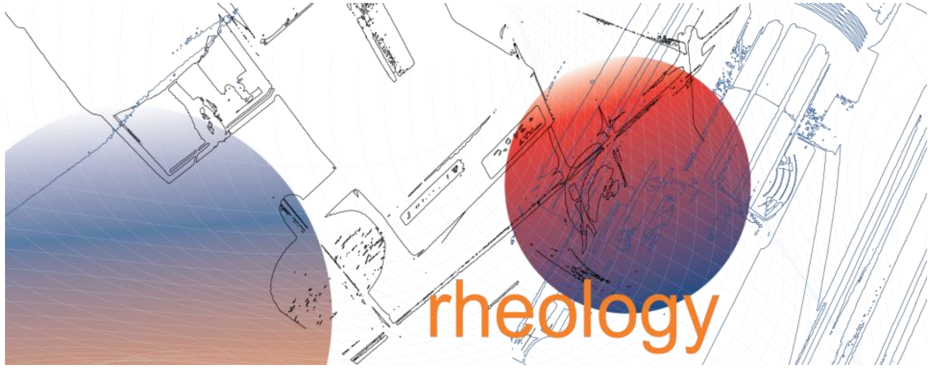


- Capture viscosity vs. temperature and time
- Isothermal time sweeps
  - 3 temperatures
  - Continue until plateau
- Calculate % cure relative to initial viscosity
- May have to eliminate initial negative slope
  - Can't have negative cure

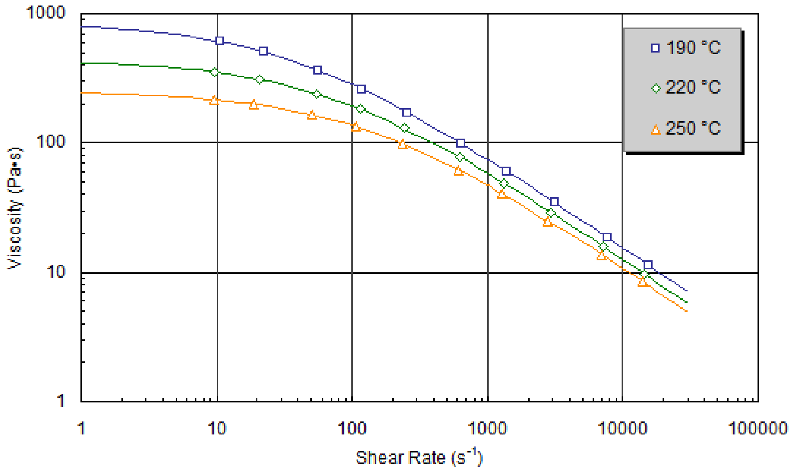
• Cure vs. Time



Modeling



- 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



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

- Evaluating Cross WLF Parameters
  - The parameters are tied to real physical behavior
  - N - measures shear thinning behavior
    - inverse of the power-law index
  - rules for N
    - 0 < N < 1
    - small N = shear sensitive
  - τ* is the critical transition stress for shear-thinning behavior
    - if τ* is large, wide Newtonian region
    - if τ* is small, narrow Newtonian region
    - τ* is small for simple linear polymers
      - eg HDPE, LDPE, PP
    - τ* is large for polymers with large side chains
      - eg. PC

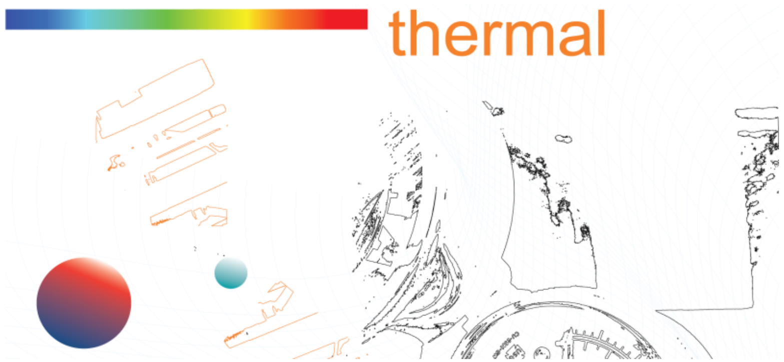
$$\eta(T,\dot{\gamma}) = \frac{\eta_0(T)}{1+\left(\frac{\eta_0\dot{\gamma}}{\tau^*}\right)^{1-n}}, \textit{where}$$

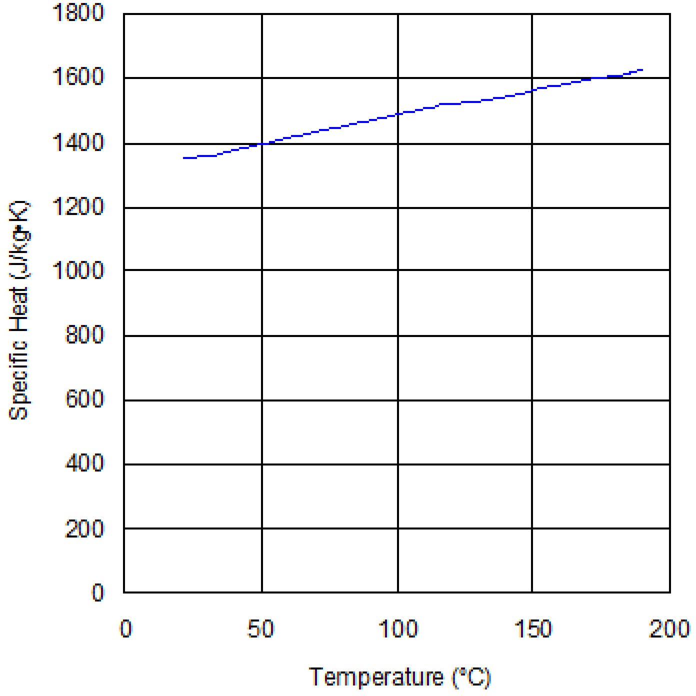
$$\eta_0(T) = D_1 \exp\left[-\frac{A_1(T-D_2)}{A_2+(T-D_2)}\right]$$

Problematic Materials

- Materials that are not liquid
  - Require high pressures
  - Reaction starts while melting
  - BMC (bulk molding compounds)
- Highly filled materials
  - Very long fibers are too long for fixtures
  - Cannot get a homogeneous sample
  - SMC
- Very fast reaction materials
  - cyanoacrylates
- UV cure materials
  - PU
- Reaction injection molding
- Foaming materials

Thermal Testing

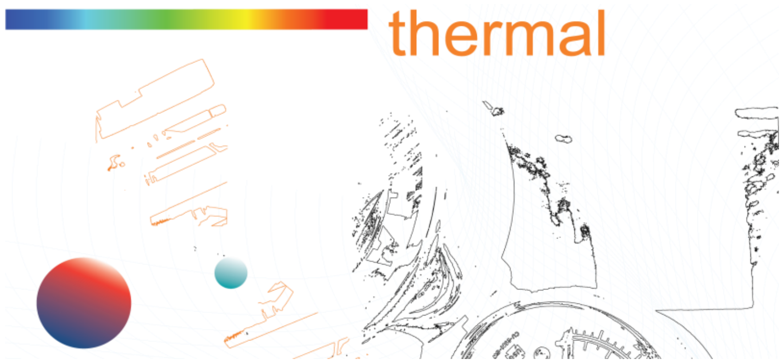




•Specific Heat

- DSC (Differential Scanning Calorimeter)
- Small samples sizes (7-15 mg)
- Performed in cooling to eliminate the curing enthalpy peak
- Enthalpy of reaction is recorded in the heating phase to but is not included in the specific heat curve.
- Some materials do not show an enthalpy peak or the peak is very small.
- The reaction peak is used to set the upper temperature for the PVT test
- Problems can arise if the material is strongly exothermic

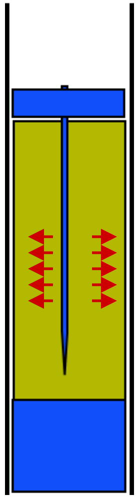
Thermal Testing

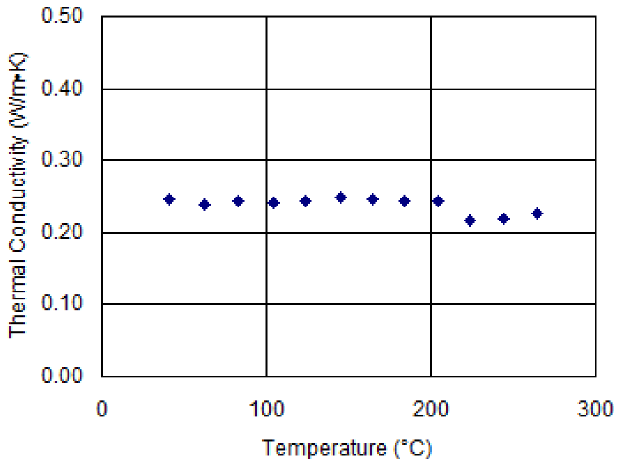


•Thermal Conductivity

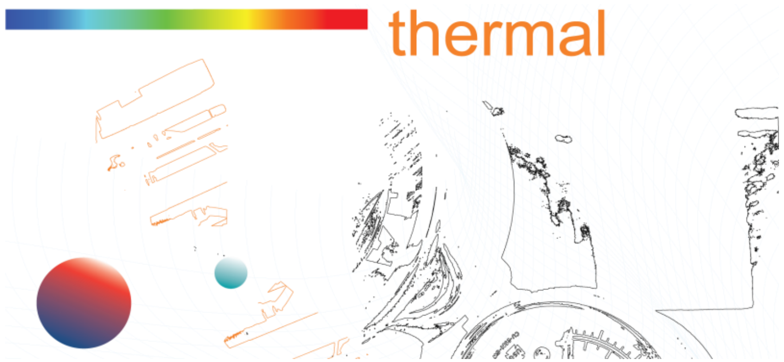
- A measure of how well a material transfers heat
  - Measured using transient line source
  - Unlike thermoplastics, the test is run from room temperature up to cure temperature
  - The barrel must be coated with silicone to prevent adhesion
  - Since the test relies on heat diffusion, exothermic material pose a problem

- Measure time to dissipate the heat pulse away from probe

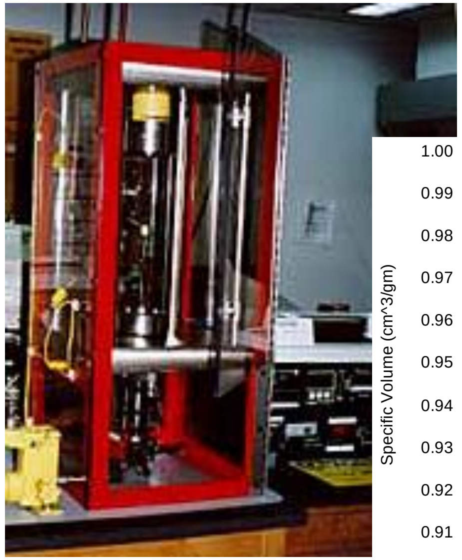


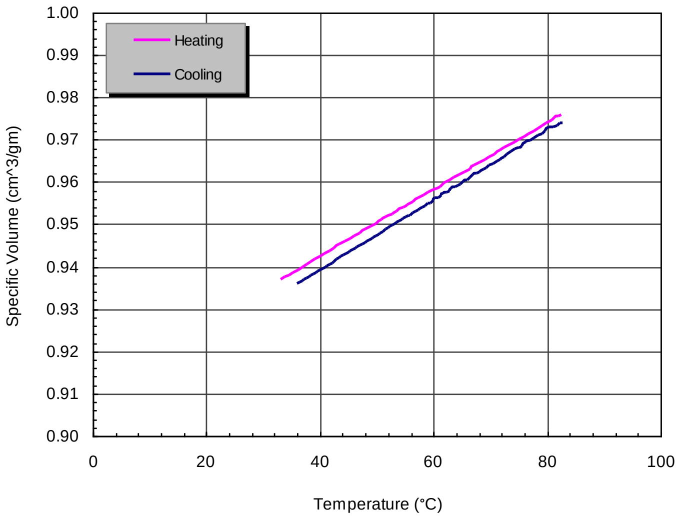


PVT (pressure, volume and temperature)

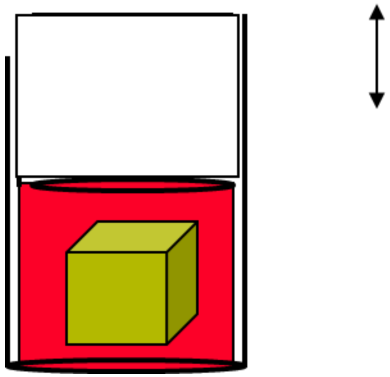


- Isobaric heating
  - Start with an uncured sample
  - Heat to just below cure temperature
  - Hold for up to two hours
  - Ensure full reaction has taken place
  - Cool back to start temperature
- The initial heat is the volumetric expansion due to heat and reaction
- The cooling is the volumetric expansion due to only thermal effect





- Cannot be done on materials the have a large amount of out-gassing
- Cannot be done on materials that begin to react as soon as they are mixed (setup time is very long)
- Can be done at multiple pressures but often not needed



expert material testing

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