Thermoplastic Material Testing for Use in Sigmasoft

DatapointLabs

technical center for materials

+

matereality

a DatapointLabs affiliate

EST 1995

ISO 17025 Quality

AMERICA'S FASTEST-GROWING PRIVATE COMPANIES Inc. 5000

Materials

Testing ¤ Data Infrastructure ¤ Productivity Software

Material Testing Expertise

Plastic
Rubber
Film
Metal
Foam
Composite
Cement
Ceramic
Paper
Wire
Fiber

- Product development / R&D support
  - CAE-centric
  - Commitment to simulation accuracy
- All kinds of materials
  - Over 1,800 materials tested each year
- All kinds of material behavior
  - Over 200 physical properties:
    - Mechanical properties
    - Thermal properties
    - Flow properties

Tensile
Compressive
Flexural
Stress-strain
Poisson's ratio
High strain rate
Bulk modulus
Fatigue
Viscoelasticity
Stress relaxation
Creep
Friction
Hyperelasticity
Thermal expansion
Thermal conductivity
Specific heat
PVT
Rheology

Customer Base

- 1200+ companies
- 11 manufacturing verticals
- Product development / R&D
- Globally available at
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Automotive
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Materials
Petroleum
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What is a Thermoplastic?

- Thermoplastics are solidified due to intermolecular bonds rather than chemical bond as in thermoset plastics
- Common thermoplastics
  - PET, PE, PVC, PP, PS, PA, ABS, etc.
- High performance
  - PEEK, PEI, PTFE, PSU, etc.

Amorphous and Semi-Crystalline

- Amorphous
  - Random molecule chains
  - Only glass transition
  - Transparent materials are typically amorphous
  - PET, PVC, PC, PMMA, PS, SAN, etc.
- Semi-Crystalline
  - Varying degree of crystallization
  - Molecules align and crystallize
  - Both glass transitions and melting point
  - PET, PP, PE, PA, PEEK, PEI, etc.
- Amorphous or Semi-Crystallinity is evident in test data

Required Testing

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

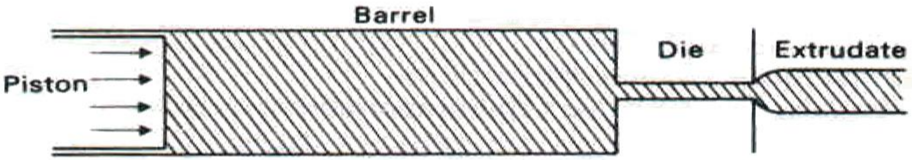
Mold Filling

Packing

Post-Mold Shrinkage Warpage

Rheometer Measurement of Viscosity

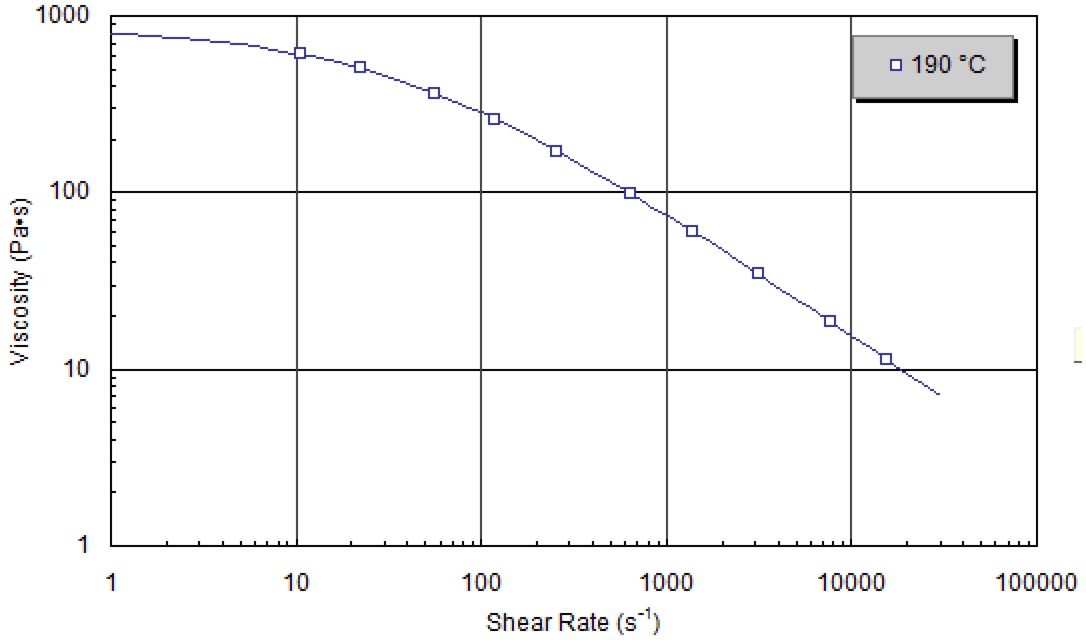
- Capillary rheometer is used
- Material is extruded through a restriction of known geometry (extremely high tolerance dies)
- Temperature and flow rate are controlled
- Pressure drop across the restriction is used to determine viscosity as a function of shear rate and temperature



Capillary extrusion rheometer

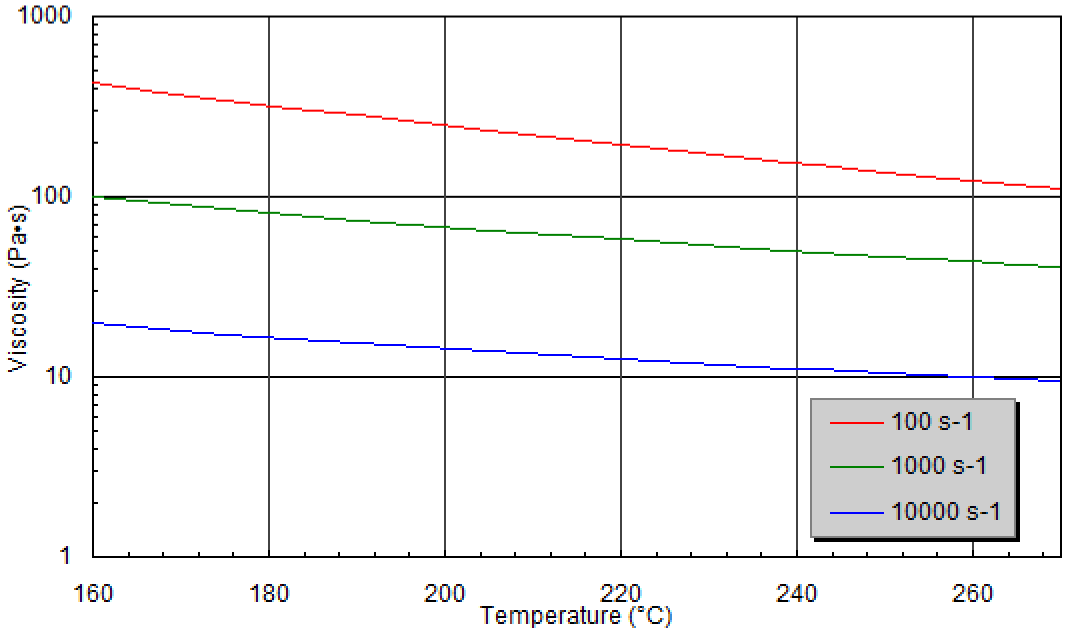
Viscosity Properties

- 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

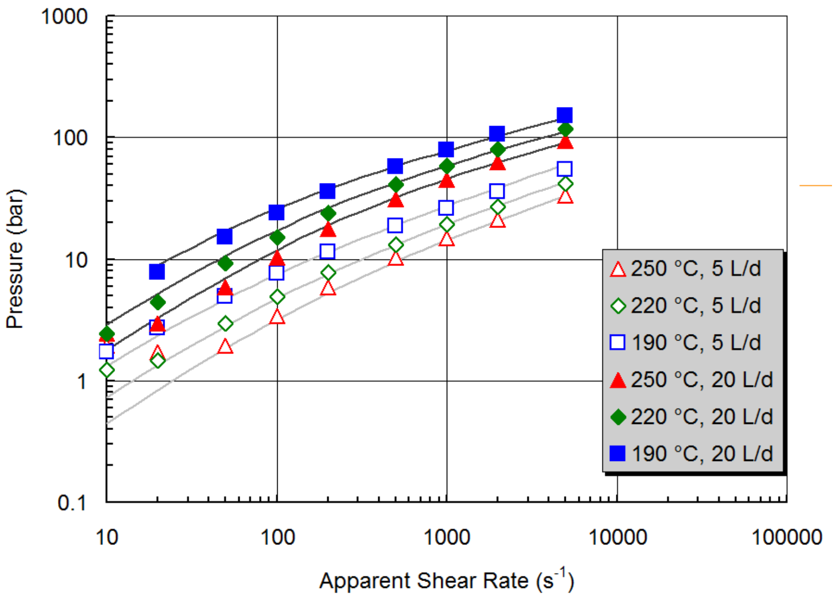
Δp = Pressure drop

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)
  - $\tau = R/2(dP/dL)$

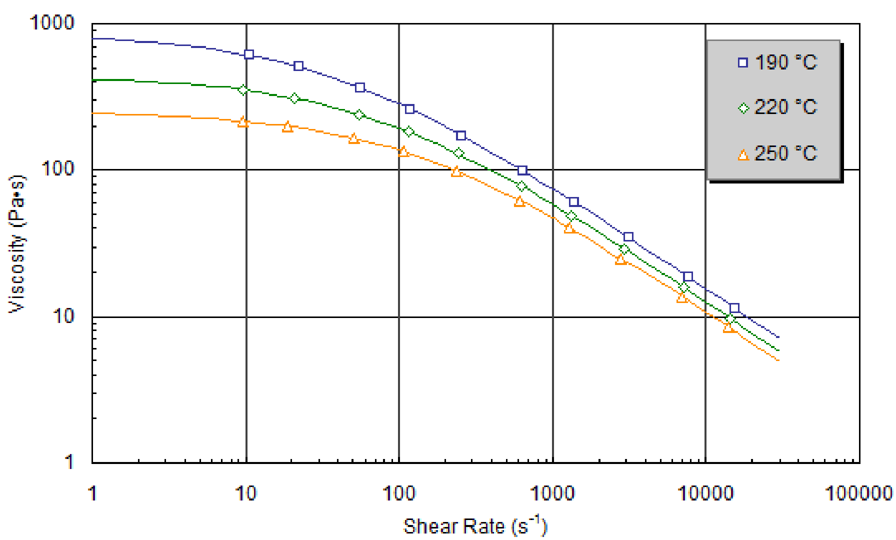


Viscosity Modeling

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

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

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



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

Evaluation of the 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

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

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

Evaluation of the Cross WLF Parameters cont.

- D1 is coupled to the WLF temperature dependency equation
  - No direct relevance
- D2 is the reference temperature
  - Theoretically where η goes to infinity
- A_1 & A_2 - WLF parameters
- A_1 defines the temperature sensitivity of viscosity
- A_2 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

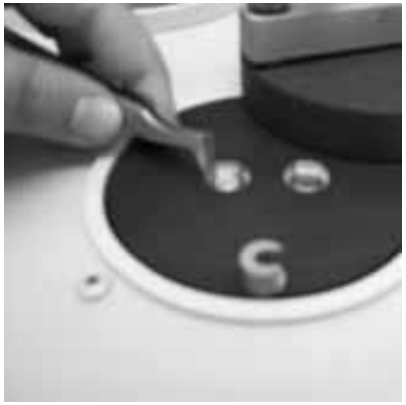
- Limited in shear rates
  - Typically test 10-10000 /s
- Residence times are longer in testing
  - Testing takes several minutes (approx. 6-10 min.)
  - Need to worry about thermal stability
- Typically testing is performed at two temperatures within the processing range and one below

Problematic Materials

- Moisture sensitive materials
  - Improperly dried materials cause reduction in viscosity
  - Over dried materials cause a rise in viscsosity
  - PET, PA, PC, 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

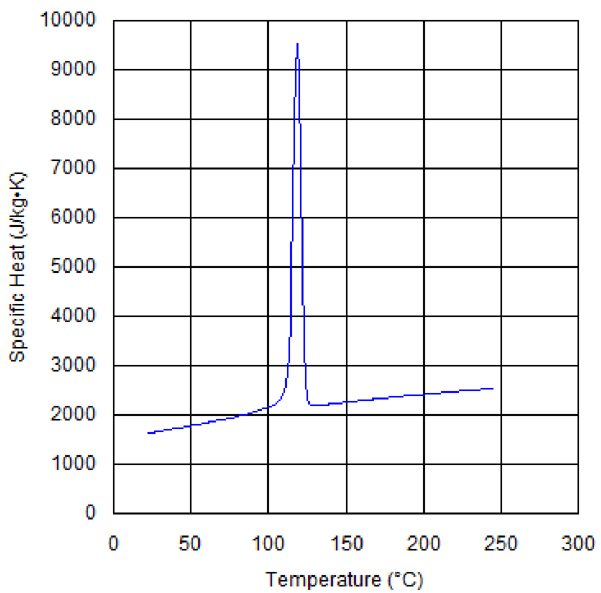
Specific Heat

- 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



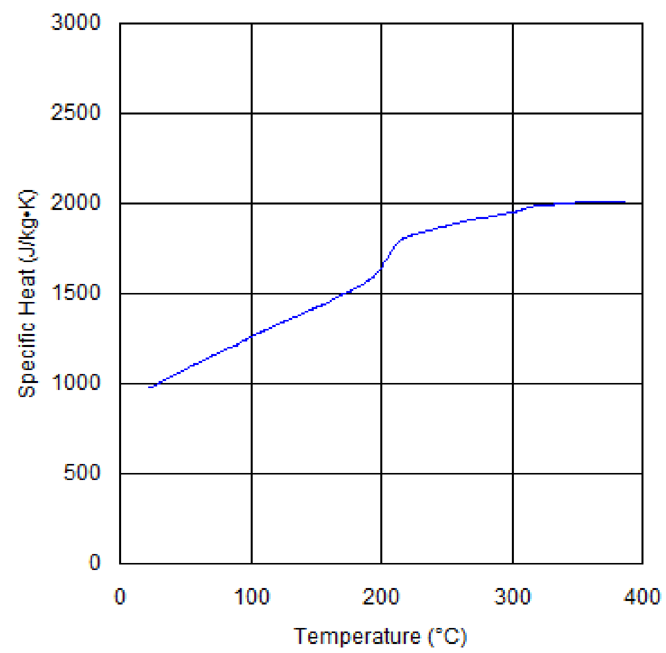
Transition Analysis

- 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 solidification of the polymer
  - The point at which the peak ends is set as the eject temperature
  - Beyond the eject temperature, no flow can tak



Transition Analysis cont.

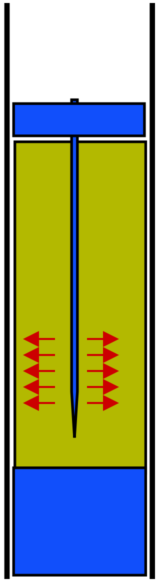
- Amorphous materials show a “knee” in the specific heat curve
  - The knee is the glass transition of the material, no crystallization takes place
  - The onset of the transition is set as the melting point to ensure complete solidification of the polymer
  - In this case the inflection point of the knee is taken as the eject temperature
  - Beyond the eject temperature, no flow can take place



Thermal Conductivity

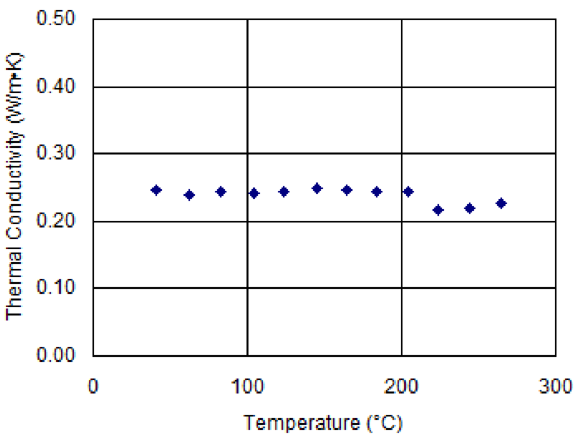
- A measure of how well a material transfers heat
  - Measured using transient line source
  - Measured in melt and solid state
  - Different behaviors for semi-crystalline and amorphous

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

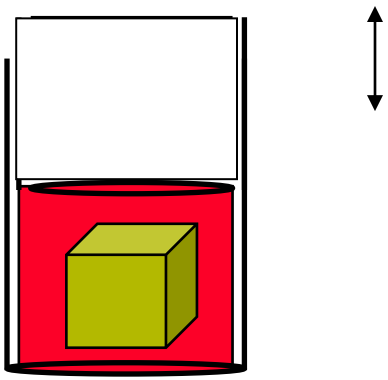


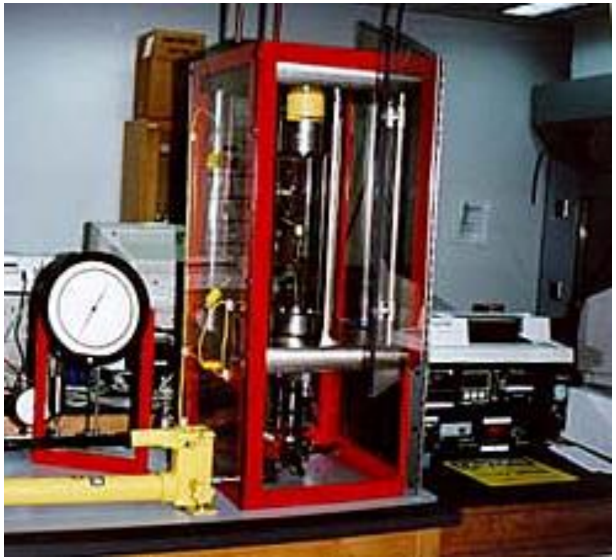
Density as a Function of PVT (Pressure Volume and Temperature

- 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

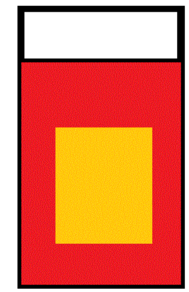
PVT Testing

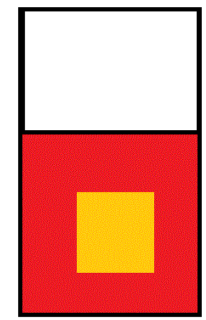
- Difficult and time consuming test
  - Initial density at ambient conditions
  - Mercury used as confining fluid
  - High temperatures and pressures
  - Complex datasets
  - True hydrostatic state



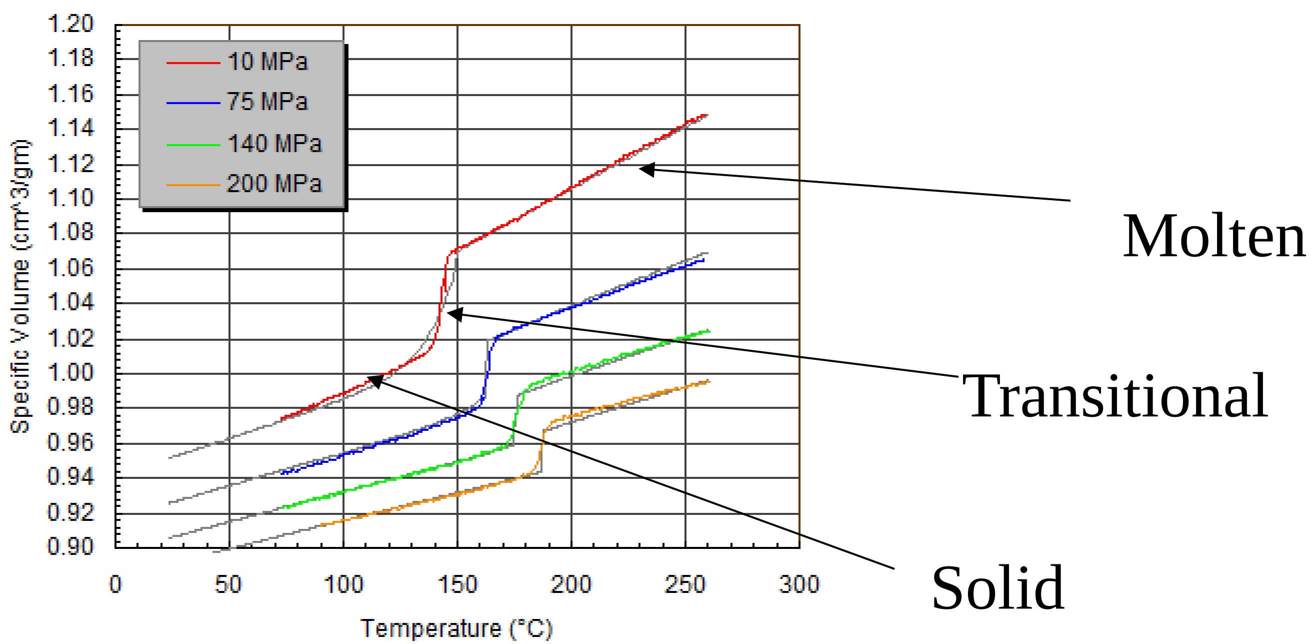


PVT Test Data



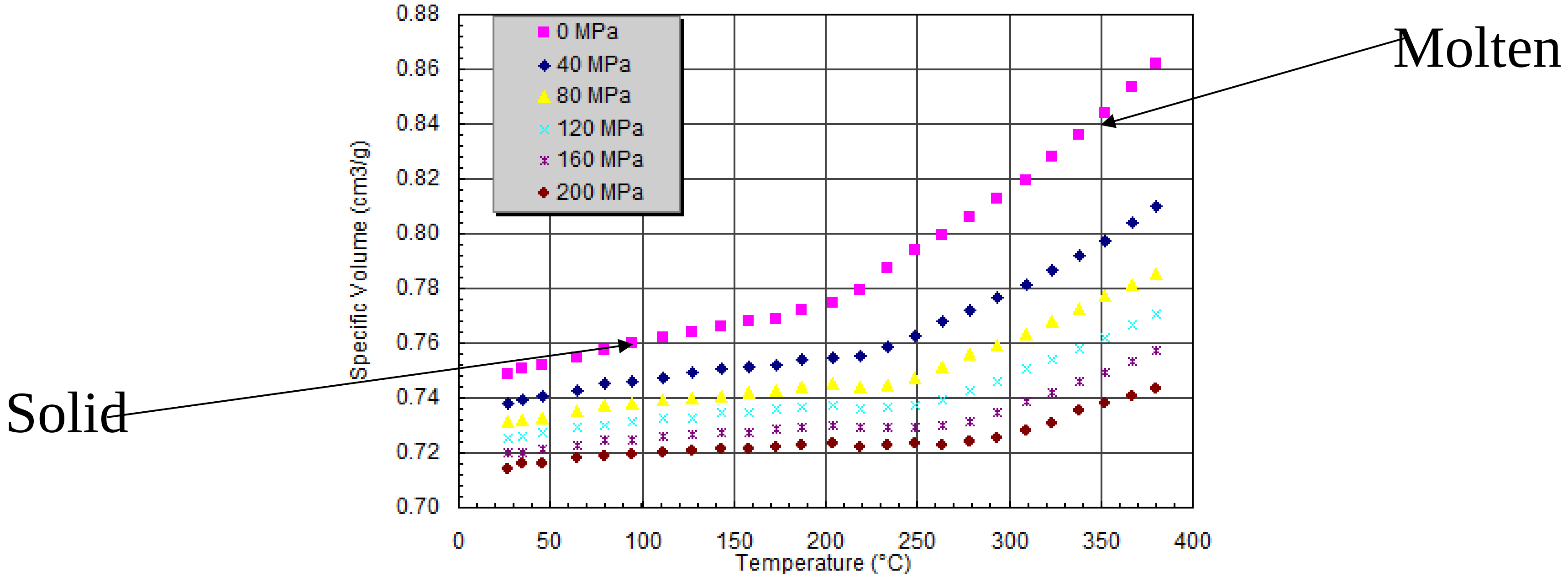


- Semi-Crystalline material
  - Transition region is critical
  - Rise in temp. = rise in spec. vol.
  - Rise in press. = drop in spec. vol.



PVT Test Data

- Amorphous material
  - Transition is not dependent on mode

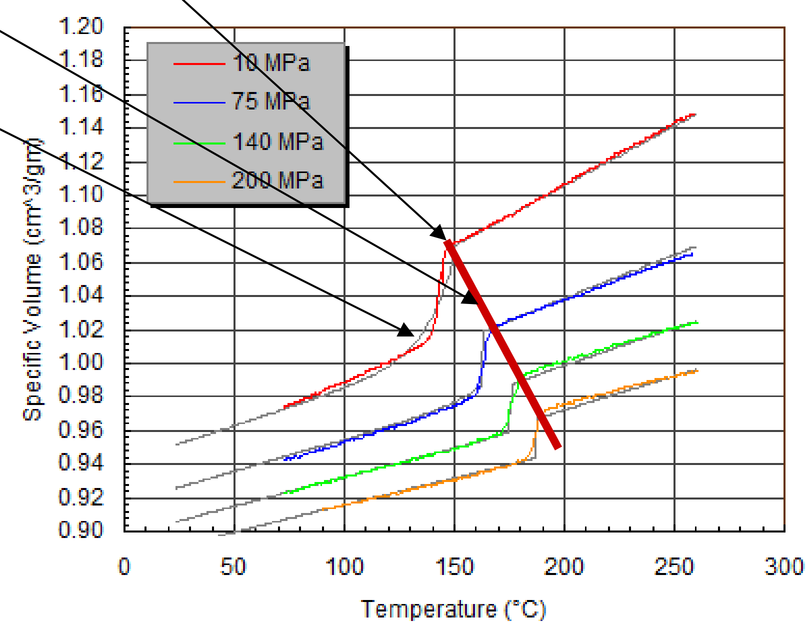


PVT Modeling

- 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 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	$m^3/kg{\bullet}K$
b3m	6.864E+07	Pa
b4m	3.209E-03	1/K
b1s	1.011E-03	m^3/kg
b2s	4.442E-07	$m^3/kg{\bullet}K$
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

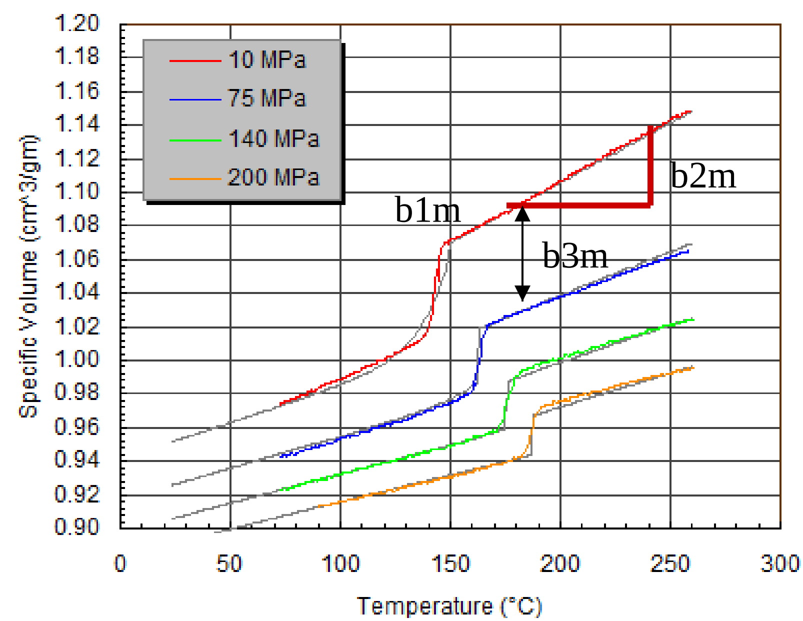


PVT Modeling cont.

- 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

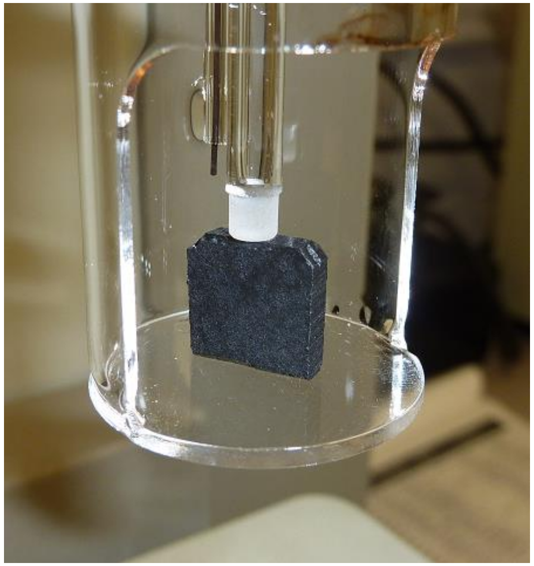


Problematic Materials

- 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



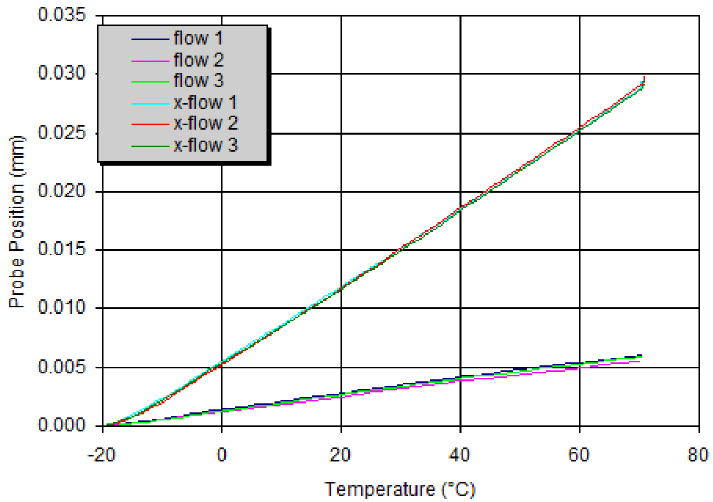
Thermal Expansion Test Data

- 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

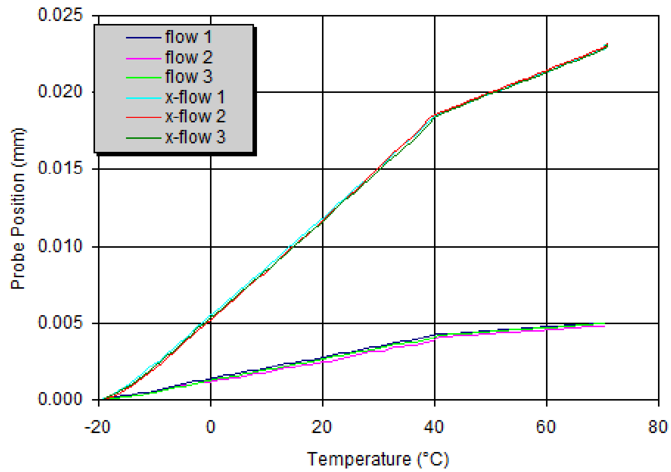
flow direction ($\alpha 1$)	
	0° to 60°C
replicate 1	6 x 10-6 / °C
replicate 2	6 x 10-6 / °C
replicate 3	6 x 10-6 / °C
average	6 x 10-6 / °C

cross-flow direction ($\alpha 2$)	
	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



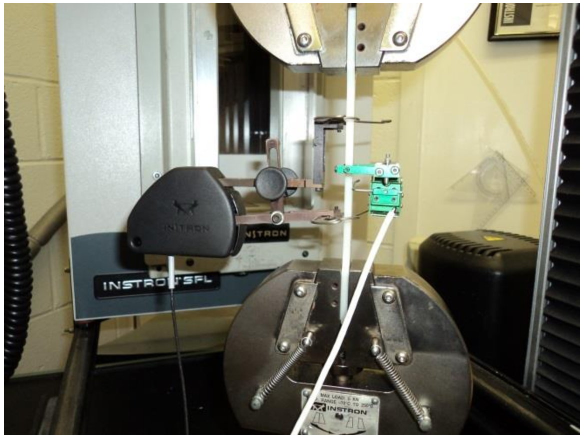
Problematic Materials

- 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



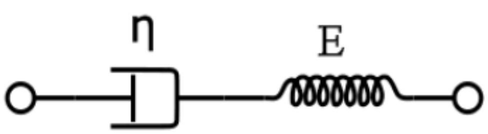
Mechanical Properties

- 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



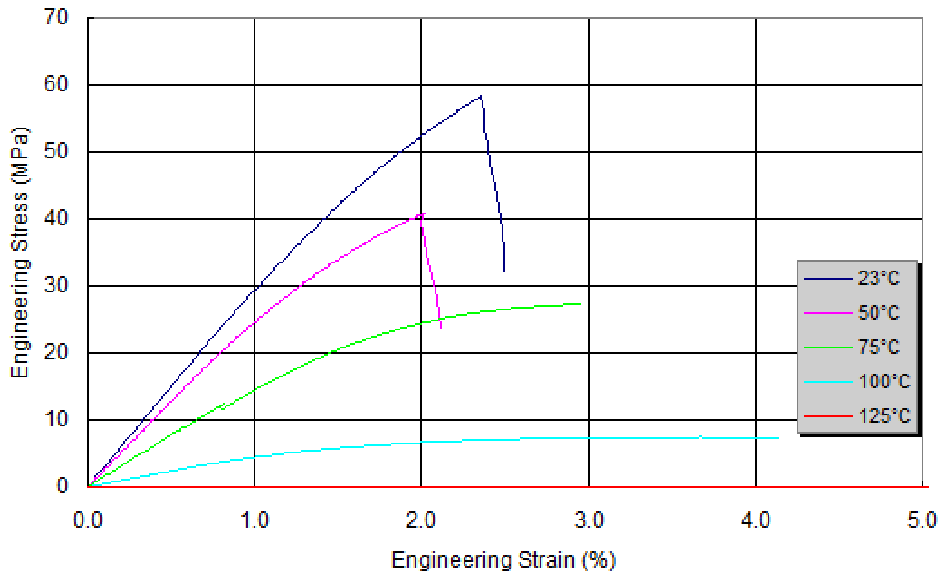
Viscoelaticity

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



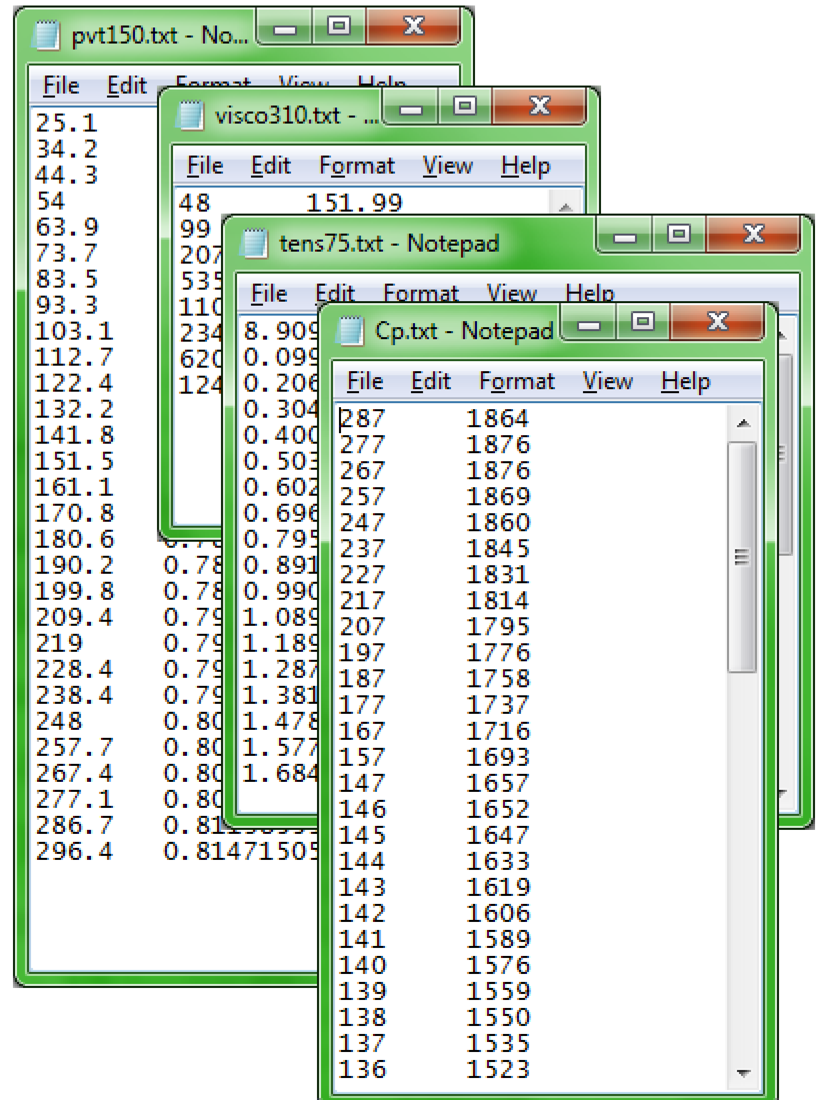
Properties

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



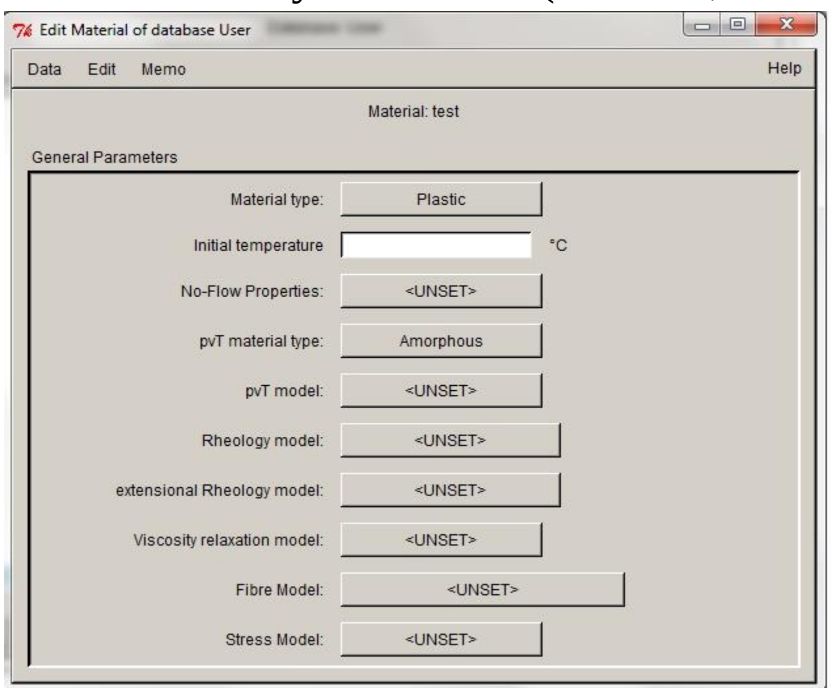
Implementation Into Sigmasoft

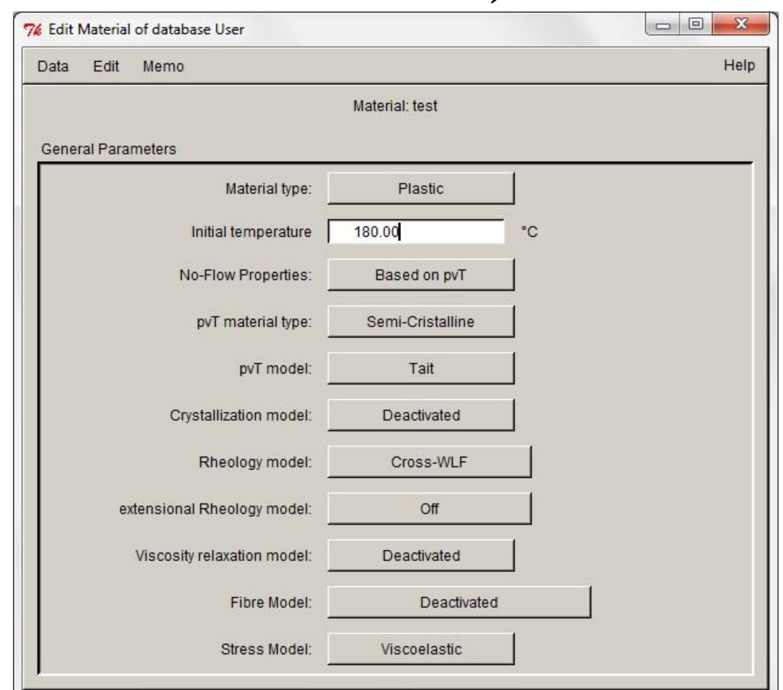
- X-Y pairs in text files
  - Viscosity (3)
  - Specific heat (1)
  - Thermal conductivity (1)
  - PVT (4-6)
  - CTE (1)
  - Mechanicals (5)
  - At least 17 input files



Implementation Into Sigmasoft

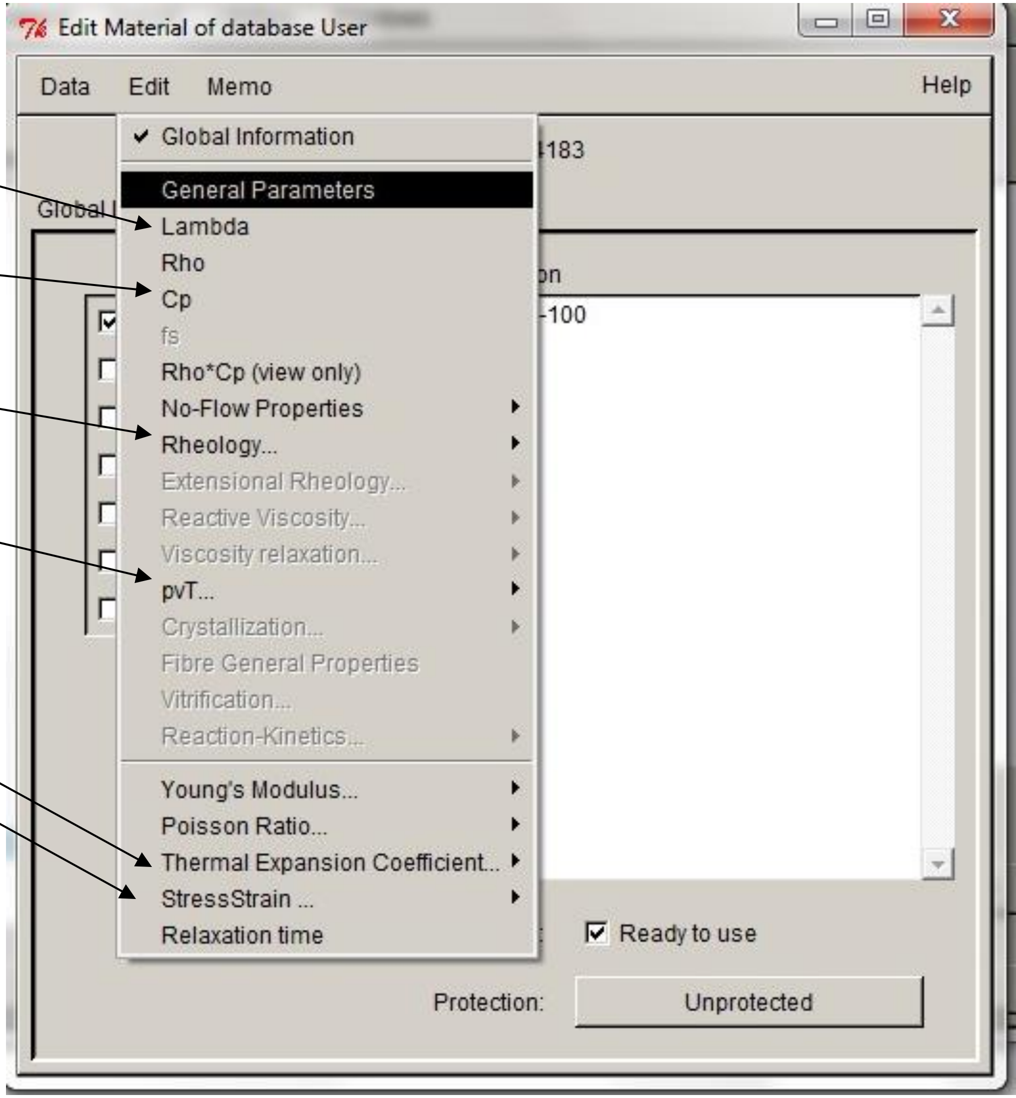
- General parameters
  - Material type → Plastic (Semi-crystalline or Amorphous)
  - Initial temperature → minimum temperature
  - Material models → PVT (Tait), Viscosity (Cross WLF), Stress (Viscoelastic), Fiber Model (Deactivated when viscoelastic is used), Crystallization (Weibull, when semi-crystalline material is selected)





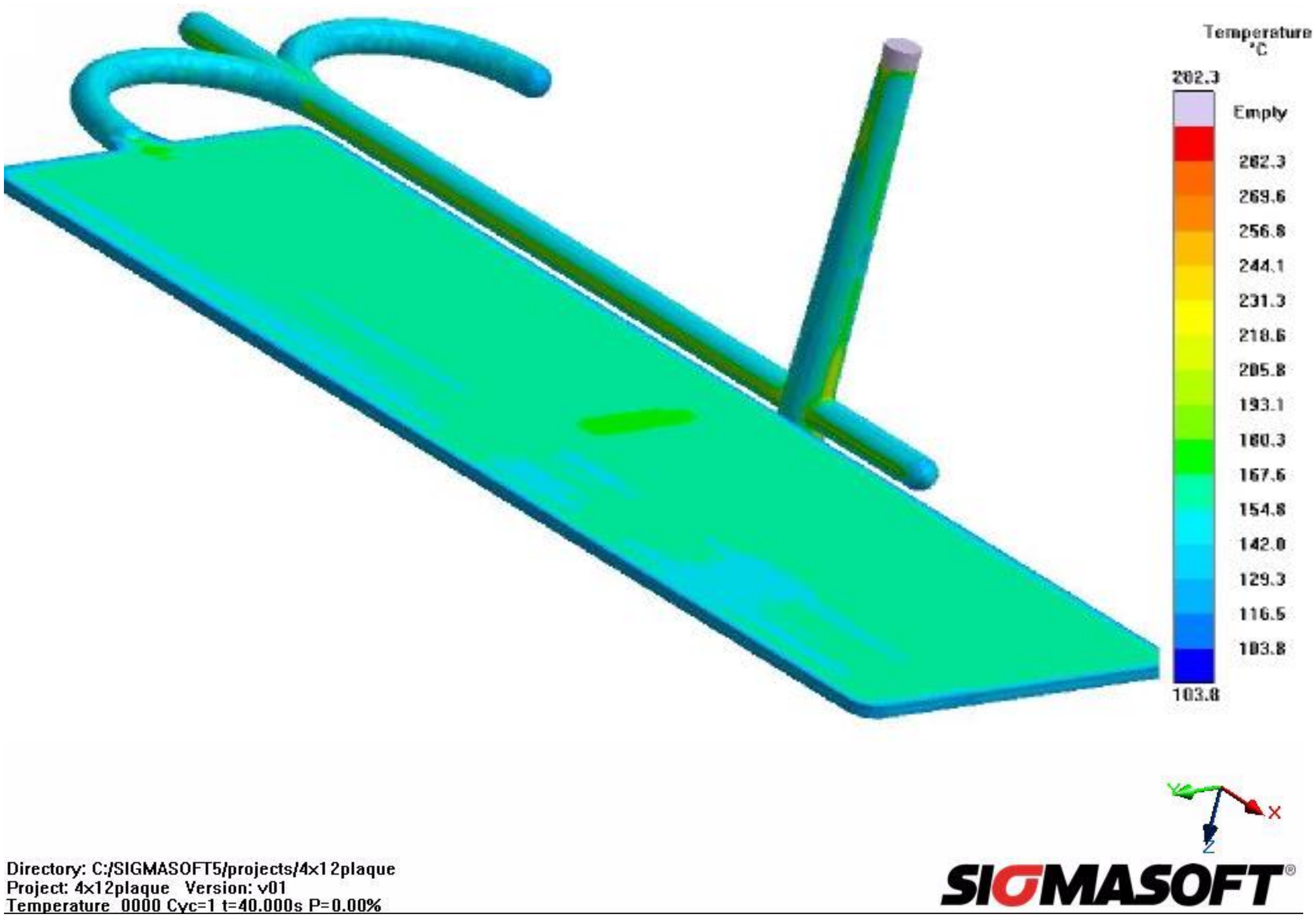
Implementation Into Sigmasoft

- Thermal conductivity
- Specific heat
- Viscosity
- PVT
- CTE
- Mechanicals
- Order of input counts
  - PVT, Cp, Lambda, Visc, CTE, mechanicals



Successful Simulation!

Testing Under $5000



Questions?

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