Material Testing Overview
(THERMOPLASTICS)
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Thermal Conductivity

Definition:
Thermal conductivity is a measure of a material’s ability to transfer heat by conduction. It is the rate of heat transfer by conduction, per unit length and degrees.

Method:
ASTM D5930, Standard Test Method for Thermal Conductivity of Plastics by Means of a Transient Line-Source Technique

Description of Method:
Thermal conductivity is measured using a transient line-source method (seen below), where a probe is inserted into the center of a molten sample, held at its processing temperature.

A line-source heater runs the length of the probe and a temperature sensor is in the middle. When thermal equilibrium is attained, a known amount of heat \( Q \) is supplied to the line-source heater and the temperature rise in the sensor is recorded over a period of time. The thermal conductivity \( k \) is calculated from the following equation:

\[
    k = \frac{QC \ln(t_2/t_1)}{4n(T_2 - T_1)}
\]

where \( T_1 \) and \( T_2 \) are temperatures at times \( t_1 \) and \( t_2 \) respectively, and \( C \) is the probe constant. Cooling scans are produced automatically by programming a range of temperatures.

Results:
Thermal conductivity is reported as a temperature scan of up to 12 points.

Precision of Method:
Intra-laboratory testing on a PS sample has shown a repeatability of 3%.

Calibration and Verification:
The K-System II unit is calibrated annually, traceable to NIST. The probe and instrument are verified on a weekly basis using polydimethylsiloxane fluid (60,000 cstk; thermal conductivity 0.16 W/m.K).
Specific Heat

Definition:
Specific heat is a measure of how much a material changes temperature with change in heat energy. It is the energy required to raise a unit mass of material by one degree (Celsius) in temperature.

Method:
ASTM E1269 Determination of Specific Heat Capacity by Differential Scanning Calorimeter
ASTM D3417 Enthalpies of Fusion and Crystallization of Polymers by Differential Scanning Calorimeter

Description of Method:
A Differential Scanning Calorimeter is used for performing specific heat measurements. A temperature scan is performed using empty pans in both chambers to establish a baseline.

A sample of a mass \( m \) is then loaded into one of the pans and the scan is repeated. The specific heat \( C_p \) is calculated from the difference in heat flow \( \Delta Q \) between the baseline and the sample needed to change the temperature by \( \Delta T \):

\[
C_p = \frac{\Delta Q}{m\Delta T}
\]

Specific heat is measured from the maximum processing temperature to 50°C, at a cooling rate of 20°C/min.

Results:
Specific heat is reported as a temperature scan of up to 12 points.

Precision of Method:
Round robin testing for specific heat has shown repeatability within a laboratory for this method to be 6%; while variation between laboratories is 9%.

Calibration and Verification:
Temperature verifications are performed at least weekly, using a heating rate of 10°C/minute, using high purity zinc and indium standards. Specific heat is verified, at least weekly, to be within 3% against a sapphire standard. The instrument is calibrated annually, traceable to NIST.
Transition Temperature and Ejection Temperature

Definitions:
Transition temperature is the temperature at which the polymer transitions from a molten state to a solid state.
Ejection temperature is the temperature at which the part has cooled sufficiently to be cleanly removed from the mold.

Method:
ASTM D3418 Transition Temperatures of Polymers by Differential Scanning Calorimetry

Description of Method:
As specified by ASTM D3418, the transition temperatures are determined from the extrapolated onset (Tf) for crystalline transitions and from the glass transition temperature (Tg) as defined by the midpoint temperature (Tm) for amorphous materials.

Results:
The transition temperature and the ejection temperature are single point values (see plots below).
Precision of Method:
Round robin testing for specific heat has shown repeatability within a laboratory for this method to be 6%; while variation between laboratories is 9%.

Calibration and Verification:
Weekly temperature verifications are performed at a heating rate of 10°C/minute, using high purity standards of zinc and indium. Specific heat is verified to be within 3% against a sapphire standard. The instrument is calibrated traceable to NIST at least annually.
Shear Viscosity

**Definition:**
Shear viscosity is a measure of a material’s resistance to flow under shear deformation. It is a measure of wall shear stress per shear rate of a flowing sample as a function of shear rate, temperature, and pressure.

**Method:**
The viscosity test developed in our laboratory adheres to the geometry and reporting requirements outlined in these standards:
- ISO-11443 Plastics - Determination of the fluidity of plastics using capillary and slit-die rheometers

**Description of Method:**
Shear viscosity is measured on an instrumented injection molding machine. The polymer is plasticized, and extruded through capillary or slit dies directly attached to the barrel of the injection unit. The flow rate is varied by adjusting the injection velocity, and the temperature and pressure drop over the die is logged during the shot. Data is collected at a range of flow rates and temperatures under processing conditions.

**Results:**
The Cross-WLF viscosity model is fit to the rheological data using a numerical, finite difference scheme. During modeling, the measured values of temperature and flow rate are used in a flow simulation to predict extrusion pressures through the die geometry. The viscosity model is adapted to minimize the difference between the measured and simulated pressures.

Moldflow uses a modified form of the Cross-WLF model to characterize the viscosity at any shear rate, temperature and pressure.

\[
\eta = \frac{\eta_o \cdot \left(1 - N\right)}{1 + \left(\frac{\eta_o \cdot \gamma}{\tau^*}\right)}
\]

\[
\eta_o = D_1 \exp \left[ -\frac{A_1 \left(T - T^*\right)}{A_2 + \left(T - T^*\right)} \right]
\]

\[
T^* = D_2 + D_3 P
\]

\[
\tilde{A}_2 = A_2 + D_3 P
\]

**Precision of Method:**
Testing on a PP sample has shown a repeatability of 8%.

**Calibration and Verification:**
Thermocouples and pressure transducers are calibrated annually. The instrument is verified against a secondary standard before testing.
**Pressure-Volume-Temperature (pvT)**

**Definition:**

pvT is the measurement of a material’s thermodynamic properties. The specific volume is measured as a function of pressure and temperature.

**Method:**

High Pressure Indirect Dilatometry

**Description of Method:**

Prior to testing, the density of the sample is determined at room temperature to use as a reference point for further measurements. Pressure-volume-temperature measurements are then made using a Gnomix apparatus that encloses the sample in a confining fluid (mercury) as seen below:

![Diagram of Gnomix apparatus](image)

The pressure in the bellows is varied over a range of 0 to 200 MPa, and transmitted to the sample by the confining fluid. The displacement of the bellows is used to determine the change in volume of the sample.

**Results:**

The pvT relationship is modeled using a modified two-domain Tait equation:

\[
v(T,p) = v_0(T) \left[ 1 - C \ln \left( 1 + \frac{p}{R(T)} \right) \right] + v_1(T,p)
\]

where \( v(T,p) \) is the specific volume at temperature \( T \) and pressure \( p \), \( v_0 \) is the specific volume on the zero gauge pressure, \( C \) is 0.0894 (universal constant), and \( B \) represents the pressure sensitivity of the material. Two temperature domains are required to model the pvT relationship, because the thermodynamic properties of polymers change at the transition to the solid state.

The volumetric transition temperature at zero gauge pressure is denoted by \( b_5 \), and the linear increase in the transition with pressure is denoted by \( b_6 \). The specific volume obtained by extrapolating the zero-isobar curve to the transition temperature is denoted by \( b_1 \). This value is the same for both domains when crossing the glass transition. However, when the material is semi-crystalline, the transition due to crystallization is accompanied by an abrupt change in specific volume, such that \( b_{1m} \) (the melt specific volume at \( b_5 \) and zero pressure) is greater than \( b_{1s} \). For each domain, the rate of change of specific volume with temperature at zero pressure is measured by the \( b_2 \) constants in the solid and melt state:

\[
V_0 = b_{1m} + b_{2m}(T - b_5) \quad \text{for } T > b_5 + b_6 p
\]

\[
V_0 = b_{1s} + b_{2s}(T - b_5) \quad \text{for } T < b_5 + b_6 p
\]

The remaining constants, \( b_3 \) and \( b_4 \), characterize \( B \):

\[
B(T) = b_{3m} \exp[- b_{4m}(T - b_5)] \quad \text{for } T > b_5 + b_6 p
\]

\[
B(T) = b_{3s} \exp[- b_{4s}(T - b_5)] \quad \text{for } T < b_5 + b_6 p
\]
In particular, a positive b4 reflects a specific volume that becomes more pressure sensitive with increasing temperature.

Typical transitions:

The pvT testing results establish the solid and melt density values that are used in Moldflow software products.

**Precision of Method:**
The instrument manufacturer states the instrument accuracy is $\pm 0.002\ \text{cm}^3/\text{g}$ at temperatures up to 250°C, and to $\pm 0.004\ \text{cm}^3/\text{g}$ at temperatures above 250°C.

**Calibration and Verification:**
Temperature, linear and pressure transducers are calibrated annually, traceable to NIST. The LVDT is calibrated before each use against a micrometer that is calibrated annually, traceable to NIST. The instrument calibration is verified against an internal standard at least once a month.
Shrinkage

**Definition:**
Shrinkage measurements characterize the difference in the dimensions of the plastic part and the mold. A series of measurements is performed to show the effects of molding conditions upon part shrinkage.

**Method:**
MPL Test Method

**Description of Method:**
Shrinkage plaques are molded on an instrumented injection molding machine. The stationary platen of the test mold is inscribed with a fine grid pattern so the linear dimensions of the part can be accurately determined. Test plaques are produced under a wide variety of molding conditions (melt and mold temperature, flow rate, hold and cooling times, hold pressures and part thicknesses)

Following molding, the samples are annealed for a nominal one week period in a controlled environment. A linear coordinate measuring device is used to determine the exact dimensions of parts molded at each set of conditions.

**Results:**
Flow simulations are performed at each of the measured molding conditions and these results are used to predict the shrinkage in the part. A regression analysis of the measured and predicted shrinkages is performed to determine the coefficients of the shrinkage model.

Shrinkage can be modeled by the Corrected Residual In-Mold Stress (CRIMS) model and the residual strain model.

**Precision of Method:**
Dimensional measurements on a reference standard have shown a repeatability of 0.1% or less.

**Calibration and Verification:**
Thermocouples and pressure transducers are calibrated annually. The instrument is verified against an internal standard before molding.
Mold Verification

Definition:
Mold verification compares the cavity pressure measured experimentally with the predictions made using the material data set established for the material.

Method:
MPL Test Method

Description of Method:
Plaques are molded on an instrumented injection molding machine. Moldings are typically performed at the recommended melt and mold temperatures, various flow rates and multiple part thicknesses.

Results:
Flow simulations are performed at each of the molding conditions using the measured flow, thermal and pvT data. The predicted cavity pressures are compared to the experimental measurements.

Precision of Method:
Testing on a PP sample has shown a repeatability of 5%.

Calibration and Verification:
Thermocouples and pressure transducers are calibrated annually. The instrument is verified against an internal standard before molding.
Tensile Modulus and Poisson’s Ratio

Definition:
Tensile modulus (\(E\)) is defined as the ratio of stress to strain in the direction of load below the proportionality limit of the material (i.e., the point at which the material begins to deform non-linearly).

Poisson's ratio (\(v\)) is defined as the ratio of the lateral contraction strain to longitudinal strain.

Method:
ASTM D638 Standard Test Method for Tensile Properties of Plastics
ASTM E132 Standard Test Method for Poisson’s Ratio at Room Temperature

Description of Method:
Specimens for tensile modulus measurement are placed in the self-aligning jaws of a tensile testing machine equipped with an extensometer and load is applied (see below). Care is taken to ensure that all data used for these measurements are taken in the elastic region of the stress-strain curves.

\[
v_{12} = \varepsilon_T / \varepsilon_L
\]

\[
v_{23} = \varepsilon_{Th} / \varepsilon_T
\]
**Precision of Method:**
Tensile modulus: To date, ASTM round robin testing has not been performed for directional measurements. Directional measurements are expected to be more precise than non-directional measurements, for which ASTM round robin testing has shown to have a variation (within laboratory) of 12% and reproducibility (between laboratories) of 96%.

Poisson's ratio: To date, no ASTM round robin testing has been performed.

**Calibration and Verification:**
The instrument and measurement devices are calibrated annually, traceable to NIST.
In-Plane Shear Modulus

Definition:
In-plane shear modulus is the change of shape produced by a tangential stress. It provides a measure of the materials' "stiffness".

Method:
Rail Shear Test

Description of Method:
This technique uses a test fixture mounted in a basic lever loading frame (see below).

Specimens are machined from the sample and notched to fit the fixture, with round end profiles designed to minimize end effects and produce a homogeneous stress field. The specimen is mounted in the test fixture and bolted in position between the rails. An extensometer is placed on the specimen between the rails with needle point arms at 45 degree angle to the direction of loading. Load is applied to the specimen and shear strain is monitored by the extensometer. This is repeated in each of the four positions (front right and left, back right and left) on the specimen.

Results:
Shear modulus \( G \) is calculated from the data measured for each position as:

\[
G = \frac{\tau}{\gamma}
\]

where \( \tau = \frac{P}{2}, \gamma = 2\varepsilon_{45^\circ} \)

\[
P = \text{Load}, \varepsilon_{45^\circ} = 45^\circ \text{ strain}
\]

Precision of Method:
To date, no round robin testing has been performed.

Calibration and Verification:
The instrument and measurement devices are calibrated annually, traceable to NIST.
Coefficient of Linear Thermal Expansion

Definition:
Coefficient of linear thermal expansion is the ratio of the change in length per degree.

Method:
ASTM D696 Coefficient of Linear Thermal Expansion of Plastics

Description of Method:
A specimen is prepared and placed at the bottom of the outer dilatometer tube with the inner one resting on the specimen (see below).

\[ \alpha = \frac{\Delta L}{L \cdot \Delta T} \]

where \( L \) is the initial length of the specimen, \( \Delta T \) is the change in temperature, and \( \Delta L \) is the change in length. Measurements are made on specimens cut from molded plaques both in the flow direction (\( \alpha_1 \)) and the transverse to flow direction (\( \alpha_2 \)).

Precision of Method:
ASTM D696 lists the variation and reproducibility of the test method upon a number of resin types which were tested in a round-robin study. The variation and reproducibility varies greatly between different resins.

Calibration and Verification:
The digital indicator, accurate to one micrometer, is calibrated annually, traceable to NIST. The temperature measurement is calibrated annually against instruments which are calibrated traceable to NIST. The apparatus operation is verified annually against a NIST copper standard.
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