Torque Ranges The most important cone-and-plate rheometer design feature is the method by which the shear stress is measured (or the strain, in a stress-controlled rheometer).

In a strain-rate-controlled rheometer, the motor that drives the cone has a typical rotation speed range of 0.001–100 rad/s. Typical cone angles range from 0.02 to 0.10 rad. Hence, according to Equation 1.60(2), the theoretical shear rate available with the rheometer is 0.01–5000 s^{-1}. However, the actual available shear range is usually much smaller as a result of torque transducer sensitivity limitations at low shear rates and flow disturbances at high shear rates (see the following).

Because the onset of flow disturbances varies considerably with the material being tested, no shear rate range is given in the feature summary at the beginning of this section. In a rate-controlled rheometer, both the torque T and the total vertical force F_N are usually calculated from the deflection of a spring measured using a displacement transducer of a differential transformer type. Care must be taken that the vertical deflection of the spring used to measure F_N does not cause a significant change in the gap between the cone and the plate. Because rotational friction from the instrument itself is detrimental to sensitivity, top-of-the-line rheometers employ air bearings on the cone drive shaft.

A given rheometer is sometimes said to cover a certain measurable range of fluid viscosities, but it is really more accurate to say that the rheometer covers a certain measurable range of torques. A typical rheometer transducer can be used accurately over a torque range covering perhaps four orders of magnitude, from 0.00001 to 0.1 N m. Alternative transducers covering both higher and lower torque ranges are also available.

For a given rate-controlled rheometer, the minimum viscosity value that can be measured can be calculated from the minimum measurable torque value, the maximum possible shear rate, and the maximum available cone-and-plate radius using Equations 1.60(2) through 1.60(4). These equations show that, for low-viscosity fluids, the torque value T, and hence measurement accuracy, can be enhanced by increasing the shear rate κ or by increasing the cone-and-plate radius R.

Selection The choice between a strain/strain-rate-controlled rheometer and a stress-controlled rheometer often depends on whether stress relaxation data or shear creep data are more desirable. However, the stress-controlled rheometer can be operated in strain-controlled mode through use of a feedback control loop that manipulates the value of the imposed torque; however, time to set strain is longer than for a strain-controlled rheometer. In addition, strain-rate-controlled rheometers should not be used to study the flow properties of a sample that might be a weak solid, because the driven rotation of the cone will damage the sample structure.

Observation of hysteresis in the viscosity curve (μ vs. κ) indicates that the structure of the sample has been damaged by flow. The properties of a fragile solid can be measured by using a strain-controlled rheometer in low-amplitude oscillatory measurements (discussed in a later section) or by using a stress-controlled rheometer. In a stress-controlled rheometer, a strain transducer is used to measure sample strain or rate of strain in response to a chosen shear stress value τ. If the sample has a yield stress value that exceeds τ, then the measured steady-state rotational speed of the cone will be zero, a result that indicates that the sample is a weak solid.

Flow Field Disturbances The goal of scientific rheometry as applied to liquids is to study the mechanical response of the liquid when subjected to a known and controlled velocity field. Unfortunately, there is no way to know beforehand whether a given rheological sample will exhibit the expected velocity field in the cone-and-plate device or any other rheometer. If the actual velocity field within the rheometer is disturbed from the expected flow pattern, then the rheometer software will probably calculate incorrect values for the viscosity and other rheological properties.

Because of flow field disturbances, it is virtually impossible to measure μ, N_1, and N_2 values with a cone-and-plate flow geometry at the high shear rate values typical in manufacturing operations (κ = 10–1000 s^{-1} for extrusion, κ = 1,000–50,000 s^{-1} for injection molding). Figure 1.60e shows a sketch of one such flow disturbance called *edge fracture*. These flow disturbances are thought to be driven by elasticity or inertia and can be delayed to higher shear rates by reducing the cone angle or the cone-and-plate radius. They do not occur when the cone-and-plate geometry is used for linear rheological measurements. This is a principal reason why linear properties G′ and G″ are more commonly measured in industrial laboratories than N_1 and N_2, even though linear rheological properties have no direct relevance to the flow conditions actually present in thermoplastic processing flows, which are usually very nonlinear.

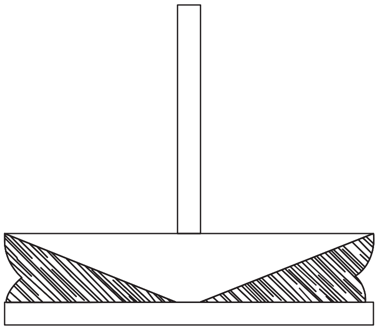


FIG. 1.60e
Flow disturbance due to edge fracture of sample in cone-and-plate instrument.

Dynamic Mechanical Analysis Linear rheological properties are measured when the strain field or velocity field only slightly perturbs the sample on a molecular level from its equilibrium state. For molten thermoplastics, linear rheological measurements have no direct relevance to polymer processing flows, because most practical manufacturing flows are highly nonlinear. However, linear rheological measurements are still very useful for characterizing the polymer molecules or making a rheological *fingerprint* of the raw material.

Linear rheological properties are most commonly measured in oscillatory experiments called dynamic mechanical analysis (DMA). For a liquid in oscillatory shear, the typical experimental setup is identical to that for simple shear flow (Figure 1.60d), with the sole exception being that the cone does not rotate continuously in one direction but, rather, alternates between clockwise and counterclockwise rotations periodically at an angular frequency ω.

The rheometer plate is held stationary, which imposes a sinusoidal shear strain on the sample, as sketched in Figure 1.60f. If ΔΦ is the maximum angular displacement of the cone, then the amplitude of the strain curve is given by

$$\gamma_{\max} = \frac{\Delta\Phi}{\alpha} \qquad \mathbf{1.60(6)}$$

where α is the cone angle. The torque necessary to keep the plate from rotating is measured and used to calculate the shear stress τ via Equation 1.60(3). Typical behavior for the shear stress τ is also plotted against time in Figure 1.60f. Note that the shear stress and the shear strain are both sinusoidal functions of time, and they both have the same angular frequency ω. However, there is a phase shift between the two curves—the strain curve lags the stress curve by a constant phase angle denoted δ.

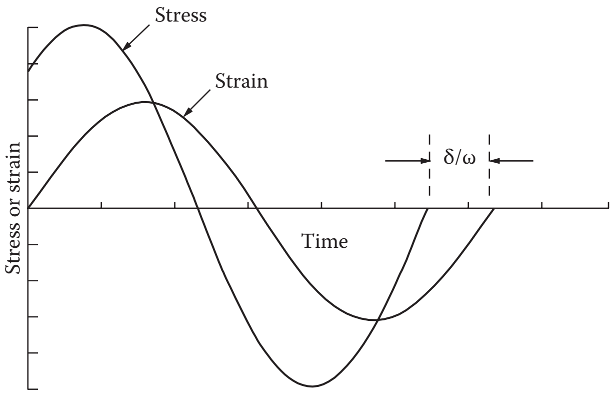


FIG. 1.60f
Typical stress and strain curves in DMA.

Viscoelastic Shear Moduli The amplitude of the shear stress curve divided by the amplitude of the shear strain curve is denoted G*, the dynamic shear modulus. Thus, the oscillatory shear experiment yields two independent rheological properties: G* and δ. To obtain linear rheological properties, one needs to check that the measured values of G* and δ are independent of $\gamma_{\max}$. An alternative set of properties are the elastic storage modulus (G′) and the viscous loss modulus (G″), given by $G' = G^* \cos\delta$, $G'' = G^* \sin\delta$.

For a cyclical shear deformation process at frequency ω, G′ is a measure of the sample's ability to store elastic strain energy, and G″ is a measure of viscous energy dissipation. The relative importance of energy dissipation and energy storage is given by the loss tangent tan δ

$$\tan\delta = \frac{G''}{G'} \qquad \mathbf{1.60(7)}$$

The value of tan δ is infinite for a purely viscous liquid, zero for a perfectly elastic solid, and intermediate between these two extremes for a real viscoelastic material. However, the mechanical behavior of most viscoelastic materials (solid or liquid) becomes more like the perfectly elastic solid as the frequency is increased, which may be explained as follows.

When a polymeric sample is subjected to a shear strain, the polymer molecules have many different modes for relaxing the stress with widely varying relaxation times. At very low oscillation frequencies, almost all of these relaxation modes are fast enough to be operative, so energy storage is minimal. Conversely, at very high frequencies, most of the polymer molecules do not have sufficient time to relax during the period of oscillation (2π/ω). Thus, the polymer molecules remain stretched throughout the entire oscillation cycle, giving rise to increased elastic energy storage and increased shear modulus G′.

Molecular Weight Distribution Polymeric materials have a wide spectrum of relaxation times, with each relaxation time corresponding to a different possible mode of mechanical relaxation. As explained in the preceding section, one can probe the mechanical relaxation processes that occur at different time scales by measuring oscillatory shear properties at various frequencies. The largest molecules in the sample contribute to relaxation processes occurring at long times or low frequencies, whereas small molecules contribute to relaxation at high frequencies.

In recent years, rheometer companies have introduced software that can be used to invert curves of G′ and G″ versus ω measured for a molten thermoplastic to give the molecular weight distribution (MWD) of the sample (Chapter 1.41). This method of estimating the MWD is more sensitive to the presence of polymer branches and high-molecular-weight *tails* in the sample than traditional solution-based methods such as gel permeation chromatography (Chapter 1.41).

However, the theory on which the inversion software is based is still evolving and thus may not work for all types of polymers. Dynamic curves measured on samples in the solid state cannot be inverted to give the MWD.

Time–Temperature Superposition Time–temperature superposition is an approximate method used by experimental rheologists to extend the effective frequency range of a rheometer in DMA experiments. As an example, suppose one is interested in predicting the in-use mechanical performance of a polymeric solid subjected to a constant stress for 1 week at 25°C. The appropriate frequency range for DMA of the solid at 25°C would be $(1\ \text{week})^{-1} \approx 10^{-6}\ \text{s}^{-1}$, which might be too low to access with a given rheometer. However, if the empirical time–temperature shift factor for the sample (a_T) is known to be 1×10^{-3} for shifting between 25°C and 90°C, then the appropriate frequency range for DMA at 90°C is $10^{-6}\ \text{s}^{-1}/a_T = 10^{-3}\ \text{s}^{-1}$, which may be accessible to the rheometer motor.

If the principle of time–temperature equivalency is valid for this sample, then the value of G* measured at 90°C and a frequency of $\omega = 10^{-3}\ \text{s}^{-1}$ is approximately equal to the value of G* for the same sample at 25°C and $\omega = 10^{-6}\ \text{s}^{-1}$. Unfortunately, this principle fails for many complex polymer materials. In particular, the principle fails if the sample has a phase transition in the temperature range between the use temperature and the testing temperature.

Conversion among Rheological Properties Linear rheological properties measured in oscillatory shear can be rigorously converted to linear rheological properties defined for stress relaxation tests, shear creep tests, or any other linear experiment. The software needed for the conversion is often supplied with the rheometer. However, the software cannot be used to calculate nonlinear rheological properties such as the non-Newtonian viscosity versus shear rate.

Parallel Disk

The principal advantage of the cone-and-plate instrument is that it imposes a uniform value of the strain rate κ on the sample in flow experiments and a uniform value of the shear strain γ in DMA experiments. This is particularly useful when analyzing liquids such as molten thermoplastics having properties that strongly depend on κ.

Unfortunately, the cone-and-plate geometry cannot be used to analyze solids. Recall that the cone, which has a truncated tip, should be slowly lowered into the sample until the truncated region is only 50–100 μm above the plate, depending on the cone. This cannot be done with solid samples. Probably, it should not be done either with liquid samples containing particles larger than a few micrometers in size, because particles of this size are large enough to disturb the flow field in the narrow gap region of the cone-and-plate instrument.

Geometry The parallel-disc rheometer geometry (Figure 1.60g) is obtained by replacing the cone in a cone-and-plate instrument with a stainless steel disc of the same diameter. All other features of the rheometer, including transducers and drive motors, remain the same. The sample must be cut or molded into a disc of uniform thickness and radius equal to the rheometer disc radius. The sample is sheared in between the two parallel stainless steel discs or plates, with the same rheometer gap at all radial positions. The parallel-disc geometry can be used to study a solid sample of any thickness, provided that the sample adheres to the surfaces of the discs. For analysis of suspensions, one can minimize wall-slip errors by choosing the gap to be much larger than the suspended particle size.

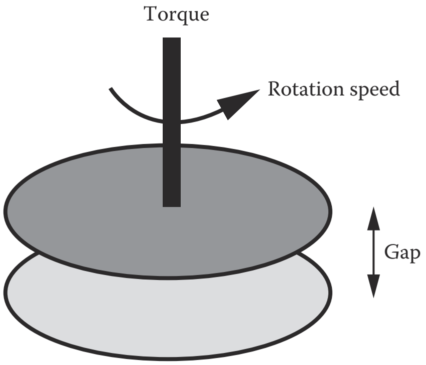


FIG. 1.60g
Parallel-disk instrument, where the "gap" = L.

The parallel-disc instrument, like the cone-and-plate, can be used to make either stress-controlled or strain-controlled measurements on the sample in steady-shear flow or oscillatory shear strain. The flow field in the instrument is expected to be equivalent to simple shear flow (Figure 1.60c) on a local level, but the local value of the shear rate κ increases with radial coordinate. The maximum value of the shear rate occurs in the sample located at the rim of the parallel discs, and is equal to

$$\kappa_{\max} = \frac{R\Omega}{L} \qquad \textbf{1.60(8)}$$

where
R is the radius of the discs
Ω is the angular speed of the rotating disc
L is the gap between the discs

Viscosity $\mu^{®}$ N_1, G′, or G″. What is this®, it should be,

Limitation The principal disadvantage of the instrument arises from the variation of the shear rate value within the sample. As a consequence, the parallel-disc instrument cannot be used to make a single-point measurement of the viscosity of a non-Newtonian liquid. This may be seen from the fluid mechanics equation used to calculate the viscosity. If the shear rate at the rim of the discs is $\kappa_{\max}$, and the torque on the discs is T, then the sample viscosity value at shear rate equal to $\kappa_{\max}$ is given by

$$\mu = T\left(2\pi R3\kappa\max\right)^{-1}\left[\frac{3 + d\ln\left(T/\left(2\pi R3\right)\right)}{d\ln\left(\kappa\max\right)}\right] \qquad \textbf{1.60(9)}$$

Equation 1.60(9) contains the derivative of the torque with respect to the shear rate at the rim, in contrast to the analogous equation (Equation 1.60(3)) used with the cone-and-plate instrument. Hence, to determine the viscosity at a single value of the shear rate, one has to measure the torque at several different rim shear rate values and then numerically evaluate the derivative in Equation 1.60(9).

Rectangular Torsion

The parallel disc instrument can be used only with solid samples that adhere to the stainless steel discs so that the *no slip* boundary condition is satisfied. *Slip* refers to the occurrence of relative motion between the steel and the sample located at the steel/sample interface. When slip occurs during DMA experiments, the computer software will use a value for the strain that is larger than the true value imposed on the sample.

One way to detect the occurrence of slip is to see if slight compression of the sample between the parallel plates has a drastic effect on the DMA results. When slip is a problem, the rectangular torsion instrument (Figure 1.60h) offers a viable alternative to the parallel-disc instrument for solid samples. The sample must be cut or molded into a thin rectangular specimen of uniform thickness.

The specimen is clamped into the instrument at both ends, and the clamping force is adjusted so that no slip can occur. During testing, one clamp undergoes low amplitude oscillatory rotations at frequency ω while the torque required to keep the other clamp stationary is measured with a torque transducer. The experimentally determined properties are the dynamic shear moduli of the sample, G′ and G″, as a function of frequency.

Torsional DMA can be used to identify the specimen glass transition temperature as the temperature at which G′ drops precipitously at fixed frequency. However, torsional DMA cannot be used to determine the MWD of the specimen.

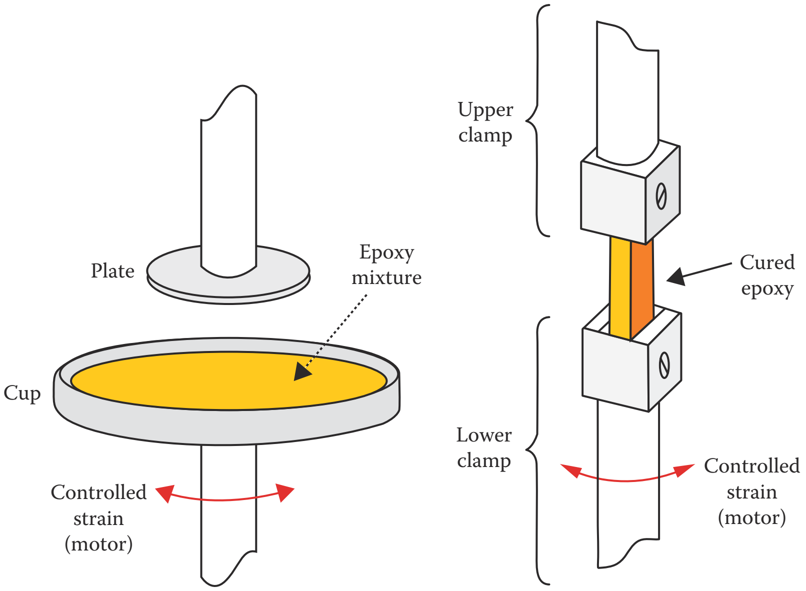


FIG. 1.60h
Rectangular torsion rheometer. (Courtesy IOP Science.)

Coaxial Cylinder

The principal advantage of the coaxial-cylinder instrument (Figure 1.60i) is a large surface area for contact between the sample and the instrument, which increases torque levels and hence is advantageous for measuring low viscosity values. However, the instrument cannot be used to measure normal stress differences and cannot easily be used with solid samples. The liquid sample completely fills the narrow gap or annulus between two concentric cylinders.

During testing, the outer cylinder (called the *cup*) is rotated while the inner cylinder (the *bob*) is held stationary. When the cup is rotated continuously, the flow field in the sample looks like simple shear flow (Figure 1.60c) on a local level. However, as with the parallel-disc instrument, the shear rate value varies with location within the sample. The sample viscosity is calculated from the measured torque on the bob. Linear shear moduli G′ and G″ can also be measured in experiments in which the cup undergoes oscillatory rotations.

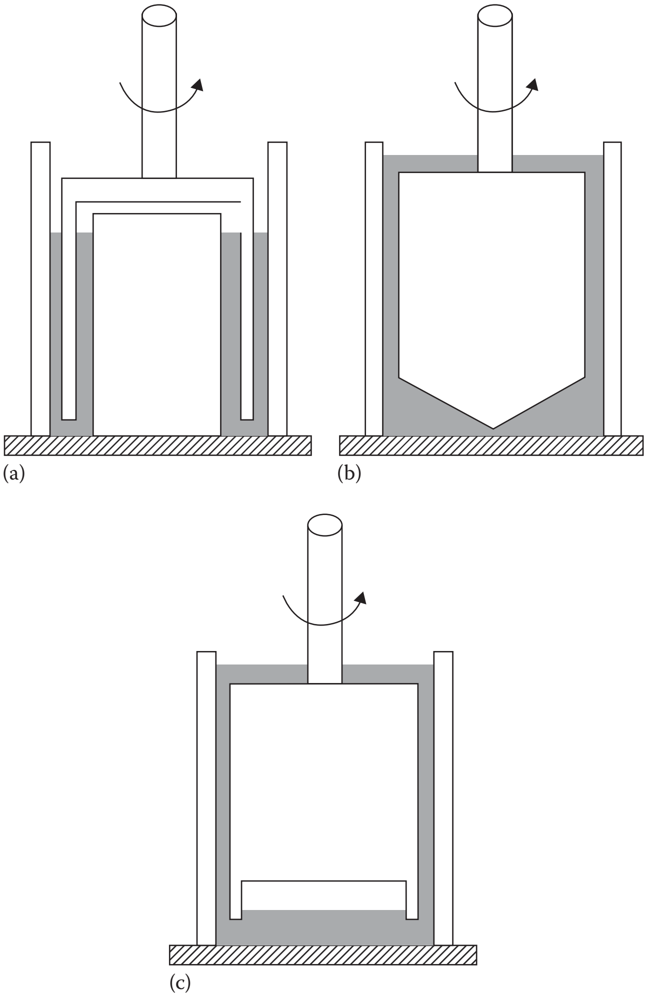

(a)
(b)
(c)

FIG. 1.60i
Three alternative cylindrical designs. (a) is the double gap, (b) the one with cone and plate at the bottom, and (c) is the hollow cavity at the bottom configuration, which serves to trap the air. (Courtesy of National Institute of Standards and Technology.)

Tension/Compression and Bending

All of the instruments considered thus far impose a shear deformation (Figure 1.60b) on the sample. However, the behavior of a solid sample during tension, compression, or bending may also be of interest, and instruments are also available for these types of deformations.

In fact, the principal difference between a so-called *rheometer* and a *dynamic mechanical analyzer* is the number of instruments available with the latter for applying various bending and tensile deformations on solid samples. The bending and tensile/compression deformations are usually applied to the specimen as an oscillatory function of time at angular frequency ω. When a small time-independent uniaxial tensile deformation is applied to a specimen, the ratio of the tensile stress to the tensile strain is defined as the linear elastic tensile modulus E.

In oscillatory experiments, one measures at each frequency the tensile storage modulus (E′) and tensile loss modulus (E″), properties which are analogous to G′ and G″, defined earlier for shear. In fact, for a material that does not undergo volume changes during deformation, the dynamic tensile moduli are exactly three times larger than the corresponding dynamic shear moduli.

Extensional Flow

The value of the extensional viscosity μ_{ex} is a measure of a liquid's resistance to stretching during flow. For commercial thermoplastics, the value of μ_{ex} is important in plastics-forming operations such as fiber spinning and blown-film extrusion.

Figure 1.60j shows an idealized representation of uniaxial extensional flow. Here, the cylinder of liquid is stretching along its longitudinal axis while contracting in the radial dimension so that the volume remains constant. To obtain a constant extension rate, the stretching force F is adjusted in such a way that the length z of the liquid cylinder increases exponentially with time t.

$$z \propto \exp(\varepsilon t) \qquad \mathbf{1.60(10)}$$

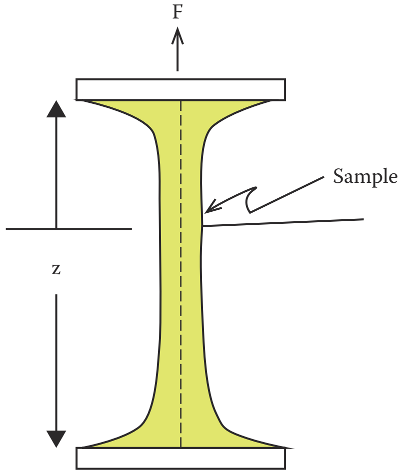


FIG. 1.60j
Uniaxial extensional flow rheometer sensor.

Here, ε is the rate of extension, analogous to the rate of strain κ in shear flow. The stretching force F and the cylinder cross-sectional area A are both functions of time. The extensional viscosity μ_{ex} is a material property defined as

$$\mu_{ex} = F/(A\varepsilon) \tag{1.60(11)}$$

Thus, for a given rate of elongation ε, the necessary stretching force increases with the elongational viscosity of the liquid μ_{ex}. Two thermoplastic samples may have identical shear viscosity curves (μ vs. κ) and completely different extensional viscosity curves (μ_{ex} vs. ε).

For some thermoplastics, the measured value of μ_{ex} calculated with Equation 1.60(11) never reaches a steady value, even though the extension rate is constant. There is little doubt that μ_{ex} is a key property for polymer processing flows that has been comparatively neglected in rheometry.

Extensional Viscosity Detectors Rheometers that have been built for measuring the extensional viscosity have used different techniques. These included the capillary breakup, rotary clamp stretching, stagnation point flow, and opposing jets techniques. However, it has proven difficult to build a robust and accurate extensional flow rheometer for industrial use.

Between 1987 and 1990, the International Committee on Rheology organized a round-robin test of extensional flow rheometers. In this test, the value of μ_{ex} was measured for an international standard fluid in various laboratories around the world. The test results were disappointing, with values measured in different laboratories varying by as much as several orders of magnitude. More recently, the filament stretching technique has found favor among academic laboratories for measurements of the extensional viscosity.

Capillary

The capillary rheometer is used to study the pressure-driven flow of a molten thermoplastic through a capillary. The viscosity of the process sample can be calculated from measurements of volumetric flow rate versus pressure drop along the capillary axis. Ordinarily, capillary rheometers are used only to calculate the viscosity; hence, these instruments are covered under the discussion of industrial viscometers (Chapter 1.71).

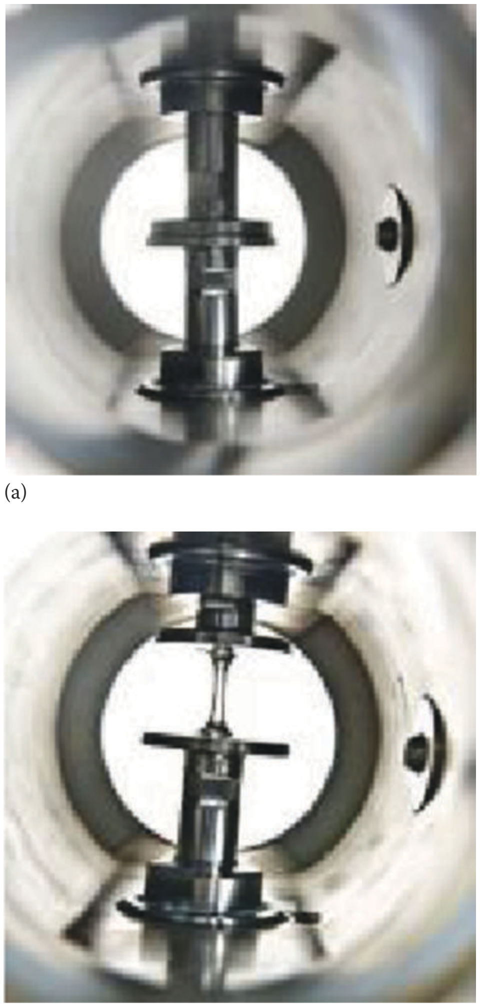
However, it is possible to use capillary rheometry to measure N_1 as well as the viscosity. This can be done by varying the length of the capillary (at fixed diameter) and noting the effect on the pressure drop within the entrance region of the capillary.

CONCLUSIONS

Molten thermoplastics have several elastic properties in addition to the viscosity that can be measured by using rheometers. True material properties can be measured only if the rheometer subjects the sample to a controlled velocity field or to a controlled deformation. Hence, the popular melt flow indexer, while useful, cannot be used to measure a true material property such as viscosity.

Probably the most significant innovation of recent years was the development of an online process rheometer for use in controlling polymer processing and similar operations, which can continuously measure the true elastic properties of a thermoplastic during processing (Figure 1.60k).

The online rheometer (OLR) continuously measures the flow properties of chemical or polymer products in the flowing pipeline, allowing the quality control of their flow properties through rheology. The flow properties of the flowing product are directly related to its pourability, viscosity, spreadability, tack, elasticity, and other textural or flow properties that can thereby be controlled quickly and online, without the need for taking grab samples for laboratory analysis.


(a)

(b)

FIG. 1.60k
The operation of the online rheometer—OLR. (a) Inside the OLR with the oscillatory squeeze-flow plates closed and (b) Inside the OLR with the oscillatory squeeze-flow plates open. (Courtesy of OnLine Rheometer Group of Rheology Solutions Pty Ltd, Bacchus Marsh, Victoria, Australia.)

SPECIFICATION FORMS

When specifying rheometers, the ISA Form 20A1001 can be used to specify the features required for the device itself, and when specifying both the device and the nature of the application, including the composition and/or properties of the process materials, ISA Form 20A1002 can be used. The following are the forms that are reproduced with the permission of the International Society of Automation.

1		RESPONSIBLE ORGANIZATION	ANALYSIS DEVICE Operating Parameters	6		SPECIFICATION IDENTIFICATIONS		
2				7		Document no		
3				8		Latest revision	Date	
4				9		Issue status		
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11		ADMINISTRATIVE IDENTIFICATIONS						40		SERVICE IDENTIFICATIONS Continued					
12		Project number		Sub project no				41		Return conn matl type					
13		Project						42		Inline hazardous area cl		Div/Zon		Group	
14		Enterprise						43		Inline area min ign temp		Temp ident number			
15		Site						44		Remote hazardous area cl		Div/Zon		Group	
16		Area		Cell		Unit		45		Remote area min ign temp		Temp ident number			
17								46							
18		SERVICE IDENTIFICATIONS						47							
19		Tag no/Functional ident						48		COMPONENT DESIGN CRITERIA					
20		Related equipment						49		Component type					
21		Service						50		Component style					
22								51		Output signal type					
23		P&ID/Reference dwg						52		Characteristic curve					
24		Process line/nozzle no						53		Compensation style					
25		Process conn pipe spec						54		Type of protection					
26		Process conn nominal size		Rating				55		Criticality code					
27		Process conn termn type		Style				56		Max EMI susceptibility		Ref			
28		Process conn schedule no		Wall thickness				57		Max temperature effect		Ref			
29		Process connection length						58		Max sample time lag					
30		Process line matl type						59		Max response time					
31		Fast loop line number						60		Min required accuracy		Ref			
32		Fast loop pipe spec						61		Avail nom power supply		Number wires			
33		Fast loop conn nom size		Rating				62		Calibration method					
34		Fast loop conn termn type		Style				63		Testing/Listing agency					
35		Fast loop schedule no		Wall thickness				64		Test requirements					
36		Fast loop estimated lg						65		Supply loss failure mode					
37		Fast loop material type						66		Signal loss failure mode					
38		Return conn nominal size		Rating				67							
39		Return conn termn type		Style				68							

69		PROCESS VARIABLES	MATERIAL FLOW CONDITIONS				101		PROCESS DESIGN CONDITIONS		
70		Flow Case Identification				Units	102		Minimum	Maximum	Units
71		Process pressure					103				
72		Process temperature					104				
73		Process phase type					105				
74		Process liquid actl flow					106				
75		Process vapor actl flow					107				
76		Process vapor std flow					108				
77		Process liquid density					109				
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79		Process liquid viscosity					111				
80		Sample return pressure					112				
81		Sample vent/drain press					113				
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Rev	Date	Revision Description	By	Appv1	Appv2	Appv3	REMARKS

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1		RESPONSIBLE ORGANIZATION		ANALYSIS DEVICE COMPOSITION OR PROPERTY Operating Parameters (Continued)	6		SPECIFICATION IDENTIFICATIONS		
2					7		Document no		
3					8		Latest revision	Date	
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5					10				

11		PROCESS COMPOSITION OR PROPERTY			MEASUREMENT DESIGN CONDITIONS				
12		Component/Property Name	Normal	Units	Minimum	Units	Maximum	Units	Repeatability
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Rev	Date	Revision Description	By	Appv1	Appv2	Appv3	REMARKS

Form: 20A1002 Rev 0

Definitions

Newtonian fluid: In a Newtonian fluid, the relationship between the shear stress and the shear rate is linear and this proportionality is the coefficient of viscosity.

Non-Newtonian fluid: In a non-Newtonian fluid, the relationship between the shear stress and the shear rate is not a constant (viscosity) and can even be time dependent. Therefore, non-Newtonian is a fluid whose flow properties differ in any way from those of Newtonian ones. Most commonly the viscosity (the measure of a fluid's ability to resist gradual deformation by shear or tensile stresses) of non-Newtonian fluids is dependent on shear rate or shear rate history. Many salt solutions and molten polymers are non-Newtonian fluids, as are many commonly found substances such as ketchup, custard, toothpaste, starch suspensions, paint, blood, and shampoo.

Peltier effect is whereby heat is given out or absorbed when an electric current passes across a junction between two materials.

Rheometer: It characterize the viscoelastic properties of polymer melts and other materials. They are laboratory instruments that are used to measure the nature of the flow of polymers and other a liquids, suspensions, or slurries when a force causes them to move. They are used for those fluid flows that cannot be defined by viscosity alone and require more parameters to be measured and controlled in order to define the polymer's flow characteristics. Rheometers are quality control tools and serve to assess the processability of resins, are used by plastics compounders, and also serve as R&D tools to help determine which resin best fits a particular process or application.

Viscosity is the fluid's resistance to gradual deformation caused by shear or tensile stresses.

Abbreviations

DMA	Dynamic Mechanical Analysis
MWD	Molecular Weight Distribution
OLR	Online Rheometer

Organization

NIST	National Institute of Standards and Technology

Bibliography

Baird, D. G., First normal stress difference measurements for polymer melts at high shear rates in a slit-die using hole and exit pressure data (2008) http://www.che.vt.edu/Faculty/Baird/Papers/2008_1.pdf.

Barnes, H. A., *A Handbook of Elementary Rheology* (2000) http://citeseerx.ist.psu.edu/showciting?cid=24183441.

Ceramic Industry, Using rheometry to improve manufacturing (2002) http://www.ceramicindustry.com/articles/print/using-rheology-to-improve-manufacturing.

Chhabra, R. P. and Richardson, J. F., Non-Newtonian flow in the process industries (1999). http://www.sciencedirect.com/science/article/pii/B9780750637701500038.

Clasen, C., Capillary breakup extension rheometry, 2010. http://cheric.org/PDF/KARJ/KR22/KR22-4-0331.pdf.

Dealy, J. M. and Wissbrun, K. F., *Melt Rheology and Its Role in Plastics Processing*, Van Nostrand Reinhold, New York, 1999. http://www.abebooks.com/book-search/isbn/0792358864/.

Gotro, J., Rheometers (2014) http://polymerinnovationblog.com/rheology-thermosets-part-2-rheometers/.

Harward Edu., Rheometry (2011) http://soft-matter.seas.harvard.edu/index.php/Rheometry.

Hassager, O., Danish Polymer Centre, Extensional rheometry (2013) http://www.dpc.kt.dtu.dk/Instrumentation/Extensional_Rheometry.aspx.

Macosko, C. W., *Rheology: Principles, Measurements, and Applications*, Wiley-VCH, New York, 1994. http://www.wiley.com/WileyCDA/WileyTitle/productCd-0471185752.html.

Malvern, Rotational Rheometry (2012) http://www.anamet.cz/sites/all/storage/school_of_rheology_part_1_rotational.pdf.

Rheology Solutions Pty Ltd (ORL), Rheology solutions (2015) http://www.onlinerheometer.com/downloads/olr004.pdf.

Schweitzer, T., A cone-partitioned plate rheometer cell with three partitions (CPP3) to determine shear stress and both normal stress differences for small quantities of polymeric fluids (2013) http://scitation.aip.org/content/sor/journal/jor2/57/3/10.1122/1.4797458.

Xu, J. et al., Use of a sliding plate rheometer to measure the first normal stress difference at high shear rates (2007) http://link.springer.com/article/10.1007/s00397-006-0156-5?no-access=true.

1.61 Sand Concentration and Subsea Pipeline Erosion Detectors

H. S. GAMBHIR and S. YADAV (2017)

Instrument types	Passive acoustic sand detector Intrusive and erosion based sand detector
Applications	Subsea or onshore pipeline monitoring
Method of detection	A. Acoustics B. Intrusive
Design pressure	Up to 9,775 psig (674 barg) for passive acoustic sensors Up to 10,790 psig (744 barg) for intrusive erosion based sensors
Design temperature	Temperature limit at pipe surface is from −40°F to +392°F (−40°C to +200°C); for instrument electronics, the limit is +10°F to +140°F (−12°C to +60°C)
Design water depth	Up to 3000 m
Unit of measurement	Micrometer (μm)
Accuracy	0.1% of full scale or better
Range	0–4 mm; metal loss resolution 5 nm
Wetted materials of construction	One of the manufacturers provides probe material AISL 316L as standard (other material on request); Erosion element material is Monel® 400
Cost	\$40,000–\$80,000 for passive acoustic type \$90,000–\$120,000 for intrusive erosion based
Partial list of suppliers	Aquip Systems PTY Ltd (A & B), http://www.aquip.com.au/products/acoustic-sand-detectors/ Clampon (A), http://www.clampon.com/ Emerson Process Management—ROXAR (A & B), http://www2.emersonprocess.com/en-US/brands/roxar/sanderosion/subseasanderosion/Pages/SubseaSand.aspx Teledyne Cormon (A & B), http://www.cormon.com/products/sand_erosion.aspx

INTRODUCTION

This chapter describes the technology behind the detection of sand and erosion in most of the oil and gas development fields. It also identifies the type of measurements, its principle of operation, and the installation and retrieval mechanism.

BACKGROUND[*,†,‡]

The production of sand particles along with the process fluid in the oil and gas subsea can lead to erosion and damage of the pipeline, process systems and clogging of filters, etc.

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[*] Bill Hedges and Andy Bodington, A comparison of monitoring techniques for improved erosion control: A field study.

[†] Birkeland R., S.E. Lilleland, R. Johnsen, N.A. Braaten, Erosion monitoring manages sand production, *Oil & Gas Journal*, September 28, 1998, http://www.ogj.com/articles/print/volume-96/issue-39/in-this-issue/general-interest/erosion-monitoring-manages-sand-production.html.

[‡] Earles, D.M., C.W. Stoesz, A.S. Amaral, B. Hughes, J.G. Pearce, H.A. DeJongh, and F.H.K. Rambow, Real-time monitoring of sand control completions (SPE 134555), Shell Exploration and Production, September 19–22, 2010.