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# Guide to Rheological Nomenclature: Measurements in Ceramic Particulate Systems

Vincent A. Hackley and Chiara F. Ferraris



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# Guide to Rheological Nomenclature: Measurements in Ceramic Particulate Systems

Vincent A. Hackley

Ceramics Division Materials Science and Engineering Laboratory National Institute of Standards and Technology Gaithersburg, MD 20899-8520

Chiara F. Ferraris

Building Materials Division Building and Fire Research Laboratory National Institute of Standards and Technology Gaithersburg, MD 20899-8621

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U.S. DEPARTMENT OF COMMERCE Norman Y. Mineta, Secretary

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# Preface

Nomenclature is fundamentally important to the progress of science and technology. Because of the rapidly growing volume of available technical information, and the speed with which this information must now be processed, it is critical to have a uniform terminology in place for describing experimental methods and instrumentation, to facilitate the exchange of material property data and for sharing technical ideas and concepts. This is especially true for the ceramics industry because of its breadth and interdisciplinary nature. The present document addresses the issue of nomenclature for one important area of ceramic science: rheology and rheometric measurements.

Ceramic suspensions, gels and pastes are the starting materials for a wide variety of applications, playing critical roles in the processing of products ranging from whitewares to concrete to paint and pigments to high-tech multilayer ceramic packages for the advanced electronics industry. Unfortunately, researchers and engineers working in these diverse fields often speak different languages, thus resulting in miscommunication and ambiguity. Even within the same field, variations in terminology are common. The need for widely accepted, uniform and precise nomenclature was emphasized recently by the Ceramic Processing Characterization Council<sup>§</sup> (CPCC), a voluntary organization of participants representing industry, instrument companies, academia and government. CPCC members identified nomenclature as a high priority issue in the areas of dispersion and rheology. In response to this need, we have initiated a series of guides to help in the interpretation of nomenclature and data relating to particle-liquid ceramic systems.

In compiling the present guide, we drew upon a number of sources, including books, review articles and published terminologies. To the extent possible, an effort was made to maintain a degree of uniformity with existing standards and conventions. To this end, definitions are generally consistent with nomenclatures published by the American Concrete Institute (ACI), the British Standards Institute (BSI), the International Union of Pure and Applied Chemistry (IUPAC) and the Society of Rheology. A complete bibliography of sources is provided.

An international ad hoc committee was formed to review the technical content and language of the nomenclature and to offer input where needed. The committee members represent a broad spectrum of experts from industry, academia and government, and include (in alphabetical order) Lennart Bergström (YKI), John Dealy (McGill University), Ed Garboczi (NIST), Bob Hunter (University of Sydney), Kamal Khayat (University of Sherbrooke), François de Larrard (Laboratoire Central des Ponts et Chaussees), Makio Naito (JFCC), Celik Ozyildirim (Virginia Transportation Research Council), Ponserril Somasundaran (Columbia University), and Abbas Zaman (University of Florida). Their efforts are gratefully acknowledged.

It is our primary hope that this document will serve as a resource to those working in particle systems applications, particularly in the ceramic sciences. It is our further hope that this document will serve to promote the establishment of a commonly recognized system of terminology throughout the ceramics community.

<sup>&</sup>lt;sup>§</sup> Formerly the Ceramic Processing Characterization Consortium

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# GUIDE TO RHEOLOGICAL NOMENCLATURE FOR LIQUID-BASED PARTICLE SYSTEMS

# 1. Introduction

Measurements and standards play an integral role in the manufacture and utilization of ceramic powders, components, coatings and composites. They enhance reliability by providing a basis for quantifying and comparing material properties during each phase of the manufacturing process, from raw materials to the finished product. Equally important is the establishment of a uniform and widely accepted nomenclature to permit the efficient exchange of scientific and technical information, and to provide a sound basis on which to standardize measurement methods and data reporting practices. This document represents the second<sup>§</sup> in a series of guides intended to serve the dual purpose of helping to establish a uniform system of terminology, geared towards the ceramics community, while also providing a convenient resource for the practicing engineer or scientist.

This document provides definitions of terms and expressions relating to the measurement of rheological properties in liquid-based ceramic particulate systems (i.e., suspensions, pastes and gels). The term *ceramic* is here broadly defined as a non-metallic inorganic material. So, although the focus is on ceramic applications, the definitions are largely generic in nature, and should be compatible with many non-ceramic applications. Equations have been used sparingly, and only where necessary for clarity or where they are integral to the subject at hand.

Besides having a glossary of basic terms, the guide is organized so that the reader can more readily locate related terms associated with specific subject areas (e.g., concrete applications or oscillatory measurements). Defined terms are indicated in bold face type. Where alternative or equivalent terms exist, they are shown in brackets adjacent to the preferred term. Terms used in definitions, and which are defined separately in the guide, are indicated in italic at their first appearance in that paragraph or section. The only exception to this rule is the term viscosity, which is not italicized when it appears by itself, due to its ubiquity throughout the text. Furthermore, an alphabetized index to the defined terms is provided.

Descriptions of instrumentation provided in this guide are generic in nature, and are presented solely for the purpose of identifying key measurement techniques and their associated nomenclature.

# 2. Fundamental Rheological Properties

**elastic** A conservative property in which part of the mechanical energy used to produce *deformation* is stored in the material and recovered on release of *stress*.

**electroviscous** The collective effects by which the presence of an electrical double-layer influences the *flow* behavior of a liquid. The double-layer is usually associated with charged

<sup>&</sup>lt;sup>§</sup> see also *Guide to the Nomenclature of Particle Dispersion Technology for Ceramic Systems*, NIST Special Publication 945, February 2000.

particles suspended in the liquid or polyelectrolytes dissolved in solution. There are three electroviscous effects:

**primary** The increase of fluid viscosity due to distortion of the double-layer during *shear*. Distortion exerts a drag, increasing energy dissipation.

**secondary** The increase of fluid viscosity due to the interaction or overlap of adjacent double-layers. Responsible for the formation of gelatin.

**tertiary** The variation in fluid viscosity that arises from geometrical changes within the fluid due to double-layer interactions. The viscosity of a polyelectrolyte solution depends on the conformation of the molecules, which in turn is affected by intramolecular electrostatic interactions between charged segments located along the polymer backbone or on side chains.

**plastic** The property of a solid body that is in the *elastic* state when the *stress* is below a critical value, termed the *yield stress*, and in the plastic state when this value is exceeded. During ideal plastic *flow*, energy dissipation and stress are independent of the rate of *deformation*.

**viscoelastic** A time-dependent property in which a material under *stress* produces both a *viscous* and an *elastic* response. A viscoelastic material will exhibit viscous *flow* under constant stress, but a portion of mechanical energy is conserved and recovered after stress is released. Often associated with polymer solutions, melts and structured suspensions, viscoelastic properties are usually measured as responses to an instantaneously applied or removed constant stress or *strain* or a *dynamic stress* or strain.

**viscoelectric** An effect by which the electric field near a charged interface influences the structure of the surrounding fluid and thereby modifies the viscosity of the fluid.

**viscoplastic** A hybrid property in which a material behaves like a solid below some critical *stress* value, the *yield stress*, but flows like a *viscous* liquid when this stress is exceeded. Often associated with highly aggregated suspensions and polymer gels.

**viscous** The tendency of a liquid to resist *flow* as a result of internal friction. During viscous flow, mechanical energy is dissipated as heat and the *stress* that develops depends on the rate of *deformation*.

# 3. Functional Forms of Viscosity

Refer to Section 11 for a complete list of symbols used the following definitions. Some viscosity terms relating to oscillatory measurements are defined both here and more explicitly in Section 8.

**apparent viscosity**,  $\eta_{app}$  The value of viscosity evaluated at some nominal average value of the *shear rate*. The apparent viscosity applies, for instance, in the *capillary method*, where a range of shear rates are employed.

**coefficient of fluidity** [**fluidity**],  $\phi$  Reciprocal of the *coefficient of viscosity*.

 $\phi = 1 / \eta$ 

**coefficient of viscosity** [viscosity],  $\eta$  The ratio of *shear stress* to *shear rate* under simple *steady shear*. The abbreviated form "viscosity" is used most often in practice, and is frequently employed without discriminating between *Newtonian* and *non-Newtonian* behavior. When the quotient is independent of shear rate (i.e., follows the Newtonian model), it is considered a material constant. When the quotient is dependent on shear rate (i.e., non-Newtonian), it should be referred to as the **non-Newtonian viscosity**.

$$\eta = \sigma/\dot{\gamma}$$

**complex viscosity**,  $\eta^*$  The frequency-dependent viscosity function determined during *forced harmonic oscillation* of *shear stress*; contains both real and imaginary parts.

differential viscosity,  $\eta_{diff}$  The derivative of *shear stress* with respect to *shear rate*.

$$\eta_{\rm diff} = \partial \sigma / \partial \dot{\gamma}$$

**dynamic viscosity**,  $\eta'$  The ratio of the *loss modulus* to the angular frequency, determined during *forced harmonic oscillation* (dynamic) measurements. The real part of the *complex viscosity*.

$$\eta' = G'' / \omega$$

infinite shear viscosity,  $\eta_{\infty}$  The high *shear rate* limiting value of viscosity. Often associated with the second *Newtonian* region in *pseudoplastic* fluids.

inherent viscosity [logarithmic viscosity],  $\eta_{inh}$  A natural logarithmic function of the *relative viscosity* reduced by the solute concentration.

$$\eta_{inh} = \frac{\ln \eta_r}{c}$$

intrinsic viscosity [limiting viscosity number],  $[\eta]$  The zero concentration limiting value of the *reduced specific viscosity*. A characteristic function for the single molecule in solution.  $[\eta]$  is equivalent to the effective hydrodynamic specific volume for the solute.

$$[\eta] = \lim_{c \to 0} \eta_{red}$$

**kinematic viscosity**, v The ratio of the viscosity of a fluid to its density.

$$v = \eta_s / \rho$$

**out-of-phase viscosity**,  $\eta''$  The ratio of the *storage modulus* to the angular frequency, determined during *forced harmonic oscillation* (dynamic) measurements. The imaginary part of the *complex viscosity*.

$$\eta'' = G' / \omega$$

**plastic viscosity**,  $\eta_{pl}$  For a *Bingham* model, the excess of the *shear stress* over the *yield stress* divided by the *shear rate*, and equal to the *differential viscosity*. For *non-ideal Bingham* materials, the differential viscosity determined in the high-shear limiting, linear portion of the *flow curve*; associated with *viscoplastic* fluids.

$$\eta_{\rm pl} = \lim_{\dot{\gamma} \to \infty} \partial \sigma \, / \, \partial \dot{\gamma}$$

reduced viscosity [viscosity number],  $\eta_{red}$  The ratio of any viscosity function to the concentration, c, of the solute or particulate phase. By normalizing viscosity in this way, concentration effects become more apparent. For instance, the reduced *specific viscosity* is:

$$\eta_{red} = \eta_{sp} / c$$

relative viscosity [viscosity ratio],  $\eta_r$  Ratio of the viscosity in a suspension or solution to the viscosity of the suspending medium or solvent.

$$\eta_r = \eta / \eta_s$$

specific viscosity,  $\eta_{sp}$  The *relative viscosity* minus unity.

$$\eta_{sp} = \eta_r - 1$$

**zero shear viscosity**,  $\eta_0$  The low *shear rate* limiting value of viscosity. Associated with the first *Newtonian* region in many *pseudoplastic* fluids.

$$\eta_0 = \lim_{x \to 0} \eta$$

# 4. Glossary of Basic Terminology

**apparent yield stress** For non-ideal *viscoplastic* materials, where the *yield stress* is indefinite, an apparent yield stress can be defined, for example, by extrapolation from the linear, high-*shear-rate* portion of the *flow curve* to the *stress* axis. (see *Bingham* relation)

**compliance** The quotient of *strain* and *stress* (e.g., *shear compliance*). The reciprocal of *modulus*.

**deformation** Movement of parts or particles of a material body relative to one another such that the continuity of the body is not destroyed, resulting in a change of shape or volume or both.

**dilatant** A property often associated with suspensions of irregularly shaped particles, in which the liquid exhibits an increase in volume while being sheared. The term is also used in common practice to mean *shear-thickening*, the increasing resistance to shear with increasing *shear rate*. It is possible for either of these two effects to exist in the absence of the other.

**dynamic equilibrium** A state in which dynamic opposing forces just balance to obtain a quasiequilibrium condition. Dynamic equilibrium is achieved during *steady shear flow* when breakdown and rebuilding of *structure* occur at similar rates, and the viscosity is constant at a given *shear rate*.

**dynamic** [oscillatory] shear flow Condition under which *stress* and *strain* vary harmonically with time during a *rheometric* experiment.

**Einstein's law of viscosity** Describes the relationship between the viscosity of a dilute dispersion and the volume fraction of the dispersed particles. The relationship is derived with two major assumptions, that the particles are solid spheres and that their concentration is very low.

$$\frac{\eta}{\eta_S} = 1 + 2.5 \phi + \dots$$

where  $\eta_s$  is the viscosity of the suspending medium and  $\phi$  is the volume fraction. The factor 2.5 is known as the **Einstein coefficient**. To describe more concentrated dispersions, higher terms in the power series have been retained and analyzed with varying success.

**equilibrium** [steady state] flow Condition under which a constant *stress* or *shear rate* is maintained for a sufficient time to allow *dynamic equilibrium* to be achieved in a fluid containing time-dependent *structure*. An equilibrium *flow curve* can be used to characterize the time-independent *flow* properties of a material.

elastic modulus [modulus of elasticity] A modulus of a body that obeys Hooke's law.

**flow** Continuously increasing *deformation* of a material body under the action of finite forces. When the force is removed, if the *strain* does not eventually return to zero, then flow has occurred.

flow curve A graphical representation of the behavior of flowing materials in which *shear stress* is related to *shear rate*.

**Hooke's law** Provides that the quotient of *stress* and *strain* (i.e., the *modulus*) is a constant. A body obeying Hooke's law cannot be *viscoelastic* nor does *flow* occur.

laminar flow *Flow* without turbulence.

**modulus** The quotient of *stress* and *strain* where the type of stress and strain is defined by the type of *deformation* employed (e.g., *shear modulus* in shear deformation).

**Navier-Stokes equations** The equations of motion for a *Newtonian* fluid model describing the balance between external forces (like gravity), the pressure force and the viscous force.

**Newtonian** *Flow* model of fluids in which a linear relationship exists between *shear stress* and *shear rate*, where the *coefficient of viscosity* is the constant of proportionality.

**no-slip** Condition in which fluid adjacent to a surface moves with the velocity of that surface. The assumption of no-slip is key to most *rheometric* measurements, especially those involving rotational devices. Slippage is a significant concern in concentrated suspensions.

**non-equilibrium flow** Condition under which *shear* is varied at a rate that does not permit *dynamic equilibrium* to be achieved. A *thixotropic loop* is the result of non-equilibrium flow conditions during shear cycling.

**non-Newtonian** Any *laminar flow* that is not characterized by a linear relationship between *shear stress* and *shear rate*.

**normal stress**,  $\sigma_n$  The component of *stress* that acts in a direction normal to the plane of *shear*.

**Peclet number**, *Pe* A dimensionless group used to compare the effect of applied *shear* with the effect of thermal (Brownian) motion;  $Pe=r^2\dot{\gamma}/D_T$ , where *r* is the particle radius and  $D_T$  is the translational diffusion coefficient. For *Pe* << 1, particle behavior is dominated by diffusional relaxation, whereas for *Pe* >>1, hydrodynamic effects dominate.

**Poiseuille flow** Laminar flow in a pipe of circular cross section under a constant pressure gradient. (see also Capillary Methods)

**Reynolds number**, Re A dimensionless group that expresses the ratio of the inertial forces to the *viscous* forces;  $Re=Dv\rho/\eta$ , where D is a characteristic dimension (e.g., particle size or pipe diameter), v is a typical fluid speed, and  $\eta/\rho$  is the *kinematic viscosity* of the fluid. The transition from *laminar* to turbulent *flow* is characterized by high Re values.

**rheology** The science of the *deformation* and *flow* of matter.

rheometric Refers to the measurement of rheological properties.

shear The relative movement of parallel adjacent layers.

**shear compliance**, *J* The ratio of *shear strain* to its corresponding *shear stress*. The reciprocal of *shear modulus*.

**shear modulus [modulus of rigidity]**, *G* The ratio of *shear stress* to its corresponding *shear strain*. The reciprocal of *shear compliance*.

shear rate [rate of shear strain],  $\dot{\gamma}$  The rate of change of *shear strain* with time  $(d\gamma/dt)$ . For liquids, the *shear rate*, rather than *strain*, is generally used in describing *flow*.

shear stress,  $\sigma$  The component of *stress* that causes successive parallel layers of a material body to move, in their own planes (i.e., the plane of *shear*), relative to each other.

**shear strain**,  $\gamma$  The relative in-plane displacement,  $\Delta x$ , of two parallel layers in a material body divided by their separation distance, *y*. Alternatively, the shear strain can be defined as *tan*  $\theta$ , where  $\theta$  is the angle of *deformation* as shown in Figure 1.

**shear-thickening** An increase in viscosity with increasing *shear rate* during *steady shear flow*. The term *dilatant* is commonly used in practice to indicate shear-thickening, although this usage is strictly incorrect.



Figure 1 Shear strain schematic.

**shear-thinning** [**pseudoplastic**] A decrease in viscosity with increasing *shear rate* during *steady shear flow.* 

**simple shear** In simple shear there is no change in the dimension normal to the plane of *shear*, and the relative displacement of successive parallel layers of a material body are proportional to their distance from a reference layer. The type of *flow* used in most *rheometric* measurements on fluids can be approximated by simple shear.

**steady shear flow** Condition under which a fluid is sheared continuously in one direction during the duration of a *rheometric* experiment.

stress Force per unit area.

**structure** In *rheology*, structure is a term that refers to the formation of stable physical bonds between particles (or chemical bonds between macromolecules) in a fluid. These bonds result in aggregate, floc, or network structure, which impacts the rheological behavior of the fluid and provides *elastic* and *plastic* properties. The term may be extended to include structural effects caused by *electroviscous* interactions, physical bonds between polymers (e.g., associative thickeners), shear-induced alignment of anisotropic particles, and radial distribution correlations in concentrated suspensions. Often, the term "structure" is invoked when little is known about the cause of observed changes in rheological properties.

**Weissenberg effect** The tendency of some *viscoelastic* fluids to *flow* in a direction normal to the direction of *shear*. The effect is manifested by behavior such as the climbing of a fluid up a rotating rod.

**Weissenberg number**, *Wi* A measure of the degree of nonlinearity or the degree to which *normal stress* differences are exhibited in a *flow*. For *steady, simple shear*, the Weissenberg number is the product of the *shear rate* and a characteristic time of the fluid. In oscillatory shear it is the product of the shear rate amplitude and the characteristic time of the fluid. In converging flows it is proportional to the *Deborah number*.

**yield response** For non-ideal *viscoplastic* materials, the *yield stress* can be indefinite and yielding may occur continuously over a narrow range of *stress* values. In this case, the behavior may be more properly termed a yield response.

yield stress,  $\sigma_y$  A critical *shear stress* value below which an ideal *plastic* or *viscoplastic* material behaves like a solid (i.e., will not flow). Once the yield stress is exceeded, a plastic material yields (deforms plastically) while a viscoplastic material flows like a liquid.

# 5. Classification of Equilibrium Flow Curves

Steady-shear flow curves for suspensions and solutions measured under equilibrium conditions may exhibit a variety of behaviors over a limited range of *shear rates*. Additionally, some materials may exhibit more than one distinct behavior over different shear rate regions of the same flow curve. Several types of behavior can be classified according to their characteristic shape. The following classification system covers the six most frequently encountered *flow* types as illustrated in the accompanying graph (Figure 2).

1. Newtonian Differential viscosity and coefficient of viscosity are constant with shear rate.

2. shear-thickening *Differential viscosity* and *coefficient of viscosity* increase continuously with *shear rate*.

3. shear-thinning [pseudoplastic] Differential viscosity and coefficient of viscosity decrease continuously with shear rate. No yield value.

4. shear thinning [pseudoplastic] with yield response Differential viscosity and coefficient of viscosity decrease continuously with shear rate once the apparent yield stress,  $\sigma_{app}$ , has been exceeded.

5. Bingham plastic (ideal) Obeys the *Bingham relation* ideally. Above the Bingham yield stress ( $\sigma_B$  in Figure 2) the *differential viscosity* is constant and is called the *plastic viscosity*, while the *coefficient of viscosity* decreases continuously to some limiting value at infinite *shear rate*.





6. **Bingham plastic (non-ideal)** Above the apparent *yield stress* the *coefficient of viscosity* decreases continuously, while the *differential viscosity* approaches a constant value with increasing *shear rate*. Extrapolation of the *flow curve* from the linear, high shear rate region (*plastic* region) to the stress axis gives the apparent *Bingham* yield stress ( $\sigma_B$ \* in Figure 2). The differential viscosity in the linear region is termed the *plastic viscosity*.

# 6. Expressions for Describing Steady Shear Non-Newtonian Flow

The expressions shown in this section are used to characterize the *non-Newtonian* behavior of fluids under equilibrium, *steady shear flow* conditions. Many phenomenological and empirical models have been reported in the literature. Only those having a direct and significant implication for suspensions, gels and pastes have been included here. A brief description of each relationship is given, sometimes with examples of the types of materials to which it is typically applied. In defining the number of parameters associated with a particular model, the term "parameter" in

this case refers to adjustable (arbitrary) constants, and therefore excludes measured quantities. Some of these equations have alternative representations other than the one shown. More detailed descriptions and alternative expressions can be found in the sources listed in the bibliography.

# Bingham

$$\sigma = \sigma_{\rm B} + \eta_{\rm pl} \dot{\gamma}$$
$$\dot{\gamma} = 0 \text{ for } \sigma < \sigma_{\rm B}$$

The Bingham relation is a two parameter model used for describing *viscoplastic* fluids exhibiting a *yield response*. The ideal Bingham material is an *elastic* solid at low *shear stress* values and a *Newtonian* fluid above a critical value called the Bingham *yield stress*,  $\sigma_B$ . The *plastic viscosity* region exhibits a linear relationship between shear stress and *shear rate*, with a constant *differential viscosity* equal to the plastic viscosity,  $\eta_{pl}$ .

# Carreau-Yasuda

$$\frac{\eta - \eta_{\infty}}{\eta_0 - \eta_{\infty}} = \left[1 + \left(\lambda \dot{\gamma}\right)^a\right]^{(n-1)/a}$$

A model that describes *pseudoplastic* flow with asymptotic viscosities at zero ( $\eta_0$ ) and infinite ( $\eta_\infty$ ) *shear rates*, and with no *yield stress*. The parameter  $\lambda$  is a constant with units of time, where  $1/\lambda$  is the critical shear rate at which viscosity begins to decrease. The *power-law* slope is (*n*-1) and the parameter *a* represents the width of the transition region between  $\eta_0$  and the power-law region. If  $\eta_0$  and  $\eta_\infty$  are not known independently from experiment, these quantities may be treated as additional adjustable parameters.

# Casson

$$\begin{split} \sigma^{1/2} &= \sigma_y^{1/2} + \eta_{pl}^{1/2} \dot{\gamma}^{1/2} \\ \dot{\gamma} &= 0 \text{ for } \sigma < \sigma_y \end{split}$$

A two parameter model for describing *flow* behavior in *viscoplastic* fluids exhibiting a *yield* response. The parameter  $\sigma_y$  is the *yield* stress and  $\eta_{pl}$  is the differential high shear (*plastic*) viscosity. This equation is of the same form as the *Bingham* relation, such that the exponent is  $\frac{1}{2}$  for a Casson plastic and 1 for a Bingham plastic.

# Cross

$$\frac{\eta - \eta_{\infty}}{\eta_0 - \eta_{\infty}} = \frac{1}{\left(1 + \lambda \dot{\gamma}^m\right)}$$

Similar in form to the *Carreau-Yasuda* relation, this model describes *pseudoplastic* flow with asymptotic viscosities at zero ( $\eta_0$ ) and infinite ( $\eta_\infty$ ) *shear rates*, and no *yield stress*. The parameter  $\lambda$  is a constant with units of time, and *m* is a dimensionless constant with a typical range from  $\frac{2}{3}$  to 1. If  $\eta_0$  and  $\eta_\infty$  are not known independently from experiment, these quantities may be treated as additional adjustable parameters.

Ellis

$$\eta = \frac{\eta_0}{1 + \left(\frac{\sigma}{\sigma_2}\right)^{\alpha - 1}}$$

A two parameter model, written in terms of *shear stress*, used to represent a *pseudoplastic* material exhibiting a *power-law* relationship between shear stress and *shear rate*, with a low shear rate asymptotic viscosity. The parameter  $\sigma_2$  can be roughly identified as the shear stress value at which  $\eta$  has fallen to half its final asymptotic value.

# Herschel-Bulkley

$$\sigma = \sigma_{v} + k\dot{\gamma}^{r}$$

A three parameter model used to describe *viscoplastic* materials exhibiting a *yield response* with a *power-law* relationship between *shear stress* and *shear rate* above the *yield stress*,  $\sigma_y$ . A plot of *log* ( $\sigma - \sigma_y$ ) versus *log*  $\dot{\gamma}$  gives a slope *n* that differs from unity. The Herschel-Bulkley relation reduces to the equation for a *Bingham plastic* when *n*=1.

# Krieger-Dougherty

$$\eta_{\rm r} = \left(1 - \Phi_{\rm m}\right)^{-[\eta]\Phi}$$

A model for describing the effect of particle self-crowding on suspension viscosity, where  $\Phi$  is the particle volume fraction,  $\Phi_m$  is a parameter representing the maximum packing fraction and  $[\eta]$  is the *intrinsic viscosity*. For ideal spherical particles  $[\eta]=2.5$  (i.e., the Einstein coefficient). Non-spherical or highly charged particles will exhibit values for  $[\eta]$  exceeding 2.5. The value of  $[\eta]$  is also affected by the particle size distribution. The parameter  $\Phi_m$  is a function of particle shape, particle size distribution and *shear rate*. Both  $[\eta]$  and  $\Phi_m$  may be treated as adjustable model parameters.

The aggregate volume fraction (representing the effective volume occupied by particle aggregates, including entrapped fluid) can be determined using this equation if  $\Phi_m$  is fixed at a reasonable value (e.g., 0.64 for random close packing or 0.74 for hexagonal close packing) and

 $[\eta]$  is set to 2.5. In this case,  $\Phi$  is the adjustable parameter and is equivalent to the aggregate volume fraction.

Meter

$$\eta = \eta_{\infty} + \frac{\eta_0 - \eta_{\infty}}{1 + (\sigma/\sigma_2)^{\alpha - 1}}$$

Expressed in terms of *shear stress*, this relation is used to represent a *pseudoplastic* material exhibiting a *power-law* relationship between shear stress and *shear rate*, with both high  $(\eta_{\infty})$  and low  $(\eta_0)$  shear rate asymptotic viscosity limits. The parameter  $\sigma_2$  can be roughly identified as the shear stress value at which  $\eta$  has fallen to half its final asymptotic value. The Meter and *Carreau-Yasuda* models give equivalent representations in terms of shear stress and shear rate, respectively. If  $\eta_0$  and  $\eta_{\infty}$  are not known independently from experiment, these quantities may be treated as additional adjustable parameters.

# **Powell-Eyring**

$$\eta = \eta_{\infty} + (\eta_0 - \eta_{\infty}) \frac{\sinh^{-1}(\tau \dot{\gamma})}{\tau \dot{\gamma}}$$

Derived from the theory of rate processes, this relation is relevant primarily to molecular fluids, but can be used in some cases to describe the *viscous* behavior of polymer solutions and *viscoelastic* suspensions over a wide range of *shear rates*. Here,  $\eta_{\infty}$  is the *infinite shear viscosity*,  $\eta_0$  is the *zero shear viscosity* and the fitting parameter  $\tau$  represents a characteristic time of the measured system. If  $\eta_0$  and  $\eta_{\infty}$  are not known independently from experiment, these quantities may be treated as additional adjustable parameters.

# power-law [Ostwald-de Waele]

$$\sigma = K\dot{\gamma}^n$$

A two parameter model for describing *pseudoplastic* or *shear-thickening* behavior in materials that show a negligible *yield response* and a varying *differential viscosity*. A log-log plot of  $\sigma$  versus  $\dot{\gamma}$  gives a slope n (the *power-law* exponent), where n<1 indicates pseudoplastic behavior and n>1 indicates shear-thickening behavior.

# 7. Time-Dependent Effects

Time-dependence includes those effects associated with transient *flow* conditions as well as those effects associated with irreversible changes that result from *shear* history.

creep The response of a material to the instantaneous application of a constant stress.

**creep function** In an applied *stress* test, where an instantaneous and constant stress is applied to a material while the *shear rate* (or *shear strain*) is measured over time, the shear rate (or strain) vs. time function is termed the *creep* function. The function  $J(t)=\gamma(t)/\sigma$  is referred to as the creep *compliance*.

**Deborah number**, *De* The ratio of a characteristic *relaxation time* of a material to the duration of the observation. In *equilibrium flow*, the effective duration of the experiment is infinity, and *De*=0. In oscillatory shear, it is the product of the frequency and the relaxation time of the fluid. In converging flows, the Deborah number is proportional to the *Weissenberg number*.

**flow hysteresis** A condition resulting from differences in the rate of energy dissipation due to *shear* history. In a typical rheometric test, *shear stress* or *shear rate* is ramped at a fixed speed up to a maximum value, then ramped back down at the same speed to the beginning. In hysteresis, one *flow curve* lies above the other, forming a continuous loop whose internal area depends on the shear and thermal history of the material, and on how rapidly the stress or shear rate was ramped. If the down-curve lies below the up-curve, then it is referred to as a *thixotropic* loop, whereas if the down-curve lies above the up-curve, then it is called a *negative thixotropic* loop.

**negative thixotropy** [anti-thixotropy] A reversible time-dependent increase in viscosity at a particular *shear rate*. Shearing causes a gradual buildup of *structure* over time.

**relaxation time**,  $\tau$  A time characterizing the response of a *viscoelastic* material to the instantaneous application of a constant *strain*.

retardation time,  $\tau$  A time characterizing the response of a *viscoelastic* material to the instantaneous application of a constant *stress*.

**rheomalaxis** An irreversible decrease of viscosity during shearing. Attributed to permanent changes in the material *structure*.

**rheopexy** An effect by which a material recovers some of its pre-sheared viscosity at a faster rate when it is gently sheared compared to when it is allowed to stand. Not to be confused with *negative thixotropy*.

**stress growth** When an instantaneous and constant *strain* (or *shear rate*) is applied to a material while *stress* is measured over time, an increasing stress vs. time or *modulus* vs. time function is termed stress growth.

**stress relaxation** When an instantaneous and constant *strain* (or *shear rate*) is applied to a material while *stress* is measured over time, a decreasing stress vs. time or *modulus* vs. time function is termed stress relaxation.

**thixotropy** A reversible time-dependent decrease in viscosity at a particular *shear rate*. Shearing causes a gradual breakdown in *structure* over time.

# 8. Oscillatory Measurements

Forced harmonic oscillation is a dynamic *rheometric* test in which both *stress* and *strain* vary harmonically with time, and both *viscous* and *elastic* parameters are derived from the material response. Such tests are almost always carried out in the **linear viscoelastic regime**, which is characterized by a linear response of *dynamic viscosity* and *elasticity* with increasing *strain amplitude*.

# Material Functions Derived from Oscillatory Tests

In a typical sinusoidal oscillation experiment, the applied *stress* and resulting *strain* wave forms can be described as follows:

 $\sigma = \sigma_0 \cos \omega t$  $\gamma = \gamma_0 \cos (\omega t - \delta)$ 

.

where  $\sigma_0$  is the stress amplitude  $\gamma_0$  is the strain amplitude  $\omega=2\pi f$  is the angular frequency *t* is time  $\delta$  is the phase lag [loss angle]

Figure 3 Sinusoidal wave forms for stress and strain functions.

The phase lag and amplitude ratio  $(\sigma_0/\gamma_0)$  will generally vary with frequency, but are considered material properties under linear *viscoelastic* conditions. For an ideal solid,  $\delta=0^\circ$ , and the response is purely *elastic*, whereas for a *Newtonian* fluid yielding a purely *viscous* response,  $\delta=90^\circ$ .

The material functions can be described in terms of complex variables having both real and imaginary parts. Thus, using the relationship:

 $\cos x + j \sin x = e^{jx}$ where  $j = \sqrt{-1}$ 

Then the stress and strain can be expressed as follows:

$$\begin{split} \sigma &= \Re \Big( \sigma_0 e^{j\omega t} \Big) \\ \gamma &= \Re \Big( \gamma_0 e^{j(\omega t - \delta)} \Big) = \Re \Big( \gamma_0 e^{-j\delta} e^{j\omega t} \Big) \end{split}$$

where  $(\gamma_0 e^{-j\delta})$  is termed the complex strain amplitude. The shear storage modulus [or storage modulus, for short], which represents the in-phase (elastic) component of oscillatory *flow*, is defined as:

G' = storage modulus = 
$$\frac{\sigma_0}{\gamma_0} \cos \delta$$

The out-of-phase (viscous) component is termed the **shear loss modulus** [or **loss modulus**, for short]:

$$G'' = loss modulus = \frac{\sigma_0}{\gamma_0} \sin \delta$$

The complex shear modulus,  $G^*$ , is then defined as follows:

 $G^* = \frac{\text{complex stress amplitude}}{\text{complex strain amplitude}} = \frac{\sigma_0}{\gamma_0} \cos \delta + \frac{\sigma_0}{\gamma_0} j \sin \delta$ 

so that:



Figure 4 Vectorial representation of moduli.

The function G''/G' measures the relative importance of viscous to elastic contributions for a material at a given frequency.

Additionally, a *complex viscosity*,  $\eta^*$ , can be defined using the complex strain rate,  $\dot{\gamma} = j\omega\gamma$ , such that:

$$\eta^* = \frac{\text{complex stress amplitude}}{\text{complex strain rate amplitude}} = \frac{\sigma_0}{j\gamma_0\omega} e^{j\delta} = \frac{G^*}{j\omega}$$

or alternatively

$$\eta^* = \eta' + j\eta$$
$$\eta' = \frac{G'}{\omega}$$
$$\eta'' = \frac{G}{\omega}$$

where  $\eta'$  is termed the *dynamic viscosity*, and is equivalent to the ratio of the stress in phase with the rate of strain ( $\sigma_0 \sin \delta$ ) to the amplitude of the rate of strain ( $\omega \gamma_0$ ). The term  $\eta''$  is referred to as the *out-of-phase viscosity*, and is equivalent to the ratio of the stress 90° out of phase with the rate of strain ( $\sigma_0 \cos \delta$ ) to the amplitude of the rate of strain ( $\omega \gamma_0$ ) in the forced oscillation.

Finally, an **absolute shear modulus** is defined as the ratio of the amplitude of the stress to the amplitude of the strain in forced oscillation (*simple shear*), or:

$$|\mathbf{G}^*| = \frac{\sigma_0}{\gamma_0} = (\mathbf{G'}^2 + \mathbf{G''}^2)^{1/2}$$

Alternatively, forced oscillation experiments can be equivalently described in terms of *compliance*, as opposed to the derivation above based on the modulus. Similar arguments lead to the following analogous terms:

**complex shear compliance**,  $J^*$  The ratio of the complex strain ( $\gamma^*$ ) to complex stress ( $\sigma^*$ ) in forced oscillation (simple shear).

shear storage compliance, J' The ratio of the amplitude of the strain in phase with the stress ( $\gamma_0 \cos \delta$ ) to the amplitude of the stress ( $\sigma_0$ ) in forced oscillation (simple shear).

**shear loss compliance**, J'' The ratio of the amplitude of the strain 90° out of phase with the stress ( $\gamma_0 \sin \delta$ ) to the amplitude of the stress ( $\sigma_0$ ) in forced oscillation (simple shear).

**absolute shear compliance**,  $|J^*|$  The ratio of the amplitude of the strain ( $\gamma_0$ ) to the amplitude of the stress ( $\sigma_0$ ) in forced oscillation (simple shear).

# 9. Measurement Apparatus

There are two common methods used for *rheometric* measurements on fluid systems: capillary (or tube) and rotational. In this section, a brief summary is given for each general method along with descriptions of common measurement devices and geometries. Measurement devices can be grouped into one of two general classifications. A **viscometer** is a device used principally for the measurement of viscosity, while a **rheometer** is a device used for the measurement of rheological properties over a varied and extended range of conditions. Only brief descriptions of these instruments and devices are provided here. For more detailed descriptions, refer to the primary sources listed in the bibliography.

# **Capillary Methods**

In capillary methods the test fluid is made to *flow* through a narrow tube as a result of hydrostatic or applied pressure. Capillary measurements are considered the most precise way of determining the viscosity of *Newtonian* and some *non-Newtonian* fluids, and are generally simpler in design and less expensive relative to *rotational* instruments. **Poiseuille's Law**, which relates the rate of flow through a capillary to the viscosity of the liquid, is the basis for the capillary method.

# **Glass Capillary Viscometer**

Widely used for measuring the viscosity of *Newtonian* fluids, including dilute solutions and suspensions, the glass capillary *viscometer* is the simplest and least expensive viscometric system available commercially. Typically, in this technique, the time required for a given volume of fluid to flow through a defined length, *L*, of glass capillary under its own hydrostatic head is measured. The flow time, *t*, is related to the viscosity using a simple relationship derived from *Poiseuille's law*, of the form:

$$\frac{\eta}{\rho} = At + \frac{B}{t}$$

where A and B are instrument constants. The second term on the right hand side is related to a kinetic energy correction, and can be ignored for long flow times. In this case, a simple linear equation results. The viscometer can be easily calibrated using a standard fluid with known viscosity at the measurement temperature. Capillaries with different diameters can be used for different viscosity ranges, while maintaining reasonable flow times.



Figure 5 Ostwald type capillary viscometer. A and B are timing marks, C is a filling mark.

# **Extrusion Capillary Viscometer**

These instruments are widely used for measuring *viscous* fluids, such as asphalt cements, polymer melts, and stable concentrated suspensions. Extrusion viscometers have the advantage of high precision and simple design, and are less subject to temperature effects that can occur during shearing of highly viscous fluids in *rotational* devices.

Extrusion viscometers use a cylindrical piston to force the test fluid from a reservoir through the capillary tube at a constant velocity. By measuring the pressure drop across the capillary as a function of *flow* rate for multiple capillaries of the same diameter, d, but differing length, L, it is possible to determine the viscosity as a function of *shear rate* (see for example Figure 6).



Figure 6 Diagram of a simple extrusion viscometer.

# **Rotational Methods**

In rotational methods the test fluid is continuously sheared between two surfaces, one or both of which are rotating. These devices have the advantage of being able to *shear* the sample for an unlimited period of time, permitting transient behavior to be monitored or an equilibrium state to be achieved, under controlled *rheometric* conditions. Rotational methods can also incorporate *oscillatory* and *normal stress* tests for characterizing the *viscoelastic* properties of samples. In general, rotational methods are better suited for the measurement of concentrated suspensions, gels and pastes, but are generally less precise as compared to *capillary* methods.

Rotational measurements fall into one of two categories: **stress-controlled** or **rate-controlled**. In stress-controlled measurements, a constant torque is applied to the measuring tool in order to generate rotation, and the resulting rotation speed is then determined. If a well-defined tool geometry is used, the rotation speed can be converted into a corresponding *shear rate*. In rate-

controlled measurements, a constant rotation speed is maintained and the resulting torque generated by the sample is determined using a suitable stress-sensing device, such as a torsion spring or strain gauge. Some commercial instruments have the capability of operating in either stress-controlled or rate-controlled modes.

# Simple Rotational Viscometer [Brookfield type]

The least expensive commercial variant of the controlledrate rotational *viscometer* is commonly referred to as a "Brookfield type" viscometer<sup>§</sup>. This device measures fluid viscosity at fixed rotation speeds by driving a measurement tool ("spindle"), immersed in the test fluid, through a calibrated torsion spring (see Figure 7). *Viscous* drag of the fluid against the spindle causes the spring to deflect, and this deflection is correlated with torque. The calculated *shear rate* depends on the rotation speed, the tool geometry and the size and shape of the sample



Figure 7 Schematic diagram of a Brookfield-type viscometer.

<sup>&</sup>lt;sup>§</sup> Certain trade names and company products are mentioned in the text or identified in illustrations in order to specify adequately the experimental procedure and equipment used. In no case does such identification imply recommendation or endorsement by National Institute of Standards and Technology, nor does it imply that the products are necessarily the best available for the purpose.

container. Conversion factors are needed to calculate viscosity from the measured torque, and are typically pre-calibrated for specific tool and container geometries. For *Newtonian* fluids the torque is proportional to the product of viscosity and rotational speed, but this proportionality is lost in the case of a *non-Newtonian* fluid. Because these instruments are robust and fairly simple to use, they have found wide application in industry, but they offer limited capabilities and precision for research-oriented applications.

# **Rotational Rheometer**

High-precision, continuously-variable-*shear* instruments in which the test fluid is sheared between rotating cylinders, cones, or plates, under controlled-stress or controlled-rate conditions, are termed rotational *rheometers*. Instruments producing *oscillatory* strains are available, and a few commercial systems permit measurement of the *normal stress*. The basic rotational system consists of four parts: (i) a measurement tool with a well-defined geometry, (ii) a device to apply a constant torque or rotation speed to the tool over a wide range of *shear stress* or *shear rate* values, (iii) a device to determine the stress or shear rate response, and (iv) some means of temperature control for the test fluid and tool. Depending on the design specifications, rheometers may also include built-in corrections or compensations for inertia, drift, and temperature fluctuations during measurement.

Most rheometers are based on the relative rotation about a common axis of one of three tool geometries: concentric cylinder, cone and plate, or parallel plates (See Figure 8).



Figure 8 Schematic diagram of basic tool geometries for the rotational rheometer: (a) concentric cylinder, (b) cone and plate, (c) parallel plate.

In the **concentric cylinder** (also called **Couette** or **Coaxial**) geometry, either the inner, outer, or both cylinders may rotate, depending on instrument design. The test fluid is maintained in the annulus between the cylinder surfaces. This tool geometry comes in several configurations, of which the three most commonly encountered are illustrated in Figure 9. The **double-gap** configuration is useful for low viscosity fluids, as it increases the total area, and therefore the *viscous* drag, on the rotating inner cylinder, and generally increases the accuracy of the measurement. The **cone** and **hollow cavity** configurations are specifically designed to reduce or account for end effects. In addition, to prevent slippage (see *no-slip*), the inner cylinder surface is sometimes serrated or otherwise roughened. The concentric cylinder geometry is typically used for the analysis of fluid suspensions.



cylindrical tool designs in cut-away view: (a) double gap, (b) cone and plate at the bottom, (c) hollow cavity at the bottom to trap air.

The cone and plate geometry consists of an inverted cone in near contact with a lower plate. The cone is usually designed with an angle of less than  $4^{\circ}$ . Either the upper or lower surface may rotate depending on instrument design. The **parallel plate geometry** can be considered a simplified version of the cone and plate, having an angle of  $0^{\circ}$ . The test fluid is constrained in the narrow gap between the two surfaces. Cone and plate and parallel plate measurement tools are most often used for highly viscous pastes, gels, and concentrated suspensions.

# **10.** Concrete and Other Coarse-Grained Materials

Ceramic materials are typically fabricated from particles in the subsieve or fine size range (i.e., less than roughly 37  $\mu$ m), though coarse agglomerates may play a role during some phase of processing. On the other hand, concrete is classified as a ceramic material, but containing a considerably broader range of particle sizes, from less than one micrometer up to about 20 mm (i.e., well into the sieve range). This wide size range results from the heterogeneous concrete composition, which includes cement (5  $\mu$ m to 60  $\mu$ m), mineral fillers (< 1  $\mu$ m to 100  $\mu$ m), fine aggregates (0.1 mm to 5 mm) and coarse aggregates (4 mm to 30 mm or higher in some special concretes) or stones.

As a result of the broad range of particle sizes and the presence of sieve-size particles, methods to measure the *flow* properties of concrete differ significantly from those methods specified for applications involving suspensions of fine particles. Test methods applied to concrete tend to be largely empirical in nature or they are scaled up versions of the techniques described in the previous section for fine particle systems. In the former case are the most commonly used tests in the industry; they generally represent an attempt to "imitate" a mode of placement or flow of the concrete during production. In the latter case are less common methods that attempt to measure fundamental rheological properties of concrete. In addition, rheological test methods for concrete tend to fall into one of four general categories:

**confined flow** The material *flows* under its own weight or under an applied pressure through a narrow orifice. The orifice is defined as an opening roughly three to five times larger than the

maximum particle size. Because coarse aggregates are often on the order of 30 mm in size, the orifice must typically be 90 mm to 150 mm in diameter. Confined flow methods include *flow cone*, *filling ability* devices, *flow test* through an opening and the *Orimet apparatus*.

**free flow** The material either flows under its own weight, without any confinement, or an object penetrates the material by gravitational settling. Free flow methods include *slump*, *modified slump*, *penetrating* rod and *turning tube viscometer*.

**vibration** The material flows under the influence of applied vibration. The vibration is applied by using a vibrating table (e.g., *Ve-Be time*), dropping the base supporting the material (*DIN slump cone test*), an external vibrator (e.g., *LCL apparatus*) or an internal vibrator (e.g., *settling method*).

**rotational rheometers** The material is sheared between two parallel surfaces, one or both of which are rotating. These tests are analogous to *rheometers* described in the previous section, except in this case the gap between surfaces must be scaled up to reflect the much larger dimensions of the concrete particles. Rotational rheometers include *BTRHEOM*, *CEMAGREF*, *two-point* or *Tattersall*, *IBB*, and *BML*.

Separate glossaries of rheological terms and test methods are provided below. For more detailed summaries of the numerous rheological tests used throughout the concrete industry, including empirical tests that are widely implemented, see Ferraris (1999) in the list of primary sources.

# Glossary of Rheological Terms

Much of the rheological work on concrete and other cementitious materials has, historically, been directed toward the practical exploitation of the material and, as a result, numerous terms are in common use that are unique to the concrete, mortar and cement industries. The most frequently encountered rheological terms are defined here.

**consistency** The relative *mobility* or ability of freshly mixed concrete or mortar to *flow*; the usual measurements are *slump* for concrete, flow for mortar or grout, and penetration resistance for neat cement paste. (see also *normal consistency, plastic consistency,* and *wettest stable consistency*)

**consistency factor** A measure of grout *fluidity*, roughly analogous to viscosity, that describes the ease with which grout may be pumped into pores or fissures; usually a laboratory measurement in which *consistency* is reported in degrees of rotation of a torque *viscometer* in a specimen of grout.

**consolidation** The process of inducing a closer arrangement of the solid particles in freshly mixed concrete or mortar, during and after placement until setting, by the reduction of voids. Consolidation is accomplished by vibration, centrifugation, rodding, tamping or some combination of these actions. This term is also applicable to similar manipulation of other cementitious mixtures, soils, aggregates or the like.

**finishing** Leveling, smoothing, consolidating and otherwise treating the surfaces of fresh or recently placed concrete or mortar to produce the desired appearance and service.

**impending slough** *Consistency* of a shotcrete mixture containing the maximum amount of water such that the product will not *flow* or sag after placement.

mobility The ability of fresh mixed concrete or mortar to *flow*.

**normal consistency** (1) The degree of wetness exhibited by a freshly mixed concrete, mortar, or neat cement grout when the *workability* of the mixture is considered acceptable for the purpose at hand. (2) The physical condition of neat cement paste as determined with the *Vicat apparatus* in accordance with a standard test method (e.g., ASTM C 187).

**plastic consistency** Condition of freshly mixed cement paste, mortar or concrete such that *deformation* will be sustained continuously in any direction without rupture.

**slump** A measure of *consistency* of freshly mixed concrete, mortar or stucco equal to the subsidence measured to the nearest 5 mm (1/4 in) of the molded specimen after removal of the *slump cone*.

wettest stable consistency The condition of maximum water content at which cement grout and mortar will adhere to a vertical surface without sloughing.

**workability** [**placeability**] That property of freshly mixed concrete or mortar that determines the ease and homogeneity with which it can be mixed, placed, *consolidated* and finished.

# **Glossary of Test Methods**

**BML** Commercial *rotational rheometer* derived from the *two-point test*. The principle of operation is identical to the two-point test, but the shape of the vane has been modified. The vane is as shown in Figure 10(c).

**BTRHEOM** Commercial *parallel plate* concrete *rheometer*. Measurements can be performed with and without applied vibration.

**CEMAGREF** Coaxial *rotational rheometer* originally developed for testing fluid lava. It has been occasionally employed to test concrete. Only one prototype currently exists.

**DIN slump cone test** A *vibration* method. A variation of the *slump cone test* in which the cone is placed on a metal sheet. After filling the cone with concrete, it is lifted and the metal support sheet is raised on one side and dropped from a pre-established height (usually about 100 mm) a specified number of times. The spread of the concrete is measured. This standard test is described in DIN 1045.

**filling ability** *Confined flow* method measuring the ability of concrete to *flow* into a predefined form. Several different molds are used: U-shaped and L-shaped are the most common. In both cases the concrete is forced by gravity to flow into and fill the mold. The volume occupied by the concrete at the end of the test is a measure of the ability of the concrete to flow, or its filling ability.

**flow cone** [V-funnel cone] *Confined flow* method consisting in filling a funnel with concrete or cement paste and determining the time for the material to *flow* out through the orifice. This time

is a measure of the material's ability to flow. There are several types of funnel, some with round and others with parallelepiped orifice and upper opening.

**flow test** Used in accordance with ASTM C1362. This test measures the *flow* of concrete or mortar into a perforated tube. This method is applicable for concrete with coarse aggregates less than 37.5 mm in diameter. The instrument consists of a hollow perforated tube that is inserted in the concrete sample, after which the material is allowed to flow into the hollow tube. The height of the concrete in the hollow tube is considered a measure of the *consistency* of the concrete.

**IBB** Commercially available *rotational rheometer*. It is derived from the *two-point test*. The principle of operation is identical, but the vane shape has been modified, and the vane moves in a planetary motion and on its axis. The vane is shaped like the letter H and is shown in Figure 10(b).

**LCL apparatus** A *vibration method*. The concrete is poured behind a gate in a large parallelepiped container. The gate is opened and the vibrator is turned on. The time for the concrete to *flow* and occupy the whole container is measured. A longer time suggests a lower ability of the concrete to flow.

**modified slump test** Modification of the *slump test* described in ASTM C143. It permits the measurement of the rate at which the concrete is slumping, which gives an estimate of the concrete *plastic viscosity*. This test was developed at NIST, but is not currently a standard method (see Ferraris (1999) in primary sources).

**Orimet apparatus** *Confined flow method* instrument consisting of a tube 600 mm in length that is closed at the bottom by a removable trap door. The time for the concrete to *flow* through the tube once the trap is released is determined. This time is a measure of the ability of concrete to flow.

**penetrating rod** *Free flow method* measuring the distance a rod of fixed mass can penetrate a concrete sample. It is a crude determination of whether the *yield stress* of the concrete is higher or lower than a predefined value. It is used to monitor concrete at the job site. A specific application of this test is the *Vicat apparatus*.

**settling method** [Fritsch test] A *vibration method*, this test measures the capability of concrete to *consolidate* under vibration. Concrete is placed in a container with an internal vibrator. The time to obtain full consolidation is measured. The full consolidation is determined when the surface of the concrete no longer moves.

**slump cone test** *Free flow method* in which a truncated metal cone, open at both ends and sitting on a horizontal surface, is filled with concrete and lifted gradually. The *slump* of the concrete is measured. This standard test is described in ASTM C143.

**turning tube viscometer** *Free flow method* consisting of a tube 60 mm in diameter and 800 mm long that can be filled with cement paste or mortar. A ball is dropped into the fluid and its velocity is measured between two points 370 mm apart. Using the Stokes equation, the viscosity can be calculated.

**two-point test** [**Tattersall rheometer**] First commercially available *rotational rheometer* for concrete. It is the first attempt to *shear* concrete at different rates and to measure the induced

*stress.* It consists of a bucket filled with concrete and a vane, shown in Figure 10(a), that rotates at controlled speeds. The torque generated during rotation is measured.

**Ve-Be time [remolding test]** Measures the ability of concrete to change shape under vibration. Concrete is placed in a *slump cone*. After the cone is lifted away, the time it takes for the concrete to remold itself into a cylinder while under vibration is measured.

**Vicat apparatus** A *free flow method*, the Vicat apparatus is a penetration device used in the testing of hydraulic cements and similar materials. The full description of this instrument and the correct procedure to determine the *normal consistency* of cement is described in ASTM C187.



Figure 10 Various vane geometries used in concrete rheometers: (a) two-point test or Tattersall, (b) IBB, (c) BML.

# 11. Units and Symbols

# **Rheometric Units**

All units should conform to the International System of Units (SI) as described by the National Institute of Standards and Technology.<sup>1</sup> The use of CGS and other non-SI units is discouraged. For convenience, SI units are shown in Table 1 in association with their common non-SI equivalents (including decimal submultiples and conversion factors where appropriate). Table 2 shows the SI-derived units with their underlying base units.

<sup>&</sup>lt;sup>1</sup> B.N. Taylor, *Guide for the Use of the International System of Units (SI)*, NIST Special Publication 811, 2<sup>nd</sup> Edition, National Institute of Standards and Technology, U.S. Department of Commerce, Washington, DC, 1995.

Quantity	SI Units	Equivalent CGS Units
viscosity	Pa·s	10 P (poise) or 1000 cP (centipoise)
kinematic viscosity	$m^2 \cdot s^{-1}$	$10^4$ St (stoke) or $10^6$ cSt (centistoke)
shear stress	Pa	$0.1 \text{ dyn} \cdot \text{cm}^{-2}$
strain	unitless	
shear rate	s <sup>-1</sup>	s <sup>-1</sup>
modulus	Ра	$0.1 \text{ dyn} \cdot \text{cm}^{-2}$
compliance	Pa <sup>-1</sup>	$10 \text{ cm}^2 \cdot \text{dyn}^{-1}$
frequency	Hz	
angular frequency	2πf	
phase angle	rad	

Table 1. SI and equivalent rheometric units.

Table 2. SI derived units expressed in terms of SI base units.

Symbol	Special Name	Other SI Units	SI Base Units
Ра	Pascal	$N \cdot m^{-2}$	$kg \cdot m^{-1} \cdot s^{-2}$
Hz	Hertz		s <sup>-1</sup>
rad	radian		$\mathbf{m} \cdot \mathbf{m}^{-1} = 1$

# List of Symbols

1	$J^*$	absolute	shear	comp	liance
		aosoiate	Silvai	comp.	nance

- $|G^*|$  absolute shear modulus
- $\omega$  angular frequency
- $\eta_{app}$  apparent viscosity
- *D* characteristic dimension
- $\tau$  characteristic time (retardation, relaxation)
- η coefficient of viscosity [viscosity]
- $G^*$  complex shear modulus
- $J^*$  complex shear compliance
- $\eta^*$  complex viscosity
- De Deborah number
- $D_T$  Diffusion coefficient, translational
- $\eta_{diff} ~~ differential ~viscosity$
- $\eta'$  dynamic viscosity (real component of the complex viscosity)
- $\phi$  fluidity
- f frequency
- $\eta''$  imaginary component of the complex viscosity
- $\eta_{\infty}$  infinite shear viscosity
- $\eta_{inh}$  inherent viscosity

[η]	intrinsic viscosity
ν	kinematic viscosity
$\sigma_{n}$	normal stress
Pe	Peclet number
δ	phase angle
$\eta_{red}$	reduced viscosity
$\eta_r$	relative viscosity
τ	relaxation time, retardation time
Re	Reynolds number
J	shear compliance
$J^{\prime\prime}$	shear loss compliance
G''	shear loss modulus
G	shear modulus
Ý	shear rate [rate of shear strain]
G'	shear storage modulus
J'	shear storage compliance
σ	shear stress
$\eta_{sp}$	specific viscosity
$\sigma_0$	stress amplitude
γ	strain
Yo	strain amplitude
$\eta_s$	viscosity of suspending medium or solven
$\sigma_y$	yield stress
$\eta_0$	zero shear viscosity

(for model-specific symbols see Section 6)

# **Bibliography**

This bibliography cites sources used in the compilation of this guide. It is not, nor is it intended to be, an exhaustive list of references available on the vast subject of rheology or rheological instrumentation. Nevertheless, it may serve as a good starting point for those who are relatively new to this field or searching for relatively fundamental information.

# **Primary Sources**

American Concrete Institute, *Cement and Concrete Terminology*, ACI 116R-90, ACI Manual of Concrete Practice 2000, Part 1, Farmington Hills, MI, 2000

British Standards Institution, Glossary of Rheological Terms, BS 5168:1975.

A.A. Collyer and D.W. Clegg, Editors. *Rheological Measurement*, Chapman and Hall, London, 1998.

- J.M. Dealy, "Official Nomenclature for Material Functions Describing the Response of a Viscoelastic Fluid to Various Shearing and Extensional Deformations," *Journal of Rheology*, **39**[1], 253-265 (1995).
- C.F. Ferraris, "Measurement of the Rheological Properties of High Performance Concrete: State of the Art Report," *Journal of Research of the National Institute of Standards and Technology*, **104**[5], 461-478 (1999).
- A. Kaye, R.F.T. Stepto, W.J. Work, J.V. Alemán and A.Ya. Malkin, "Definition of Terms Relating to the Non-Ultimate Mechanical Properties of Polymers," IUPAC Recommendations 1997, *Pure and Applied Chemistry*, **70**, 701-754 (1998).

# **Other Sources**

- H.A. Barnes, "Shear-Thickening ("Dilatancy") in Suspensions of Nonaggregating Solid Particles Dispersed in Newtonian Liquids," *Journal of Rheology*, **33**[2], 329-366 (1989).
- M. Bohdanecky and J. Kovář, Viscosity of Polymer Solutions, Elsevier, Amsterdam, 1982.
- E.C. Bingham, "An Investigation of the Laws of Plastic Flow," U.S. Bureau of Standards Bulletin, 13, 309-353 (1916).
- F.R. Eirich, Editor. *Rheology Theory and Applications*, Vol. 1, Academic Press, NY, 1956; Vol. 3, 1960; Vol. 4, 1967.
- J.W. Goodwin, "Rheology of Ceramic Materials," Ceramic Bulletin, 69[10], 1694-1698 (1990).
- P.C. Hiemenz and R. Rajagopalan, *Principles of Colloid and Surface Chemistry*, 3rd Edition, Chapter 4, pp. 145-192, Marcel-Dekker, 1997.
- R.J. Hunter, "Rheology of Colloidal Dispersions," Chapter 18 in *Foundations of Colloid Science*, Vol. 2, Oxford University Press, NY, 1989.
- J. Mewis and A.J.B. Spaull, "Rheology of Concentrated Dispersions," *Advances in Colloid and Interface Science*, **6**, 173-200 (1976).
- D. Quemada, "Models for Rheological Behavior of Concentrated Disperse Media under Shear," pp. 571-582 in Advances in Rheology, Vol. 2: Fluids, Universidad Nacional Autonoma de Mexico, Mexico, 1984.
- H.N. Stein, "Rheological Behavior of Suspensions," pp. 3-48 in *Encyclopedia of Fluid Mechanics*, Vol. 5: Slurry Flow Technology, Gulf Publishing Company, Houston, 1986.
- J.R. Van Wazer, J.W. Lyons, K.Y. Kim, and R.E. Colwell, *Viscosity and Flow Measurement A Laboratory Handbook of Rheology*, Interscience Publishers, New York, 1963.
- R.W. Whorlow, Rheological Techniques, Ellis Horwood Ltd., Chichester, 1980.

A.A. Zaman, *Techniques in Rheological Measurements: Fundamentals and Applications*, Instructional Module, NSF Engineering Research Center for Particle Science and Technology, University of Florida, Gainesville, 1998.

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