

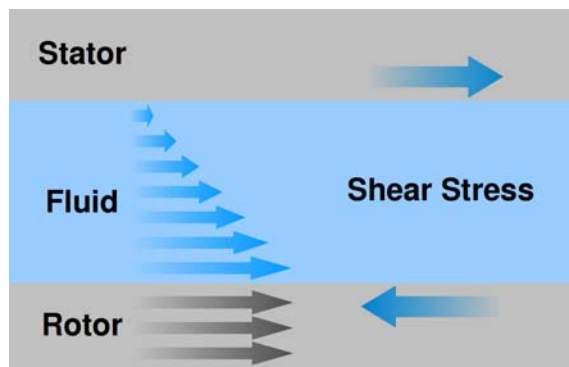
Lenterra RealShear™ Sensors for High-Shear Mixers

Lenterra is introducing a new class of instrumentation to measure wall shear stress to provide critical insight for high-shear mixing applications.

High-Shear Mixing

High-shear mixers are being increasingly used across numerous industries, including for the production of pharmaceuticals, food, and cosmetics.¹ They can provide significantly shorter mixing cycles to radically improve throughput compared with conventional mixers. HSMs are typically of the rotor-stator type (see figure to the right), in which one element (the rotor) rotates in close proximity (as small as 0.2 mm) to a stationary element (the stator). Mix components that pass between them experience high shear stress.

The high levels of shear stress are most often used for particle size reduction to produce an emulsion or dispersion with desirable characteristics, such as viscosity, granularity, or in the case of food products qualities like spreadability or “feel on the tongue.” In pharmaceutical manufacturing, high-shear mixers can additionally facilitate crystallization by creating supersaturated liquids, and can be used to produce particular drug polymorphs during mixing.



High-shear mixer dynamics. Fluid and rotor velocities are shown as well as the wall shear stress induced on the mixer components by the fluid.

Shear Stress

Shear stress is a force that acts on an object in a direction that is parallel to its surface. The amount of shear stress is determined by the viscosity of the fluid flowing across it and how much the fluid velocity varies with varying distance from the wall (known as the “velocity gradient” or “shear rate”). If the shear rate is known, the wall shear stress measured by a RealShear sensor can be used to measure viscosity. In high-shear mixers the shear rate can be calculated using the known geometry of the rotor-stator and the rotation rate of the rotor.

Challenges

Though high-shear mixers are powerful tools, there are a number of challenges that process engineers face when using them:

- **Overprocessing**

For most processes, there is a maximum shear stress at which ideal particle size is achieved, and exceeding that can lead to wasted processing time and/or heat buildup that can destroy high-value components, such as active pharmaceutical ingredients (APIs). In some cases, it is necessary to add undesirable processing aids to protect sensitive biological components.

¹ S. Shelley, “High-shear mixers: still widely misunderstood,” *Chemical Engineering Progress*, 11, 7-12, 2007.

▪ Underprocessing

If the shear stress or mixing time is insufficient, substandard product can be produced. Inhomogeneities and nonideal particle sizes can be the result. Pharmaceutical process engineers, in an effort to avoid overprocessing, may instead underprocess, leading to failed batches.

▪ Scale Up

Typically new products and processes begin in small, benchtop batch mixers. Scaling up from these small mixers to larger industrial mixers can be a very difficult process due to the complexities of the fluid dynamics. The trial-and-error process of scale-up can be frustrating and require a tremendous number of man-hours and, particularly in pharmaceutical mixing, the waste of extremely costly ingredients.

These problems could be mitigated if it were possible to directly measure the shear stress produced during the mixing process. If for example the moment when the maximum ideal shear stress were achieved for a given batch was known, no further wasteful or potentially destructive mixing would be performed. Knowledge of shear stress could provide critical insight during a scale-up operation. Rather than engaging in a costly series of trial-and-error experiments, process engineers would be able to rapidly replicate the shear conditions in laboratory benchtop mixers in the full-sized mixers on the production floor. Until now, however, instrumentation to measure shear stress in high-speed mixers has simply been unavailable.

Lenterra Shear Stress Sensor

Lenterra's new RealShear™ series of sensors (featuring patented technology) now make it possible to directly measure wall shear stress in real time. This critical knowledge enables:

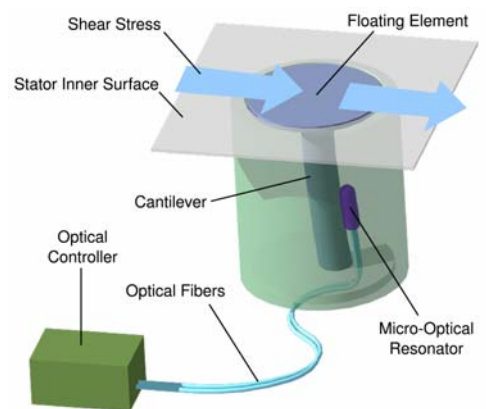
- Rapid scale-up from the high-shear mixing laboratory to the production floor replacing the costly and time-consuming trial-and-error process.
- Operator or automatic intervention to prevent spoiled batches
- Precision tuning of process parameters to aid crystallization or drug polymorph production, and to eliminate need for processing aids
- Avoidance of over-processing of products, resulting in improved throughput
- Reduction in post-production testing due to more precisely controlled processing
- Higher control over the process resulting in higher quality and uniformity of product



The RealShear™ Sensor

Sensor Features

- Direct, precision measurement of shear stress over a wide range of sensitivity
- In-line operation with no disruption of process flow or alteration of rotor-stator geometry – sensor is mounted flush with stator wall
- Bidirectional sensitivity
- Chemically resistant construction
- Wide operating temperature range from -30°C (-20°F) up to 200°C (390°F)
- Self-calibrated for temperature variations
- Fiber optic sensing means no electromagnetic interference and no ignition hazard
- Fast measurement rate (up to 10 kHz) for detailed analysis of dynamic flows
- Turn-key operation when coupled with a Lenterra optical controller and software.



The Lenterra Shear Stress Sensor concept

Shear Stress and Viscosity

Shear stress and viscosity are interrelated through the velocity gradient of a fluid. The RealShear™ sensor directly measures wall shear stress, but if the velocity gradient is independently known, viscosity can be calculated making it an on-line, real-time viscometer. For high-shear mixers, the known rotor geometry and velocity can be used for this calculation. This powerful capability removes the need for further costly instrumentation when viscosity needs to be monitored. Measuring viscosity in this way uses the same principle as “cup-and-bob” viscometers in which a cylinder is rotated inside a cup, submerged in a fluid. Unlike typical off-line viscometers however, RealShear™ sensors provide continuous viscosity measurements during mixing operation without the necessity to remove material from the batch.

Principle of Operation

A floating element that is in direct contact with the fluid under test, is attached to a cantilever beam which deflects in response to shear stress applied to the floating element surface, and transmits a force to a micro-optical resonator (see diagram). This resonator (typically made of silica glass) has an optical spectrum with peaks centered at particular light wavelengths. These resonances can be recorded by sending light to the resonator, and measuring the light that returns from it. When the cantilever deflects, the micro-optical resonator attached to the cantilever experiences strain, causing a shift in its resonant optical wavelength. This measurable shift is proportional to the shear stress.

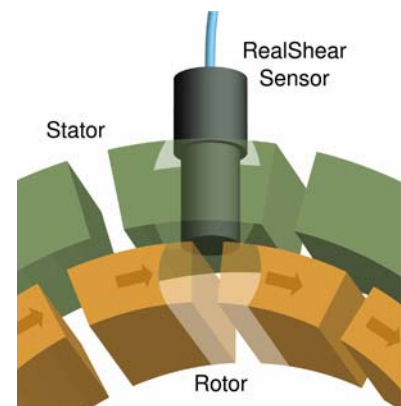
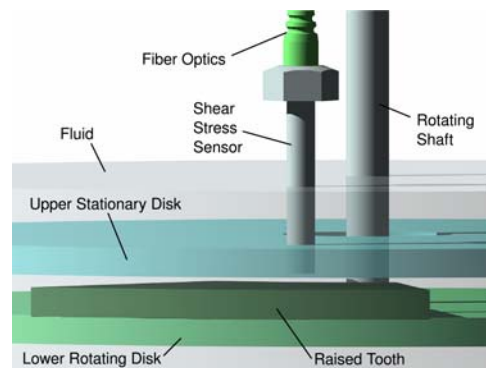


Diagram showing positioning of sensor for shear stress measurement.

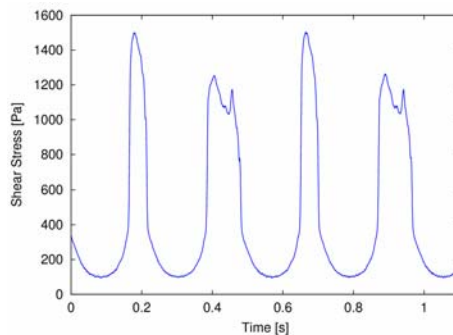
Simulated Mixer Tests

Lenterra's shear stress sensors have been tested in a rotating disk experimental setup to simulate conditions in rotor-stator mixers. The sensor is mounted vertically in a threaded hole in a 9" diameter stationary disk such that the face of the sensor is flush with the bottom surface of the disk. A second disk is below it by a variable distance (several millimeters), and is attached to a rotating shaft. This second disk has two raised portions (teeth) located opposite from one another on the disk, are 30 and 67 degrees in angular extent, and are 0.9 and 1.2 mm from the upper stationary disk, respectively. The assembly is immersed in a testing fluid which exerts shear stress on the sensor face when the lower disk is rotating.



The rotating disk testbed

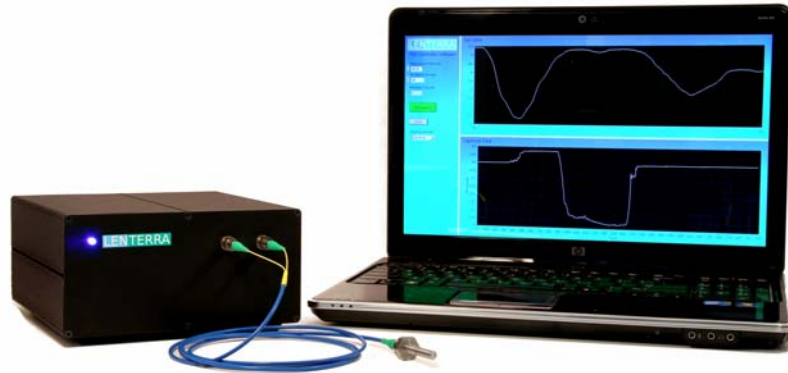
This plot shows shear stress data measured by the sensor using glycol as the test fluid with the lower disk rotating at a rate of 122 RPM. Two full rotations are shown in which the small and large tooth pass by the sensor twice each. As expected, the smaller gap (0.9 mm) above the smaller tooth results in a stronger peak shear stress (1500 Pa at approximately $t = 0.2$ and 0.65 seconds) compared with the larger tooth (1200 Pa at $t = 0.4$ and 0.9 seconds with a gap of 1.2 mm). Note the fine detail captured during the large tooth passes revealing complexities in the fluid dynamics.



Shear stress data obtained in the rotating disk testbed

Complete Measurement System

The RealShear™ sensor is available in a wide range of sensitivities to best match a particular application. Optical controllers are also available from Lenterra specifically designed to interrogate the micro-optical resonators that make up the sensitive elements inside the sensor. The controller communicates with a PC running Lenterra-designed software that enables an operator to view and store real-time shear stress data.



The complete wall shear stress measurement system

About Lenterra

Lenterra specializes in the development and manufacture of sensor instrumentation based on a range of patented optical- and ionized gas/plasma-based technologies. The company provides solutions for a wide range of industries and applications, including pharmaceutical manufacturing, industrial mixing, oil and gas production, and environmental monitoring.



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