Rheological Analysis under Pressure

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ABSTRACT

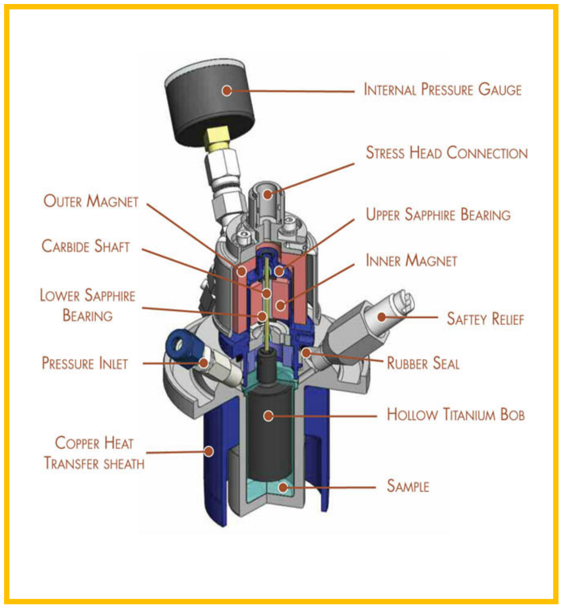
Typical rheological tests are all performed under atmospheric pressure conditions. However, in certain applications such as oil drilling, people need to evaluate the visco-elastic properties of materials under high temperature as well as high pressures. In addition, to evaluate high temperature viscosity of some volatile solutions, one will need a sealed vassal to prevent evaporation and also to hold the sample at higher than the atmospheric pressure. In this paper, we introduced a pressure cell unit, which is used on TA Instruments AR and DHR series rheometers. This pressure cell accessory is capable of measuring the visco-elastic properties of samples up to 150°C with a pressure as high as 2000psi (138 bar). This accessory can be a very helpful tool for industries such as oil drilling, food, petroleum or pharmaceuticals that need high temperature and high pressure rheological measurements.

THE PRESSURE CELL TECHNOLOGY AND SPECIFICATIONS

The Pressure Cell TA offers to all AR and DHR rheometer users is a sealed vessel that can be pressurized up to 138 bar (2,000 PSI), over a temperature range of –10°C to 150°C. It can be used either in self pressurizing mode, in which the pressure is produced by the volatility of the sample, or by externally applying the pressurization, typically with a high pressure tank of air or nitrogen gas. All necessary plumbing and gauges are included as a manifold assembly.

The Pressure Cell is ideal for studying the effect of pressure on rheological properties, as well as studying materials that volatilize under atmospheric pressure. This option is available for the AR and DHR series rheometers.

The pressure cell option is used with the Peltier Concentric Cylinder jacket. At the top is an outer magnet assembly that houses strong rare earth magnets and attaches to the stress head. At the bottom is a stainless steel cup, to withstand high pressure, surrounded by a copper sheath for excellent heat transfer. Also connected to the cup are the pressure inlet, internal pressure gauge, and pressure relief valve. The rotor assembly houses the inner magnet attached to a low friction carbide shaft, supported above and below by sapphire bearings. A hollow titanium cylindrical rotor is attached to the bottom of the carbide shaft. When the upper magnet assembly is lowered over the rotor assembly, the inner and outer magnets couple, levitating the rotor assembly. The rheometer's motor movement is then transferred to the rotor through a strong magnetic coupling to drive the cylinder inside the vessel and deform the sample. This innovative high-powered magnetic coupling and low friction bearing design allows for both steady shear and dynamic measurements.



The specifications for the standard pressure cell concentric cylinders are:

Stator inner radius:	14.00 mm
Rotor outer radius:	12.75 mm
Cylinder immersed height:	44.00 mm
Gap:	3500 mm (recommended)
Backoff distance:	56000 mm
Geometry inertia:	67.00 mN.m.s^2 (approximate)
Sample volume:	9.5 ± 0.5 ml
Temperature range:	-10 to 150°C
Maximum applied pressure:	138 bar (2000 psi)
Maximum pressure (self-pressurising):	5 bar (72.5 psi)
Seal construction:	DuPont® Kalrez™

PRESSURE CELL APPLICATIONS

The pressure cell accessory is a powerful tool and has been widely used on variety of industries such as oil drilling, petroleum, food, pharmaceutical and other chemical processing. The following case studies exhibit some of the examples where the pressure cell accessory is used.

Case Study -1

To better understanding the ability of motor oil to provide necessary lubrication under different environmental conditions, it is important to know its viscosity over a range of temperature and pressures. Figure 1 shows the viscosity study results of a motor oil under different pressure conditions. The tests were performed at temperature of 20°C and pressure of atmospheric; 1000 psi (69 bar) and 2000 psi (138 bar). The results show that the viscosity of the motor oil increases under pressurized conditions.

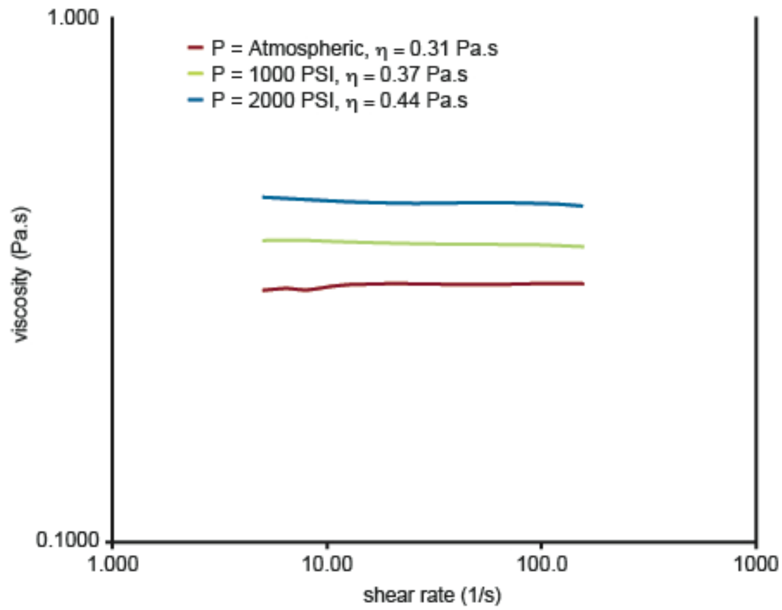


Figure 1 Effect of pressures on the viscosity of motor oil

Case Study -2

Figure 2 shows the viscosity test result of a sugar syrup at a broad temperature range. The measurement temperature range was set from 30°C up to 150°C. When the measurement temperature is higher than 90°C to 100°C, it is necessary to use a sealed vessel to prevent any evaporation and also to hold the pressure that built up as the temperature increases. The pressure cell unit is an ideal tool for this kind of applications.

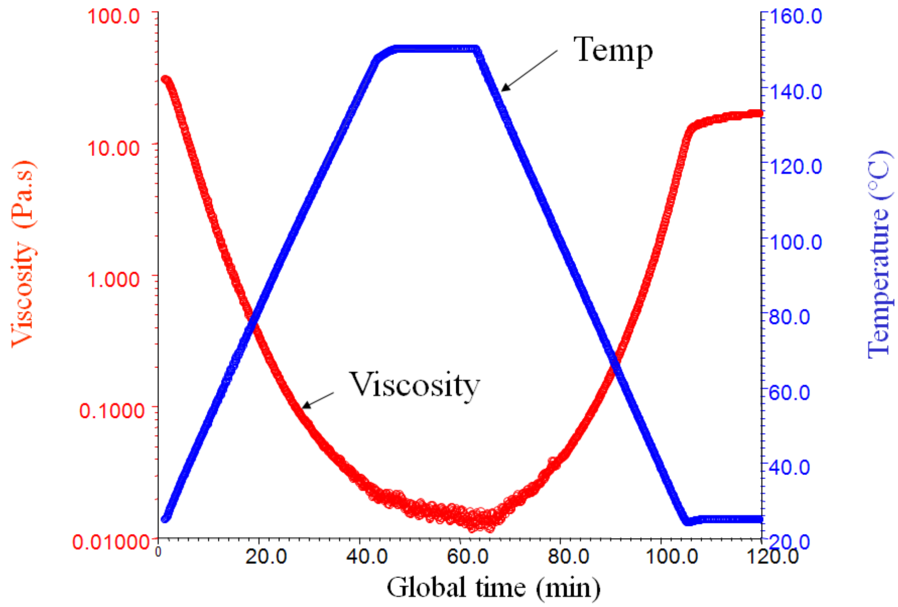


Figure 2 Viscosity of a sugar syrup as a function of temperature

Case Study -3

The pressure cell accessory is not only eligible to perform flow (i.e. viscosity) measurements, it can also be used to study the visco-elastic properties of materials (G'; G" and tan d) by doing oscillatory tests.

Figure 3 shows the time-temperature super position measurement results of a sugar syrup sample. Frequency sweep tests were conducted at temperatures of 62°C, 72°C, 82°C and 92°C. The master curve was generated using reference temperature of 92°C. The TTS analysis results fit well with the WLF model (shown in figure 4).

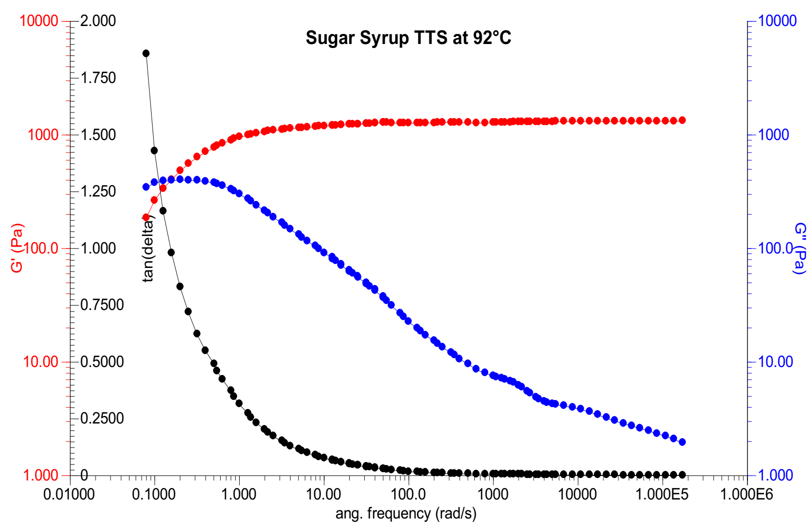


Figure 3 TTS master curve of a sugar syrup at temperature of 92°C

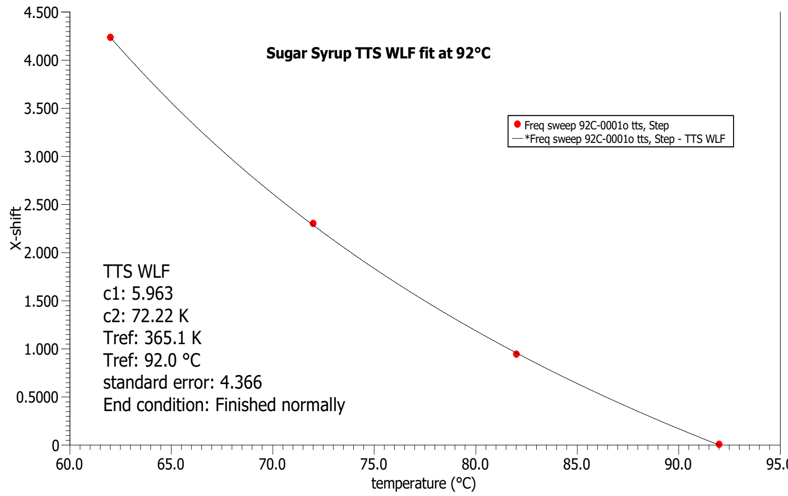


Figure 4 The TTS WLF fit of a sugar syrup at temperature of 92°C

Case Study -4

Figure 5 shows the test result of monitoring a gelation reaction of a hydrogel under high temperature and high pressure. The chemical cross-linking reaction only starts at a temperature of 100°C or higher. In order to prevent water from boiling and evaporating, an external pressure of 300psi was apply to the system during the measurement. The test result shows that this sample gelled over time, the sol-to-gel transition happened after about 44 minutes of reaction.

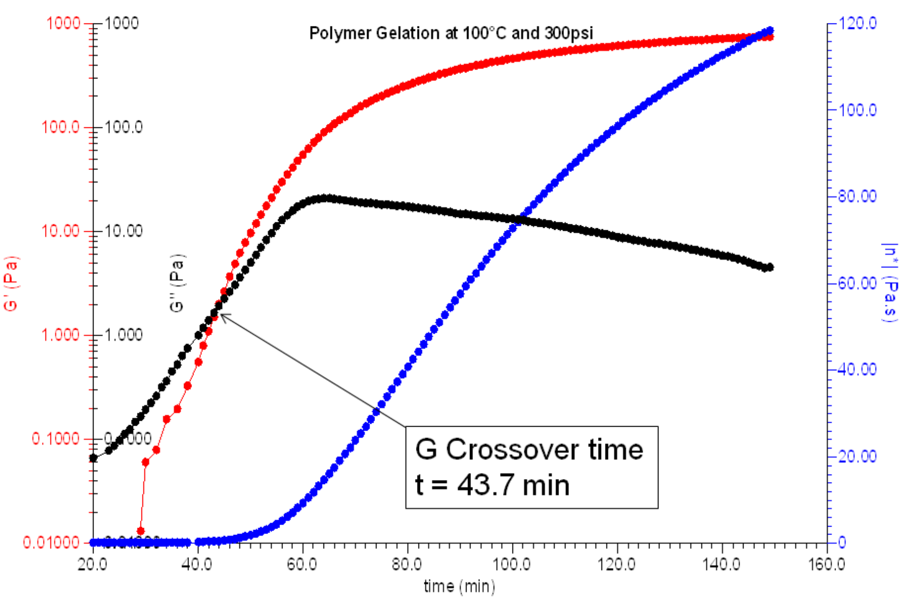


Figure 5 Monitoring hydrogel gelation at temperature of 100°C and pressure of 300psi