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Controlled-stress rotational rheometry : An historical review

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Abstract

The recent renaissance in controlled-stress rheometry has meant that more and more commercial models of this type of instrument have appeared in the (rheological) marketplace and many papers now deal with the results obtained by their use. It is therefore both timely and appropriate that this mode of rheometry should be reviewed for the sake of new and old users who are probably not be aware of its development. The history of controlled-stress measurements is therefore given, and the particular efforts of the late Jack Deer in the 1970s are chronicled, and then the later developments that have made it possible that such low torques can now be applied and such low rotational speeds measured. These have been mostly in the areas of air bearing and optical disc technologies. The typical results now obtained are illustrated.

Keywords : controlled-stress, rheometry, Deer

1. Introduction

It is no exaggeration to say that the modern developments in controlled-stress rheometry have brought about a revolution in the field of rheological measurements. For this reason, it seems both timely and appropriate to review progress in this area. We need not consider those aspects and artefacts of rheometry common to both controlled-strain and controlled-stress rheometer geometries, e.g., wide-gap geometry calculations, slip, inertia, edge effects, but we do need to consider those unique things which make the controlled-stress mode so beneficial.

2. Early history

When trying to measure viscosity as a function of the flow conditions in simple shear, the advantages of rotational-type viscometers - compared with the tube-flow geometries with modifications such as U-tube viscometers (see Barnes, 2002) - were soon realised, since they gave the possibility of easy variation of shear-rate or shear-stress for a given sample, as well as the time of shearing, with the obvious sample-containing benefit and easier temperature control.

Since commercial versions of rotational viscometers were introduced in the early decades of the twentieth century, there were always both controlled-stress and controlled-rate instruments on the market. The earliest commercial controlled-strain viscometer is that due to MacMichael (1915; 1918), and this type of instrument was available right up until WWII. The instrument was manufactured by Eimer and Amend of New York, USA, and it incorporated a 60-mm diameter disc, 5-mm thick suspended in a rotating cup, or various cylinders, driven at 20 rpm, see Fig. 1. Viscosity was usually quoted in MacMichael degrees read form the graduations on the disc for measuring the twist on the torsion wire. The instrument had good temperature control, and worked efficiently for viscosities in the 0.05 − 250 Pa.s range. Many improvements were made to the original design, particularly by extending the speed range to 10 − 38 rpm, giving a shear-

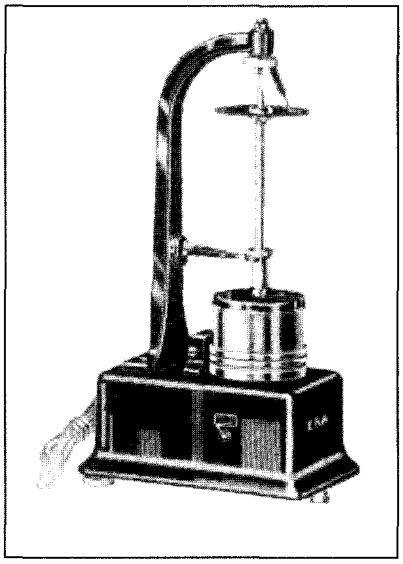

Fig. 1. The MacMichael controlled-strain viscometer.

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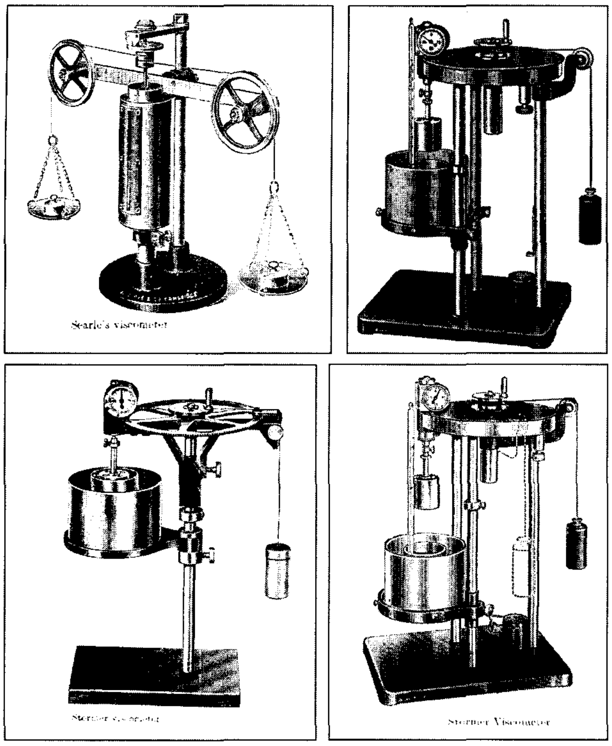
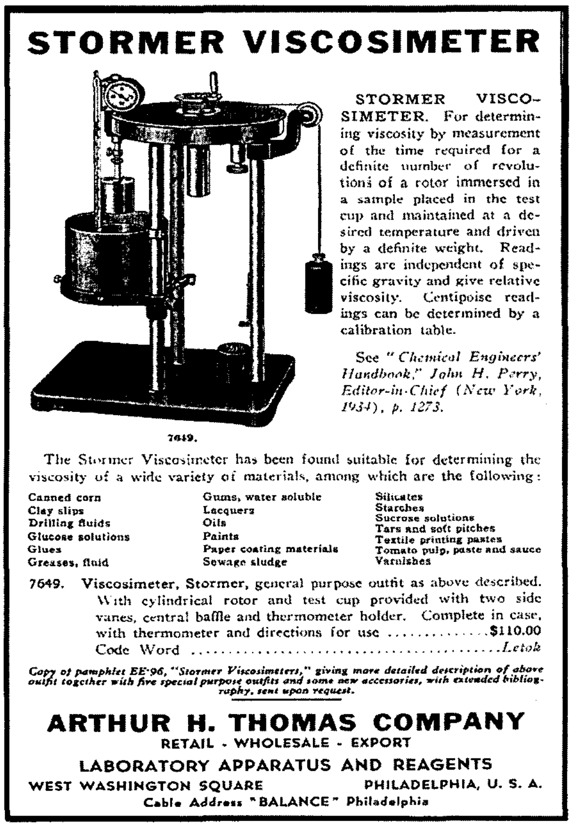


Fig. 2. Early commercial controlled-stress viscometers (including the Searle, 1912 and Stormer, 1909 versions).



STORMER VISCOSIMETER

STORMER VISCOSIMETER. For determining viscosity by measurement of the time required for a definite number of revolutions of a rotor immersed in a sample placed in the test cup and maintained at a desired temperature and driven by a definite weight. Readings are independent of specific gravity and give relative viscosity. Centipoise readings can be determined by a calibration table.

See "*Chemical Engineers' Handbook,*" *John H. Perry, Editor-in-Chief (New York, 1934), p. 1273.*

7649.

The Stormer Viscosimeter has been found suitable for determining the viscosity of a wide variety of materials, among which are the following:

Canned corn
Clay slips
Drilling fluids
Glucose solutions
Glues
Greases, fluid
Gums, water soluble
Lacquers
Oils
Paints
Paper coating materials
Sewage sludge
Silicates
Starches
Sucrose solutions
Tars and soft pitches
Textile printing pastes
Tomato pulp, paste and sauce
Varnishes

7649. Viscosimeter, Stormer, general purpose outfit as above described. With cylindrical rotor and test cup provided with two side vanes, central baffle and thermometer holder. Complete in case, with thermometer and directions for use $110.00
Code Word ....*Letok*

Copy of pamphlet EE-96, "Stormer Viscosimeters," giving more detailed description of above outfit together with five special purpose outfits and some new accessories, with extended bibliography, sent upon request.

ARTHUR H. THOMAS COMPANY
RETAIL - WHOLESALE - EXPORT
LABORATORY APPARATUS AND REAGENTS
WEST WASHINGTON SQUARE PHILADELPHIA, U. S. A.
Cable Address "BALANCE" Philadelphia

Fig. 3. A Stormer viscosimeter advertisement from around 1934.

rate range of around 1~10 s^{-1}, with a measured shear stress up to about 300 Pa.

The contemporary commercial controlled-stress equivalents were the Searle and Stormer instruments: Fig. 2 shows all these instruments, see Searle (1912) and Stormer (1909). This was commercialised by the Pye company of Cambridge, England. Stormer's design was also commercialised by Arthur H. Thomas of Philadelphia, see Fig. 3; inner cylinders and fork-shaped paddles were available, and there was good temperature control.

One obvious result of these two kinds of instruments meant that flow curves were plotted in two ways. Either with the shear rate or the shear stress plotted along the x-axis as the controlling factor, or else the shear rate. Then on the y-axis, the resulting shear stress or the shear rate were plotted. The prevalence of stress-controlled machines meant that most early flow curves had the stress plotted along the x-axis.

The Stormer viscometer was still readily available into the 1960s, with a range of cylinders, flag-type vanes, etc. The maximum loading was 2 kg, the string length was about 1 m, wound onto a drum of radius 14.25 mm, and the gear ratio is 11:1. This would result in a shear stress of around 2000 Pa, and the probable minimum shear stress was probably not much less than then about ten times lower than this value. Various forms of Stormer viscometer are still available and are used for measuring the consistency of paints, ASTM D562 (Consistency of paints Stormer viscometer), and is also used for petroleum products.

Obviously the ease of producing of shear stress with weights and pulleys meant that these instruments had their attraction in terms of simplicity and economy, the only other necessity being a stopwatch to time the rotation rate! However, such methods had severe limitations in terms of friction of the pulley bearings and cylinder supports that dictated the minimum possible shear stress. Also, the limited length of the string holding the weights meant that the time of application of the stress was limited and it was difficult to know if steady state had been reached!

The next steps in the development of viscometers in general moved beyond these instruments, and are reviewed by the present authors (Barnes *et al.*, 1999) where we see that while commercial controlled-strain instruments evolved, commercial controlled-stress instruments remained static.

3. Some home-made controlled-stress creep instruments

The use of the controlled-stress mode was well known in the area of solids and soft solids testing and was particularly useful in carrying out low-stress, long-term tests called creep tests. These showed that very slow mechanisms operated that resulted in all kinds of materials, and

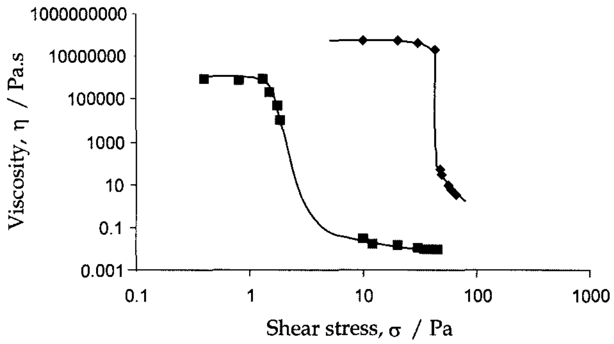


Fig. 4. Rehbinder and Michajlows 1961 data for 10 and 20% aqueous Bentonite, showing the now familiar precipitate drop in viscosity for a relatively small increase in shear stress.

for soft metals such as lead were easily measured by hanging weights on wires (see Evans and Wilshire, 1993). There was then interest in performing such tests for highly structured liquids that appeared to have a yield stress, below which no flow at all occurred. A number of workers therefore constructed their own instruments. The well-known commentators on viscometers and rheometers, van Wazer *et al.* (1963) pointed out in the early 1960s that creep experiments could be performed using a Stormer viscometer, and that the deformation could be magnified using a light beam and mirror arrangement.

Rehbinder used a home-made creep apparatus and produced the kind of flow curves that are now commonplace when using modern controlled-stress instruments, but then were quite rare, e.g., Rehbinder and Michajlow (1961). Typical results are shown in Fig. 4. We note that the shear rates involved in measuring the low-stress viscosity plateau were at or lower than 10^{-6} s^{-1}.

Philip Sherman [then at Unilever Research Laboratory, Welwyn, England] described a concentric-cylinder viscometer converted to a controlled-stress instrument, where the cylinders were ribbed to avoid slip Sherman (1970). The inner cylinder was hung from a light torsion wire, and driven by a weights-and-pulleys system. The angular deflection of the inner cylinder was followed by the reflection of a light beam off a mirror mounted on the inner cylinder, thrown onto an extended scale. Even more sensitive readings could be made using an electronic rotational-displacement transducer. Sherman reported creep measurements on melted ice-cream at 20°C, where steady state took up to about 5 minutes, and the viscosities were then around 10^3 Pa.s. The typical applied stresses were as low as 0.5 Pa. This compared to about 100 minutes for ice-cream at −11°C measured on a parallel-plate instrument, with a viscosity of around 10^7-10^8 Pa.s. A Burgers viscoelastic flow model applied, with typical values for G_1 of 15.6 Pa, η_2 around 520 Pa.s and a relaxation time of 33.4 s. These types of measurements were used to distinguish good and bad ice-cream mixes.

Harnett *et al.* (1992) more recently built a similar apparatus and found that the steady-state creep viscosity of various kinds of butter at 10°C varied from about 2 to10×10^{11} Pa.s. The creep was monitored for 17 h.

Other efforts made in this area include the following:

- Zimm and Crothers (1962) who used a rapidly rotating magnet to produce a constant torque on a steel pellet contained in the inner, glass, cylinder of their viscometer: stresses as low as 10^{-5} Pa in measurements on dilute aqueous solutions.
- Williams (1969) who used the stator of an a.c. induction motor instead of a rotating magnet so that the stress could be varied readily by altering the current amplitude.

4. Modern commercial controlled-stress developments

The modern revival in controlled-stress rheometry is due almost entirely to one man the late Jack Deer, who died in January 2003. While working as a technician at the London School of Pharmacy in the mid-1960s, Jack Deer, at the instigation of various colleagues, re-engineered an old Weissenberg Rheogoniometer to operate in a controlled-stress mode in order to carry out creep experiments, as others were doing at the time, see above.

Soon a dedicated instrument was made driven by an air turbine as the source of controlled stress, see Fig. 5, where

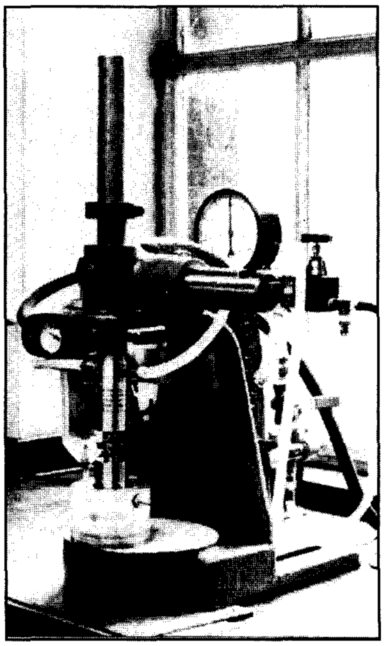


Fig. 5. The pre-commercial air-bearing Deer instrument.

the applied torque depended on the flow-rate of the air through the turbine. Deer was joined by Peter Finlay in about 1968, who added the first rudimentary electronics to the instrument. A provisional patent was taken out on the instrument, but it was allowed to lapse, and a full patent was never obtained - this accounts for the later proliferation of controlled-stress instruments from different manufacturers. Using a pre-commercial version of the air-turbine rheometer in the creep mode, it was found that the creep (*i.e.* zero shear) viscosity of wool fat BP at 25°C over a shear rate range of $1.76-6.14\times10^{-6}$ s^{-1} was constant (to within a small experimental error) at a value of $\sim 2\times10^{6}$ Pa.s. Torques up to 6×10^{-3} N.m could be applied with bearing friction torque of the order 4×10^{-6} N.m, see Davis *et al.* (1968) and Davis (1969).

The School of Pharmacy allowed Deer to exploit his idea commercially, and about 1969 he entered into a business arrangement that gave him 10% of sales value of any instruments sold. The Deer Rheometer Marketing company was set up, with Chris Musgrove as the sales driving force and manufacturing was subcontracted to Lucky Engineering in Clacton.

In the first advertising material for the new rheometer, it was claimed that with such an instrument the application of a known and controlled-stress provides the rheologist with vital information of this critical region in the form of a creep curve. These claims could be made because they had introduced a 'specially designed air bearing ... and an air turbine drive system for the application of torsional stress that is independent of rotational speed ... throughout the operational range'. (Air bearings and air turbines had been and are still used for dentists drills, but of course at much higher speeds, hence the whine!) The original air turbine was soon replaced by the electric drag-cup motor.

The rotation was measured using a snail cam mounted on the rotating shaft, which was fitted with an inductive pickup. The rotation rate was measured using a sectored disc cutting an light beam, see Fig. 6. In the late 1970s "Integrated Petronics" microprocessor-based instrument was introduced with improved electronics.

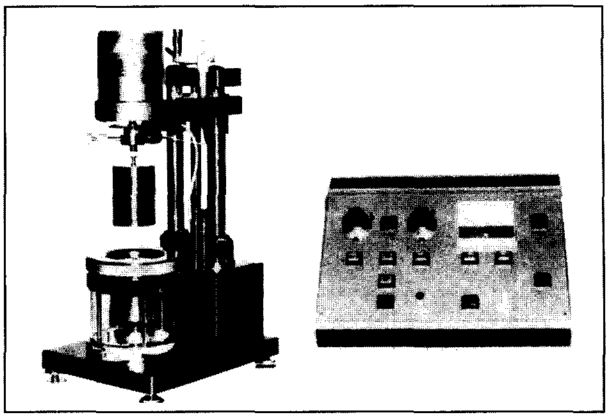


Fig. 6. The mark 1 Deer rheometer.

About 1980 Deer dissociated himself from Deer Rheometer Marketing and approached Neville Charrington (who got his PhD at the School of Pharmacy in 1958) who ran a company called Carri-Med, to see if I would be interested in helping him to market a separate instrument. The result was the early 1980s Carrimed CSR controlled-stress instrument controlled by an Apple micro-computer. This represented the first modern concept computer-driven, automated instrument, with flow-curve, creep and oscillation capability. Initially the oscillatory drive was in the form of a digital sine-wave generator which plugged into the drive electronics to give a torque sine-wave output on the instrument, but there was no simple way of analysing the data generated however. This was quickly replaced with an Apple II computer-based system which generated the sine wave and analysed the resulting stress and strain waveforms, allowed for inertia effects and outputted results in terms of loss and storage moduli.

In 1990, TA Instruments Inc. began as a stand-alone

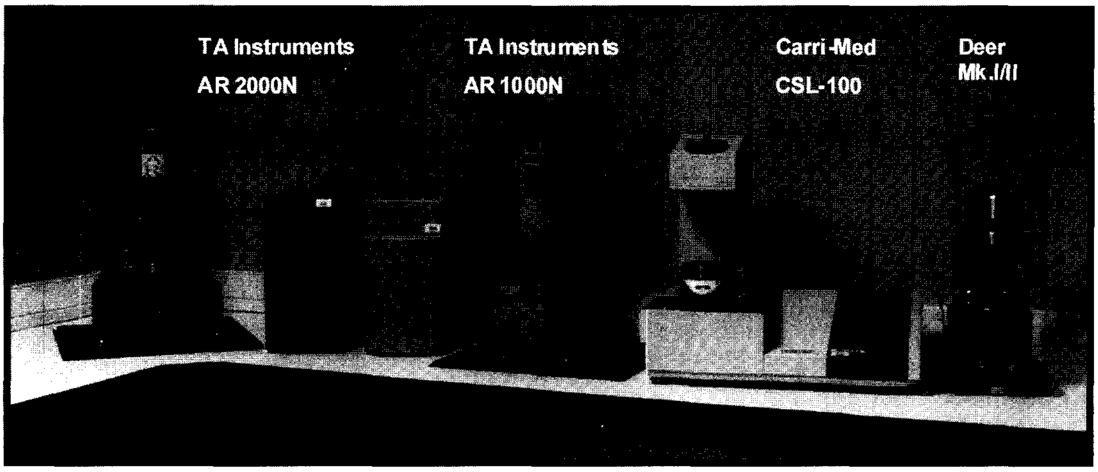


Fig. 7. The controlled-stress family tree, the Deer and its linear descendants.

company and in 1993 acquired Carri-Med Plc., and in 1996, TA Instruments became a subsidiary of the Waters Corporation, suppliers of equipment for permeation chromatography.

The development of this line of instruments is illustrated in Fig. 7.

In 1986 Bohlin introduced their CS controlled-stress instrument and around about the same time Krüss brought out their VE2. By the 1990s, many manufacturers had controlled-stress instruments at prices which rivalling controlled-speed, mid-price rheometers.

Now in the twenty-first century, top-of-the-range instruments have reached the point where few users will use even a small fraction of the instrument potential; the technique has matured; technical differences between makes are insignificant in practice, the real differentiator is now technical, software, and service support.

5. Special controlled-stress rheometer features

Air bearings

Various ball-bearing arrangements have been used to suspend and centre the rotating elements of rheometers and viscometers. Sometimes jewel or needle bearing methods of suspension were used, with the consequent low but by no means zero friction, while some home-made, non-commercial instruments have used thin-wire suspensions (e.g., Sherman). The introduction of the air-bearing as a suspending and centring method brought a radical improvement in the operation of all kinds of rheometers. For instance, the Wiessenberg Rheogoniometer used a radial air-bearing to centralise the upper spindle which was suspended from the torsion wire/bar and which held the upper geometry member, such as a cone, a plate or a cylinder. However, as the torque-sensing part of the instrument was only free to move through a very small angle, the bearing was simple in design and relatively undemanding from the manufacturing point of view.

The big step forward was the bearing used in the early Deer rheometer which provided axial as well as radial location, and this method allowed the suspended geometry to rotate continuously, 'floating on air' and rotating with minimum resistance.

For the ideal controlled-stress rheometer, when applying a nominally zero torque, the drive system should not rotate. But the very low level of achievable dynamic friction meant that even the slightest asymmetry in the radial air flow in the bearing resulted in rotation! This was known as the 'windmill' effect. The first solution to this was to apply a manually adjustable torque to try to offset the torque generated in the air bearing, the so-called 'bias' value. This was a significant help, but it required a skilled operator. The next step was for the instrument's electronics to perform the job automatically, and this was the situation until the mid 1990's.

By then, the performance of the electronics and transducers used in controlled-stress instruments had reached the point where using a single torque value to offset bearing asymmetries was no longer adequate, and that a 360° 'map' was produced of how residual torque varied with rotation, leading to more a more refined mapping procedures.

In parallel with this were a multitude of refinements to the design and manufacturing procedures of air bearings, but also in this respect the limits of what could be achieved with jet-type air bearings with their concomitant internal turbulence were being approached.

Various manufacturers have introduced air bearings where the air was introduced via a porous surface rather than through individually machined orifices (jets). In this situation, the relative freedom from turbulence led to an order-of-magnitude improvement in residual torque and its uniformity in rotation, extending the lower limits of usable stress values to the point where the low shear Newtonian region could be achieved with a wide range of materials.

Drag-cup motors

The requirement that a motor should produce a torque that is independent of the rotational speed is fully met by so-called drag-cup motors. These are non-contact and have no brushes, thus no friction, making them ideal for controlled-stress rheometry.

Sophisticated control systems now mean that because the position of the rotor is known very accurately, a feed-back system can be used to control the stress and then the system can be run as a controlled strain, as a given strain rate, or an oscillatory strain, or even a steady strain and monitor the stress, *i.e.*, a step-strain test.

The drag-cup motor came into its own when operated by microprocessor electronics capable of dealing with all of its quirks and technical difficulties. Combining this with the modern high-performance microcomputer to automate operation and analyse data, and one has the necessary ingredients for the modern revolution in affordable rheometers.

The drag-cup motor has some very desirable characteristics, particularly the smooth nature of the torque which is generated by the magnetic 'drag' of a magnetic field rotating typically at thousands of rpm, on a thin-walled light metal 'cup' - hence the name 'drag-cup'. It cannot however be assumed to have perfectly linear behaviour with torque or speed but these are easily dealt with by the drive electronics and an adequate calibration technique.

Another feature is that when the design is carefully optimised using modern computer-based engineering techniques, the moving parts of the drive system (the cup) can be very light indeed, in fact the limit is the difficulty of manufacturing the cup with thin enough walls. This results in the highly desirable result of very low inertia, which offsets the apparent disadvantage of the controlled-stress design where the inertia of the whole drive system affects the measurement, whereas in a typical controlled strain instrument, only the inertia of the torque sensing components affect the results. Minimising the inertia is the controlling factor for

- the maximum frequency in oscillatory testing and
- the response time for step changes in stress.

Residual inertial effects can be accounted for by the appropriate theory, see Baravian and Quemada (1990).

Optical encoders

Once controlled-stress instruments were available with good drives and low friction, the next challenge was the measurement of lower and lower levels of rotation and rotation rate. In modern instruments this is assured by the use of very finely discriminating optical encoders. Values of resolution as low as ~0.5 μ rad can now be made. Stable electronics can process the signals from the encoders to give measurement of rotation rates as low as 10^{-8} rad/s.

Producing controlled-stress or controlled strain on the same instrument

All modern controlled-stress instruments collect and log precise measurements of the torque, position and rotation rate of the spindle, and are thus able to be used for creep, flow curve and oscillatory experiments. However, if one has sophisticated electrical control system in conjunction with these data-logging facilities, it is, in principle, possible to use various rapid electronic feedback strategies in order to produce other mode of testing, *i.e.*,

- controlled-strain, so that various strain/time patterns can be achieved, so by increasing the rotational position linearly to produce steady-state flow or in an oscillatory fashion to produce a sinusoidal strain pattern. Logging the stress signal needed to achieve these particular patterns is the akin to measuring the stress in a controlled strain experiment.
- applying and holding a given rotational position and monitoring the torque, then monitoring the stress needed to hold the particular value of strain is akin to performing a stress-relaxation experiment.
- loop tests with a triangular shear-rate/time profile, etc, to mimic thixotropic loop tests for measuring thixotropy (however, this is not always desirable, since both time and shear rate are varying).

Of course, having such a sophisticated control system means that, not only can any experiment can be performed, but, in principle, *any form of electrical motor* could be used to produce *any* of these modes of testing, as long as, as we have said, we have the necessary precise values of torque, position and rotational speed. In fact, Paar Scientific claim that their use of electrically commutated motors, rather than a drag-cup motor can fulfil this purpose. These motors consist of permanent magnets mounted on the spindle surrounded by a set of coils. The coils carry a current that interacts with the magnets to induce a torque on the spindle, making it a variation of the synchronous motor. The torque is measured directly via the motor current. Because the current/torque relationship is inherently linear, these motors control stress quickly and accurately. This linearity also simplifies the electronics needed to feed position information from the encoder back to the motor, when adjusting the torque. The motor can therefore control position and speed well. The Paar-Physica company use these kinds of motors to drive their hybrid rheometers that they claim can produce either a controlled-stress or a controlled-strain capability. However, manufacturers of the drag-cup motor based rheometers method claim that using permanent magnets mounted on the rheometer spindle will greatly increase the mechanical inertia of the system, which is then large compared with the small amount of extra inertia from a thin metal drag-cup. Also, it can be argued that the linearity of torque versus drive current becomes a drawback when dynamic ranges of millions to one are sought, the drag cup motor with its quasi square law relationship between current and torque needs only a 1000:1 current range to achieve a million to one torque range.

6. A summary of the evolution of parameters in commercial instruments

Application of controlled shear stress has over the years been performed using :

- Weights and Pulleys (as in the original Stormer/Couette viscometer),
- Air Turbine + air bearing on a modified Rheogoniometer by Deer,
- Drag-cup motor + air bearing: Deer, TA (Carri-med), Bohlin and Rheotec International (now Krüss), and
- Electronically commutated motor + low friction (Physica) mechanical bearing.

Measurement of strain and strain rate (shear and shear rate) in these instruments was achieved by :

• Timing of rotations (Stormer)	~ 10^{-1} rad
• Slotted disk optical system and a snail-cam/inductive transducer	~ 10^{-3} rad
• Optical Encoder	~ 10^{-5} rad
• Enhanced Optical Encoder	~ 10^{-6} rad

The move to lower and lower shear stresses and shear rates made possible by these improvements is shown in table 1 for a series of rheometers which are linear descen-

Table 1. Some typical commercial controlled-stress rheometer specifications (see Barnes *et al.*, 1999).

Date	~ 1970	~ 1978	~early 1980s	~ late 1980s	~ 1999
Instrument	Air Turbine Rheometer	Deer Rheometer Mark 1	Carrimed RheometerMark 1	Carrimed CSL 100	TA Inst. AR 1000
Torque (N.m)					
Min.	10^{-4}	10^{-5}	10^{-6}	10^{-6}	10^{-7}
Max.	10^{-2}	10^{-2}	10^{-2}	10^{-2}	0.1
Resolution	10^{-4}	10^{-5}	10^{-7}	10^{-7}	10^{-9}
Ang. Veloc. (rad/s)					
Min. (creep)	–	–	–	–	10^{-8}
Max.	50	50	50	50	100
Resolution	–	–	10^{-2}	10^{-4}	–
Creep (strain)					
Resolution	2×10^{-2}	2.5×10^{-3}	$2.5 \times 10^{-}$	10^{-5}	6.2×10^{-7}
Max.	–	–	–	–	1300

Manufacturer	Models	Website
Bohlin	CS, CS50	Bohlin.co.uk
Brookfield	R/S	Brookfieldengineering.com
Haake	Rheostress 1 and 600	Therm.com
Physica	MCR series	Anton-paar.com
Reologica	Stresstech, Stresstech DMA	Reologica.se
Rheometrics*	SR5/ 2000/5000, ARES	Tainst.com
TA(Carri-med)	AR 500/1000/2000	Tainst.com

*Now owned by TA Instruments.

dants of Jack Deer's original instrument.

Developments to control the shear history of the sample were :

- Ramp control of torque by programmer,
- Ramp control of torque by computer,
- Preshear programming linked to ramp control.

Controlled-stress rheometers on the market today

The most well-known controlled-stress rheometers available today are shown in Table 1.

7. Advantages of controlled-stress rheometry

Creep testing

Creep testing refers to the application of a given stress to a material and the monitoring of the subsequent deformation. Its value had long been known for testing solid-like materials and there had been considerable developments in the area of experimentation and the concurrent theoretical explanation. Even for soft-solid-like materials such as ice-cream and soap had been studied. In all these cases the typical response was an immediate deformation on the application of the stress, and then the movement towards a

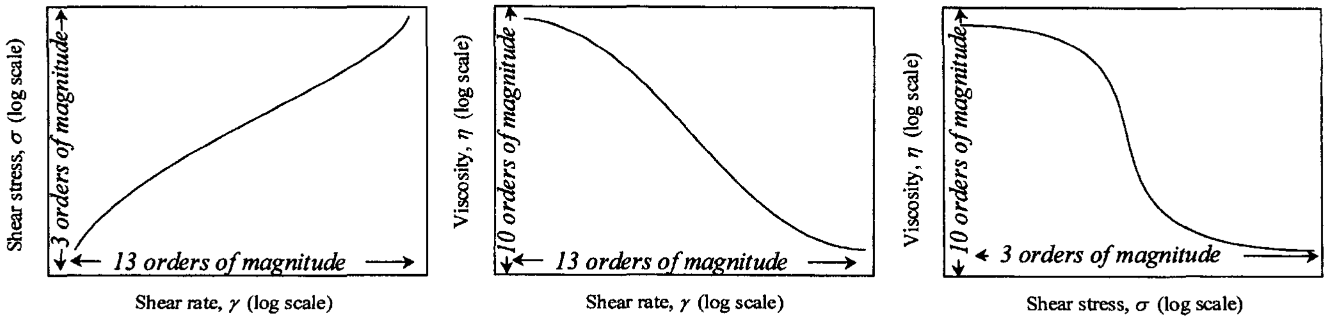


Fig. 8. Various ways of presenting flow data to illustrate the ranges of stress and shear rates that need to be generated and/or measured.

steady-state, but very slow continual deformation, so slow that it was called creep. If the test had been conducted on materials made up into long rods, then the immediate deformation was a Youngs modulus and the eventual slope of the steady state motion gave an extensional viscosity.

Complete flow curve

Even a cursory examination of a typical shear-thinning, non-Newtonian flow curve of shear stress versus shear rate will show the advantage of the controlled-stress method, see Fig. 8. (This might be a polymer solution such as Carbopol or a flocculated suspension of small particles.)

If we now examine the curves in Fig. 8, we see that trying to span the complete range of behaviour from the low-shear-Newtonian plateau to the high-shear-Newtonian plateau would require us to be able to generate either 13 decades of rotation speed, or three decades of stress. Then in the former case we would have to be able to measure 3 decades of generated stress and in the latter case, we would need to measure 13 decades of speed! In the early part of the last century, the possibilities in terms of speed and torque generation were very limited, so typically only a decade or so of stress and a few decades of rotational speed were possible.

The logarithmic gear box used in the Sangamo Weissenberg Rheogoniometer in the 1960s and 1970s went some way to produce a wide range of shear rates. With the available geometries, this instrument could produce shear rates over the typical range 10^{-3} to 10^{3} s^{-1}. The torque produced was measured via the small twist of a torsion strip on which the geometry was suspended. The lower rotation speeds were not applied immediately, and relied on the gear wheels engaging this was a major disadvantage and severely limited the lowest speed.

As we will see, modern developments have made it more and more possible that we are able to fulfil these generation and measurement requirements.

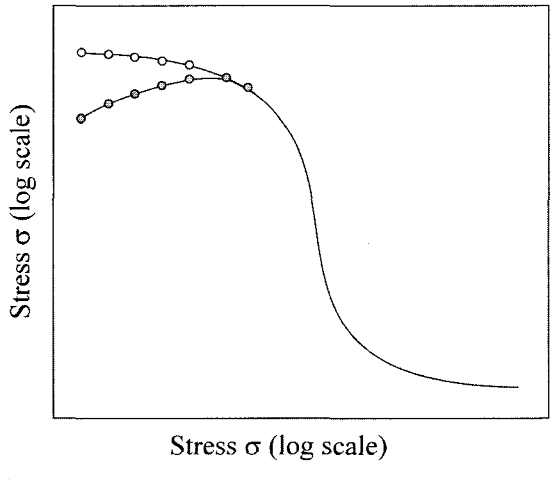


Fig. 9. Creep viscosity artefact.

A small problem

The typical strain response in time of a structured liquid to the application of a low stress is shown in Fig. 9. The short-term elastic-like response soon gives way to slower and slower movement until at last a minimum value of residual strain rate is achieved. However, it is difficult to know when the steady-state, minimum strain rate is actually achieved, especially at very low strain rates. In a typical creep experiment the stress is incrementally increased and the steady-state condition is sensed and the minimum rotation rate is recorded. If the conditions set up in the rheometer software that decide when steady-state has been achieved are not strictly enough set, steady state is thought to be achieved too early. The condition gets progressively worse as the set stress is decreased and the condition shown in Fig. 9 is sometimes seen. Tighter conditions need to be set to overcome this problem.

- filled points, data obtained at too-short test times,
- open points correct points obtained using longer test times.

A very useful output

The end result of the innovations present in the new generation of controlled-stress rheometers

- very low stable applied stress signal,
- very low friction, and
- very low rotation and rotation-rate measurement capability

Means that we have now a unique instrument that exactly matches the requirements of measuring the complete flow curves of many structured liquids.

Having the ability to measure at very low shear stress and measure any resulting flow (shear rate) also means that the controlled-stress instrument has been indispensable in resolving the yield-stress debate. When plotted on a linear basis, most flow curves for structured liquids indicate a yield stress when the flow curve is extrapolated to zero shear rate. This intercept on the stress axis becomes lower and lower as lower and lower shear rates are attained. When the very low stresses available on the latest controlled-stress instruments are applied to such liquids, we always see that steady-state flow is eventually achieved, albeit at very low shear rates, indicating very high (but finite) viscosity.

Having access to the lower Newtonian plateau often allows one to investigate and predict the physical stability of suspensions and emulsions where the particles of the dispersed phase might sediment or cream with time. For the very small particles often found in commercial products, the relevant stresses are quite small, and the viscosity of the suspending phase might be very large. The modern controlled-stress rheometer is therefore ideal for such a

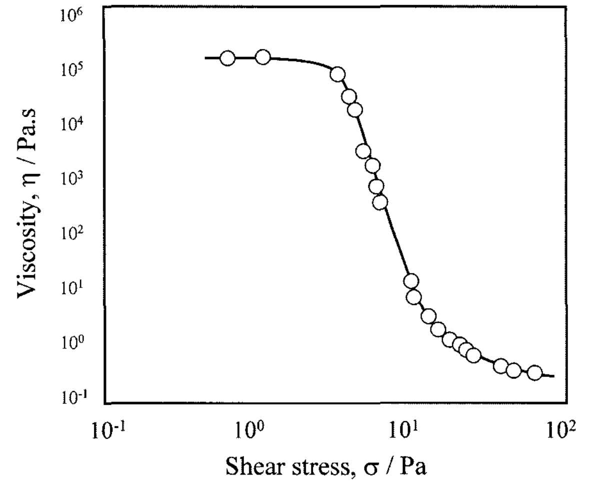


Fig. 10. The flow curve of a commercial sample of a liquid abrasive cleaner, measured by one of the authors (HAB) in 1983, using a Deer Rheometer Mark II.

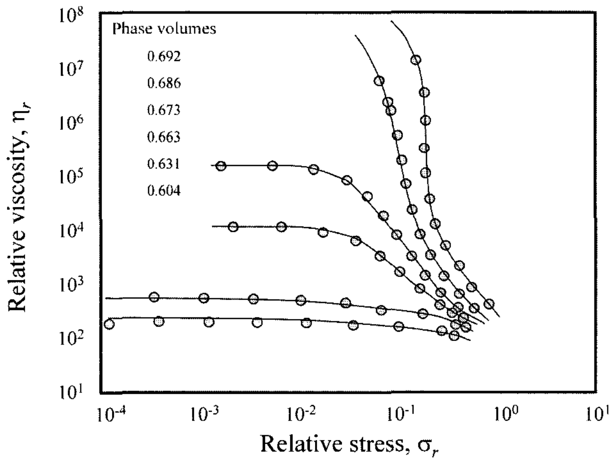


Fig. 11. Flow curves of latices : Buscall (1992).

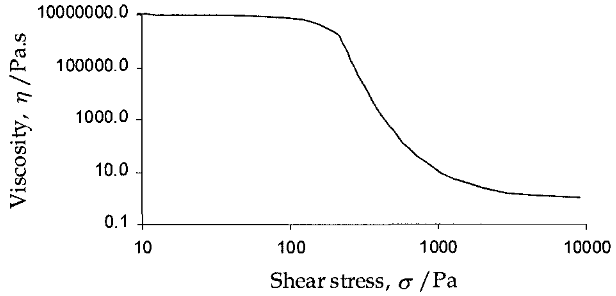


Fig. 12. Flow curve of a typical automobile lubricating grease.

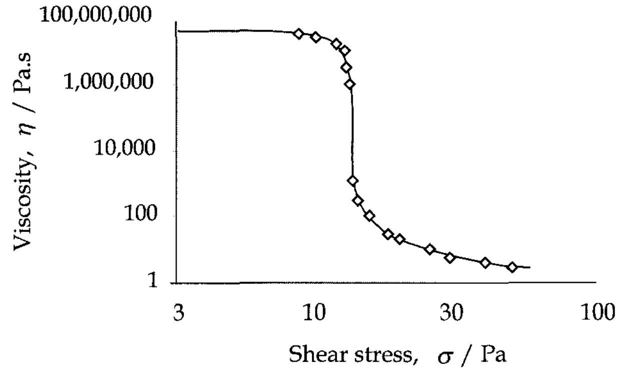


Fig. 13. Flow curve for molten chocolate.

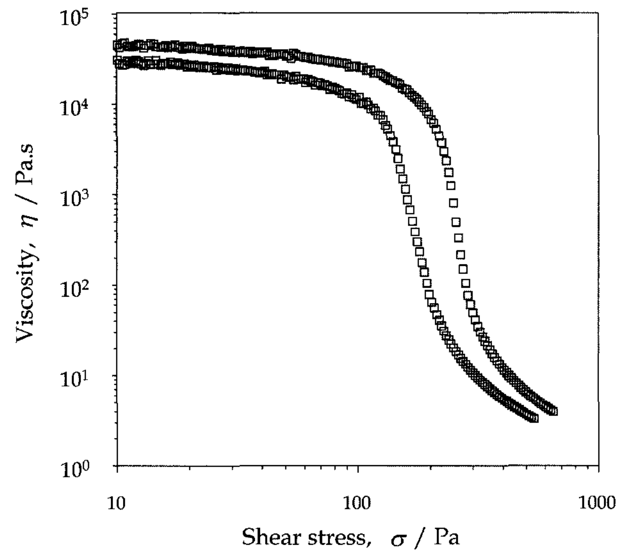


Fig. 14. Flow curve for two mayonnaises (data from Jason Stokes).

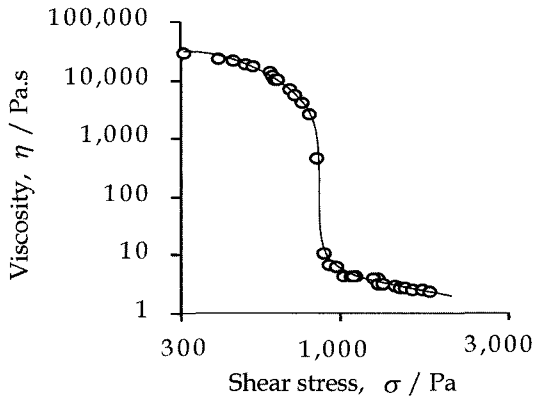


Fig. 15. Flow curve for flocculated ink.

study, see Salt (1990).

Some typical results

The end result of all the developments in controlled-stress is the ability to apply very low stresses and to measure very low shear rates. The usual result of this is that many workers have been able to span the whole range of behaviour of highly structured liquids that show very high viscosities at low stresses and low viscosities at high stresses, with the transition from the one kind of behaviour to the other occurring over quite a narrow range of stresses. Miscellaneous examples of this are shown in Fig. 10−15.

8. Conclusions

Although controlled-stress instruments were always available alongside their controlled-stress counterparts, they eventually lagged behind in terms of sophistication and lack of commercial exploitation, having given way in the postwar period to instruments such as the Brookfield and Haake viscometers and the Weissenberg Rheogoniometer. However, following the pioneering work of Jack Deer at the London School of Pharmacy in the 1960s, great strides were made, and sophisticated controlled-stress instruments resulted. They have now come of age and stand side-by-side with their controlled-strain equivalents in the marketplace. Given their ability to measure at very low stress and shear rates they have certain advantages for many structured liquids and are the rheometer of choice in certain areas.

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