30 YEARS OF PROGRESS IN VISCOMETERS AND RHEOMETERS

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Abstract

The history of commercial viscometry and rheometry over the last thirty years is summarised. In terms of those instruments that have established themselves in a lasting position in the field, we highlight the importance of a sound original idea, good marketing, and constant improvement as technology advances. Instruments in this area fall into three categories: simple, low-cost instruments to measure viscosity or at least an *equivalent* Newtonian viscosity; multi-speed instruments often able to record data on another display or storage device, and rheometers, i.e. instruments capable of a rotational shearing action, as well as one of the following: oscillation, creep and normal force measurement to give some measure of viscoelasticity.

The advances in technology in many areas over the past 30 years have all fuelled the rapid development of instruments that has been nothing short of astonishing. Of especial importance has been the continuous developments of electric motors, and transducers, as well as the increases in speed and memory and reductions in price of digital processors and microprocessors. With this increased computing power perhaps with instruments using complex flow regimes it may be as easy to analyse data as with simple shearing now. There will always be, however, those who want to go to higher frequencies, lower deformations, etc., to meet those challenges of measuring the rheology of materials at the limits.

Kurzfassung

Die Geschichte der kommerziellen Viskosimetrie und Rheometrie der letzten dreissig Jahre wird vorgestellt. An Hand von Geräten, die sich in der Rheometrie und Viskometrie selbst einen Platz geschaffen haben, wird die Bedeutung einer originellen Idee, eines guten Marketing und einer stetigen technischen Verbesserung diskutiert. Die Instrumente lassen sich in drei Gruppen einteilen: einfache, preisgünstige Geräte zur Messung der Viskosität oder einer zumindest der newtonschen Viskosität äquivalenten Viskosität; Geräte mit einstellbarer Geschwindigkeit und Datenausgabe; und Scherrheometern mit einer der folgenden Eigenschaften: Oszillations-, Kriechversuch- und Normalspannungsmessungen zur Bestimmung der viskoelastischen Eigenschaften einer Probe.

Die technologischen Fortschritte der letzten dreissig Jahre hat dabei zur Entwicklung von Instrumenten geführt, die durchaus Überraschungen bieten. Die stetige Entwicklung der elektrischen Antriebe, der Messwertaufnehmer sowohl die Erweiterung des Geschwindigkeitsbereiches, des Speicherplatzes und die Preisstürze bei Prozessoren und Mikroprozessoren sind hierbei von besonderer Bedeutung. Durch Kombination der gestiegenen Rechenleistung mit Messgeräten für komplexe Strömungsbereiche wird es in Zukunft vielleicht möglich sein, diese komplexen rheologischen Eigenschaften so einfach zu untersuchen, wie es heutzutage für die einfache Scherströmung üblich ist. Natürlich werden die Rufe nach höheren Geschwindigkeiten, kleineren Deformationen, usw. auch in Zukunft nicht verstummen, um auch die rheologischen Eigenschaften in Grenzbereichen untersuchen zu können.

Résumé

On résume l'histoire de la viscosimétrie et de la rhéométrie commerciales durant les trente dernières années. Parmi les appareils reconnus sur le marche, nous évoquons plus particulièrement ceux qui présentent une idée originale, un bon marketing et un développement constant . Les instruments répondant à ces critères se répartissent en trois catégories: les instruments simples et de faible coût permettant de mesurer une viscosité ou tout au moins un équivalent de viscosité newtonienne; les appareils à vitesse multiple souvent capables par ailleurs de visualiser ou d'enregistrer des données; et enfin, les rhéomètres, c.à.d. les appareils permettant d'appliquer des cisaillements en rotation et offrant l'une des possibilités suivantes: mesures en mode oscillatoire, fluage, mesures de forces normales pour évaluer la viscoélasticité des matériaux.

Les progrès technologiques dans de nombreux domaines depuis les 30 dernières années ont évidemment contribué au rapide développement d'instruments. Les développements constants au niveau des moteurs électriques et des capteurs, l'accroissement de la rapidité et de la capacité de mémoire des processeurs digitaux et des microprocesseurs, ainsi que la baisse de leurs prix, ont tout spécialement contribué à ces progrès. Avec cette puissance de calcul de plus en plus importante, il sera peut-être possible d'analyser les données pour des écoulements complexes aussi facilement que de nos jours pour un cisaillement simple. Il y aura toujours, cependant, ceux qui, en voulant atteindre des fréquences plus élevées, des déformations plus petites etc., tenteront de défier les limites actuelles des techniques de mesure des propriétés rhéologiques des matériaux.

Key Words:
Viscometer, rheometer, history.

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Date	~ 1970	~ 1978	~ early 1980's	~ late 1980's	~ 1999
Typical Instrument	Air Turbine Rheometer	Deer Rheometer	Carrimed Mk1	Carrimed CSL 100	TA Instruments AR 1000
Torque (Nm)					
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}
Angular velocity (rad/s)					
min. (creep)	-	-	-	-	10^{-8}
max.	50	50	50	50	100
Resolution	-	-	10^{-2}	10^{-4}	-
Creep (strain)					
max.	-	-	-	-	1300
Resolution	$2*10^{-2}$	$2.5*10^{-3}$	$2.5*10^{-4}$	10^{-5}	$6.2*10^{-7}$

Table 1: Some typical commercial controlled-stress rheometer specifications.

- Air Turbine + air bearing on a modified Rheogoniometer by Deer,
- Drag Cup motor + air bearing: Deer, TA (Carrimed), 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 falling weight (Stormer) 10^{-1} rad
- Slotted Disk (optical system) with Snail Cam and 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 descendants of Jack Deer's original instrument. Developments to control the shear history of the sample were:

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

7 RECENT DEVELOPMENTS

Welcome recent developments in rheometers over the last decade include:

- Automatic system checks and self calibration of inertia, etc., with electronic auto-zeroing and auto-ranging.
- Non-Newtonian wide-gap correction for concentric cylinder instruments.
- Inertia correction for ramped results.
- Gap/temperature compensation. (It is interesting to note that Ferranti-Shirley compensates for gap changes in a temperature ramp by heating a collar around the lower plate; this was from the early 70's. Recently a rheometer user took daily recordings of the contact micrometer reading of their Carri-Med and the laboratory temperature, summer and winter. The graph of ambient temperature against micrometer reading was surprisingly linear!) To some extent this is answered by Bohlin and Rheometrics monitoring normal force or temperature; this is called 'autotensioning'.
- Automatic selection of the best model for curve-fitting data.
- Data-handling protocols for data transfer to main-frames.
- Mathematical curve fitting especially non-linear models such as the Cross, Bird-Carreau, etc.
- Computer interfaces as standard as in all scientific instrumentation.

8 A VISION OF THE FUTURE

How can this class of instruments be improved? We suggest the following as some possible improvements, readily achievable in the near future:

- An instrument system that automatically recognises the inserted geometry, possibly by some bar-code recognition.
- Algorithms to test for thixotropy and to use the results to design a method to obtain further more comprehensive results.
- Use of non-Newtonian standard liquids and self-checking.
- Check for wall-slip using data from different cylinder sizes.
- Universal procedures to operate on all instruments, *i.e.* open systems of software.
- Magnetic levitation for geometry suspension to eliminate the need for an air supply.

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9 CONCLUSION

The advances in technology over the past 30 years has been nothing short of astonishing, especially with the continuous increases in speed, memory and reductions in prices of Personal Computers. This has been particularly true in the area of controlled-stress rheometers, see Barnes [5]. With increased computing power perhaps with instruments using complex flow regimes it will be as easy to analyse data as it is with simple shearing now. There will always be, however, those who want to go to higher frequencies, lower deformations, etc., to meet those challenges of measuring the rheology of materials at the limits.

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Erratum

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NONEQUILIBRIUM THERMODYNAMICS – A TOOL FOR APPLIED RHEOLOGISTS: ERRATUM

Author: Hans Christian Öttinger

p 20: Equation 4: $M(x) = M(x)^T$ instead of $M(x) = -M(x)^T$
p 24: Ten lines before the CONCLUSIONS: Ref. [9] should be Ref. [10]
p 24: Inside CONCLUSIONS: Line #9: 'loose' should read 'lose'
p 25: The photo was taken in 1997 (and not in 1998)

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MICRORHEOMETRY FOR STUDYING THE RHEOLOGY AND DYNAMICS OF POLYMERS NEAR INTERFACES: ERRATUM

Authors: Gavin J.C. Braithwaite and Gareth H. McKinley

The manuscript should be supplemented by the following table:

Parameter	Value		Comment
Plate Size (diameter)	0.013 m		5 mm-0.02 m possible.
Gap Range	0.5-500 μm		Practical lower limit ~ 5 μm.
Sample Size	0.07-40 μL		Governed by parameters above.
Temp. Range	Ambient		The design has been specifically considered with higher (~ 200° C) temperatures in mind – since we messure the gap absolutely thermal expansion is not a problem.
Stress Range	8 mPa-1 kPA		This is drive current limited so different drive electronics would significantly incress the range.
Dynamic Range (oscillatory)	< 1 mHz-500 Hz		Governed by the stiffness of the springs and mass of surface. Note resonance currently at 50 Hz.
Velocity Range (steady shear)	~ 1 nm/s-1 mm/s		The lower limit at the moment is drift limited.
	Gap = 1 μm	**Gap = 500 μm**	
Strain Range (gap dependent)	0.5 %-5000 %	0.001 %-10 %	
Strain Rate Range	$2 \times 10^{-6} s^{-1}$-$2 s^{-1}$	$1 \times 10^{-3} s^{-1}$-$1000 s^{-1}$	
Viscosity Range	4 mPa.s-500 x 10^6 Pa.s	8 x 10^{-6} Pa.s-1 x 10^6 Pa.s	

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