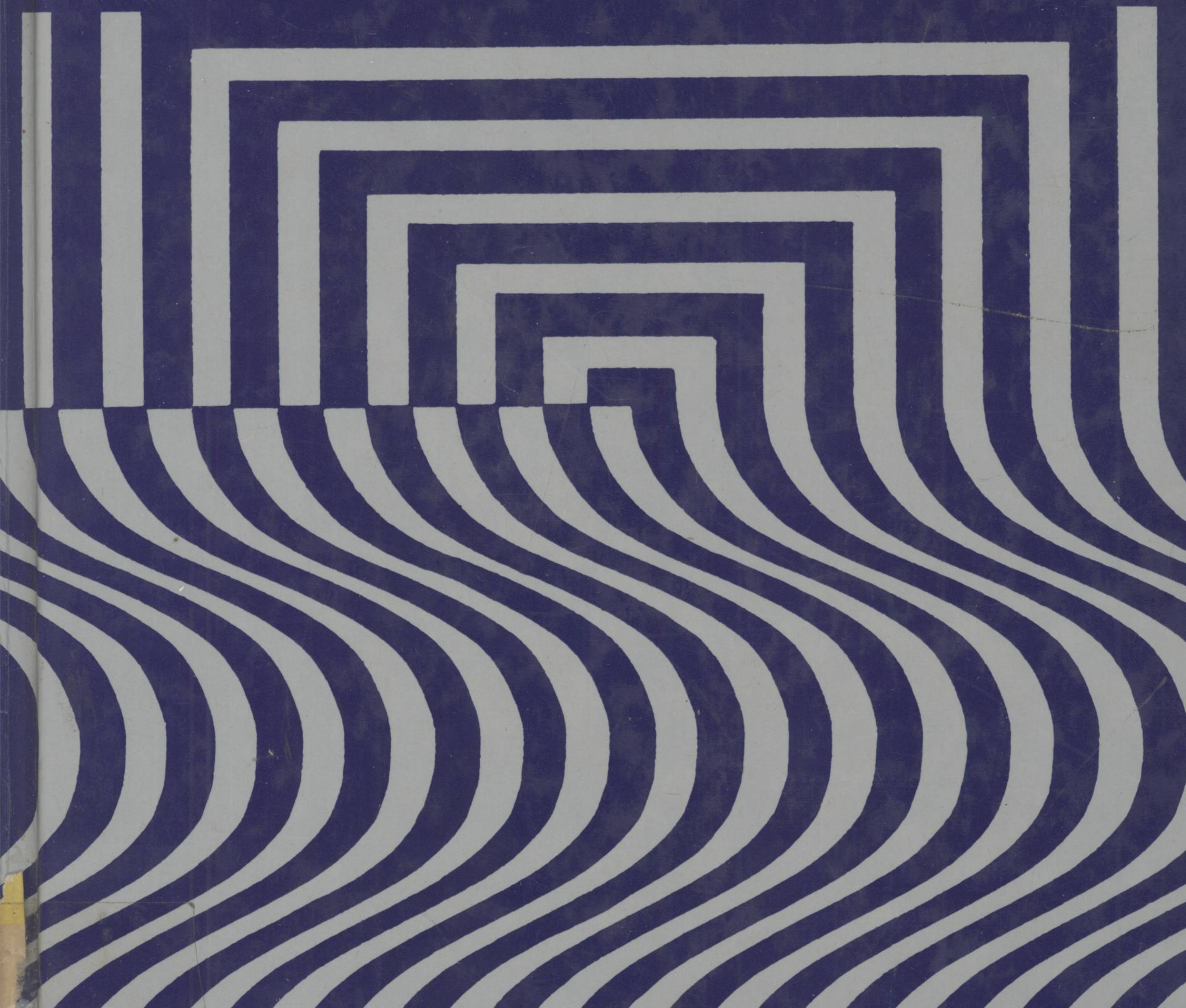
ELLIS HORWOOD SERIES IN PHYSICS AND ITS APPLICATIONS

RHEOLOGICAL TECHNIQUES

second edition

R.W. Whorlow



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Author's preface

My interest in a unified treatment of rheological experimental methods began over thirty years ago when I established a postgraduate course entitled 'Rheological Techniques' at Battersea College of Technology. The audience included chemists, physicists, biologists, pharmacists, polymer and paint technologists, and others. In this book I have given a much fuller and up-to-date discussion of techniques but have assumed that the readership would cover a similar wide range of interests. This assumption has influenced both the choice of examples of instruments and the level of the mathematical analyses.

I have attempted to give an account of the principles of all the important, and many of the less important, rheological test methods. In some cases particular instruments have been described in detail, but more usually the sections of instruments used to measure pressure, torque, displacement, frequency, etc. have been considered separately. Thus a rheologist faced with a new problem in measurement will be helped to select the most appropriate component parts to assemble to form a complete instrument. Those with more orthodox requirements may find a suitable commercially available instrument among those listed in the Appendix.

Since the first edition there has been considerable progress in methods of measuring elongational viscosity and a complete new chapter has been devoted to this topic. Also, in this edition more attention has been paid to normal stress measurement, particularly to the recently developed methods not covered in Professor K. Walter's monograph (*Rheometry*, Chapman & Hall, 1975). The now widespread use of computers to control instruments and to analyse data is referred to repeatedly throughout the book. The criterion for inclusion of an instrument in this book is that it should be capable of providing values of stress over a range of known strains or strain rates. Thus 'single point' instruments, for example penetrometers or flow cups, have, in general, been excluded.

Most of the formulae used to evaluate experimental results have been derived from basic physical principles. The derivations are not always rigorous mathematically but are intended to give an understanding of the origin of many widely quoted formulae without recourse to primary sources. In particular, expressions used in the analysis of viscoelasticity measurements have been presented in a consistent notation for the entire frequency range. Where appropriate, free use has been made of complex numbers and of the solutions of differential equations, but tensor analysis has not been used. The terminology is in general consistent with the *British Standard Glossary of Rheological Terms* (BS 5168:1975, British Standards Institution).

The information presented in this book has been accumulated, from sources too numerous to mention, over many years. However, my task in obtaining up-to-date information would have been far more difficult without the entirely voluntary work

of the Honorary Editors and abstractors of *Rheology Abstracts* and the Appendix could not have been produced without the cooperation of the many companies listed. Finally I should like to thank my wife for all her help and encouragement.

R. W. Whorlow

Preamble: on computers in rheology

Imagine a visitor to a current exhibition of rheometers who had not been to such an exhibition since 1980, the year of publication of the first edition of *Rheological Techniques*. His or her immediate reaction would be: 'The computer has taken over!'

Computer control is essential for some rheological measurements and very convenient for many others. Computer evaluation of results is also essential with some experimental techniques, but for most it is simply very convenient. Either use can very easily trap the unwary into drawing false conclusions. One of the potential disadvantages of the use of computers in rheology has been put succinctly by Baker *et al.* (1988): it 'deters operators from thinking'!

When a computer is used to analyse results it is essential to be able to obtain an output of raw data after an absolute minimum of electronic manipulation in order to get the 'feel' of the experiment in some typical, and untypical, situations. Odd behaviour at, for example, the extremes of shear rate, frequency, temperature, etc., at points where there is a change of range, or with slightly unusual samples is more likely to be obvious in the raw data. Calculation of the required results by independent means in a few cases and comparison with the output from the computer is invariably instructive; it will either show up the deficiencies in the program or, with luck, give the operator confidence that the computer is doing what he thinks it is doing.

There are many reasons why the software provided with a rheometer may not do quite what the purchaser expects. Apart from 'bugs' (which, of course, only show up with his rather unusual materials!), many questions may not be answered clearly by the suppliers. Is the empirical formula used to characterise the sample appropriate for this material over the whole range? Will one inaccurate point at the bottom of the range be discarded, or retained and contribute disproportionately to the calculated intercept? How is the phase difference between two harmonic signals found and will the computer 'notice' if one is by no means harmonic? Is the data being smoothed before being differentiated and is any useful information being lost as a result? After some work on the raw data other similar questions are likely to be asked.

When the computer is used to control the apparatus, rather different questions arise. These may be straightforward to answer but nevertheless important—the effectiveness of temperature control can be checked by independent thermometers. A common action of a computer controller is to change the rotation rate (or flow rate) stepwise when the torque (or pressure) becomes sufficiently steady, for example, changes by less than $x\%$ in 10 s. Results obtained in this way can be very misleading unless the time-dependence of the torque has been followed for the same type of material for longer times. If the torque falls quickly, within a few seconds, to a steady value the computer program is sensible. If it falls slowly and steadily, at less than $x\%$ in 10 s, for a long time, the measured torque will not represent a steady value.

In cases where computer control is crucial to the operation of the apparatus, for example when very rapid changes in flow rate or strain are to be made, it may only be possible to test the apparatus as a whole by using well characterised materials—Newtonian fluids, steel springs, or the viscoelastic materials which have been used for large-scale inter-laboratory comparisons in recent years.

Computer hardware and software are outside the scope of this book. Anyone contemplating purchase or use of a rheometer of any type with an associated computer should read the survey by Baker *et al.* (1988) on 'Computer control and data processing in extrusion rheometers'.

REFERENCE

Baker, F. S., Carter, R. R. and Privett, G. J. (1988) In: Collyer, A. A. and Clegg, D. W. (eds), *Rheological Measurement*, Elsevier Applied Science, pp. 151–188.

List of symbols

Conventional symbols used for coordinate directions, etc., and symbols used only immediately after they have been defined are not included.

A	Cross-sectional area
a	Tube radius. Rod radius. Thickness of strip
a_T	Shift factor
B	Magnetic flux density
b	Breadth of slit die. Breadth of strip. Shape factor
C	Stress-optical coefficient
c	Torsional rigidity. Wave velocity
c_g	Group velocity
$D^* = D' - \mathrm{j}D''$	Complex tensile compliance
D	Die-swell ratio
d	Thickness of strip (bending)
E	Young's modulus
$E^* = E' + \mathrm{j}E''$	Complex Young's modulus
e	Potential difference (moving coil)
e'	Potential difference (clamped coil)
F	Force
f	Frequency
G	Torque per unit length
G'	Shear modulus (Hookean materials). Shear storage modulus (viscoelastic materials)
G''	Shear loss modulus
G^*	Complex shear modulus
G_{T}	Total torque. Bending moment
g	Acceleration of free fall
$H(\tau)$	Modulus density function
h	Thickness (compression tests). Thickness of slit die
I	Moment of inertia. Second moment of area of cross-section
$J^* = J' + \mathrm{j}J''$	Complex shear compliance
J_{g}	Glassy state compliance
J_{p}	Reduced compliance
K	Bulk modulus. Thermal conductivity
$K^* = K' + \mathrm{j}K''$	Complex bulk modulus
k	Coefficient in power law equation. Radius of gyration of cross-section
$L(\tau)$	Compliance density function
l	Length. Plate spacing (rotational instruments)

l_w	Length of wire in coil
m	Mass
N,n	Exponents in power law equations
N_1,N_2	First and second normal stress differences
P	Isotropic pressure. Total pressure difference
p	Pressure gradient
Q	Mechanical Q-factor
R	Cone radius. Disk radius
R_1,R_2	Inner and outer cylinder radii
Re	Reynolds number
r	Arbitrary radius. Reflection coefficient
S	Surface or interfacial tension
T	Kelvin temperature. Period
T_j	Jet thrust
t	Time
U	Plastic viscosity
u	Velocity of fluid element
V	Volume. Volume flow rate
v	Mean fluid velocity. Velocity of bounding surface or of mass, etc.
W	Wall shear stress. Force on beam
w	Weight per unit length
Z	Electrical impedance
Z_0	Electrical impedance of stationary coil
Z_M	Mechanical impedance (Chapter 6). Mechanical impedance per unit area (Chapter 7)
$Z_c = R_c + jX_c$	Characteristic impedance
z_0	Attenuation constant
δ	Phase lag of strain relative to stress
ε	Extensional strain. Tube end correction
Γ	Propagation constant
γ	Angle of shear. Shear strain
γ_0	Strain amplitude
η	Viscosity (in shear)
$\eta^* = \eta' - j\eta''$	Complex viscosity
η_E	Extensional viscosity
θ_0	Cone–plate angle
Λ	Logarithmic decrement
λ	Wavelength
ν	Kinematic viscosity
Π	Poisson's ratio
ρ	Density. Radius of curvature (bending)
σ	Stress
σ_Y	Yield stress
σ_0	Stress amplitude

τ	Retardation time. Relaxation time
ϕ	Fluidity
Ψ_1, Ψ_2	First and second normal stress coefficients
ψ	Coefficient in power law equation
Ω	Angular velocity of cylinder, cone, etc.
ω	Angular velocity of fluid element. Angular frequency

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