A New QC Instrument technology for Asphalt

Asphalt Institute meeting

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SCIENTIFIC INSTRUMENTS

Modern Rheometers

- The science of rheology is now well able to characterise bitumen at a development/R&D level
- BUT
  - Modern rheometers are almost universally controlled stress based.
  - This means
    - Expensive
    - Delicate
    - Need PC/compressed air/water circulator
    - Skilled lab operator

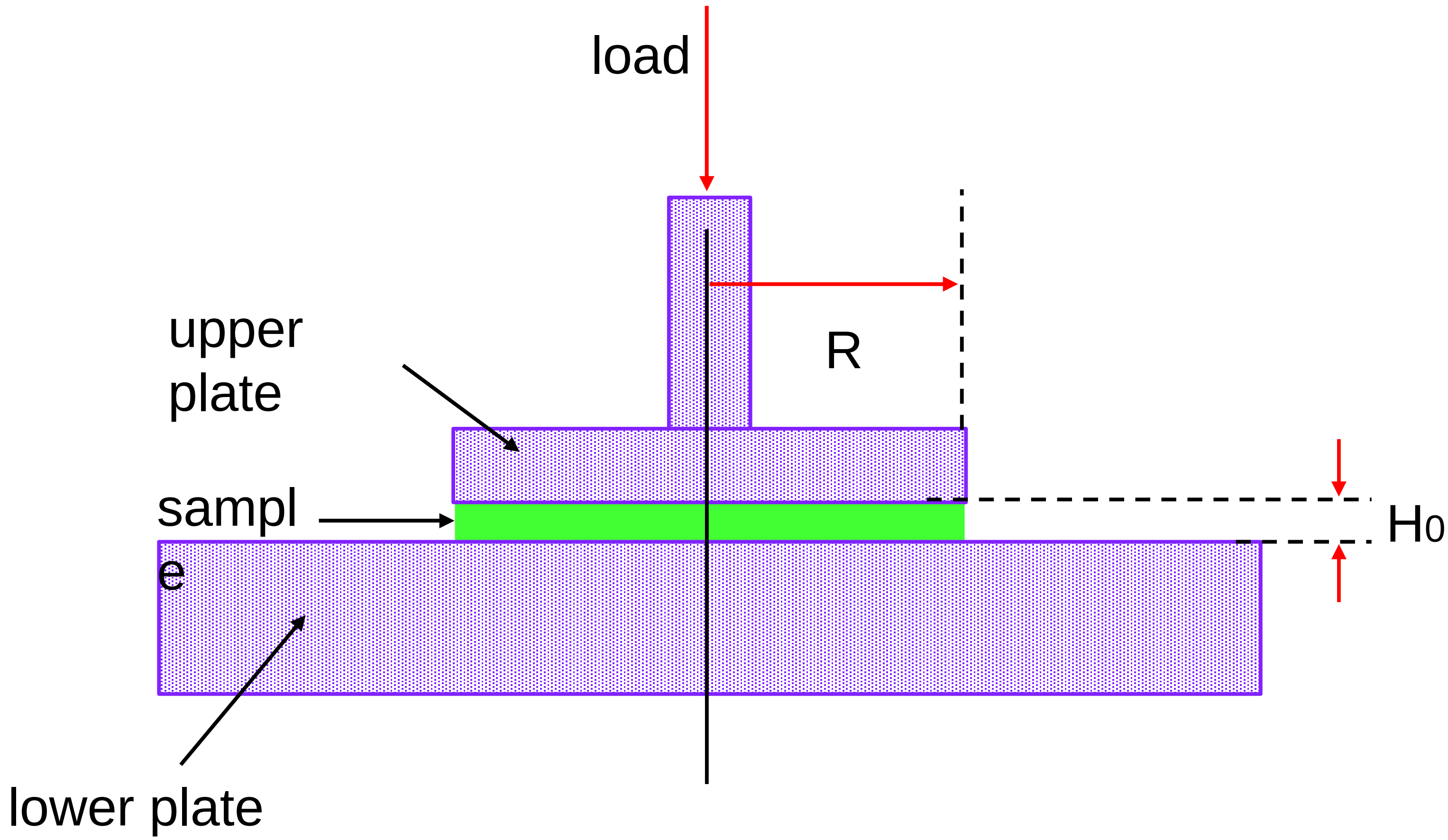
Rheometer principles

- The use of rotational drive systems in rheometers is almost universal.
- It is
  - widely accepted and understood
  - the theory underlying these instruments is straightforward
  - But the accompanying technology of:- optical encoders, air bearings, ECM & drag cup motors etc. is responsible for the price/complexity/delicacy.
- For a genuine low cost, robust instrument, something new is needed!

Oscillatory Squeeze flow

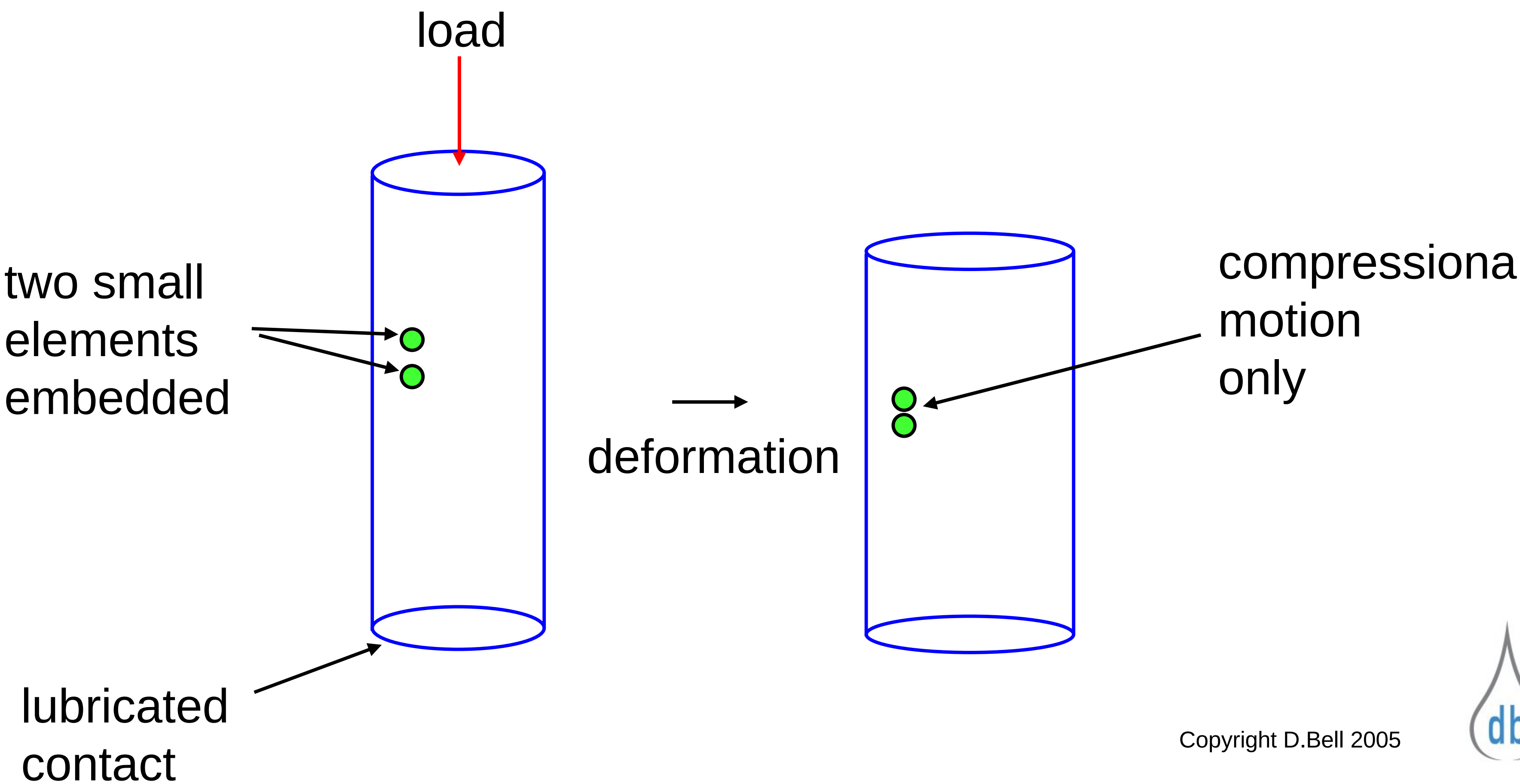
- The need for straightforward, easy to analyse theories was important in the days of hand calculations.
- But maths has moved on and the PC can deal with almost any conceivable mathematical complexity in far less time than an experiment takes.
- So now a simpler, cheaper to make, drive system can be used even if the analysis is more complex.
- Hence OSF !

A Simple compressional rheometer



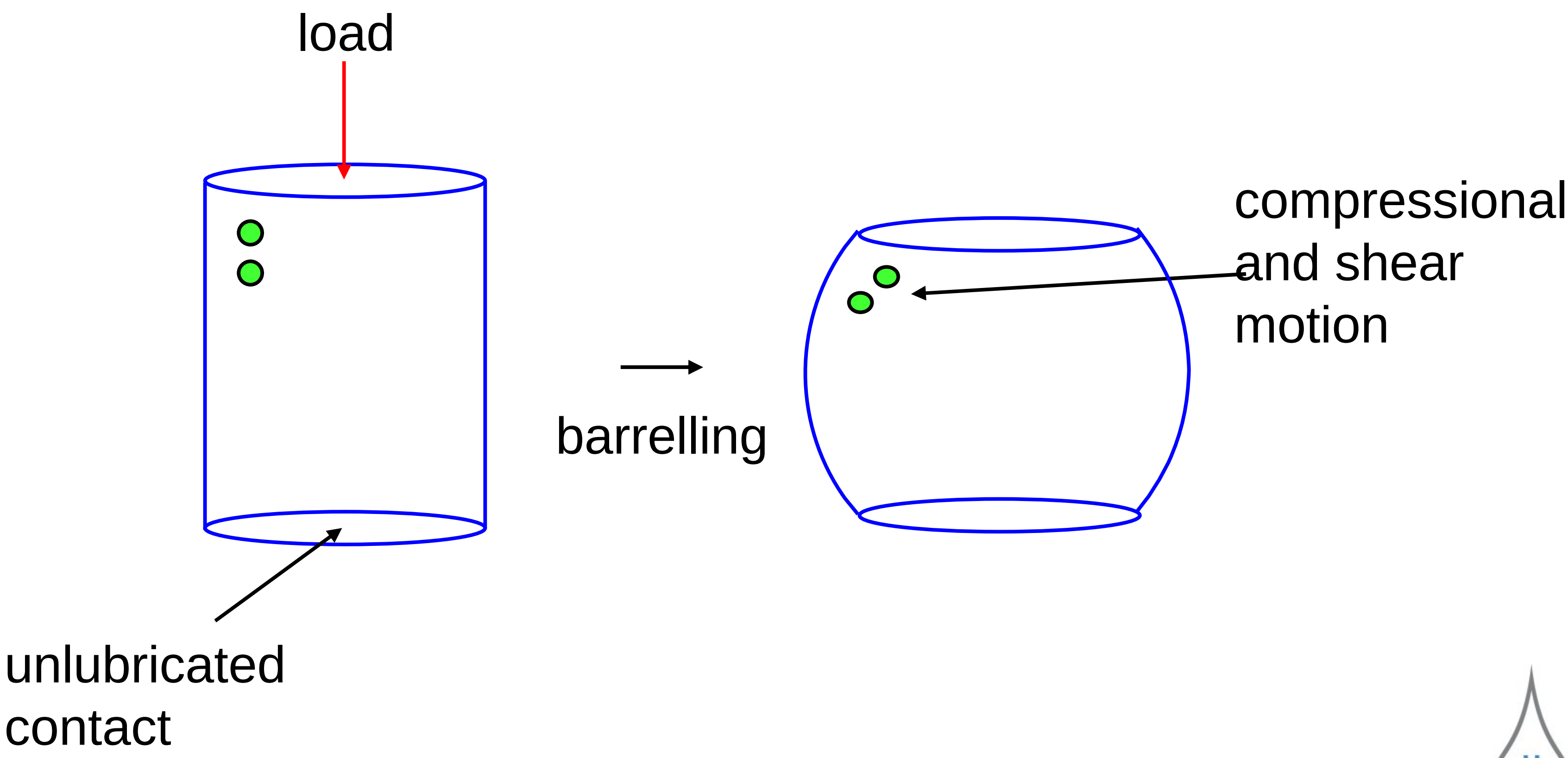
Cylinder of high aspect ratio or with boundary slip

Gap, H >> radius, R



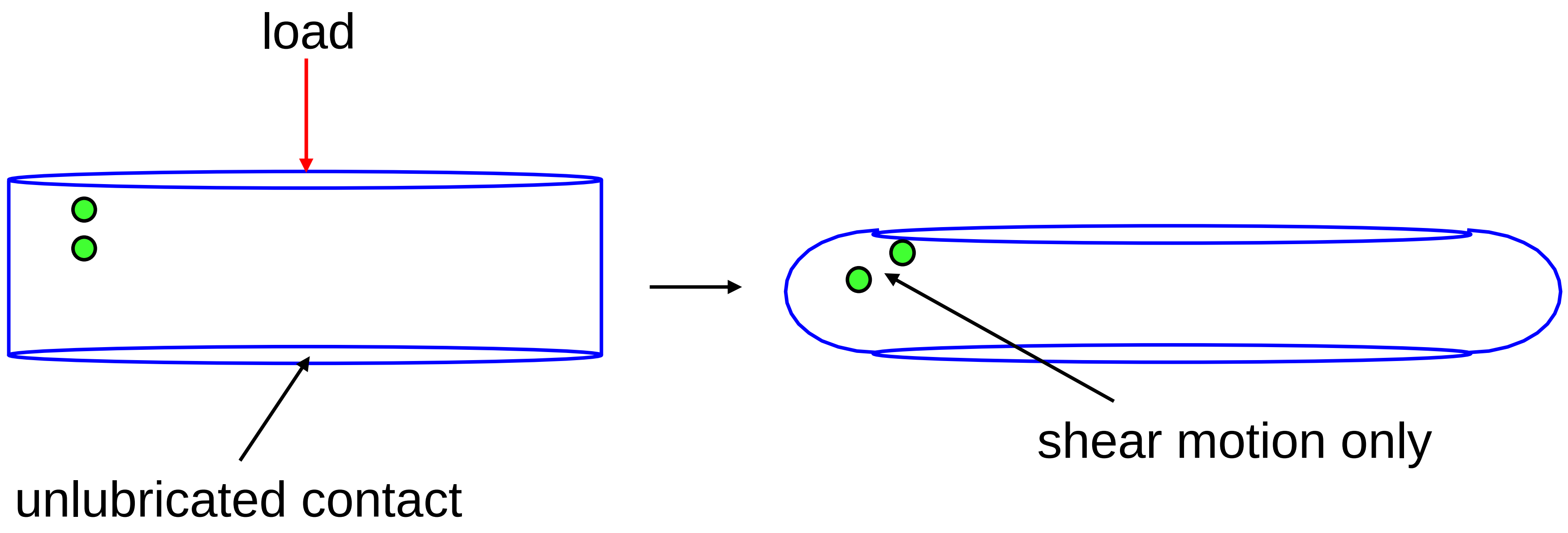
Cylinder of intermediate aspect ratio (no-slip)

Gap, H ~ radius, R



Cylinder of low aspect ratio (disc): no slip

Radius, R >> gap, H



Advantages of simple compressional rheometer

- Easy to construct
  weight on a plate

- Easy to operate
  measure displacement over time

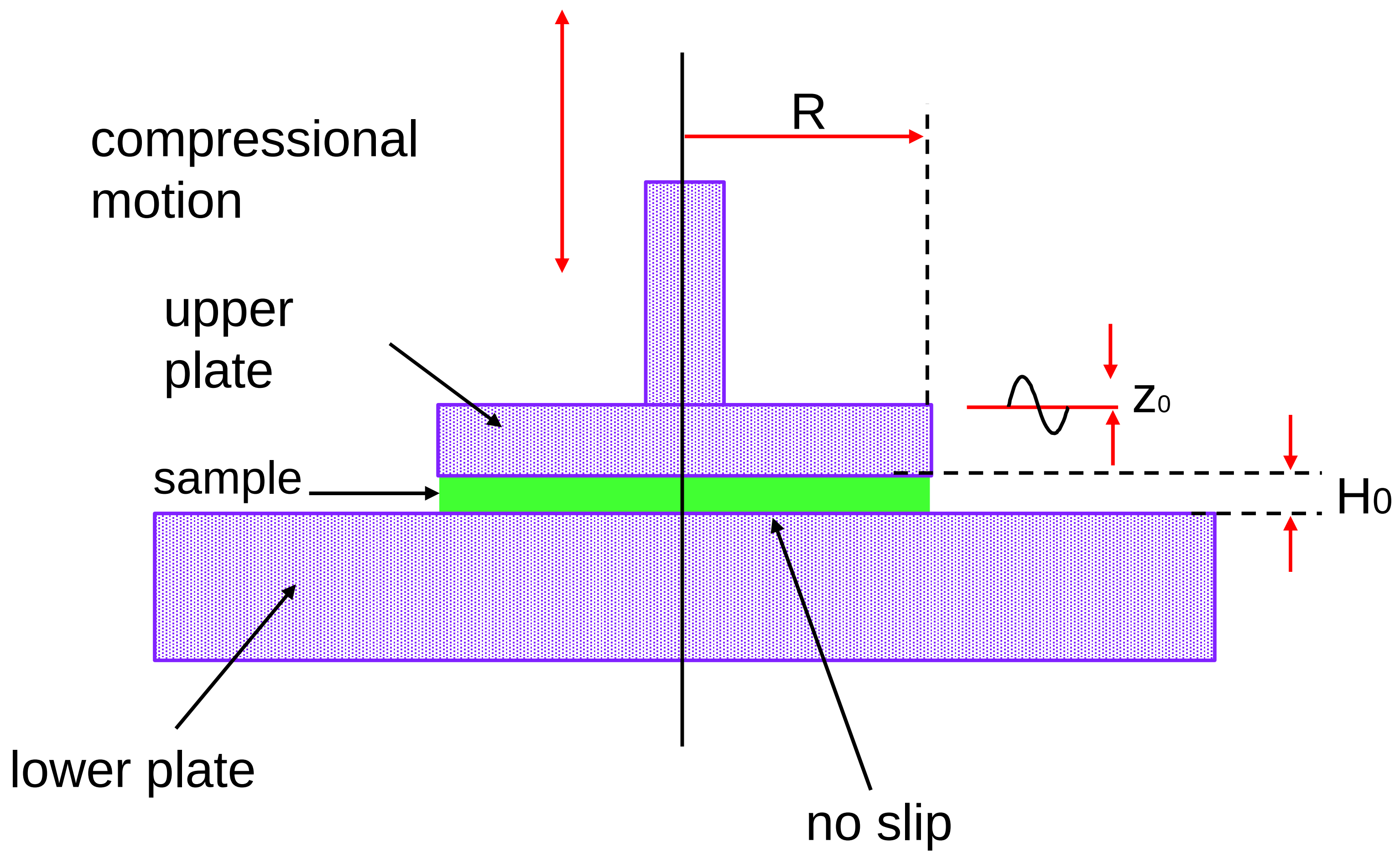
- Range of shear rates
  high strain rates
  low shear rate at large gaps
  high shear rate at small gaps

- So long history
  - Stefan, 1874

Disadvantages of simple compressional rheometer

- Inhomogeneous stress field
  - components of both shear and compressive stresses
  - range of shear and compressive stresses
- Limited travel
  - single pass
  - cf. infinite travel for rotational instruments

Operation of the CP50



Basics of OSF theory (1)

Consider two infinite, parallel, plates at *z = 0* and *z = H(t)* where *r, q, z* is the usual cylindrical polar coordinate system and *t* is time. The lower plate is stationary and the upper plate is assumed to oscillate with frequency *w*, about *z* = *h* (constant). Hence:

$$H(t) = h + \varepsilon e^{i\omega t}, \qquad (1)$$

where *e* is the amplitude of the oscillation, which is assumed to be small. *e* is real and the real part of (1) is implied.

A fluid is contained between the plates. The velocity components for the fluid are all assumed to have an $e^{i\omega t}$ dependence:

$$v_r = u\, e^{i\omega t},\ v_\theta = v\, e^{i\omega t},\ v_z = w\, e^{i\omega t}, \qquad (2)$$

where *u, v* and *w* may be complex.

Basics of OSF theory (2)

The pressure field is written as: $p = P\, e^{i\omega t}$. (3)

The normal force F, required to generate the flow is assumed to have the form:

$$F = F_0\, e^{i(\omega t + c)}, \quad (4)$$

where c is referred to as the phase lag.

In the 'Compressional', F_0 and ω are specified and ε and c are measured (i.e. it is a *stress*-controlled instrument). We wish to relate ε and c to relevant rheometric properties of the fluid.

Basics of OSF theory (3)

It follows that the governing equations of motion are, to first order in e:

$$i\omega\rho u = -\frac{\partial P}{\partial r} + \eta^*\left(\frac{\partial^2 u}{\partial r^2} + \frac{1}{r}\frac{\partial u}{\partial r} + \frac{1}{r^2}\frac{\partial^2 u}{\partial \theta^2} + \frac{\partial^2 u}{\partial z^2} - \frac{2}{r^2}\frac{\partial u}{\partial \theta} - \frac{u}{r^2}\right) \qquad (7)$$

$$i\omega\rho v = -\frac{1}{r}\frac{\partial P}{\partial \theta} + \eta^*\left(\frac{\partial^2 v}{\partial r^2} + \frac{1}{r}\frac{\partial v}{\partial r} + \frac{1}{r^2}\frac{\partial^2 v}{\partial \theta^2} + \frac{\partial^2 v}{\partial z^2} + \frac{2}{r^2}\frac{\partial v}{\partial \theta} - \frac{v}{r^2}\right) \qquad (8)$$

$$i\omega\rho w = -\frac{\partial P}{\partial z} + \eta^*\left(\frac{\partial^2 w}{\partial r^2} + \frac{1}{r}\frac{\partial w}{\partial r} + \frac{1}{r^2}\frac{\partial^2 w}{\partial \theta^2} + \frac{\partial^2 w}{\partial z^2}\right) \qquad (9)$$

while conservation of mass implies that:

$$\frac{\partial (ru)}{\partial r} + \frac{\partial v}{\partial \theta} + r\frac{\partial w}{\partial z} = 0 \qquad (10)$$

Basics of OSF theory (4)

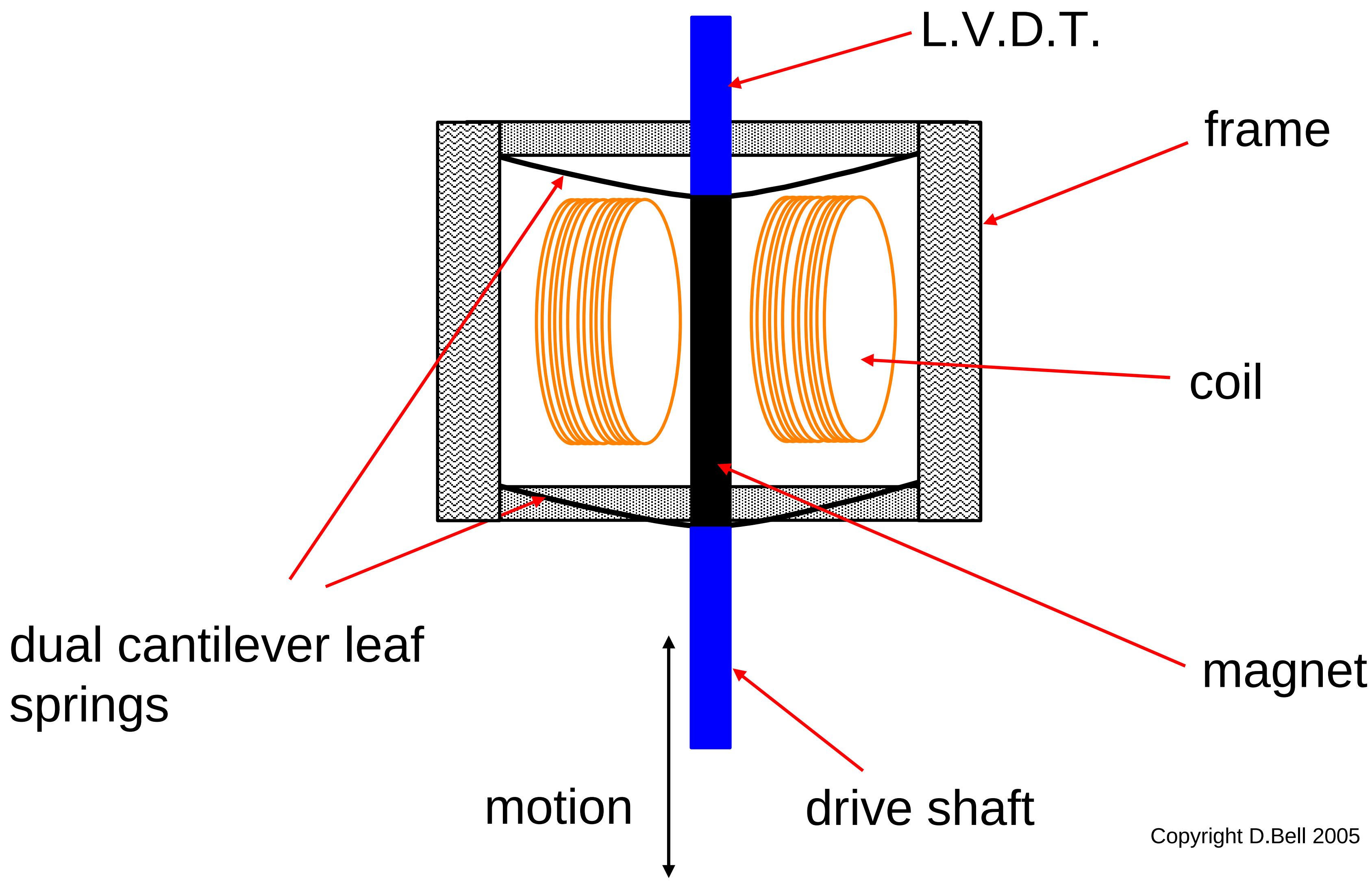
After mathematical manipulation and solving the equaitions, the **Elastic** and **Viscous** modulii are shown to be given by:

$$G' = \frac{2h^3 F_0 \cos \omega c}{3\pi \varepsilon a^4} + \frac{\omega^2 \rho h^2}{10}$$

and

$$G'' = \frac{2h^3 F_0 \sin \omega c}{3\pi \varepsilon a^4}$$

Schematic of the actuator assembly



- It actually looks like this

- It is compact, robust, easy to operate electrically, and needs no compressed air!

Calibration

The Physics of the basic “mass on a spring” instrument is simple and easy to characterise very precisely and can be calibrated from first principles with just a weight

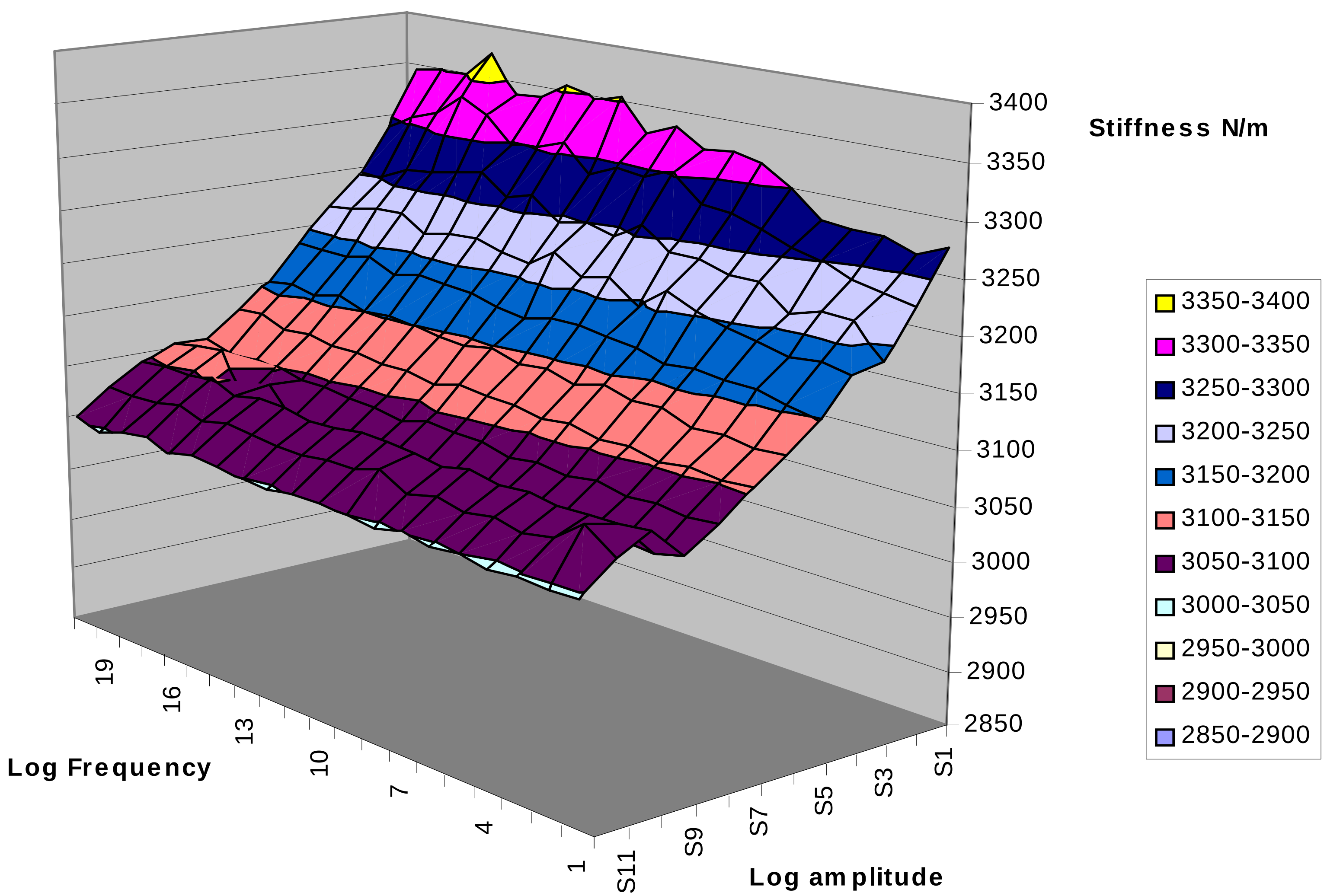
1. Lower plate Lead screw stepper motor is actuated to force a defined movement of the suspension, this calibrates the displacement sensor and d/a converter
2. A calibrated weight is hung from the suspension, the resulting deflection of the suspension is measured by the displacement sensor, and hence the spring constant is known. Then the drive transducer is held at the null position by the drive electronics, and the force system is calibrated.
3. The electronics then sweep a range of frequencies to detect the resonance frequency, which enables the mass of the moving parts and the damping factor to be calculated

Now the instrument can measure accurately force and displacement. This is all carried out by a utility built into the instrument which is accessed by a PC through a serial port.

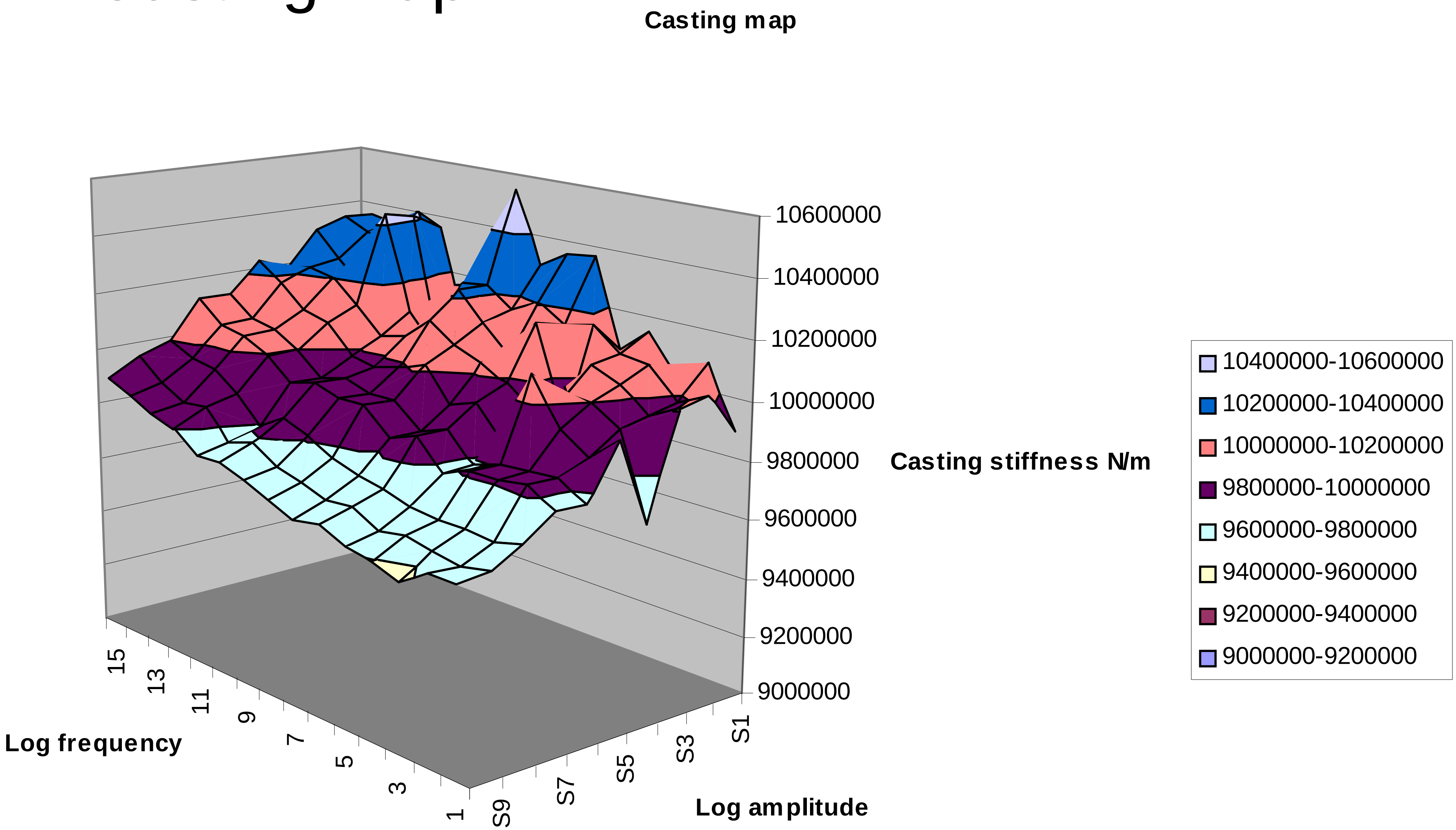
Mapping

- The ultimate sensitivity and accuracy can be improved by “mapping the precise behaviour of the suspension system.
- At high forces, the range can be extended to stiffer materials by allowing for the deflection of the casting which can also be mapped.
- These mappings are carried out by firmware utilities built into the instrument, and are easy to carry out.
- For a given instrument, the maps are consistent over time

Suspension Map



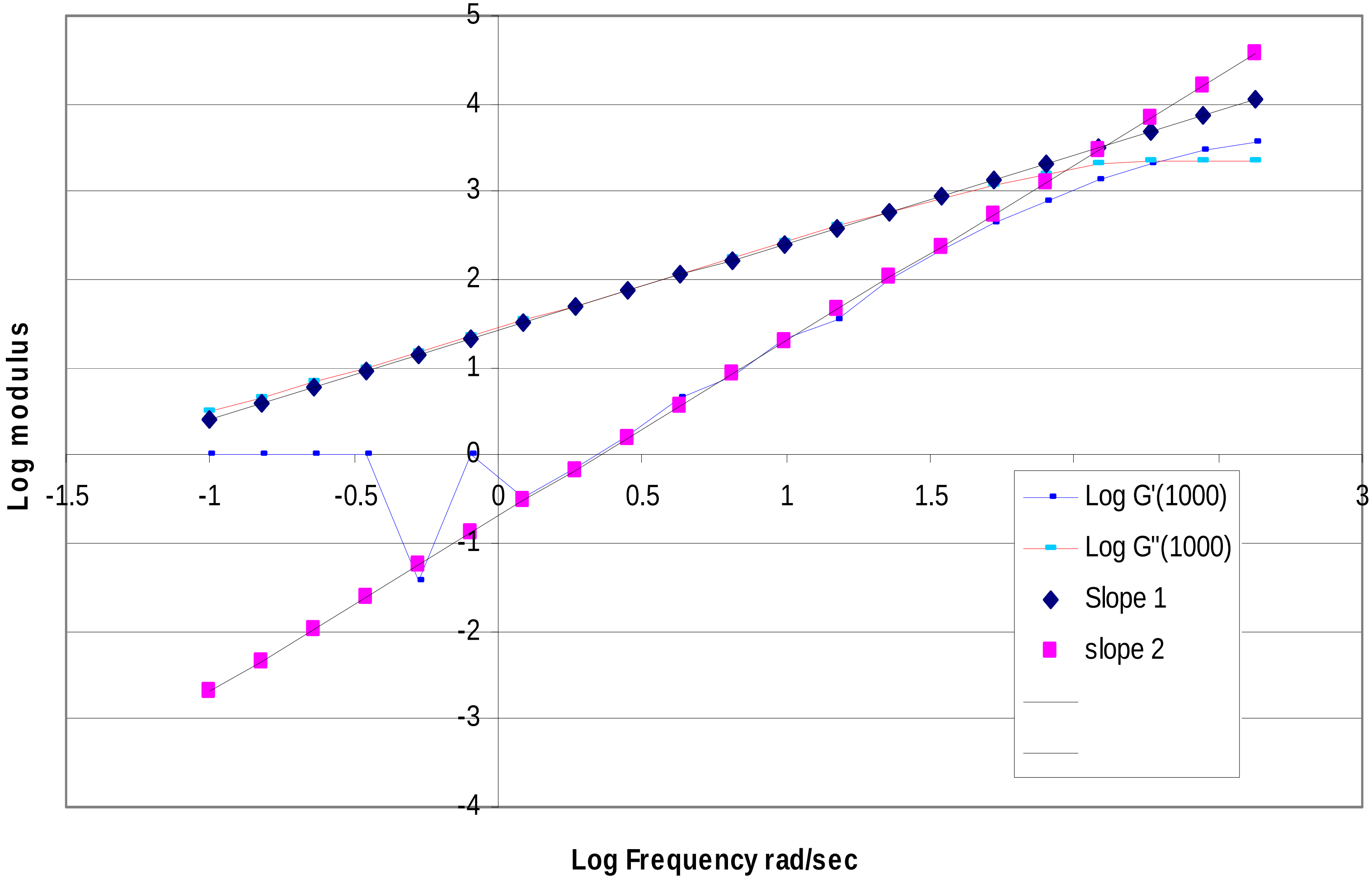
Casting Map


Casting map
10600000
10400000
10200000
10000000
9800000
9600000
9400000
9200000
9000000
Casting stiffness N/m
Log frequency
15
13
11
9
7
5
3
1
S9
S7
S5
S3
S1
Log amplitude
10400000-10600000
10200000-10400000
10000000-10200000
9800000-10000000
9600000-9800000
9400000-9600000
9200000-9400000
9000000-9200000

The complete instrument

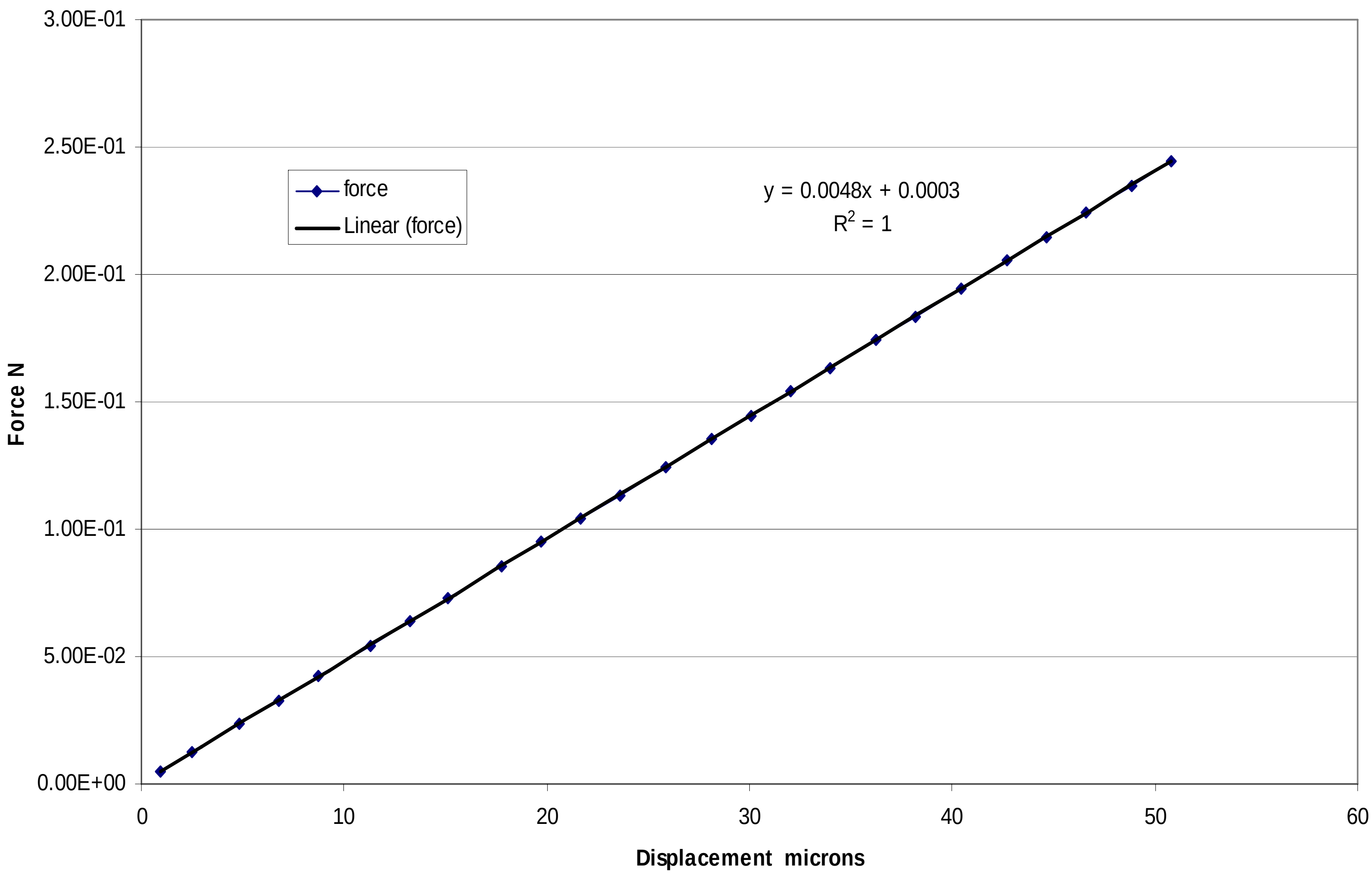
- Compact
- Self contained
- Robust
- Completely automated once loaded
- Low cost compared with Rotational instruments that can also measure G*, G’ & G”
- Can measure Penetration and softening point in **14 minutes!**

30,000 centistokes silicone



A pure elastic sample !

“Strain Sweep” for steel spring



The Gerschkoff relationship

- David Gerschkoff proposed a pragmatic relationship between Bitumen dynamic properties (G*, G' & G") in his paper:-

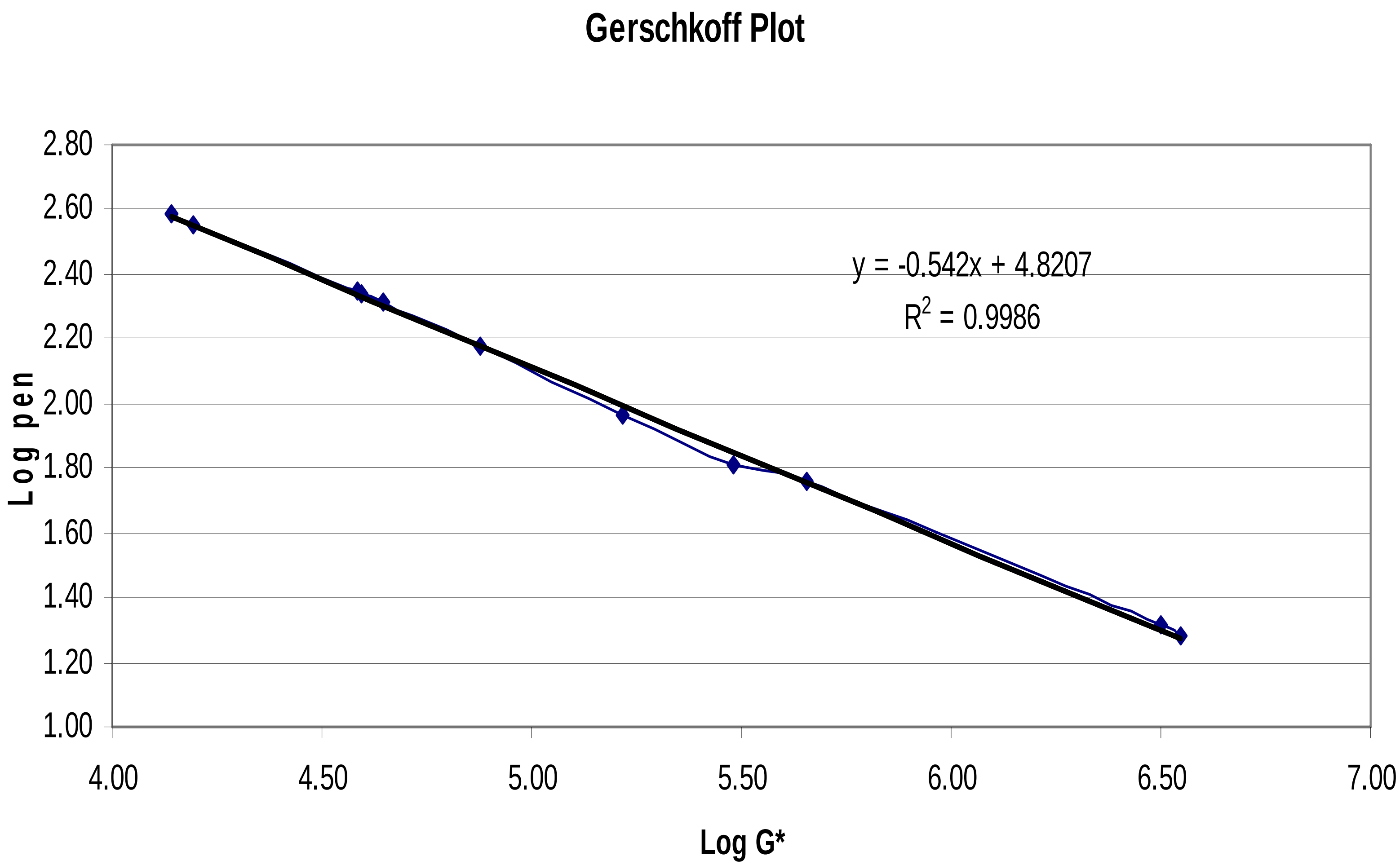
  "Polymer Modified Bitumens -performance in empirical and rheological test".

  Gerschkoff D First European Workshop on the rheology of Bituminous binders April 1995 paper 34

The relationship is of the form:-

$$\text{Log pen} = K.\log G^* + c$$

A typical plot using CP 50 data

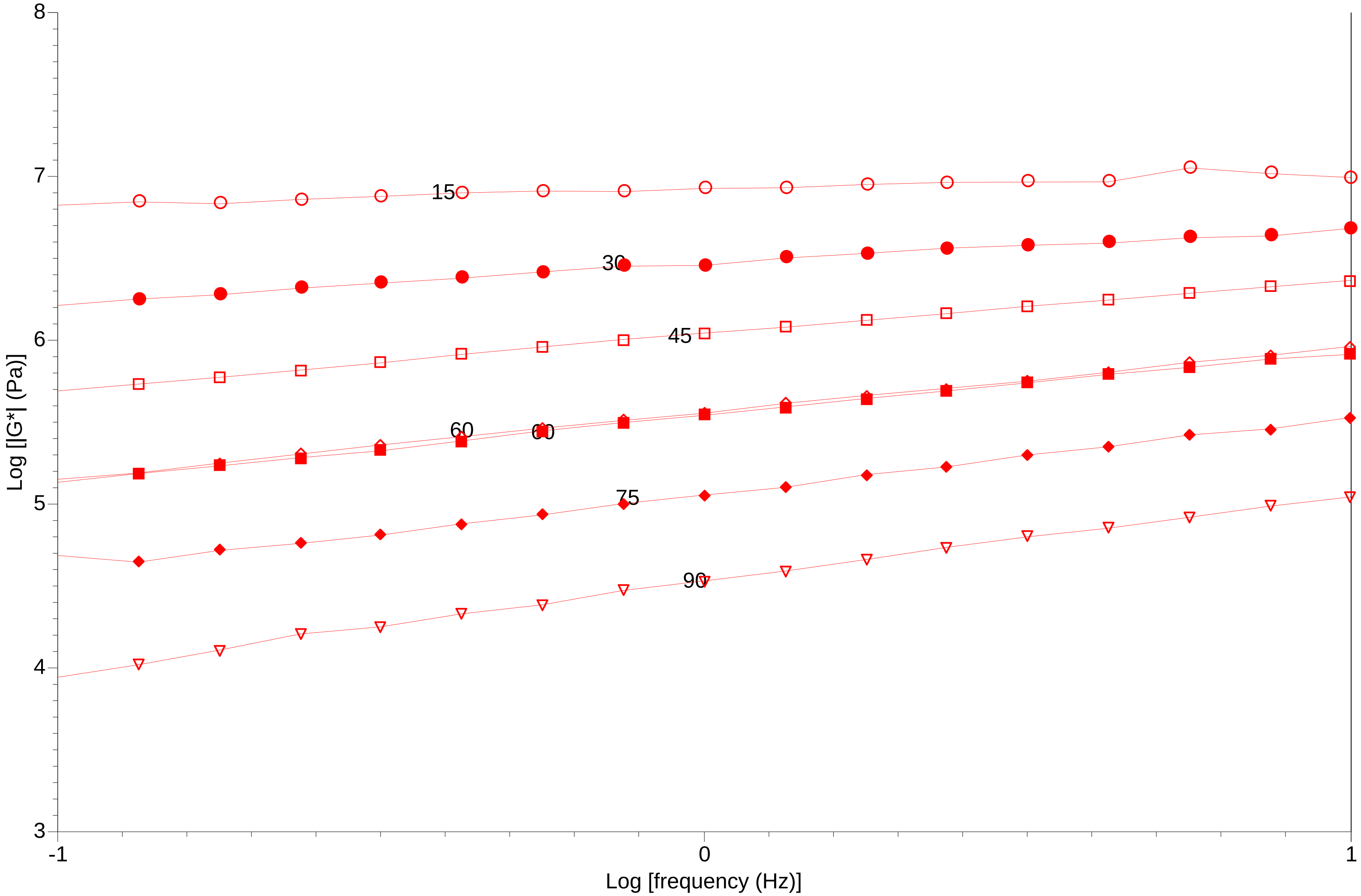


Quality Control Rheometer

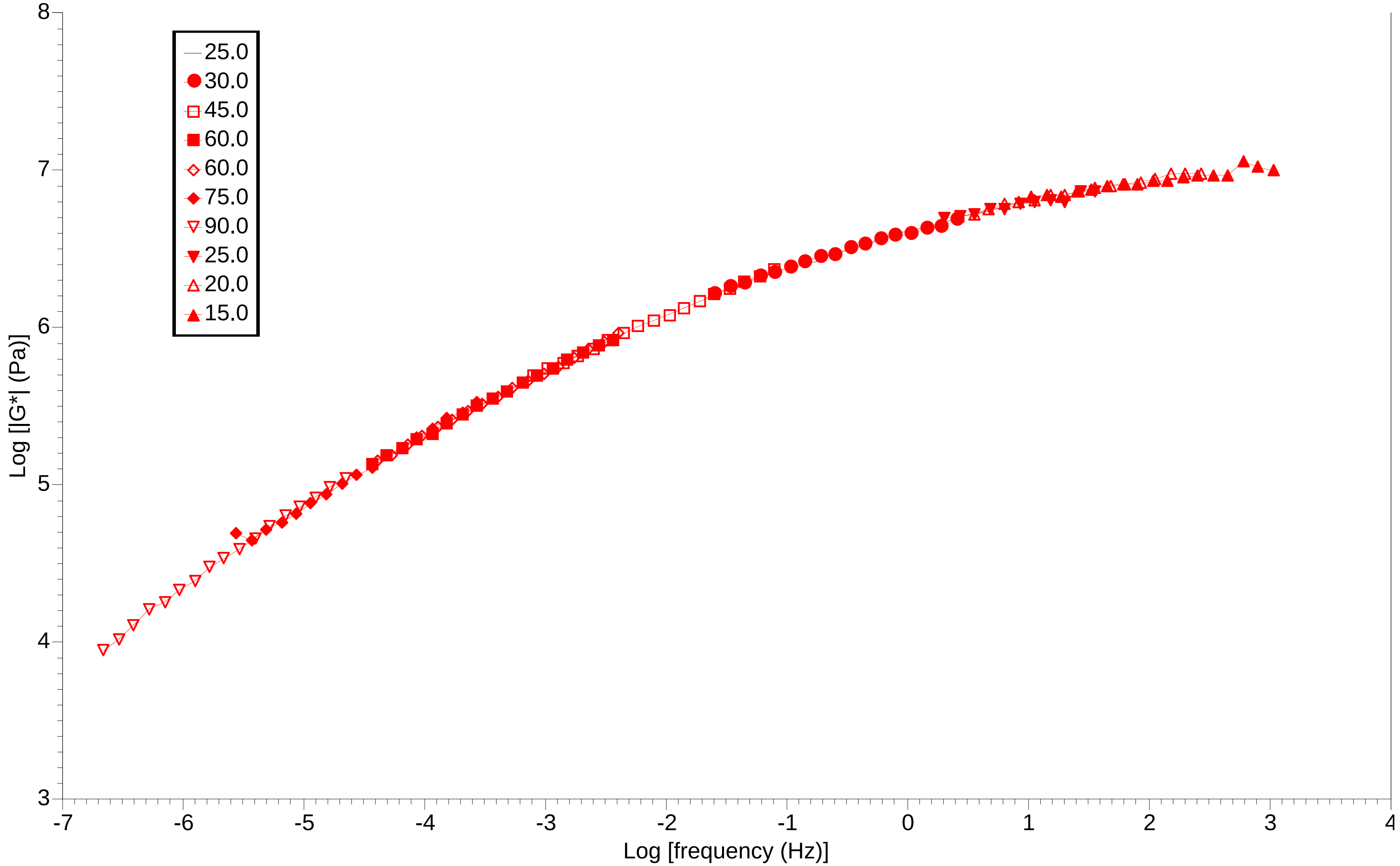
PC control capability

- Although the design of the CP50 enables it to be operated as a stand alone QC instrument,
- It can also be interfaced to a PC and operated in the wide range of “Laboratory” rheometer modes e.g.
- Frequency sweep
- Temperature sweep
- Stress or strain sweep
- And use any rheological variable:-
- G’,G” G* G*/sin delta etc

Isothermal data for ES roofing grade



TTS master curve for ES roofing grade



Summary

- as it is fundamentally a stress controlled dynamic rheometer (DSR), it can measure G*/sine δ and any conventional shear modulus, on straight or Polymer modified binder
- as the CP50 can also do simple squeeze flow it should be well suited to cumulative creep tests
- low running costs, does not need any services other than electrical power. It comes with everything you need.
- No delicate air bearing!
- Proven in use in a factory QC environment.
- The instrument can be operated “standalone “ with built in printer for result output.
- Ideal for monitoring binder processing in real time!

Summary (2)

- Can be operated as a conventional rheometer by PC using dedicated control and analysis software (included), but PC is not necessary for automatic operation in QC mode.
- Easy to calibrate from first principles (calibration kit included)
- Can measure properties of PMB's, and any other non standard products just as easily as straight bitumens
- Based on a sound mathematical theory
- The instrument has been shown to be capable of meeting the CEN repeatability standards for Bitumen