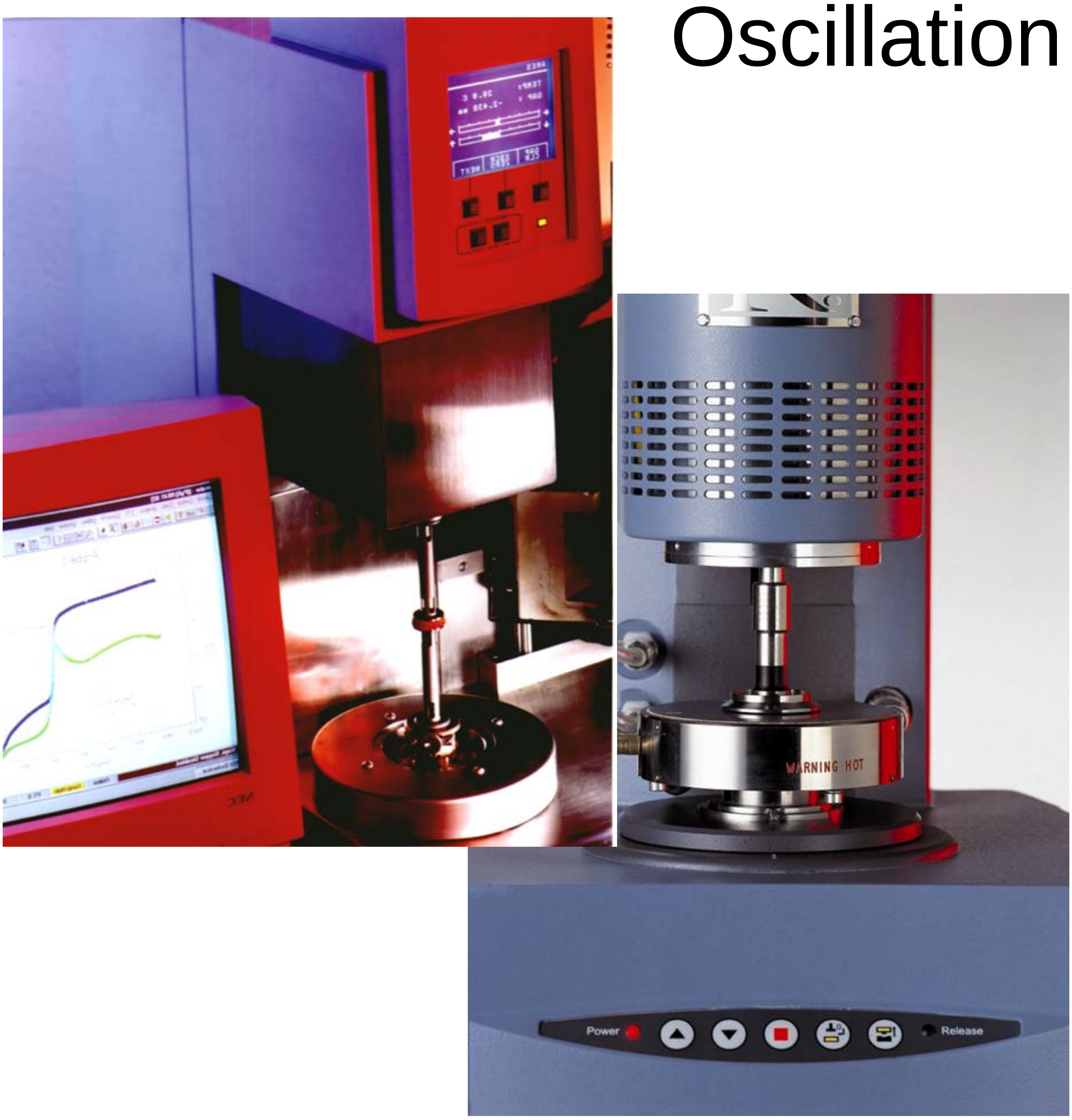
Understanding Instrument Compliance Correction in Oscillation



by A.J. Franck, TA Instruments

Instrument Radial Compliance Correction during Dynamic Mechanical Testing

Scope

Linear viscoelastic measurements are often taken for granted. Many users are not aware that systematic errors such as instrument compliance may significantly corrupt the test results, especially at high frequency when the sample becomes stiff in comparison to the instrument itself. Instrument compliance effects can be demonstrated, when running high modulus materials in different size plate geometries. Figure 1 shows the complex modulus of PIB measured at -20°C using 8mm and 25mm plates[1]. With increasing frequency, the complex modulus of the test run with 25mm plate geometry deviates significantly from the result obtained with the 8mm plate geometry. In the same graph, the measured sample deformation is plotted versus frequency. While the strain amplitude for the test performed with 8mm parallel plates is virtually constant and close to the 2% command value, does the actual sample strain for the tests performed with 25mm plates, decrease with frequency. This shows that for these tests, the deformation amplitude applied by the actuator is not fully transferred onto the sample. The smaller the sample strain in relation to the command strain, the higher the compliance effect of the instrument.

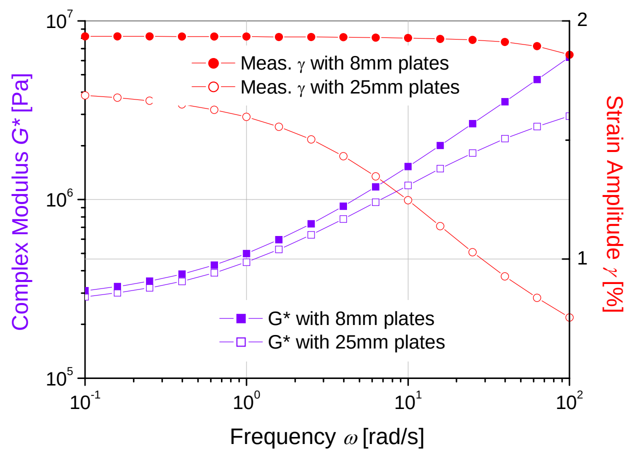


Figure 1: Poly-isobutylene measured in oscillation in parallel plate geometry at –20 °C

What is instrument compliance?

The true shear strain applied to the sample is always lower than the command (motor) strain because the test fixtures and the torque transducer are also deformed by the stress, required to shear the sample. If the sample/geometry configuration is stiff compared to the instrument, instrument compliance effects become significant and need to be corrected[2]. Figure 2 exhibits the typical configuration for a rheometer with separate torque transducer. Since the torque measurement system has a finite stiffness, the angular displacement $\varphi_T(t)$ is not zero. Similar does the test fixture itself deform slightly by the amount $\varphi_g(t)$, due to the applied torque. The final sample deformation is $\varphi_s(t)=\varphi_m(t)-(\varphi_T(t)+\varphi_g(t))$ with the test fixture compliance defined as $C_g=\varphi_g(t)/M_m(t)$ and the transducer compliance $C_T=\varphi_T(t)/M_m(t)$. Both compliance contributions become important when the sample is stiff compared to the instrument itself.

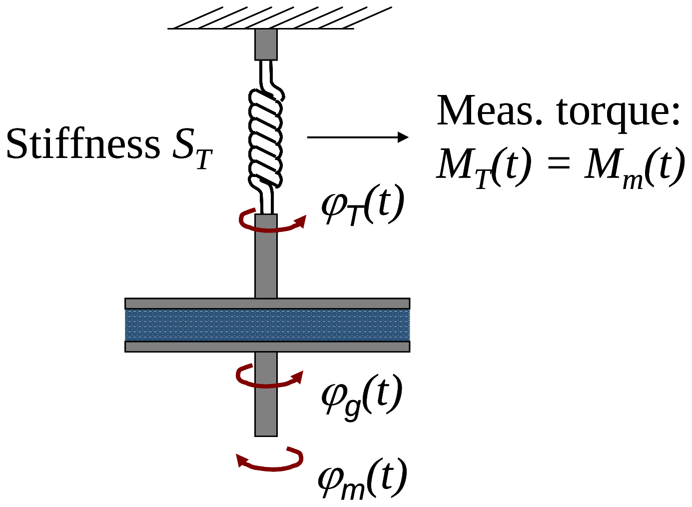


Figure 2: Schematical representation of a rheometer with separate torque transducer

$$G' = \frac{G'_a\left(1 - C_g\frac{G'_a}{k_g}\right) - G''_a\left(C_g\frac{G''_a}{k_g}\right)}{D} \qquad (1)$$

$$G'' = \frac{G''_a}{D}$$

$$tg\,\delta = \frac{tg\,\beta}{1 - C_g\left(\frac{G'_a}{k_g}\right)\left(1 + tg^2\beta\right)} \qquad (2)$$

$$D = \left(1 - C_g\frac{G'_a}{k_g}\right)^2 + \left(C_g\frac{G''_a}{k_g}\right)^2 \qquad (3)$$

$$\frac{|\varphi_s|}{|\varphi_m|} = \sqrt{D}$$

Table 1: Compliance correction for the dynamic moduli G' , G" and loss tanδ(3)

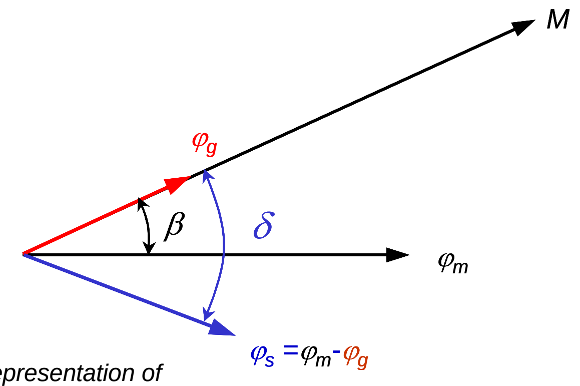


Figure 3: Vector representation of the tool compliance

Since the test fixtures are usually made of steel and deform very little under the imposed stress, they behave purely elastic during the measurement and C_g is a constant, real number. The FRT transducer in the ARES has a response time controlled by the servo loop and therefore the compliance changes with frequency and is a complex number.

Note that the FRT also has an axial compliance. The axial compliance does only marginally affect the modulus, but has a significant influence on the transient normal force measurement.

For both rheometers, AR and ARES compliance corrections are necessary, when stiff samples are measured. Since the AR has no transducer, only the test fixture compliance needs to be corrected.

Test fixture compliance in AR & ARES rheometers

For the AR rheometer and the ARES, assuming quasi-infinite stiffness for the transducer – thus transducer deflection is negligible, the angular displacement of the motor and the sample torque can be represented as vectors in the complex plane as shown in figure 3. The total motor angular displacement is φ_m, the test fixture displacement due to the tool compliance C_g is φ_g. The true angular displacement for the sample φ_s is the vector sum.

$$\varphi_s = \varphi_m - \varphi_g \qquad (1)$$

Since C_g is a real number, φ_g is in phase with the sample torque M and not the angular displacement φ_m.

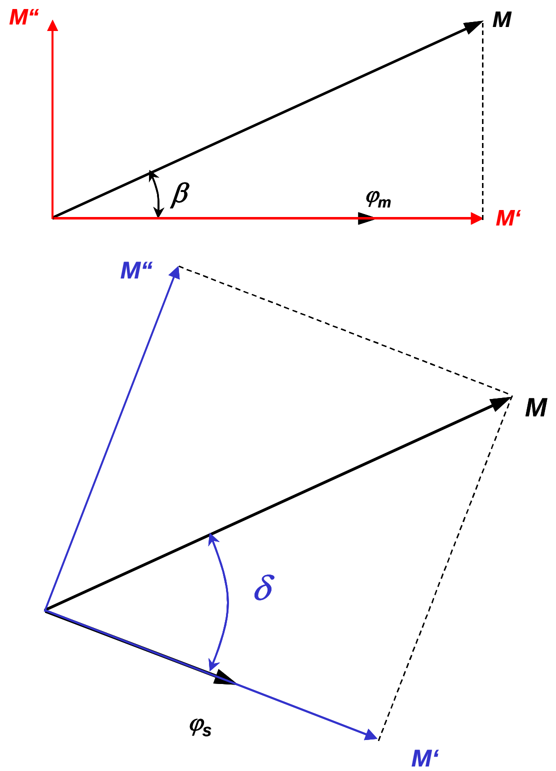


Figure 4: Decomposition of the torque a) in reference to the motor strain vector and b) in reference to the sample strain vector

Since

$$\varphi_g = C_g \cdot M$$

$$M = S^* \cdot \varphi_m = (G_a^* / k_g) \cdot \varphi_m$$

$$\varphi_g = C_g \cdot (G_a^* / k_g) \cdot \varphi_m \qquad (2)$$

*S** is the “apparent” stiffness and G_a* the “apparent” dynamic modulus; k_g the geometry constant. Figure 4 shows the real and imaginary torque contributions of the apparent modulus G_a*. The true dynamic modulus *G** is given by:

$$M = (G^* / k_g) \cdot \varphi_s \qquad (3)$$

When inserting eqs. (2) and (3) into (1), the true modulus can be written as:

$$G^* = \frac{G_a^*}{(1 - C_g (G_a^{*}/k_g)} \qquad (4)$$

The equations for the true storage and loss modulus are calculated from *G*=G’+iG”* and shown in table 1. The real and imaginary torque contribution of the true modulus *G** are shown in figure 4. It should be noted, that the tool compliance, also a real number does affect the modulus and the phase of the sample dynamic modulus *G**.

Transducer compliance for the ARES rheometer

The FRT “Force rebalance Transducer” uses a servo control to drive the upper plate back to its zero position, when a torque is applied[4]. The FRT transducer therefore can be considered to be quasi-infinite stiff. However during high frequency testing of stiff samples, the servo will not correct instantaneously[5], the compliance of the transducer increases and cannot be neglected anymore. For the sake of simplicity, the test fixture compliance is omitted in the following. The true sample deformation can be represented by the difference of the motor and transducer angular displacement (Figure 5):

$$\varphi_s = \varphi_m - \varphi_T \qquad (5)$$

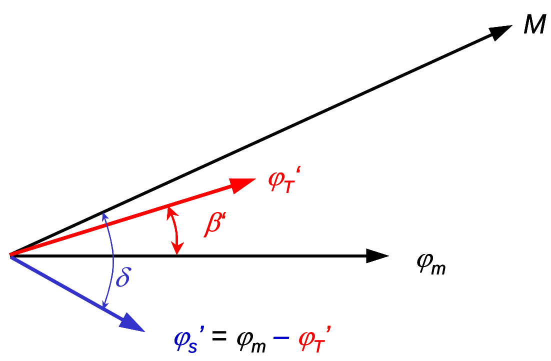


Figure 5: Vector representation of the FRT transducer compliance

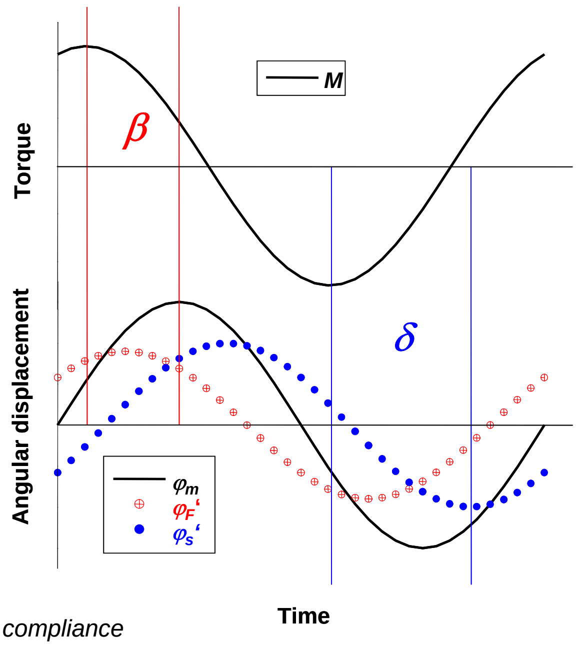


Figure 6: Real time compliance correction in the time domain

with $\varphi_T = C_T M$; φ_T is the transducer angular displacement. In contrast to the tool compliance C_g, is the transducer compliance C_T a complex number and not in phase with the torque anymore. Since the phase offset β' is not known and varies with frequency and sample stiffness, it is preferable to determine the sample deformation φ'_s directly in the time domain by subtracting the transducer $\varphi_T(t)$ angular displacements from the raw motor $\varphi_m(t)$ displacement (Figure 6). This is referred to as real time compliance correction. The advantage of this approach is that the instrument compliance is eliminated during the raw data sampling. Since it is virtually impossible to measure the deformation of the sample directly, the correction of the test fixture compliance has to be implemented as discussed in the previous section.

The compliance of the FRT transducer as a function of frequency is a characteristic of the transducer itself. Figure 7 shows the angular displacement φ_T of the 1KFRTN1 transducer as a function of frequency for 3 different test scenarios. The transducer displacement scales with the measured torque and bottoms out around 20 nrad. This is the angular position resolution of the servo encoder. The transducer compliance calculated from the transducer angular displacement and the torque, is only a function of the oscillation frequency and increases linearly with frequency and levels off around 100 rad/s. At 8 rad/s, the compliance of the 1KFRTN1 transducer is approximately 10 mrad/Nm.

Correction of test fixture compliance

The test fixture compliance correction factor can be defined as the ratio of the apparent sample stiffness G_a^*/k_g and the tool stiffness $1/C_g$. If accurately known, the tool compliance can be readily corrected with the equations in table 1. Usually the compliance correction is small, but can become significant when the product of sample stiffness and test fixture compliance is large.

In figure 8 the loss modulus is severely underestimated if the

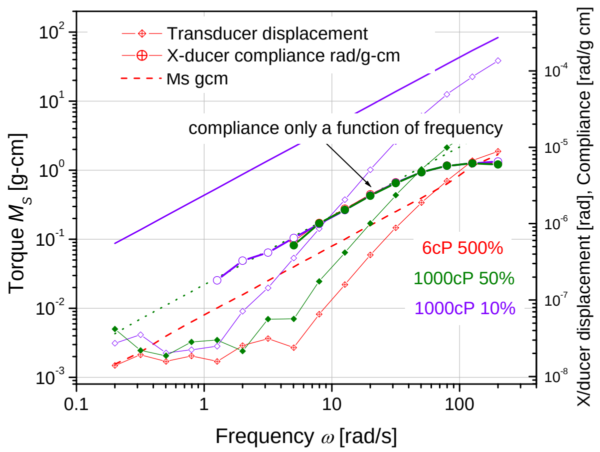


Figure 7: 1KFRTN1 transducer compliance as a function of the test frequency

compliance effects are not taken into account. The error for the storage modulus is relatively small since the nu-

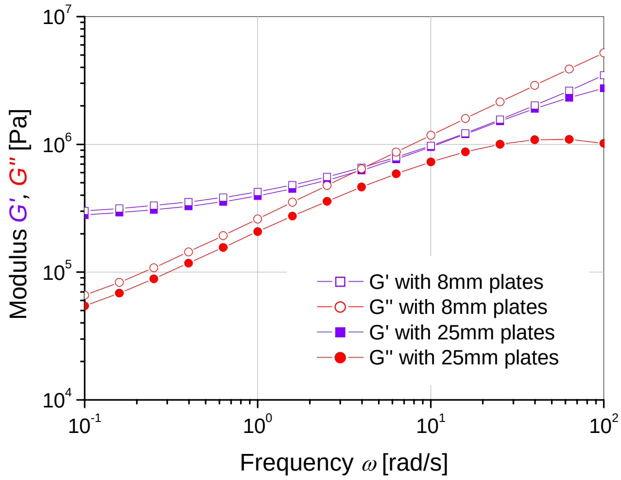


Figure 8: G' and G" of poly-isobutylene measured at –20°C before compliance correction

merator of eq.1 in table 1 is always smaller than G_a' and the denominator D is smaller than 1. Note, that the correction for the loss factor $tan\delta$ is non linear in respect to the apparent loss factor.

Since the sample stiffness depends on the dynamic modulus and the sample geometry, the geometry can be chosen, such as to eliminate

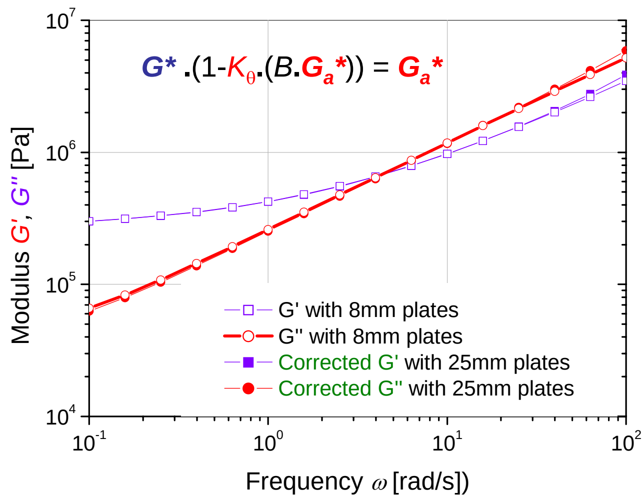


Figure 9: G' and G" of poly-isobutylen measured at –20°C after compliance correction

instrument compliance. If the sample modulus is below 0.4 MPa, the compliance correction for the 1KFRTN1 transducer, when using 25mm plates can be omitted. The plateau modulus G_{eN} for most polymers however is higher. With 8 mm plates, the modulus limit is about 100 times higher i.e. 40 MPa, because the sample stiffness is proportional to the 4th power of the plate radius:

$$S_s^* = G^* \frac{\pi R^4}{2h} \qquad (6)$$

As such, the data obtained with 8mm plates in figure 1, represent the true storage and loss modulus and can be used to calibrate the compliance of the 25mm tool fixture. With eqs.1&2 from table 1, the tool compliance C_g can easily be calculated using the apparent moduli obtained with the 25mm plates and the data obtained with the 8mm plates as true moduli. A tool compliance of 7 mrad/Nm is obtained for the 25mm plate test fixture. The corrected 25mm plate data match very well the 8mm plate data, shown in figure 9. The correction of the 8mm plate data shows no change – which implies the validation of the FRT transducer correction.

Conclusion

Neglecting instrument compliance can lead to significant errors in linear viscoelastic measurements in the plateau and transition region. Without tool compliance correction, the upper limit for the complex modulus is 0.4 MPa when using 25mm plates, 1 mm gap for the ARES. Tool compliance is a common problem for all rheometers, with or without separate torque transducer, when stiff samples are being tested. Figure 10 shows the ratio of measured and command strain (FRT compliance) and the ratio of corrected and measured strain (Tool compli-

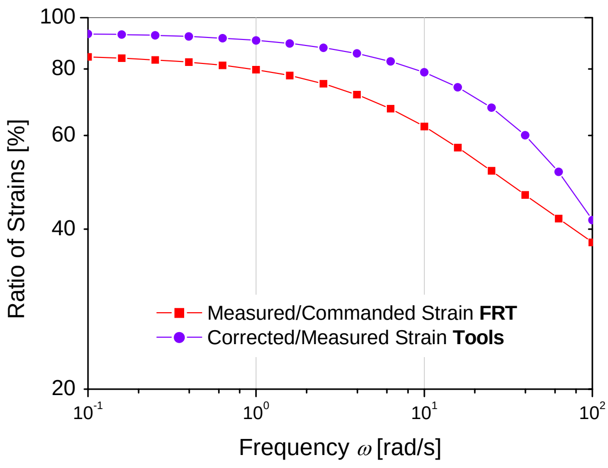


Figure 10: Ratio of measured/ command strain and corrected/ measured strain

ance) as a function of frequency for the PIB at -20°C using 25mm plates, 1mm gap. The ratio representing the FRT compliance is only slightly higher than the ratio for the tool compliance. This means for the ARES rheometer, that when compliance effects are detected and the measured strain is significant smaller than the command strain, the tool compliance correction needs to be active in order to obtain the correct dynamic moduli. The Orchestrator software allows the correction of test fixture compliance in addition to transducer compliance from software version 7.00 on .

References

1. Liu, C.Y.; Bailly, C.; Yao, M.L.; Garritano, R.; Franck, A. presented at the SOR in October 2006, Portland, Maine

2. Gottlieb, M.; Macosko, C.W.; Rheol. Acta **21**, 90 (1982)

3. Sternstein, S.S.; Adv. Chem. Series **203**, 123 (1983)

4. Franck, A.; presented at the conference "New techniques in Experimental Rheology", September 1985, Reading, UK

5. Mackay, M.E.; Halley, P.J.; J.Rheol.**35**, 1609 (1991)

Keywords: radial compliance, compliance correction, FRT transducer, plateau modulus, compliance