



Normal Force Measurements on the AR 1000-N Rheometer PART II PRACTICAL TIPS

Introduction:

When viscoelastic samples undergo a steady shear they can generate a force normal to the plane of the shear. This is commonly called the normal force (or normal stress if the correct geometry factors are considered). For more information on the theory of normal force please refer to RH062.

The normal force transducer on the TA Instruments AR 1000-N Rheometer uses the latest high sensitivity load cell technology. This provides a very robust, virtually deflectionless measurement system capable of detecting normal forces in the range 0.01N to 50N. The transducer measures the raw force but this can be expressed via the software as a normal stress once the appropriate geometry factors are used.

There are several ways in which this normal force transducer can be utilized, namely:

- loading under normal force control
- monitoring of normal forces generated during loading
- measurement of normal forces generated during shear

While it is simple to measure these forces on the AR 1000-N, an understanding of some of the practical considerations needs to be born in mind to prevent erroneous results.

Normal forces generated during loading:

When a sample is loaded and compressed between the upper and lower geometries there can be a significant normal force measured by the transducer. This force usually decays once the compression ceases.

Loading Under Normal Force Control

Tip 1:

Even delicately structured materials can exhibit normal forces during compression. Without the closure under normal force control option such samples should be loaded using the exponential close option of the Automatic Gap Setting system

using a small compression velocity (of the order of $100\mu\text{m s}^{-1}$.) With an AR 1000-N using the closure under normal force control option is advisable if structure preservation during loading is of importance. The user defined peak normal force will depend upon the sample under test and some experimentation may be required before optimum values can be set. The compression velocity should again be of the order of $100\mu\text{m s}^{-1}$. The total time for the geometry gap to be set will depend upon the peak normal force value entered, the rate at which the normal force increases during compression and the initial compression velocity set. The ideal situation is to have the rate of head descent match the rate at which the normal force can decay so that the head always continues down and never actually has to stop.

Tip 2:

Once the final gap set position is reached you should then wait for the normal force to either decay to zero or at least wait for equilibrium to be reached. However with some samples, such as polymer melts, this can take hours. In such cases you can either activate the '*wait for normal force*' option found under the '*pre-experiment*' step of any procedure. Simply toggle the option on and enter the required value. The experiment will not start until the normal force decays below this value but still consider the fact that the normal force may continue to decay after the experiment has begun. Depending upon the subsequent test you are running reproducibility of results could be compromised if you do not wait for the normal force to decay completely.

Tip 3:

When you load stiff samples and the level of normal force generated during loading is high, and potentially will take a long time to decay, it is advisable to set the initial gap to be slightly less than the final gap you wish to use. (of the order of $100\text{-}200\mu\text{m}$.) (Use the '*enter gap*' option on the manual screen.) Load the sample in the normal manner but then increase the gap to the required gap before starting the experiment. This should remove the retained normal force. This is only advisable on thick or stiff samples. Remember to ensure that the geometry details contain the correct dimensions before you start the experiment.

Monitoring Normal Force During Loading

Tip 4:

To actually record the normal forces generated during loading you need to set up a creep procedure as this is the only technique that will log data without shearing the sample. There are several ways to do this but a suggested method is outlined below. Remember you may need to adjust times depending upon the type of samples being measured and how quickly the normal force is changing.

1. Set the 'Automatic gap options' to load either under normal force control or with linear closure, and set the compression velocity to be $200\mu\text{ms}^{-1}$.
2. Toggle **ON** the 'override wait for gap' option found in the 'Pre-experiment step options' under the 'Experiment Options' menu on the main menu bar.
3. Set up geometry details, etc. in the normal manner and load the sample but do not close the gap yet.
4. Move the head down slowly, using the 'enter gap' button until it is just touching (or literally just above by a few microns) the sample.
5. Raise the head 1mm and read this gap position in the manual page.
6. Enter this gap as the compression distance in the 'Automatic gap options'.
7. Set up a creep procedure with the following parameters:
 - a) Remove *Pre-experiment step*
 - b) First *creep step* set up as:
Temperature: as appropriate
Applied stress: 0 Pa
Time: 5s (or as the distance you backed off in step 5 above divided by the compression velocity (i.e. $1000/200 = 5$ s in this case))
 - c) Second *creep step* set up as:
Temperature: as appropriate
Applied stress: 0 Pa
Time: however long you wish to monitor the normal force for.
8. Ensure that the geometry details has the correct final gap set position (Particularly important to check if you have been using the 'enter gap' button and have a parallel plate attached.)

9. Set the 'creep graph options' to time on the x axis and normal force on the y axis.
10. Reset the normal force in the manual page or ensure you have the 'reset zero force before run' option, found under 'Pre-experimental step options' under the 'Experiment options'.
10. Start the experiment in the normal way.
11. When the dialogue box, asking whether you want to set the correct gap, appears select **yes** and the experiment will begin immediately and the rheometer head will move down.
12. Once the experiment has finished, save and display the data in the usual way.

Measurement Of Normal Force During Shear

Tip 5:

Strictly speaking normal forces are generated as a result of shearing 'elastic' samples. Therefore measurement of normal forces is only relevant using the flow technique. The normal force can be related to the amount of elasticity in a sample and therefore a correlation (analogous to the Cox-Merz relationship) between normal force, measured under steady shear, and oscillatory elastic modulus can be made (1).

Polymer melts are typical samples that will exhibit normal force under shear but measuring such samples at shear rates above 10s^{-1} , with a rotational rheometer, tends to be difficult because of secondary flows causing the sample to be expelled from the gap.

Negative Normal Forces:

Negative normal forces are a true rheological phenomena and should always be considered. Literature exists explaining this phenomena and the situations and types of samples that might exhibit negative normal forces.(2)

References:

1. *Introduction to Rheology* (Barnes, Hutton and Walters) Chapter 6 - Page 111
2. *Introduction to Rheology* (Barnes, Hutton and Walters) Chapter 4 - Page 67

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