UNDERSTANDING NORMAL FORCE CONTROL ON AN AR-SERIES RHEOMETER

Fred Mazzeo, Ph.D.
TA Instruments, 109 Lukens Drive, New Castle, DE-19720, USA

ABSTRACT

Normal force control is an additional option for the AR500 & AR1000 and standard on the AR2000 rheometers. This feature is important for many applications, from ensuring that contact is maintained between the geometry and sample during compression control or keeping solids taut in tension control. This brief will describe how normal force control operates, explain the normal force control parameters, cite specific conditions to apply when testing materials requiring normal force control and discuss specific examples.

INTRODUCTION

WHEN SHOULD NORMAL FORCE BE USED?

Normal force control should be used when materials expand and/or contract during testing. If these changes in volume occur, the material will either cause a normal stress field within the material or shrink away from the testing geometry, either situation may cause error into the results. Generally, volume changes within a material often occur by a change in temperature, solvent/moisture loss or when the material gels. In order to convey the use of normal force completely, the remainder of this paper will associate the volume change of a material with a change in temperature, or simply, its coefficient of expansion. The most widely used test mode to control normal force is in the oscillation mode and the procedure type is a temperature ramp.

> NOTE: Normal Force Control is disabled when using cone & plate type geometry, since correct rheological results depend on the truncation gap to remain constant when using this type of geometry.

HOW DOES NORMAL FORCE CONTROL WORK?

The air bearing system contains a thrust air bearing, which virtually eliminates upward movement of the drive shaft, allowing any volumetric change of the sample to be sensed by the normal force transducer. The normal force transducer is located below the bottom testing assembly, i.e. below the fixed bottom stage (AR1000 & AR500) or below the

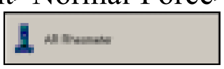
Smart Swap➢ connector (AR2000). When the material undergoes a change in volume, assuming the material is stiff enough to translate the resultant force, the normal force transducer will detect a change in normal force and the linear ball slide will increase or decrease the gap accordingly. This will result in the normal force conditions, set-up prior to the start of the procedure, to be maintained.

> NOTE: During the oscillation normal force control is disabled. When the oscillation has completed, all data is collected and the normal force will be adjusted accordingly

CONTROLLING NORMAL FORCE

Normal force can be activated on either a *Global/Macroscopic* scale, maintaining the specified conditions prior to, during and after the testing, or *Microscopic* scale, maintaining the specified conditions when the test is started.

Global/Macroscopic Control

In order to activate normal force control after loading the sample and prior to the start of the procedure, the user must either go to the main tool bar and choose Instrument>Normal Force>Control Normal Force or view the Instrument status page by choosing  and then choosing the Normal Force Control (NFC)  button.

> NOTE: This NFC button is only active when the Instrument Status page is viewed.

Microscopic Control

The normal force control can be automatically turned on at the instant the procedure is started by activating it in the conditioning step under the Normal force tab, shown in Figure 1. In order for microscopic control to be activated, the box labeled 'Use current instrument settings' must be unchecked, and check the box 'Active'.

Figure 1

Global/Macroscopic to Microscopic Control

Another method to control normal force is to activate both the global/macroscopic mode and the microscopic control with different control conditions. This will automatically change the normal force settings from global/macroscopic control to microscopic control.

Turning off Global/Macroscopic Control when the Test is started

The global/macroscopic control can be automatically turned off when the procedure is started by de-selecting both the 'Use current instrument setting' and the 'Active' boxes located within the conditioning step of the procedure.

Control Settings

Upon performing either global/macroscopic or microscopic control, the following choices will appear (Figure 2).

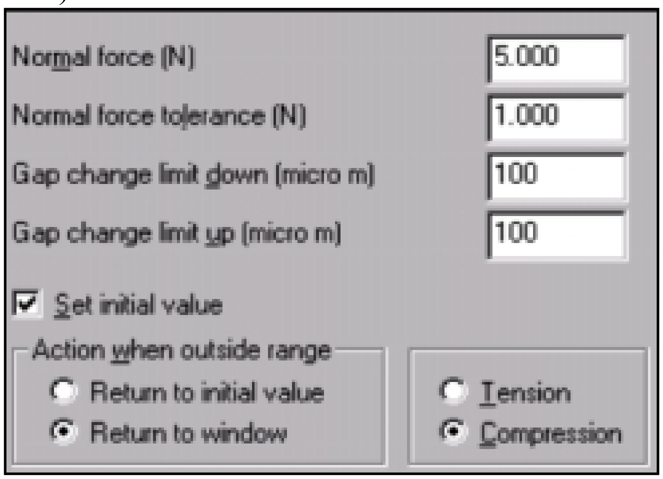


Figure 2

In order to activate the normal force control, the 'Active' box must be selected. The 'Normal force' box is where the user can set the value applied to the sample by the rheometer. The 'Normal force tolerance' is the allowed normal force range the force value can vary without any adjustment of the instrument. The 'Gap change limit down' and 'Gap change limit up' is the amount, in micrometers, that the head will move to maintain the set force value. Once either of these limits is exceeded, normal force control will be temporarily de-activated. To determine the allowable range that the instrument will travel to control normal force, one must add or subtract the up and down limits, respectively, from the current gap value when the normal force control is activated, in either the Global/Macroscopic or the Microscopic mode. If it is necessary to apply the 'Normal force' immediately, check the box 'Set initial value'. If unchecked, the normal force will be only controlled within the tolerance window.

The section labeled 'Action when outside the range' maintains the normal force within the tolerance range while the normal force control is activated. 'Return to initial value' maintains the value set in the 'Normal force' box at all times. The 'Return to window' selection allows the instrument to maintain a value of 'Normal force' plus or minus the 'Normal force tolerance' (this option is recommended to use). The last section shows an option to either control the normal force in 'Tension' or in 'Compression'. This setting will apply the force either in an upward or downward direction, respectively. A negative force will signify a tensile force and a positive force signifies a compressive force, when viewed within the software.

GENERAL NORMAL FORCE CONTROL CONDITIONS

Table I: Recommended Values for Torsion (Tension Mode)❷

Sample Type	Normal Force	Normal Force Tolerance	Gap change (Down)	Gap change (Up)
Soft thermoplastic above Tg @ RT–	2 N	1.75 N	2000 microns	5000 microns
Stiff thermoplastic below Tg @ RT–	2 N	1.75 N	2000 microns	5000 microns
Elastomer above Tg @ RT–	1 N	0.5 N	2000 microns	5000 microns
Stiff thermoset below Tg @ RT–	5 N	4 N	1000 microns	5000 microns

Table II: Recommended Values for Parallel Plate (Compression Mode)❷

Sample Type	Normal Force	Normal Force Tolerance	Gap change (Down)	Gap change (Up)
Isothermal test–	1 N	0.2 N	10 -15% gap	10 -15% gap
Cure (Starts as solid) –	5 N	6 N	10 -15% gap	10 -15% gap
Cure (Starts as liquid)*	0.5 N	2 N	10 -15% gap	10 -15% gap
PSA *	1 N	3 N	10 -15% gap	10 -15% gap

❷ NF CONDITIONS ARE SHOWN AS EXAMPLES AND MAY NEED ADJUSTMENT BASED ON THE FIRST SET OF RESULTS
– SET INITIAL VALUE
* DO NOT SET THE INITIAL VALUE

EXAMPLES

Measuring Samples in Tension

Figure 3 shows an oscillatory temperature ramp on ABS performed at 5°C/min, frequency = 1 Hz, Strain = 0.025% under a tensile normal force control of 3.0 N ⑥ 2.5N with up and down limits equal to 2000❍m. The thick dashed line indicates –3.0 N while the thin dashed line shows the upper tolerance limit equal to 0.5N. In this example, the normal force tolerances were exceeded during most of the test; this is explained in the endnote[☛]. In order to achieve stored normal force data within the programmed conditions, one may consider using a slower ramp rate and a higher frequency.

The test was set to begin at a temperature of –125°C using the global/macroscopic mode activated after loading at room temperature. The glassy region for the ABS is found between -125°C and ~ -100°C and the need for normal force control is shown. During the glass transition, the coefficient of thermal expansion increases, which results in a larger increase in the sample dimensions. This transition increases the amount of deflection on the normal force gauge and the normal force value increases at a much larger rate, much faster than instrument can control the normal force. This results in a faster gap change in this region, shown by the change in slope of gap vs. temperature. Once the transition is complete, the rheometer regains control of the normal force conditions. This large increase in the value of the normal force is again shown as the ABS sample begins to soften, shown by the dramatic decrease in G'.

During this transition, the ability for the lower modulus material to deflect the strain gauge is less, and the normal force conditions are easily achieved. Since the conditions of normal force are met, the gap will not increase, shown by the constant gap value over the temperature range between 120°C-170°C. Beyond 170°C the normal force begins to rise and normal force control is again activated, shown by the large increase in gap.

At approximately 185°C, the gap remains constant, while the normal force is increasing beyond the set conditions. This is because the normal force control is de-activated, as the gap change limit up value has now been exceeded. This sample was loaded at room temperature and normal force control was then activated. At this time, the current gap was approximately 45900❍m. This gap value when the normal force control was activated is taken as the point to add or subtract the up and down limits within the programmed conditions. The up and down limits were set to 2000❍m each, in this case. This explains why normal force control was de-activated at ~47900❍m.

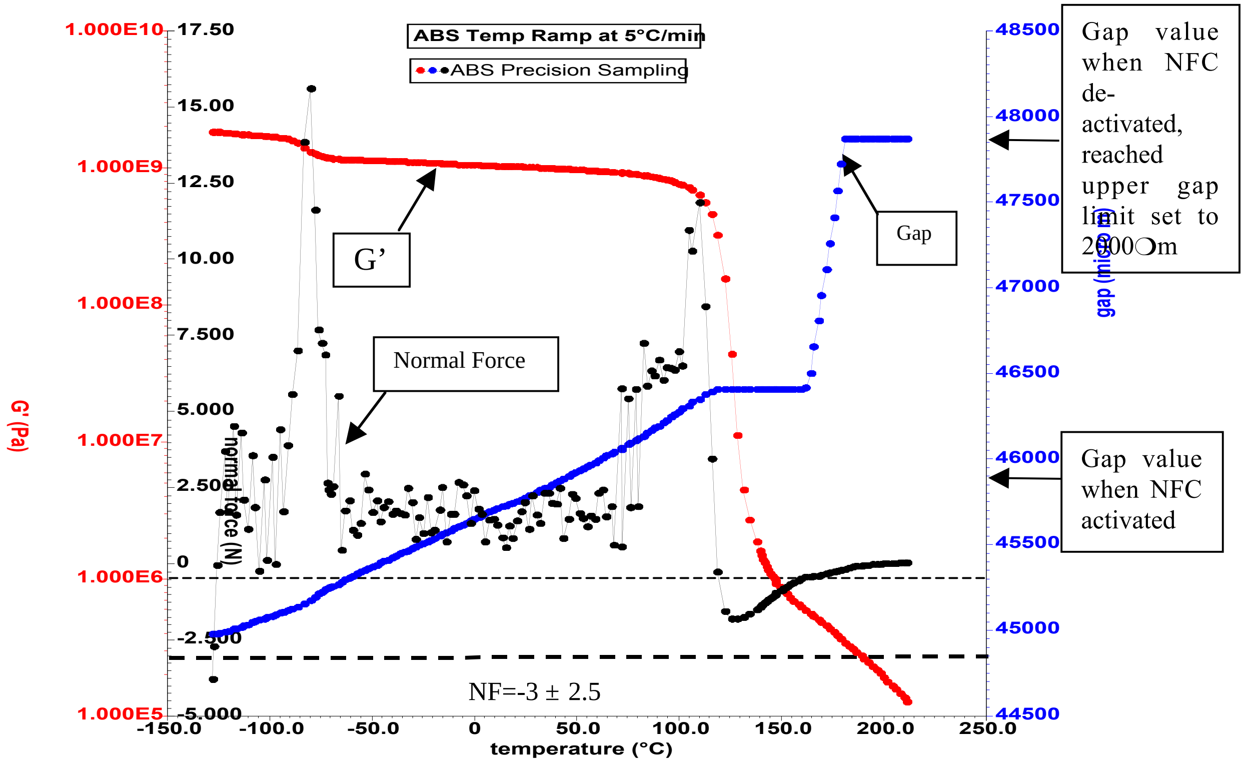


Figure 3

Measuring Samples in Compression

Samples that exhibit contraction throughout the experiment will move away from the measuring plate geometry or when using the torsion assembly. Therefore, it is necessary to use normal force control in the compression mode. The normal force conditions for the powder resin cure, shown in Figure 4, are 10.0 N ⑥ 1.0N with up and down limits equal to 5000❍m and 100❍m, respectively. This option to set the initial value was selected.

Once the test began, the normal force was adjusted to meet the upper tolerance range of 11.0 N of force. As the temperature was increased, the material began to soften at about 53°C. The gap was adjusted accordingly to maintain the normal force conditions up to ~65°C. (Please refer to the endnote, which explains why the normal force appears to be outside of normal force control tolerances☛.) At ~65°C, the normal force control is de-activated, since the gap change down limit in the NFC conditions was exceeded. This prevented the material from being pushed out of the gap as it softened. Between ~65°C and ~115°C, the gap change rate is due to the gap-compensation correction. This correction accounts for the expansion of the measuring devices as the temperature is changed.

At ~100°C, the powder resin begins to cure, shown by an increase in the elastic modulus, G'. During the cure, the material begins to expand shown by an increase in the normal force, even though gap-compensation is activated. However, since the normal force control is de-activated because the down limit was exceeded, the gap change continues to follow the gap-compensation correction factor. This continues until the normal force upper tolerance limit is exceeded, which re-activates the normal force control. This is shown to occur at ~115°C, where the gap is increased to maintain the NFC conditions.

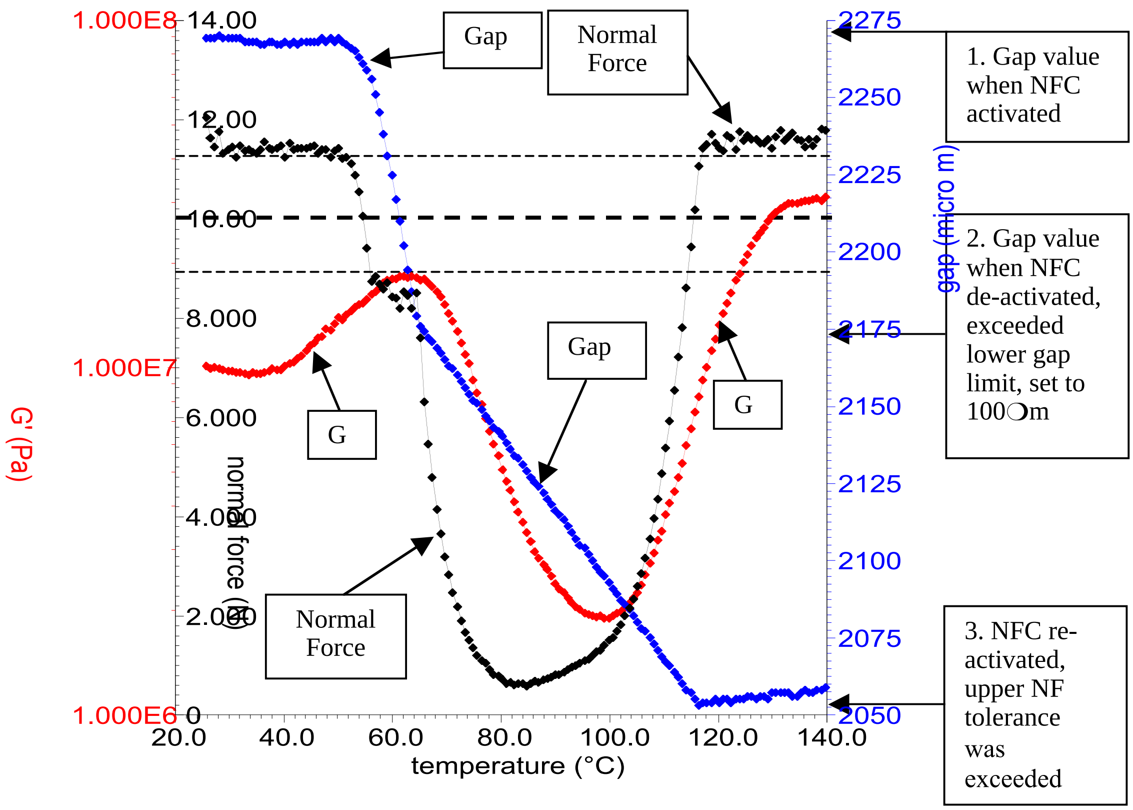


Figure 4

☛ **NOTE:** In any oscillation procedure, normal force can only be controlled when the test is not oscillating. During the time between oscillations, normal force control is activated and the normal force conditions are achieved. When oscillation is occurring, normal force is disabled and, because the material's dimensions are possibly changing, for example, when the material is being heated, the strain gauge will deflect and the normal force value may increase beyond the controlled conditions. When the oscillation completes, all data is collected, including the current normal force value. Since the normal force value is collected while the normal force control is de-activated, the normal force values collected may become larger than the normal force control conditions. To verify that normal force control is working properly, one must look at the normal force value displayed on the LCD screen located on the instrument tower between oscillations. By doing so, one can notice that the normal force is meeting the set criteria.