

# Issues with Normal Force Control During Curing

- Performing a curing test on a polymer system
- At room temperature, the normal force is held steady at  $\sim 9\text{N}$
- Once the temperature increases to  $149^\circ\text{C}$ , the normal force drops to  $-4\text{N}$
- On cooling back to room temperature, the normal force goes as low as  $-23\text{N}$
- Specified normal force in the procedures stays at  $+9\text{N}$
- Similar temperature ramp on sapphire did not have any issues
  - Points to a problem with the decreasing gap
- Moving profile should not limit gap

s979483 - Remote Desktop Connection

20200107 EFC550 curing - Anton Paar RheoCompass™

Home

Measuring Set

Presentation

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Test

Measurement

Interval

Insert

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Move left

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Set Variable

Add

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Raw Data

Get waveform

Get MultiDrive variables

Profiles and events: Default

Measuring method: Shear rheological measurement

Create command file

Start

Stop

Operation

Project - Data Browser

Search...

Deselect all

Apply auto-display

This project contains 6 tests in total:

20200108 ECF550 curing 7-10-90

Pre-Cure Holds 1

Curing 1

20200114 ECF550 curing 20-20-90

Pre-Cure Holds 1

Curing 1

20200116 ECF550 curing 120-120-90

Pre-Cure Holds 1

Curing 1

20200108 ECF550 curing 8-10-90

20200128 ECF550 curing 40-40-90

Pre-Cure Holds 1

Curing 1

20200204 Sapphire

Measurement 1

Measurement 2

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My Apps

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RheoFinder

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Test definition 1

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Diagram

Measurement

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Measurement

Curing

End

Shear rheological measurement

|                | Points | 7        | 33 Points | 8             | 13 Points | 9     | 240 Points<br>5 s | 10       | 33 Points | 11            | 13 Points |
|----------------|--------|----------|-----------|---------------|-----------|-------|-------------------|----------|-----------|---------------|-----------|
| $\gamma$       |        |          |           |               |           |       |                   |          |           |               |           |
| $\gamma$       | %      | 0.005    | %         | 0.0005...0.01 | %         | 0.005 | %                 | 0.005    | %         | 0.0005...0.01 | %         |
| f              | Hz     | 0.1...30 | Hz        | 1             | Hz        | 1     | Hz                | 0.1...30 | Hz        | 1             | Hz        |
| T              | °C     | 25       | °C        | 25            | °C        | 25    | °C                | 25       | °C        | 25            | °C        |
| F <sub>N</sub> | N      | 8.97     | N         | 8.97          | N         | 8.97  | N                 | 8.97     | N         | 8.97          | N         |

Test

Diagram: gap,N

Diagram: Eta\*

Table

View 1

Diagram: G', G''

QuickSapphire

Details

Name, Info

Picture, Title, Buttons

Set Values

Data Generation

Event Control

Options

Harmonics

Lab User 898-1668

Anne Rheology

10:18 AM 2/5/2020

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20200107 EFC550 curing - Anton Paar RheoCompass™

Control Panel - CTD1000 Parallel Plate Configuration

Dashboard - CTD1000 Parallel Plat...

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20200204 Sapphire

Measurement 1

Measurement 2

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Test definition 1

Start

View

Diagram

Measurement

Pre-Cure Holds

Measurement

Curing

End

Shear rheological measurement

|                |                   |                |                            |                |                   |                |                          |                |                           |                |                   |
|----------------|-------------------|----------------|----------------------------|----------------|-------------------|----------------|--------------------------|----------------|---------------------------|----------------|-------------------|
| 1              | 60 Points<br>5 s  | 2              | 13 Points                  | 3              | 120 Points<br>5 s | 4              | 33 Points                | 5              | 13 Points                 | 6              | 120 Points<br>5 s |
| $\gamma$       | 0.005 %<br>f 1 Hz | $\gamma$       | 0.0002...0.005 %<br>f 1 Hz | $\gamma$       | 0.005 %<br>f 1 Hz | $\gamma$       | 0.005 %<br>f 0.1...30 Hz | $\gamma$       | 0.0005...0.01 %<br>f 1 Hz | $\gamma$       | 0.005 %<br>f 1 Hz |
| T              | 149 °C            | T              | 149 °C                     | T              | 149 °C            | T              | 149 °C                   | T              | 149 °C                    | T              | 149 °C            |
| F <sub>N</sub> | 8.97 N            | F <sub>N</sub> | 8.97 N                     | F <sub>N</sub> | 8.97 N            | F <sub>N</sub> | 8.97 N                   | F <sub>N</sub> | 8.97 N                    | F <sub>N</sub> | 8.97 N            |

Test

Diagram: gap,N

Diagram: Eta\*

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View 1

Diagram: G', G''

QuickSapphire

Details (multi-selection)

Name, Info

Picture, Title, Buttons

Set Values

Data Generation

Event Control

Options

Harmonics

Lab User 898-1668

Anne Rheology

10:17 AM  
2/5/2020

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Home

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Presentation

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Dashboard - CTD1000 Parallel Plate Configuration

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20200204 Sapphire

Measurement 1

Measurement 2

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My Apps

Project

RheoFinder

Setup

Test

Test definition 1

Start

View

Diagram

Measurement

Pre-Cure Holds

Measurement

Curing

End

Shear rheological measurement

| Points   | 11                                   | 13 Points<br>120 s                                           | 12                                                   | 300 Points<br>5 s                                            | 13                                                   | 13 Points                                                    | 14                                                   | 21 Points                                            | 15                                                   | 360 Points<br>5 s                                    |
|----------|--------------------------------------|--------------------------------------------------------------|------------------------------------------------------|--------------------------------------------------------------|------------------------------------------------------|--------------------------------------------------------------|------------------------------------------------------|------------------------------------------------------|------------------------------------------------------|------------------------------------------------------|
| $\gamma$ | <div><div>%</div><div>Hz</div></div> | <div><div>0.0005...0.01</div><div>%</div><div>Hz</div></div> | <div><div>0.003</div><div>%</div><div>Hz</div></div> | <div><div>0.0005...0.01</div><div>%</div><div>Hz</div></div> | <div><div>0.003</div><div>%</div><div>Hz</div></div> | <div><div>0.0005...0.01</div><div>%</div><div>Hz</div></div> | <div><div>0.003</div><div>%</div><div>Hz</div></div> | <div><div>0.003</div><div>%</div><div>Hz</div></div> | <div><div>0.003</div><div>%</div><div>Hz</div></div> | <div><div>0.003</div><div>%</div><div>Hz</div></div> |
| $f$      | <div><div>1</div><div>Hz</div></div> | <div><div>1</div><div>Hz</div></div>                         | <div><div>1</div><div>Hz</div></div>                 | <div><div>1</div><div>Hz</div></div>                         | <div><div>1</div><div>Hz</div></div>                 | <div><div>1</div><div>Hz</div></div>                         | <div><div>0.1...10</div><div>Hz</div></div>          | <div><div>1</div><div>Hz</div></div>                 | <div><div>1</div><div>Hz</div></div>                 | <div><div>1</div><div>Hz</div></div>                 |
| $T$      | <div><div>°C</div></div>             | <div><div>149</div><div>°C</div></div>                       | <div><div>25</div><div>°C</div></div>                | <div><div>25</div><div>°C</div></div>                        | <div><div>25</div><div>°C</div></div>                | <div><div>25</div><div>°C</div></div>                        | <div><div>25</div><div>°C</div></div>                | <div><div>25</div><div>°C</div></div>                | <div><div>25</div><div>°C</div></div>                | <div><div>25</div><div>°C</div></div>                |
| $F_N$    | <div><div>N</div></div>              | <div><div>8.97</div><div>N</div></div>                       | <div><div>8.97</div><div>N</div></div>               | <div><div>8.97</div><div>N</div></div>                       | <div><div>8.97</div><div>N</div></div>               | <div><div>8.97</div><div>N</div></div>                       | <div><div>8.97</div><div>N</div></div>               | <div><div>8.97</div><div>N</div></div>               | <div><div>8.97</div><div>N</div></div>               | <div><div>8.97</div><div>N</div></div>               |

Test

Diagram: gap,N

Diagram: Eta\*

Table

View 1

Diagram: G', G''

QuickSapphire

Details (multi-selection)

Name, Info

Picture, Title, Buttons

Set Values

Data Generation

Event Control

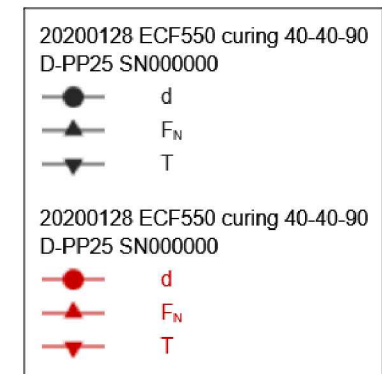
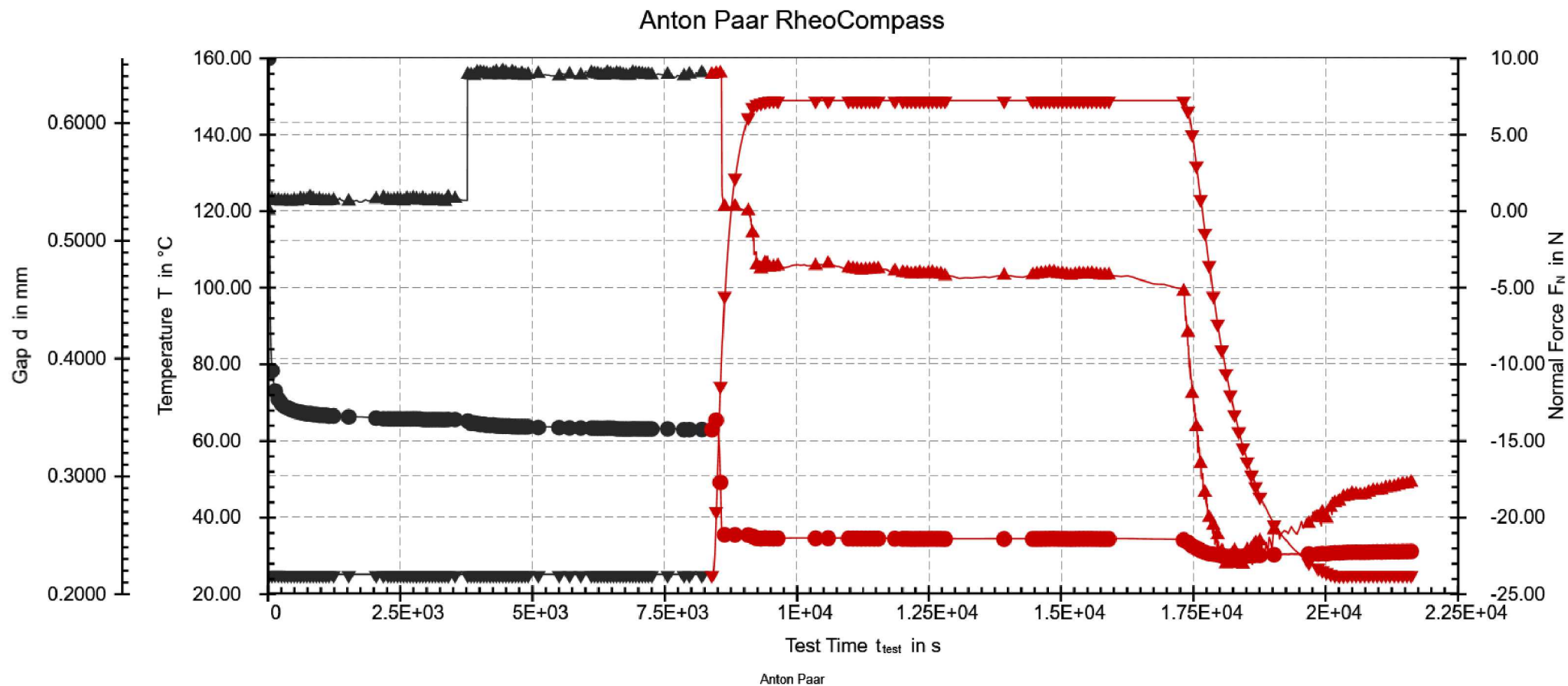
Options

Harmonics

Lab User 898-1668

Anne Rheology

10:21 AM 2/5/2020





A 1mm sapphire disk run through the same thermal cycle easily maintains the correct normal force

s979483 - Remote Desktop Connection

20200107 EFC550 curing - Anton Paar RheoCompass™

Home Measuring Set Presentation Window Test Measurement

Interval Insert Append Move left Move right Add Delete Set Variable

Get waveform Get MultiDrive variables Raw Data

Profiles and events: Default Measuring method: Shear rheological measurement

Create command file Measuring Definition Start Stop Operation

Project - Data Browser

Search... Deselect all Apply auto-display

This project contains 6 tests in total:

- 20200108 ECF550 curing 7-10-90
  - Pre-Cure Holds 1
  - Curing 1
- 20200114 ECF550 curing 20-20-90
  - Pre-Cure Holds 1
  - Curing 1
- 20200116 ECF550 curing 120-120-90
  - Pre-Cure Holds 1
  - Curing 1
- 20200108 ECF550 curing 8-10-90
- 20200128 ECF550 curing 40-40-90
  - Pre-Cure Holds 1
  - Curing 1
- 20200204 Sapphire
  - Measurement 1
  - Measurement 2

References Auto-Display

My Apps

Project

RheoFinder

Setup

QuickSapphire

Test Definition 1

Start

Measurement

Measurement

End

Shear rheological measurement

|                | 1                    | 2                  | 3                  | 4                  |
|----------------|----------------------|--------------------|--------------------|--------------------|
| Points         | 60 Points            | 60 Points          | 240 Points         | 240 Points         |
| Time           | 5 s                  | 5 s                | 5 s                | 5 s                |
| T              | T 23 °C              | T 23 °C            | T 149 °C           | T 25 °C            |
| F <sub>N</sub> | F <sub>N</sub> 0.9 N | F <sub>N</sub> 9 N | F <sub>N</sub> 9 N | F <sub>N</sub> 9 N |

Test Diagram: gap,N Diagram: Eta\* Table View 1 Diagram: G', G\* QuickSapphire

Details (multi-selection)

Data points

Number: 240

Duration: Constant

Point: 5 s

Interval: 1200 s

Set value

Variable: Profile: Constant Value:

Additional settings

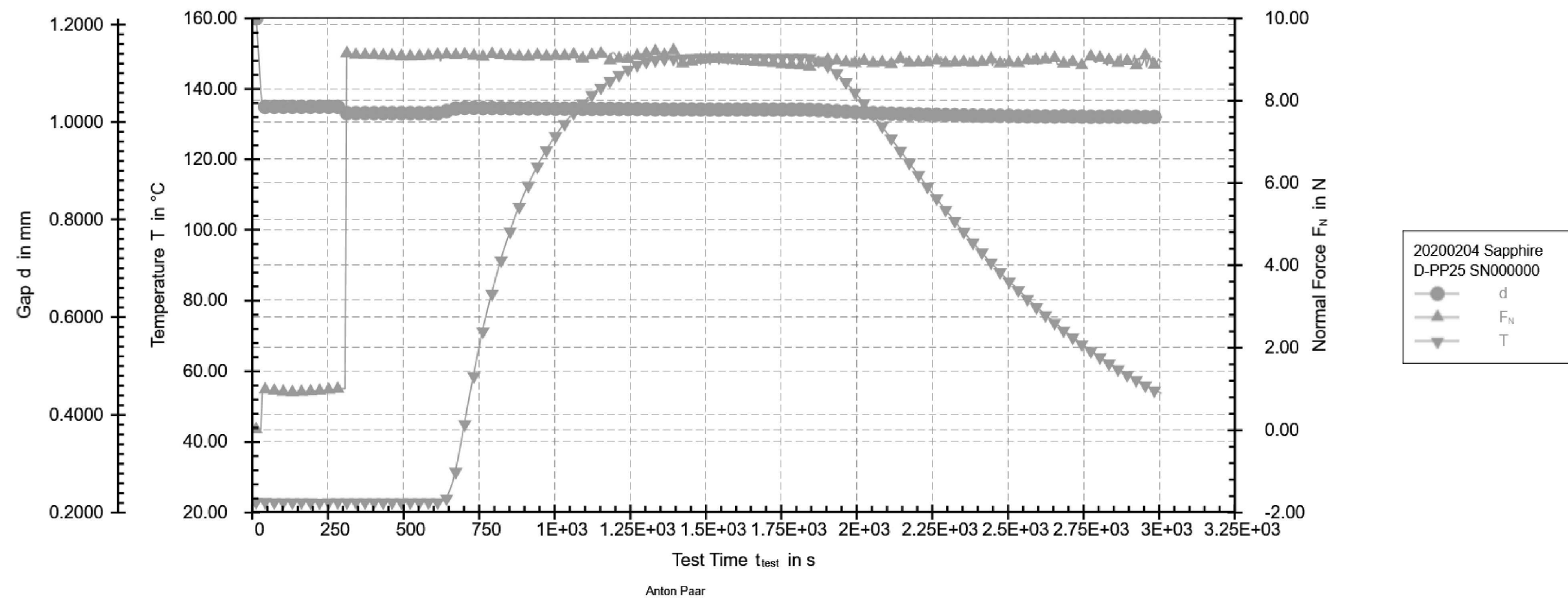
Name, Info Picture, Title, Buttons Set Values Data Generation Event Control Options Harmonics

Lab User 898-1668

Anne Rheology

10:16 AM 2/5/2020

Anton Paar RheoCompass



Nothing in the past like moving profile should have issues at these gaps

Home

Measuring Set

Presentation

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New

Duplicate

Refresh

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Export certificate

New

Delete

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Remove user

Change or reset password

Add role membership

Remove role membership

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User

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Calibrations

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Measuring systems

User administration

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20200107 EFC550 curing - Anton Paar RheoCompass™

Control Panel - CTD1000 Parallel Plate Configuration

Dashboard - CTD1000 Parallel Plate...

Moving Profiles

Search...

80 items (including hidden items)

| Moving profile name                  | Origin of creation    | Is the default moving profile | Last modified         | Last modified b |
|--------------------------------------|-----------------------|-------------------------------|-----------------------|-----------------|
| Application   Pressure cell (PR)     | Created by Anton Paar | no                            | 4/10/2019 11:15:25 AM | anton.paar      |
| Application   Interfacial (IRS)      | Created by Anton Paar | no                            | 4/10/2019 11:15:09 AM | anton.paar      |
| Application   Alloys (CTD 1000)      | Created by Anton Paar | no                            | 4/10/2019 11:15:05 AM | anton.paar      |
| Application   Asphalt                | Created by Anton Paar | yes                           | 4/10/2019 11:14:59 AM | anton.paar      |
| Service   radial compliance deter... | Created by Anton Paar | no                            | 4/10/2019 11:11:50 AM | anton.paar      |
| Viscoelastic                         | Created by Anton Paar | yes                           | 1/11/2019 9:31:09 AM  | anton.paar      |
| Viscoelastic   set start angle       | Created by Anton Paar | no                            | 1/11/2019 8:52:31 AM  | anton.paar      |
| Viscoelastic   lift & rotate         | Created by Anton Paar | no                            | 1/11/2019 8:52:25 AM  | anton.paar      |
| AMG Solid bar   SRF   touch con...   | Unknown origin        | no                            | 9/6/2018 12:18:48 PM  | 898-1668        |
| Tension   SRF, upper   rigid sam...  | Created by Anton Paar | no                            | 6/25/2018 3:09:39 PM  | anton.paar      |
| Reactive   UV curing   set gap &...  | Created by Anton Paar | no                            | 6/25/2018 3:09:39 PM  | anton.paar      |
| Paste like   lift & rotate           | Created by Anton Paar | no                            | 6/25/2018 3:09:39 PM  | anton.paar      |
| Paste like                           | Created by Anton Paar | no                            | 6/25/2018 3:09:39 PM  | anton.paar      |
| Melts   set start angle              | Created by Anton Paar | no                            | 6/25/2018 3:09:39 PM  | anton.paar      |
| Melts   lift & rotate                | Created by Anton Paar | no                            | 6/25/2018 3:09:38 PM  | anton.paar      |
| Melts                                | Created by Anton Paar | no                            | 6/25/2018 3:09:38 PM  | anton.paar      |
| Low-viscosity   set start angle      | Created by Anton Paar | no                            | 6/25/2018 3:09:38 PM  | anton.paar      |
| Low-viscosity   lift & rotate        | Created by Anton Paar | no                            | 6/25/2018 3:09:38 PM  | anton.paar      |
| Low-viscosity                        | Created by Anton Paar | no                            | 6/25/2018 3:09:38 PM  | anton.paar      |
| AMG Solid bar   SRF   gap contr...   | Unknown origin        | no                            | 2/13/2018 9:58:49 AM  | 898-1668        |

Moving Profiles Details - Paste like for ROT+F+LIFT

Lift drive settings

Minimum position: 0.001 mm

Maximum position: 10 mm

Force control hysteresis: 0.25 N

Timeout gap control: 0 s

| Position [mm] | Velocity [µm/s] | Force [N] |
|---------------|-----------------|-----------|
| 0             | 10              | 25        |
| 5             | 10              | 25        |
| 125           | 10              | 1         |

Use default values

Name, Info

Lift drive, action

Rot drive, action

During Measurement

Detect position

Lab User 898-1668

Anne Rheology

10:39 AM 2/5/2020