APPLICATION**NOTE**

Paints – Rotational Rheometry

How Does the Drying of Paints Influence their Rheological Properties? The Immobilization Cell

Claire Strasser and Senol Gezgin, Applications Laboratory

Introduction

The Immobilization Cell is used with the Kinexus rotational rheometer to characterize the rheological properties of a paint or coating while it is dried on a substrate.

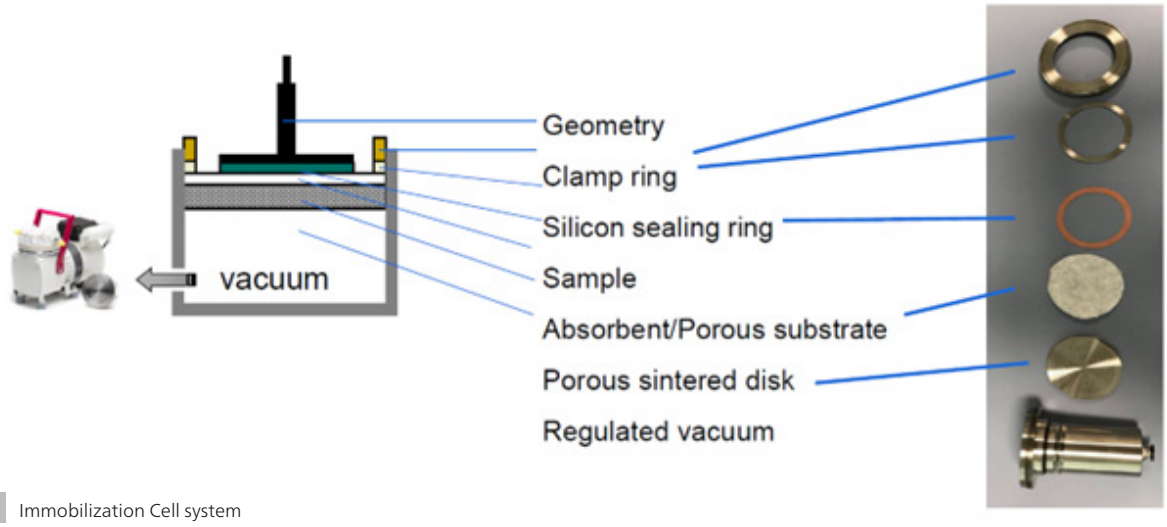
Such a test is useful to determine the effects of

- Solids content
- Porosity of the coating substrate
- Thickness of the coating substrate
- Water retention additives
- Applied pressure drop

on the rheological properties of a material.

Figure 1 shows the Immobilization Cell system.

A measurement is made by placing the sample on a substrate positioned on a porous sintered disk and a vacuum applied below to initiate the dewatering process. An upper geometry (cone or plate) of up to 45 mm in diameter can be used, and real-time measurements made in rotation (viscometry) to characterize the sample's immobilization kinetics, or oscillation to detect changes in the viscoelastic properties.



1 Immobilization Cell system

NGB · Application Note 304 · EN · 0124 · Technical specifications are subject to change.

NETZSCH-Gerätebau GmbH
Wittelsbacherstraße 42 · 95100 Selb · Germany
Phone: +49 9287/881-0 · Fax: +49 9287/881505
at@netzsch.com · www.netzsch.com

NETZSCH

Measurement Parameters

In the following, the rheological properties of a wall paint were measured during drying. Table 1 details the measurement parameters.

LVER – Linear Viscoelastic Range

- The LVER is the amplitude range where strain and stress are proportional.
- In the LVER, the applied stresses (or strains) are insufficient to cause structural breakdown of the structure and hence microstructural properties are being measured.

Remarks on:

Selection of the shear strain: The shear strain of 0.5% was selected because it is in the linear viscoelastic region (LVER) and so does not lead to a breakdown of the sample´s structure. This was determined by means of an amplitude sweep experiment (results not displayed). Of course, the sample changes during the measurement because it is drying, so that its LVER may also change. A look at the harmonic distortion curve revealed that the sample remained in the LVER during the entire measurement.

The normal force applied during the measurement: A gap of 1 mm was chosen for the test, but to make allowance for the shrinkage that was expected from the drying of the sample, a small normal force was applied to ensure contact was maintained between the upper plate and the sample with the change in gap during the test. This technique prevented the sample from being ejected as maintaining a normal force resulted in the reduction in gap size matching the sample's shrinking.

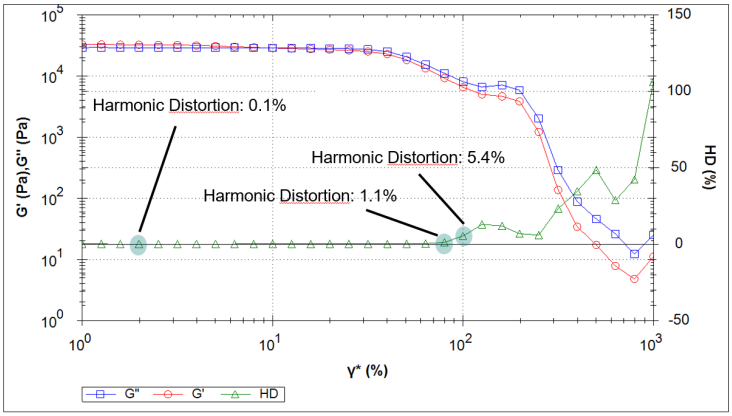
Table 1 Measurement Parameters

Device	Kinexus ultra+ rotational rheometer
Type of test	Oscillation, time sweep
Geometry	PP40 (plate/plate, diameter: 40 mm)
Gap at measurement start	1 mm
Normal force during measurement	0.5 N
Frequency	1 Hz
Shear strain	0.5%

Harmonic Distortion

Within the LVER, input oscillation frequency is the same as the output oscillation frequency. Beyond the LVER, we get **harmonic distortion**. The input oscillation breaks down to higher (i.e., harmonic) frequency responses.

As the strain goes out of the LVER, the harmonic distortion increases. It can be easily displayed in the NETZSCH rSpace software.



NGB · Application Note 304 · EN · 0124 · Technical specifications are subject to change.

NETZSCH-Gerätebau GmbH
Wittelsbacherstraße 42 · 95100 Selb · Germany
Phone: +49 9287/881-0 · Fax: +49 9287/881505
at@netzsch.com · www.netzsch.com

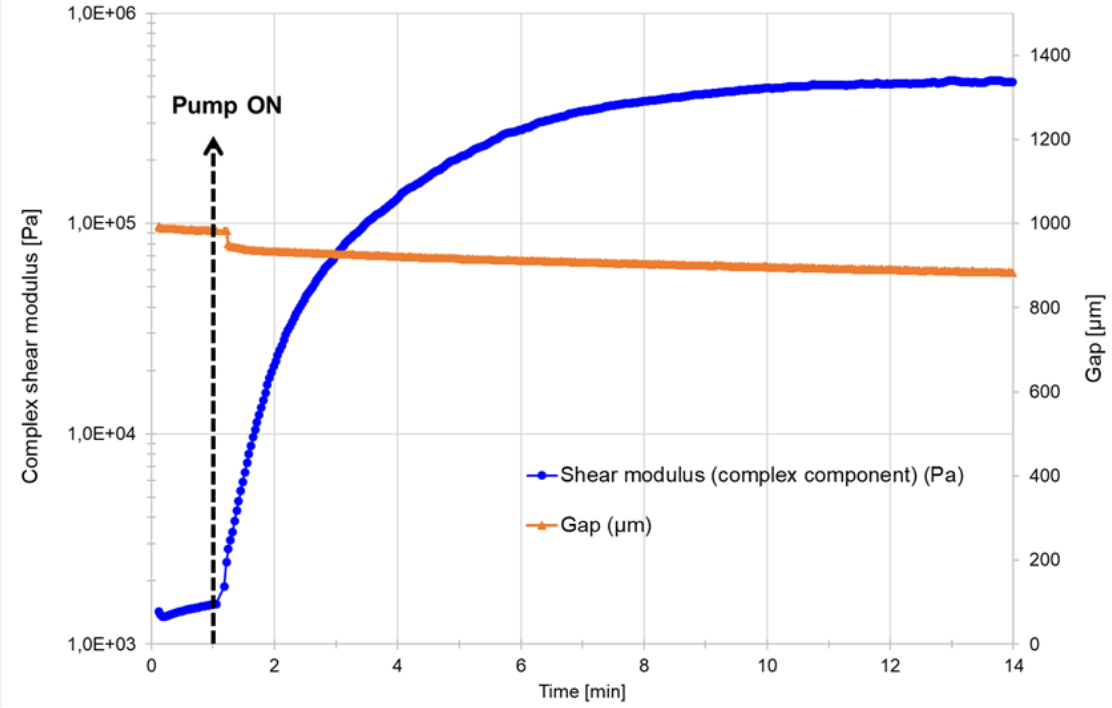
Measurement Results

Figure 2 displays the complex stiffness and the gap measured during drying of the wall paint.

After a 1-minute equilibration in which an oscillation was applied without vacuum, the pump was switched on and dewatering of the paint began. This resulted in a three–decade increase in the complex modulus (stiffness) within 11 minutes, while the sample shrank by more than 10%. After this time, the complex modulus as well as the gap plateaued, indicating the end of the drying process.

A demonstration of setting up the Immobilization Cell can be seen in this video:

How to use the immobilization cell.mp4



2 Complex stiffness and gap during paint drying

NETZSCH-Gerätebau GmbH
Wittelsbacherstraße 42 · 95100 Selb · Germany
Phone: +49 9287/881-0 · Fax: +49 9287/881505
at@netzsch.com · www.netzsch.com