Tracking UV Curing Fast Reactions in a Rheometer Using the Fast Oscillation Mode

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Introduction

Curing, hardening, crosslinking, crystallization, melting, all these processes involve a drastical change of a material's mechanical properties due to the build-up or destruction of a structure. Thus, rheological methods are the perfect tools to characterize these processes. In order not to influence the build-up or destruction of a structure, the preferred rheological method is an oscillation test with a suitable small amplitude.
Typically, these kinds of tests are done with an oscillation frequency of 1 Hz, which limits the data rate to one data point per several seconds. Even increasing the frequency to 10 Hz yields only 1 - 2 data points per second. For very fast processes like e.g. the curing of a super glue or a fast UV curing material, which happens within around 1 second, this speed is not sufficient to yield enough data point to describe the curing process.

The Fast Oscillation Mode

To be able to describe fast processes with enough data points, the Thermo Scientific HAAKE MARS offers the so-called Fast Oscillation Mode. While the classical oscillation mode requires at least one full period of oscillation consisting of a large number of raw data points before a data point can be calculated (Fig. 1), the fast oscillation mode uses a different approach.

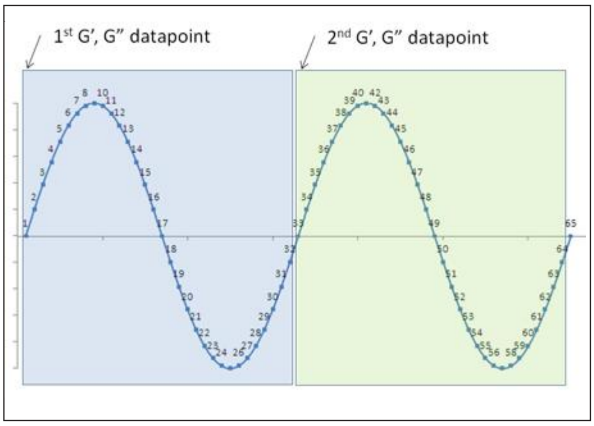

Fig. 1: Schematic illustration of the classical oscillation mode. A full period is always needed to calculate a data point.

The fast oscillation mode uses the continuous stream of raw data points. For every new raw data point an old raw data is discarded and the interval used for evaluation shifts step by step along the raw data coming in (Fig. 2).

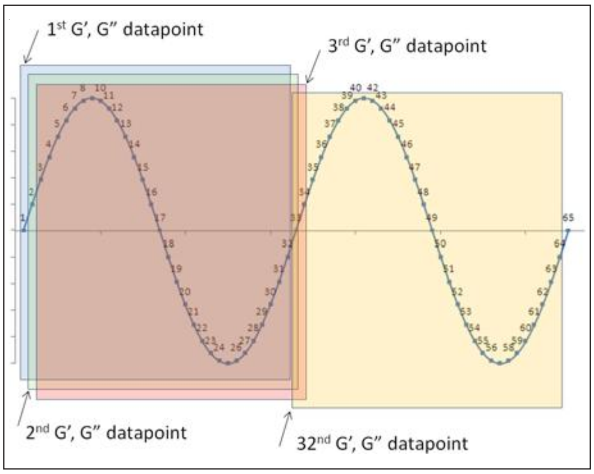

Fig. 2: Schematic illustration of the fast oscillation mode. The rheometer continuously collects new raw data points while continuously discarding the oldest raw data points. A window of one period length is moving along the stream of raw data making higher data rates possible.

Except for the first data point, every following data point is not calculated based on a brand new raw data set but on an updated raw data set. With this approach new data points can be calculated up to 500 times per second i.e. every 2 ms. How to set up such a test method is described in [1].

UV Curing

Due to the high reaction rates involved, UV curing processes are typical examples for rheological tests, which demand a very high data rate. For the test described here, the Upper Plate UV Tool (Fig. 3) has been used, which can be combined with any standard lower plate temperature module. With this combination the quality of the temperature control is not affected in spite of the fact that the sample is exposed to UV light through a transparent geometry.
A UV curing dental filling material has been put into a 20 mm plate/plate geometry. After 60 s the UV light was turned on for 10 s. Within little more than 10 s the storage

Fig. 3: The HAAKE MARS III equipped with the Upper Plate UV Tool (left). The sample is exposed to the UV light from the top through a UV transparent rotor (right). For these photos the sample hood usually used for safety reasons and better temperature control has been removed.

modulus G' increased by a factor of 1000 (Fig. 4). Using the Fast Oscillation Mode, the quickly changing properties of the curing material could be tracked without any gaps in the data. As can be seen in the magnified part of the data in Fig. 4, the data points are collected with a constant spacing of 2 ms giving enough room to monitor even much faster processes.

Summary

With its Fast Oscillation Mode the HAAKE MARS III is capable of following even extremely fast changes in sample properties with oscillation tests. Data rates up to 500 points per second or in other words 1 point every 2 ms are possible as has been shown using a UV curing material.
A UV curing process can be simulated in the rheometer in a very reproducible way due to the use of the Upper Plate UV Tool, which leaves the temperature control unaffected. Subsequently, the test conditions are properly controlled making even the comparison of reactions with different heat of reaction possible.

References

[1] Cornelia Küchenmeister, Jint Nijman and Kiyoji Sugimoto, Thermo Fisher Scientific application note V246 "Measuring fast UV curing materials using oscillatory rheometry"

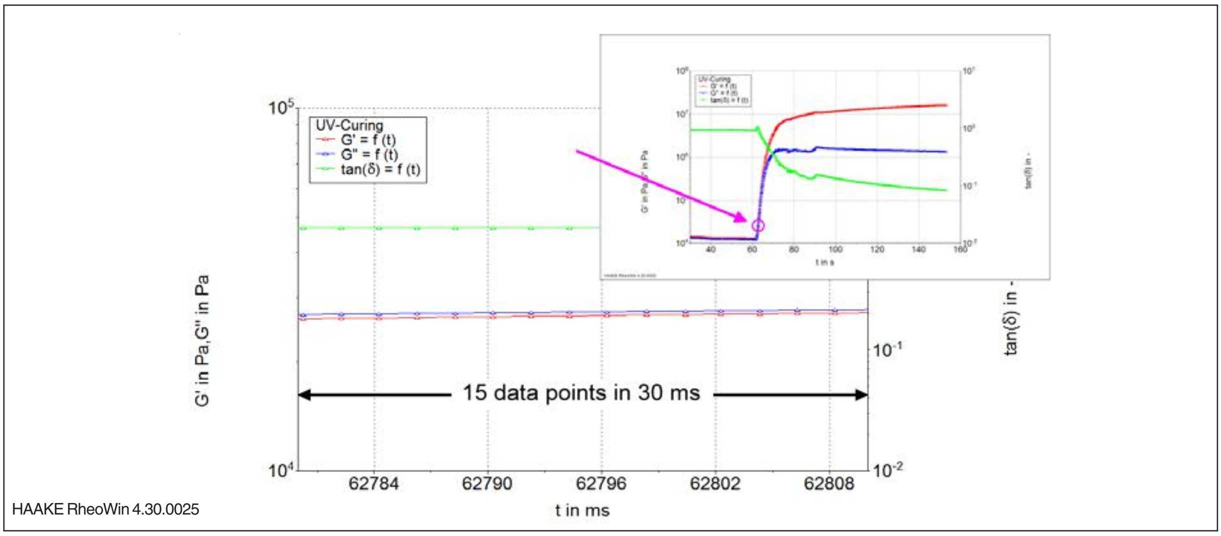

Fig. 4: UV curing of a dental filling material. The storage modulus rises within roughly 10 s by a factor of 1000 (small picture). The Fast Oscillation Mode has been used with its maximum speed of 1 data point every 2 ms to trace the material's properties without missing the quickly changing details.

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