Publishable JRP Summary Report for IND05 MeProVisc
Dynamic Mechanical Properties and Long-term Deformation Behaviour of Viscous Materials

Overview

Many European idustries now incorporate viscous materials into their products to reduce weight and costs, and to improve performance. However, viscous materials deform through time, and changes in their dimensions and properties caused by deformation may affect product performance. Industry therefore require methods to accurately measure the long-term deformation and properties of viscous materials to ensure they are used effectively. This project developed new methods to measure the shape, mechanical properties and deformation rate of viscous materials. Project outputs were adopted by industry, researchers, and equipment manufacturers, and were fed into International Organisation for Standardisation (ISO) committees as the basis for new standards.

Need for the project

A wide range of industrial products incorporate viscous materials (polymeric components), to improve cost-effectiveness, reduce weight and improve performance. For example, polymeric components can be used to manufacture lighter cars, reducing fuel consumption and CO_2 emissions, whilst also improving mechanical efficiency in components such as bearings and drive belts. However, the lifetime and performance of polymeric components is limited by their long-term deformation behaviour, as progressive deformation may alter their properties. Yet, before this project, prevailing measurement methods only measured short-term behaviour and assumed long-term stability - no methods were available for accurately measuring the long-term properties of viscous materials. To develop higher-performance, more competitive products, European industry therefore required methods to accurately measure the behaviour of viscous materials throughout their lifetime.

Specifically, more stable measurement instruments were required that could reduce measurement drift (a reduction in instrument accuracy over time) in order to distinguish drift from genuine nano-level deformation changes. New standardised measurement methods could then be developed using the new high-stability instruments, traceable to SI measurement unit definitions. The accurate interpretation of measurement results often requires complex modelling, yet the current models were too simplistic to simulate long-term deformation. New analysis techniques were required to better understand accuracy and uncertainty in long-term measurements of deformation. Suitable materials, with established properties, were also needed, to be used as reference materials from which measurement instruments could be calibrated.

Scientific and technical objectives

The goal of this project was to develop methods to accurately measure long-term changes in the dimensions and properties of viscous materials. Objective 1 developed methods to differentiate measurement drift from genuine long-term changes in material properties. Based on this, objective 2 then developed methods to measure a range of properties of viscous materials, including indentation creep testing. Objective 3 developed new quantitative methods to analyse the results from objective 2:

1. To develop traceable measurement methods of instrument drift in order to clearly distinguish instrument drift from material creep
2. To develop and validate methods for the measurement of dimensional and mechanical properties of viscous materials when in contact or stressed. These properties include: shape or dimension, elastic modulus (complex or multi-component) and viscosity (time constant)

Report Status: PU Public

EMRP
European Metrology Research Programme
Programme of EURAMET
The EMRP is jointly funded by the EMRP participating countries within EURAMET and the European Union

3. To develop new analysis methods (standard protocols & algorithms) for the measurement of dimensions and mechanical properties of viscous materials to demonstrate modulus measurements from all methods.

Results

1. To develop traceable measurement methods of instrument drift in order to clearly distinguish instrument drift from material viscosity.

Measurement systems are subject to drift, becoming increasingly less accurate through prolonged use. To measure changes in the properties of viscous materials over time, it is therefore necessary to be able to distinguish measurement drift from genuine measured change. Industrial users therefore require a highly-stable measurement system that can used in industrial settings and that can be traced to measurement standards. The objective was achieved through the development of a new, highly-stable, optical interferometer system, with low-noise, high-sampling rate electronics, suitable for use in industrial settings. Optical interferometers combine and overlap light-waves to intensify the waves, creating high-resolution, accurate images, suitable for measuring changes in the shape and dimensions of viscous materials. The stability of the optical interferometer was assessed and calibrated using an X-ray interferometer at NPL, the UK's National Measurement Institute (X-ray interferometers are more accurate but less suitable for industrial conditions than an optical interferometers). The optical interferometer demonstrated a measurement drift of 0.58 nanometres (approximately the width of two atoms) over a 64 hour period, a drift rate of one proton width per second – suitably accurate for measuring long-term changes in the properties of viscous materials. The optical interferometer system was made portable, to ensure it can be used to make *in situ* measurements. Tactile probe profilometry is used in industry to measure micro surface features and other small height changes in materials, to control manufacturing and quality assurance processes. Such measurements are made using a probe, and the weight of the probe must be carefully controlled to ensure it doesn't damage the sample or cause sample build-up on the probe itself, both of which distort measurements. This is particularly difficult to achieve for polymers that can deform under the probe. New correction algorithms were successfully developed for profilometer probes, which enabled correction for distortions created during soft material surface mapping. The project team also developed well-characterised step height change reference materials and organised a comparison exercise where in which they were used by profilometry instrument manufacturers to assess instrument performance. A tactile probe profilometer ISO standard is in preparation.

2. To develop and validate methods for the measurement of dimensional and mechanical properties of viscous materials when in contact or stressed. These properties include: shape or dimension, elastic modulus (complex or multi-component) and viscosity (time constant).

After the reduction in measurement drift achieved in objective 1, changes in the mechanical and dimensional properties of viscous materials can be measured. Indentation creep testing, a technique that measures properties of a sample under pressure, was used to measure changes in shape and dimension, and to assess the elasticity and viscosity of samples. The objective was achieved through the development of a capability at NPL for long-duration, ultra-stable indentation creep measurements, using a UNHT nano-indenter (from Anton Paar Ltd). Displacement rates as low as 20 femtometres per second over a 66 hour period were demonstrated (less than two atomic nucleus widths per second), one of the longest nano-indentation creep tests ever performed.

3. To develop new analysis methods (standard protocols & algorithms) for the measurement of dimensions and mechanical properties of viscous materials to demonstrate modulus measurements from all methods.

Current analysis methods are not sophisticated enough to accurately measure the properties of viscous materials over time. New algorithms and models are needed that can convert results obtained in objective 2 into data that can be compared with results from other testing instruments (such as acoustic testing and dynamic mechanical analysis), which are traceable to SI unit definitions. This will ensure measurements from indentation creep testing can be traced to SI the units of length (the metre), mass (the kilogram) and time (the second). The objective was achieved, as new, time-enabled contact mechanics analysis methods were developed and validated for both dynamic (oscillating) and creep-compliance experiments. Open-source publication of the algorithms and source-code is intended, in order to facilitate direct use in ISO standards and rapid uptake in test instruments. The improved analytical models were developed and incorporated into

SIOMEC software by the Saxonian Institute of Surface Mechanics for the analysis of mechanical surface measurements such as instrumented indentations, scratch tests and tribology tests.

In addition, the project undertook initial work to identify suitable candidate reference materials that could be used to validate and calibrate apparatus used for long-term experiments for other users. Eight different polymeric materials were selected, and samples were distributed to project partner institutions for mechanical property testing. The new analysis methods were applied to the obtained data from macroscopic tests and both oscillating dynamic indentation and indentation creep tests.

Actual and potential impact

Dissemination of results

To promote the uptake of the methods and materials developed, project outputs were shared broadly with scientific and industrial end-users: 15 papers were published in international scientific journals (listed in the following section), including *Philosophical Magazine*, *Wear*, *Journal of Applied Polymer Science*, *Measurement Science and Technology*. Over 50 presentations were given at national and international conferences, meetings and seminars. 9 presentations were given to standards committees, including ISO/TC/SC3, ISO/TC/SC2, and CEN/TC 155 WG26. Discussions were held in these meetings on the development of new standards for measuring viscous materials, and have been progressed as new work items (ISO-14577 Part 6 and Part 7). 18 trainings sessions/workshops were held on testing viscous materials. Measurements were contributed to a study of the ultra-violet induced degradation of polypropylene, and have been included in a report by the UK Materials Knowledge Transfer Network. Stakeholder meetings were held in September 2012 at the PSE 2012 conference in Germisch-Partenkirchen, Germany; April 2013 at NPL, Teddington, UK; October 2013 at the ECI Nano-Mechanics conference in Olhao, Portugal.

To demonstrate the use of the methods developed here, two industrial case-studies were written; "*Correction of step height measurement obtained using commercially available tactile probe methods*" and "*Nano-Indentation Visco-Elastic Characterisation of Elastomers for Creep Prediction of Automotive Timing Belts".*

Early impact on industry

Mahr, a leading manufacturer of measurement equipment in the plastics processing industry, took part in the profilometry comparison exercise in objective 1. Results from this exercise, along with the project's correction algorithm, have allowed Mahr to develop a new 'low load' profilometer suitable for viscous materials, which that can accurately measure a sample without altering or damaging it, and without material accumulating on the probe. The profilometer correction algorithms have also been incorporated in a German Institute for Standardization (DIN) standard, ensuring wider access to these techniques for instrument manufacturers and will ultimately lead to improved profilometry data for their customers.

Within objective 2, the project successfully developed a new method for measuring the creep properties of viscous materials, measuring creep rates in the 20 femtometres per second range. This method is being used by SDS Limited, who design, manufacture and install water management systems, to better understand the long-term performance of their underground structural products, which must maintain their dimensional stability over decades of use. Long-term deformation measurements on materials used in the construction of underground water storage chambers are helping SDS to develop more durable and longer-lasting products. Additionally, SDS's involvement in the project has allowed them to better understand and influence the work of standards committees, allowing SDS to help shape the development of other test methods relevant to their product range through CEN and ISO committees.

Detailed analyses of measurement errors and new calibration procedures were developed for a range of instruments used throughout the project. Results were shared with the instrument manufacturers, such as Anton Paar and Micro Materials Ltd, at a workshop held in conjunction with the final project meeting at PTB in Germany. Anton Paar has used these results to develop a dedicated instrument specifically for the polymer testing market. Anton Paar have also upgraded their nano-test control and data processing software across their range to incorporate improved correction and mechanical property determination routines, ensuring their customers can benefit from the greater understanding of microscale testing that has resulted from the project.

The Saxonian Institute of Surface Mechanics has used the results from objective 3 to enhance their range of software products, including algorithms and models. The software is widely acclaimed, and used by the indentation and scratch measurement communities for the study of surface properties of a wide range of materials. This software is a powerful tool for designers to use across a huge range of applications, confident in the knowledge that their designs will result in more durable products.

Two new work items have been proposed and accepted by ISO TC164/SC3I for hardness testing of materials. The development of these standards will ensure widespread uptake of the test and analysis methods developed in this project.

Potential future impact on industry
The new methods developed in this project will allow scientific and industrial researchers to accurately measure the properties of viscous materials, leading to the development and widespread use of polymeric components in lightweight, high-performance and low-cost products. The methods developed can also be applied to additional measurement needs, including creep testing of polymers, rubbers and composite materials, commonly used in the aerospace, automotive and wind-power sectors. The techniques developed could be used to develop new innovations in these industries, such as the replacement of thermoset matrix composite materials with more durable thermoplastic matrix composites. Ultimately, the techniques developed here will be used to develop new materials and products across a broad range of industries, supporting European industrial competitiveness.

List of publications

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JRP start date and duration:	1st October 2011, 36 months
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The EMRP is jointly funded by the EMRP participating countries within EURAMET and the European Union