Title: Dynamic mechanical properties and long-term deformation behaviour of viscous materials

Abstract

The drive to reduce the cost and weight (light-weighting) of manufactured goods is increasing polymer use across a diverse range from cars to optical lenses to packaging. There is also a desire to reduce environmental impact through reduction in the energy consumed, reduction in packaging and increased recycling. Viscous materials such as polymers change shape and dimension when contacted and are also subject to changes over time. Manufacturing often introduces gradients in the material property and so high-resolution property and deformational behaviour maps are needed to improve design, development and production quality control.

Joint Research Projects (JRPs) submitted for this topic should aim to address the development of validated, highly accurate methods to measure parameters such as drift, dimension, mechanical properties (e.g. modulus, viscosity) and long-term deformation behaviour, which are required to design stable structures, to predict component lifetime and performance and to correct for time-dependent deformation induced by tactile scanning instruments.

Conformity with the Work Programme

This Call for JRPs conforms to the EMRP 2008, section on "*Metrology R&D for applied and fundamental metrology"* related to *Industry* on pages 13, 14, 39 and 42.

Keywords

Dimensional metrology, dimensional stability, polymer properties, creep, viscosity, modulus, new materials, structural connections, precision engineering, cost-efficiency.

Background to the Metrological Challenges

Industry requires high precision engineering parts with predictable and stable dimensions and 'tools' that are very stable in dimension and/or alignment. Such tools may comprise complex lens assemblies and metrology systems, incorporating junctions such as bolts, kinematic contacts and adhesives that are viscous. Material properties and construction design rules are needed to enable improved stability and reduced realignment downtime. In the wider industry, components increasingly contain viscous materials and yet are lifetime and performance limited by long-term deformation behaviour, e.g. car timing-belts. Manufacturing often introduces gradients in the material property and so high-resolution property and deformational behaviour maps are needed to improve design, development and production quality control. This is possible by using contact probe methods such as instrumented indentation testing (IIT) but requires validated viscous contact mechanics.

High creep and/or viscous materials change dimension over time in response to internal or applied stress, causing time dependent errors in static dimensional measurements. The contact stress applied to viscous materials by scanning/contact instruments, scanning probe microscopes (SPM) causes significant deformation, resulting in error and uncertainty in dimensional measurements. This is a problem for high-precision parts (e.g. valves, seals, micro-mouldings, polymeric camera lenses). Such deformation behaviour can also cause drift or instability in the structures and instruments used in high-end processing or process control, where accurate registration of parts or very small tolerances are required [1,2]. Substantial cost savings would result from better long-term alignment and dimensional stability providing better average performance and longer intervals between instrument realignments and increased component or system shelf life. The required stability time-scales are application dependent but can be extremely long. SPM/profilometry systems may only require hours, but photolithography systems already require weeks. Plastic actuators in common dispensers (e.g. asthma inhalers) and instruments awaiting launch into space require a shelf life of years.

Mechanical property measurement of viscous materials by static contact methods is similarly compromised. Corrections for instrument drift are not possible when these are combined with creep rates due to viscosity of the material contacted. There is a need for valid material property and component dimension data to input into models to predict the dimensional instability of parts due to manufacturing residual stresses and/or in-service conditions. This is essential for a comprehensive Design Failure Mode Effect Analysis (DFMEA); without which the adoption of innovation in industry becomes impossible. For example, the auto industry is increasing its use of polymers and composites in cars, but this sector requires a high level of confidence in the design of new components to avoid the risk of in-service failure resulting in the cost and PR damage of a product recall.

Recycled polymers have properties that are different to virgin feedstock and which change every recycling circuit they undergo. The ability to measure actual properties of changing blends of recycled and reprocessed materials would close the polymer usage cycle, enabling true re-use and recycling.

BIPM and the Versailles Project on Advanced Materials and Standards (VAMAS) have agreed to cooperate to improve the traceability of materials property measurement. The ad hoc CIPM WG on material property measurements has outlined the need for an improved metrology framework for reference materials and standardization [3, 4]. Mandates also exist in ISO/TC164/SC3 and ISO/TC61/SC2 to develop indentation-based methods for high spatial resolution polymer property measurement [5].

Extremely high-resolution traceable displacement sensors have been developed by several NMIs based on combined x-ray and optical interferometry]. Fabry-Pérot displacement metrology has been demonstrated on a laboratory level to have 10 pm accuracy with sample extensions of 50 mm on a minute timescale but long-term drift measurement in this regime is now required. Commercially available optical interferometer systems achieve accuracies at sub nm-level, mostly limited by environmental circumstances and by instrument-dependent measurement nonlinearities.

Calibrated and non-linearity corrected capacitance-based displacement sensors are able to achieve similar uncertainties to optical interferometers and are the transducer of choice for most instrumented indentation test (IIT) systems. IIT systems typically are able to measure the quasi-static indentation (elastic) modulus and indentation hardness of elastic-plastic materials with uncertainties of a few %. In fact practical displacement resolutions are usually limited by vibrational noise to ~0.2 nm even in a quiet environment. Standard IIT systems are sensitive to thermal expansion and have to use an elastic (non-creeping) contact to measure and correct for the displacement drift associated with local thermal instabilities. Older instruments have differential thermal expansion coefficients of the order of 1000 nm K^{-1} and require heavy insulation.

Although highly suited to sensitive drift and creep measurements, IIT instruments are unable to distinguish between instrument drift and viscous contact creep. Very recent instrument designs minimise thermal expansion coefficient by a zerodur construction and active feedback on the surface reference probe. With environmental sensitivity minimised the ability to distinguish creep from drift is much improved. However, contact with viscous materials still causes problems both for determining the contact stiffness and the area of the contact, making elastic modulus and hardness measurements using quasi-static indentation cycles highly procedurally dependent.

In general two approaches have been used to obtain the visco-elastic properties of polymers: dynamic or oscillating (ac) methods such as dynamic mechanical analysis (DMA) and static creep measurements under constant load. Dynamic methods such as DMA are most commonly used to determine relative macro property changes as a function of temperature. They have poor absolute modulus accuracy and provide only sample-averaged, single frequency property values. IIT can adopt an identical oscillating approach, which enables a dynamic contact stiffness to be measured and mapped with high spatial resolution but require validated analysis and measurement methods. Static creep experiments are affected by additional displacements due to thermal/instrument drift and will include permanent visco-plastic creep as well as recoverable visco-elastic creep. AC IIT methods have high resolution This could be achieved by transferring the better physical models used in uniaxial and indentation creep methods from the time to the frequency domain. This would provide Finite Element modelling compatible materials parameters. CRMs are required to significantly improve measurement reproducibility.

Thus the main challenges relate to:

- Determination of dimensions over time in response to internal or applied stress
- Mechanical property measurement of viscous materials
- Determination of differences in properties between recycled polymers and virgin feedstock.

Scientific and Technological Objectives

Proposers should address the objectives stated below, which are based on the PRT submissions. Proposers may identify amendments to the objectives or choose to address a subset of them, in order to maximise the overall impact, or address budgetary or scientific / technical constraints, but the reasons for this should be clearly stated in the JRP protocol.

The JRP should aim to provide better understanding of mechanical properties of polymers leading to improved dimensional measurements and production quality control.

The specific objectives are to:

1. Validate measurement of structure/instrument dimension/alignment drift over industrially relevant periods preferably using non-contact methods.
2. Derive design rules for achieving excellent intrinsic structure or instrument stability and develop specifications to achieve metrology platforms mostly insensitive to environmental fluctuations.
3. Measure viscous properties of selected materials and connections that are representative of various industrial needs.
4. Provide underpinning metrology concerning contact with a viscous material to enable measurement and prediction of creep rates in contacts and stressed materials over both metrological and industrially relevant frequencies or time scales.
5. Develop standard test protocols to demonstrate equivalence of complex modulus values obtained by oscillating indentation contact and those obtained by indentation creep and also with independent methods such as uniaxial tensile creep and dynamic mechanical analysis.
6. Enable European guidelines and standards development addressing modulus measurement of viscous materials in bulk and coating form and contact methods for the dimensional measurement of viscous materials.

Proposers shall give priority to work that meets documented industrial needs and that which supports transfer into industry e.g. by cooperation and/or by standardisation.

Proposers should establish the current state of the art, and explain how their proposed project goes beyond this.

Potential Impact

Proposals must demonstrate adequate and appropriate participation/links with the “end user” community. This may be through the inclusion of unfunded JRP partners or collaborators, or by including links to industrial/policy advisory committees, standards committees or other bodies. Evidence of support from the “end user” community (eg letters of support) is encouraged.

Where a European Directive is referenced in the proposal, the relevant paragraphs of the Directive identifying the need for the project should be quoted and referenced. It is not sufficient to quote the entire Directive per se as the rationale for the metrology need. Proposals must also clearly link the identified need in the Directive with the expected outputs from the project. In your JRP submission please detail the impact that your proposed JRP will have on any Directives.

You should also detail other impact of your proposed JRP as detailed in the document “Guidance for writing a JRP”

You should detail how your JRP results are going to:

- feed into the development of urgent standards through appropriate standards bodies eg CEN

- transfer knowledge to the automotive, aerospace, packaging, pharmaceutical, construction and general manufacturing sectors.

You should also detail how your approach to realising the objectives will further the aim of the EMRP to develop a coherent approach at the European level in the field of metrology. Specifically the opportunities for:

- improvement of the efficiency of use of available resources to better meet metrological needs and to assure the traceability of national standards
- the metrology capacity of Member States and countries associated with the Seventh Framework Programme whose metrology programmes are at an early stage of development to be increased
- outside researchers & research organisations other than NMIs and DIs to be involved in the work

Time-scale

The project should be of 3 years duration.

Additional information

The references were provided by PRT submitters; proposers should therefore establish the relevance of any references.

[1] "Roadmap Precisietechnologie", Innovation-driven research program (IOP) on precision technology, SenterNovem, www.senternovem.nl, Utrecht, The Netherlands, 22 January 2004.
[2] International Technology Roadmap for Semiconductors (ITRS), www.public.itrs.net.
[3] "Evolving Need for Metrology in Material Property Measurements", Report of the CIPM ad hoc Working Group on Materials Metrology (WGMM), BIPM, 2008.
[4] Memorandum of Understanding on Cooperation between the BIPM and the VAMAS, June 2008.
[5] ISO/TC 164 Business Plan "Mechanical Testing of Metals", 2004.