EMPIR Call 2014 – Industry and Research Potential

Selected Research Topic number: **SRT-i23**
Version: 1.0

Title: Reliable micro mechanical test data for modelling advanced materials

Abstract

Accurate mechanical property measurement of micro- / nano-scale test pieces is essential to underpin the development of materials' modelling to achieve a halving of development lead times for advanced materials. This is essential for maintaining a competitive edge for European manufacturing. Production and measurement of micro- / nano-scale test pieces has advanced in the past five years, but with minimal information on uncertainty budgets, and measurements made at ambient (room) temperatures. Systematic evaluation is needed on the scale and effect of damage induced by manufacture, force and displacement measurement errors caused by poorly known compliance and friction effects, or from the effects of residual stresses within the test pieces.

Keywords

Micro-mechanical testing, focused ion beam, low force measurement, micro-specimen temperature measurement

Background to the Metrological Challenges

Development of new materials and components in advanced materials (such as alloys capable of withstanding higher temperatures and corrosive conditions for the aerospace and power generation sectors and higher strength steel and aluminium alloys for the construction and transport sectors) often takes many years of iterative development with a gradual refinement of the materials' microstructure as the effects of changing composition and processing conditions on the microstructure, and the behaviour of the microstructure in use, is gradually understood. This time factor results in high development costs, and an inability to respond to the rapidly changing market reduces industry competitiveness [1]. To develop "designer" materials, where the required performance is built in to the materials' structure from inception, will lead to highly desirable improvements in the cost and time of the materials' development cycle.

The development of modelling techniques such as crystal plasticity finite element modelling and other simulation methods have the potential for dramatically reducing costs [2, 3]. However, these models lack essential accurate input on material properties data, which is measured at the microstructural scale (e.g. as a function of crystal orientation, dislocation content or grain boundary structure). Mechanical behaviour at elevated temperatures is required for modelling real applications, but so far, such measurements have not been made above ambient temperatures.

The development of focused ion beam (FIB) microscopes has enabled the manufacture of a range of mechanical test pieces with critical dimensions between 500 nm and 10 µm over the past 10 years. A FIB produces a focused beam of gallium ions, used to mill material around the region of interest in a material's microstructure. Control of the beam can produce pillars, cantilever beams or micro-tensile specimens, which can then be compressed / bent / pulled in situ, or outside the microscope, by a variety of nano-mechanical test systems. This information can be used to relate the mechanical properties to the structure. However, such measurements are prone to a wide variety of errors, of which a primary concern is the effect of damage to the microstructure by the milling action of the gallium ions; FIB milled pillars have been shown to reduce yield strengths. The effect of uncertainty of dimensions can be estimated, but little is reported on the consistency of dimensions along a specimen's entire length, such as the cross section of a cantilever beam or the uniformity of pillar diameters.

The full metrological framework for micro-mechanical testing needs to be defined with major effort put into understanding the conflicting information on the effects of ion beam machining on the measured stresses during deformation. Comparative studies of different materials are impossible in the absence of standards for

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these measurements; and currently the manufacturing methods and subsequent measurement techniques used are highly individual, with comparisons between these techniques being negligible.

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 proposal.

The JRP shall focus on the development of metrological procedures for the measurement of the mechanical properties of advanced materials on the nano- and micro-scale.

The specific objectives are

1. To determine the accuracy and uncertainty of measurements of the mechanical properties of micromechanical test pieces produced by focused ion beam microscopy and other techniques from a representative range of advanced materials.
2. To develop methods for the measurement of the depth of ion beam damage and its relationship to changes in the elastic modulus and initiation of plastic deformation in both ductile and brittle materials using techniques such as microfocus X-ray diffraction methods and electron microscopy and correlated with Monte Carlo simulations of ion implantation. Techniques to relate nano- and microscale measurements of the 3D microstructure of the materials using slice and view incorporating 3D Electron Backscatter Diffraction (EBSD) should also be addressed.
3. To develop methods for measurement and control of temperature on the localised micro specimens during deformation, enabling rapid fluctuations in temperature to be sensed on very small volumes during mechanical testing.
4. To develop traceable low force measurement methods (at loads of < 10 µN) in order to evaluate friction and compliance effects during contact between probes < 1 μm^2 in area, to compare these methods with techniques such as Atomic Force Microscopy (AFM) force volume mapping, and to develop dimensional measurement techniques to determine specimen profiles on undercut surfaces when the access to these surfaces is limited to regions < 20 µm x 20 µm.
5. To engage with industry that manufactures and or / exploits advanced materials to facilitate the take up of the technology and techniques developed by the project for the accurate measurement of the mechanical properties at micro/nano-scale, to support the development of new, innovative products based on advanced materials thereby enhancing the competitiveness of EU industry.

Proposers shall give priority to work that meets documented industrial needs and include measures to support transfer into industry by cooperation and by standardisation. An active involvement of industrial stakeholders is expected in order to align the project with their needs – both through project steering boards and participation in the research activities.

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

EURAMET expects the average EU Contribution for the selected JRPs to be 1.5 M€, and has defined an upper limit of 1.8 M€ for any project.

EURAMET also expects the EU Contribution to the external funded partners to not exceed 30 % of the total EU Contribution to the project. Any deviation from this must be justified.

Any industrial partners that will receive significant benefit from the results of the proposed project are expected to be unfunded partners.

Potential Impact

Proposals must demonstrate adequate and appropriate participation/links to the “end user” community, describing how the project partners will engage with relevant communities during the project to facilitate knowledge transfer and accelerate the uptake of project outputs. Evidence of support from the “end user” community (e.g. letters of support) is also encouraged.

You should detail how your JRP results are going to:

Address the SRT objectives and deliver solutions to the documented needs,

- Drive innovation in industrial production and facilitate new or significantly improved products through exploiting top-level metrological technology,
- Improve the competitiveness of EU industry,
- Feed into the development of urgent documentary standards through appropriate standards bodies,
- Transfer knowledge to the Advanced Materials sector.

You should detail other impacts of your proposed JRP as specified in the document "Guide 4: Writing Joint Research Projects"

You should also detail how your approach to realising the objectives will further the aim of EMPIR to develop a coherent approach at the European level in the field of metrology and include the best available contributions from across the metrology community. 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 EURAMET Member States whose metrology programmes are at an early stage of development to be increased
- organisations other than NMIs and DIs to be involved in the work

Time-scale

The project should be of up to 3 years duration.

Additional information

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

[1] Cummings, P. T., & Glotzer, S. C.: http://www.wtec.org/sbes-vision/RDW-color-FINAL-04.22.10.pdf2010

[2] Eureka Cluster Programme Proposal - Metallurgy Europe Road Map 2014-2020

[3] Materials Genome Initiative, http://www.whitehouse.gov/sites/default/files/microsites/ostp/materials_genome_initiative-final.pdf