European Partnership on Metrology
Call 2022 – Digital Transformation, Health, Integrated European Metrology, Normative and Research Potential

Selected Research Topic number: **SRT-n11**
Version: 1.0

Title: Traceability improvement for instrumented indentation testing

Abstract

Instrumented indentation testing (IIT) is a widespread mechanical characterisation technique that, for its flexibility, speed, and non-destructiveness, meets the quality control requirements of Industry 4.0 and the European Green Deal. IIT achieves thorough, bulk-to-local, mechanical characterisation of many properties, such as Young's modulus, and stress-strain curves, which conventionally require costly destructive tests. Traceability is key to the industrial adoption of IIT. However, as noted by ISO/TC164/SC3-WG4, ISO 14577 presents several calibration methods that are impractical and ambiguously defined. By investigating measurement uncertainty influence factors, calibration methods and traceability will be improved.

Keywords

Instrumented indentation test, Nano-indentation, Depth-sensing indentation, Calibration, Measurement uncertainty, Traceability chain, Reference materials, Testing platform, Surface topography

Background to the Metrological Challenges

To cope with the more stringent demands of customers for enhanced performance and customisation, novel manufacturing processes, such as additive manufacturing and advanced materials, including innovative composites and coatings, are being developed. This development is contextualised in Industry 4.0, Sustainability, and European Green Deal framework. Furthermore, the need to manage big data requires flexible and fast quality inspection that relies on thorough, accurate and precise characterisation methods. Amongst several product properties, the surface characterisation is core to control the manufacturing process, as surfaces may feature distinctive manufacturing signatures, and to engineer the product functionality. In particular, the mechanical properties of technological surfaces are critical in determining the tribological, wear and fatigue behaviour. Within this framework, the European Green Deal calls set a path for the development and the widespread adoption of reliable and traceable non-destructive characterisation methods to achieve zero-defect manufacturing and zero waste, while developing bio-compatible and recycled materials. The availability of characterisation methods that do not require ad-hoc shaped specimens, are essential to achieve these strategic objectives. IIT is one of the most appealing mechanical characterisation techniques, it consists of a semi-destructive test, which requires limited sample preparation and can be performed on the final product. It allows thorough multiscale mechanical characterisation, i.e. ranging from sub-micrometre domain to bulk properties, in terms of hardness, creep, relaxation, Young's modulus and stress-strain (as reported by ISO/TR 29381:2008) behaviour.

Traceability of IIT is a key requirement to provide users with confidence in comparing the results of the tests, although performed on different IIT machines and under different experimental conditions. Traceability is obtained by calibrating the IIT machine according to ISO 14577-2:2015. Amongst the factors governing the measurement uncertainty of mechanical characterisation, the frame compliance and the area shape function parameters are the most important. Current ISO standard outlines several methods for machine calibration, which are based on different hypotheses and whose description is not prescriptive enough and seems based on good practice: this makes traceability difficult to demonstrate for this characterisation technique.

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 protocol.

The JRP shall focus on the traceable measurement and characterisation of instrumented indentation testing and support its industrial adoption for quality control.

The specific objectives are:

1. To develop, in accordance with 009-ISO TC164 SC3 WG4 and 010-ISO TC164 SC3 WG4, new practical calibration methods for improving the traceability of IIT while taking into account critical influence factors, such as frame compliance, area shape function and indentation force calibration. To develop methods to evaluate the measurement uncertainty of the calibration methods for IIT platforms listed in the ISO 14577-2:2015.
2. To assess the effect of the most relevant influence factors in the calibration of IIT platforms by developing methods to propagate uncertainty contributions and by investigating reference materials with long-term mechanical stability, with high homogeneity and less pile-up/sink-in effects for calibration at room and high temperatures. To dynamically characterise IIT in relation to industrial applications and investigate, as per ISO 14577-1:2015, three ranges of IIT (nano, micro and macro), as per ISO 14577-1:2015.
3. To develop a new, simplified, more rigorous and practical calibration framework for IIT platforms including the evaluation of measurement uncertainty for dynamic mechanical characterisation. To develop high accuracy and direct method to be exploited for higher traceability chain level calibration based on Atomic Force Microscope (AFM) measurements for the indenter area calibration, whilst an additional industry-oriented method, which does not require the use of an AFM, should also be developed.
4. To improve traceability for IIT, by simplifying the calibration method and defining clear calibration conditions for application ranges. To clarify if the route (when and how) towards direct calibration is possible.
5. To contribute to the standards development work of the technical committee ISO/TC 164, in particular to the revision of the ISO 14577, to ensure that the outputs of the project are aligned with their needs, communicated quickly to those developing the standards and to end-users, and in a form that can be incorporated into the standards at the earliest opportunity.

The proposed research shall be justified by clear reference to the measurement needs within strategic documents published by the relevant Regulatory body or Standards Developing Organisation or by a letter signed by the convenor of the respective TC/WG. EURAMET encourages proposals that include representatives from industry, regulators and standardisation bodies actively participating in the projects. The proposal must name a “Chief Stakeholder”, not a member of the consortium, but a representative of the user community that will benefit from the proposed work. The “Chief Stakeholder” should write a letter of support explaining how their organisation will make use of the outcomes from the research, be consulted regularly by the consortium during the project to ensure that the planned outcomes are still relevant, and be prepared to report to EURAMET on the benefits they have gained from the project.

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

EURAMET expects the average EU Contribution for the selected JRPs in this TP to be 0.8 M€ and has defined an upper limit of 1.2 M€ for this project.

EURAMET also expects the EU Contribution to the external funded beneficiaries to not exceed 30 % of the total EU Contribution across all selected projects in this TP.

Any industrial beneficiaries that will receive significant benefit from the results of the proposed project are expected to be beneficiaries without receiving funding or associated 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,

- Feed into the development of urgent documentary standards through appropriate standards bodies,
- Transfer knowledge to the aerospace, automotive and biomedical sectors.

You should detail other impacts of your proposed JRP as specified in the document “Guide 4: Writing Joint Research Projects (JRPs)”

You should also detail how your approach to realising the objectives will further the aim of the Partnership 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] *010 ISO TC164 SC3 WG4 traceability indentation testing frame compliance*

https://www.metpart.eu/go/need10