

Title: Improvement of standards related to Instrumented Indentation Test for advanced materials characterisation

Abstract

Instrumented indentation testing (IIT) is a mechanical characterisation technique that, due to its flexibility, speed and non-destructiveness, meets the quality control requirements of Industry 4.0 and the European Green Deal. IIT achieves high resolution local characterisation of many material properties, that conventionally require costly and toxic destructive tests on large scale pieces. However, traceability will be key to the industrial uptake of IIT and substantial normative research is required to update ISO 14577 (metallic materials: instrumented indentation test for hardness and materials parameters). Therefore, proposals addressing this SRT should investigate the factors that influence the measurement uncertainty, in order to enhance and develop robust and practical calibration methods, which should improve the traceability of IIT for use in the characterisation of advanced materials. Data, methods, guidelines and recommendations should be provided to CEN/TC 352 [1] and ISO/TC 164 [2] as a contribution to the revision or development of ISO 14577.

Keywords

AFM-based IIT, calibration, coating testing, dynamic IIT (DIIT), high-temperature testing, instrumented indentation testing (IIT), measurement uncertainty, mechanical characterisation, pile-up, stress, testing platform, traceability chain, vacuum

Background to the Metrological Challenges

New approaches to manufacturing are being developed, which align with Industry 4.0 and sustainability initiatives like the European Green Deal. This emphasises the need for fast and accurate quality inspection methods like instrumented indentation testing (IIT), which is used for the non-destructive surface characterisation of mechanical properties, like hardness, modulus and residual stress, in zero-defect / zero-waste manufacturing to control manufacturing processes and product functionality. IIT is used to optimise manufacturing processes, to characterise microstructures, to assess the mechanical properties of multi-layer materials and to replace destructive tests for average bulk properties. Advanced IIT measurement protocols are available, which cover dynamic measurements and extend from room to high-temperature applications.

IIT results need to be traceable to ensure confidence in comparisons, and this is achieved through calibration (ISO 14577-2). However, the use of different calibration methods has led to a lack of compatibility in these mechanical characterisations. The frame compliance and the area shape function parameters are the most important factors, which govern the measurement uncertainty of the mechanical characterisation. Calibration methods for the frame compliance need to be developed for use at the macro range. Therefore, the applicability of IIT needs to be extended, and the main influence factors need to be addressed. The calibration and verification methods that are used by indentation machines need to be improved by reducing the bias and uncertainty caused by frame compliance, the area shape function and by correcting the effects of pile-up and sink-in. Standardised data exchange formats and soft-gauges are not yet available and need to be developed in order to improve comparability between methods and IIT machines. Overall, the metrological performance of IIT needs to be enhanced for the mechanical characterisation of advanced materials. This information needs to be provided to ISO/TC 164/SC 3 to be considered for use in the revision of ISO 14577-1, on a test method, and ISO 14577-2, on the verification and calibration of testing machines.

Dynamic instrumented indentation testing (DIIT) has recently been standardised for applications in the linear elastic field of materials, which is useful for the traceable calibration of frame compliance and indenter area

function. DIIT has also been exploited for coating characterisation, but the dynamic response of the indentation platform still needs to be calibrated, which requires clear traceability and uncertainty propagation. The effects of fundamental calibration on the frequency response of AFM-based IIT and DIIT platforms has not been reported, nor have the effects of the uncertainty in the cantilever geometry and the indirect calibration of the dynamic response. These aspects need to be investigated to enable the traceable quantitative evaluation of the mechanical surface response and to ease pathways to traceability. Alternative coating models have been proposed, but these need to be assessed, and approaches developed, for the characterisation of coatings thinner than 100 nm. The indentation size effect also needs to be addressed with a focus on applications in the semiconductor industry. In conclusion, new methods for AFM-based IIT and DIIT platforms, across the nano, micro and macro ranges, need to be developed with improved metrological performance and traceability, and this needs to be provided to ISO/TC 164/SC 3 to be considered for use in the revision of ISO 14577-4, on a test method for metallic and non-metallic coatings, and ISO 14577-5, on linear elastic DIIT.

IIT testing is now possible under extreme conditions, such as high temperatures and in vacuum. However, a comprehensive database of material properties, under these conditions, needs to be developed for use in predictive modelling, which require redefined measurement procedures and calibration methods. Challenges include testing under vacuum, or restricted conditions, and managing heat flow and the thermal / thermo-chemical compatibility of the indenter and the substrate. Contact drift also poses a significant challenge, which leads to measurement errors. Therefore, local thermal effects on the indenter and the sample surface need to be evaluated by considering the indenter material, geometry and other factors. In addition, a procedure to design and calibrate force, displacement, frame compliance and indenter tip materials and geometry needs to be developed for application at up to 1100 K. High-temperature candidate reference materials have been identified, but they need to be evaluated up to 1100 K, in vacuum conditions, to see if they are suitable for use in calibration and verification at multiscale. In addition, these approaches need to be validated in industrial applications. This input needs to be provided to ISO/TC 164/SC 3 to be considered for use in the development of ISO/AWI 14577-6, on instrumented indentation test at elevated temperature, and ISO 14577-3, on the calibration of reference blocks, needs extending to cover testing at up to 1100 K in a vacuum.

ISO14577:2015 struggles to measure the properties of stressed materials or those with pile-up around the indenter, which affects the accuracy of the indentation modulus and hardness measurements. Although IIT could be used to evaluate stress fields, it requires these properties to be separated, which has so far led to the draft standard being downgraded to a technical specification. Recent research has shown that a corrected hardness result could be achieved by using a reference elastic-modulus value, but no method has been agreed. Therefore, how the IIT measurement of residual stress, and stress change is affected by the main influence factors needs to be investigated. These advancements also need to be demonstrated through relevant applications in automotive manufacturing technology. This metrological input is required by ISO/TC 164/SC 3 to be considered for use in the further development of ISO/TS 19096:2023, on instrumented indentation test for hardness and materials parameters: evaluation of stress change using indentation force differences. ISO 14577 needs to be extended to include materials that are stressed or pile up when indented.

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 proposal shall focus on the metrology research necessary to support standardisation related to instrumented indentation testing for advanced materials characterisation.

The specific objectives are

1. To enhance the metrological performance of IIT for the mechanical characterisation of advanced materials. This should include improvement of the calibration and verification methods that are used by indentation machines: specifically by reducing the bias and uncertainty caused by frame compliance, the area shape function and the effects of pile-up and sink-in. The applicability of IIT should also be extended, and the main influence factors addressed, across the nano, micro and macro ranges. In addition, standardised data exchange formats and soft-gauges should be developed in order to improve comparability between IIT machines (ISO 14577-1,-2).
2. To provide traceable methods for AFM-based IIT, and for directly calibrating dynamic IIT (DIIT) platforms across the nano, micro and macro ranges. These methods should be used to improve metrological performance, and traceability, for the characterisation of coatings (ISO 14577-4) and for DIIT (ISO 14577-5). In addition, alternative coating models should be assessed, and approaches developed, for the characterisation of coatings that are thinner than 100 nm (ISO 14577-4). The

indentation size effect should be addressed with a specific focus on applications in semiconductor industry.

3. To investigate the effects of temperature and vacuum on key IIT factors such as the force and displacement scale, frame compliance, area shape function and contact temperature (ISO/AWI 14577-6). Local thermal effects on the indenter and the sample surface should also be evaluated by considering the indenter material, geometry and other factors. High-temperature candidate reference materials should be evaluated up to 1100 K, in vacuum conditions, to see if they are suitable for use in calibration and verification (ISO 14577-3). In addition, these approaches should be validated through relevant applications in the nuclear, aerospace or other industries.
4. To investigate how the evaluation of residual stress, and stress change, by IIT, is affected by the main influence factors (e.g., methods to evaluate the strain field, the convolution of the material microstructure, the indentation size effect and local plasticity) (ISO/TS 19096:2023). In addition, these advancements should be demonstrated through relevant applications in automotive manufacturing technology.
5. To contribute to a revision of ISO 14577 by providing the data, methods, guidelines and recommendations, which are necessary for the standardisation of IIT tests for hardness and metallic material parameters, to CEN/TC 352 and ISO/TC 164. Outputs should be in a form that can be incorporated into the standards at the earliest opportunity and communicated through a variety of media to the standards community and to end users.

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 convener 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.

Proposers should note that the programme funds the activity of researchers to develop the capability, not the required infrastructure and capital equipment, which must be provided from other sources.

EURAMET expects the average EU Contribution for the selected JRPs in this TP to be 1.0 M€ and has defined an upper limit of 1.3 M€ for this proposal.

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 proposal's 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,
- Facilitate improved industrial capability, or improved quality of life for European citizens in terms of personal health, protection of the environment and the climate, or energy security,
- Transfer knowledge to the materials characterisation sector.

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 Metrology 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.

Timescale

The project should be of up to 3 years duration.

Additional information

The links provided in this section are only correct at the time of publication up until the end of the Call year.

These references have been provided by EURAMET.

- [1] *003 CEN TC 352 Traceability Improvement for Instrumented Indentation Testing.*
<https://www.metpart.eu/go/need03>
- [2] *019 ISO TC164 Traceability and Uncertainty improvement Instrumented Indentation Test (IIT).*
<https://www.metpart.eu/go/need19>