Versailles Project on Advanced Materials and Standards

Calls for Participation – December 2024

VAMAS

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VAMAS

Surface Chemical Analysis
Technical Working Area 2

Project A44

Quantitative Imaging Using NanoSIMS for Biological Materials

Objective

The aim of this international interlaboratory comparison is to validate a protocol for quantifying concentrations of drugs and metabolites using Nanoscale Secondary Ion Mass Spectrometry (NanoSIMS).

The work forms the first steps towards a documentary standardised protocol for NanoSIMS and relates to ISO TC 201 (Surface Chemical Analysis).

Background

NanoSIMS has found great utility in providing high spatial resolution (< 50 nm), but only semi-quantitative, mapping of drugs and metabolites at a subcellular scale. However, it is necessary to develop a robust method to carry out quantitative measurements to expand the usability of the data.

The team at AstraZeneca have developed a protocol to prepare test materials that have been used to generate calibration curves for bromine and iodine. These curves allowed the researchers to successfully provide absolute quantification of halogen-containing analytes in fixed and resin-embedded biological samples at a high spatial resolution. The interlaboratory study will help to validate the protocol, develop best practices and provide confidence in NanoSIMS measurements.

Standardization Needs

The pre-standardisation need addressed here is the interlaboratory comparison of results obtained when a protocol for quantification of analyte in a biological context is used.
There are currently no standardised procedures for quantitative imaging of biological materials using NanoSIMS.

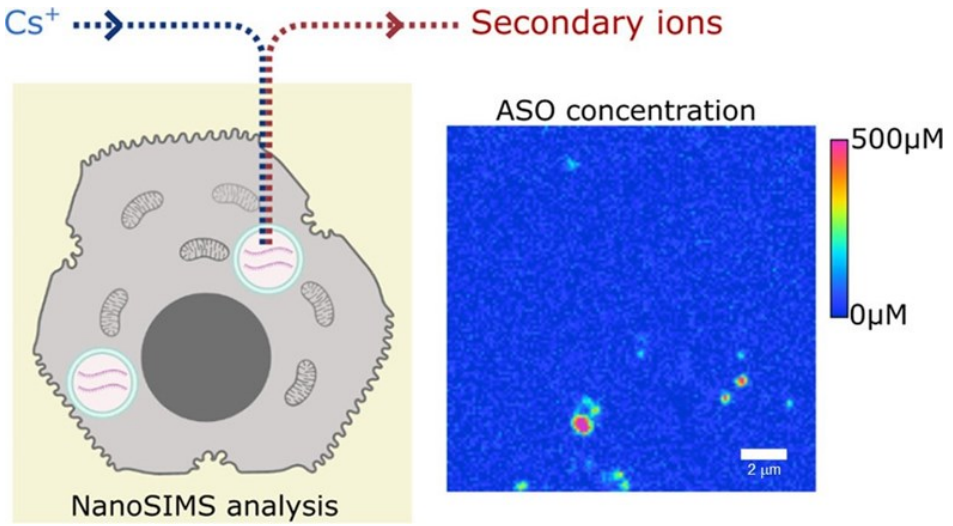
Relevant Standards Committees

ISO/TC 201 Surface Chemical Analysis

Work Programme

- Protocol for NanoSIMS measurement will be prepared.
- Samples will be prepared by project leadership and distributed to participants along with protocol.
- The results of the analysis will be compared with uncertainty evaluations.
- The project started in August 2024 for a duration of 24months.

Call for Participation



NanoSIMS schematic and Hue Saturation Intensity image showing concentration of Antisense Oligonucleotide within a primary human hepatocyte (Becquart et al, Anal. Chem, 2022)

Deliverables and Dissemination

- Publication in a peer-reviewed scientific journal and presentations in conferences.
- A draft international report of a protocol of direct quantification of analytes in biological samples using NanoSIMS to be submitted to ISO TC 201.

Volunteers Welcome

Participants will fund their own involvement (approx. 4 days work).

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August 2024

VAMAS

Surface Chemical Analysis
Technical Working Area 2

Project A45

Comprehensive file format for the measurement and analysis of surface chemical analysis data

Objectives

The purpose of this collaborative research is to identify specifications and considerations that should be reflected in a comprehensive data format that describes data, metadata, and workflows related to the measurement and analysis of surface chemical analysis instruments and software. The achieved results of this interlaboratory comparison study will be used in future ISO/TC 201 standard proposals.

Background

There is a wide variety of equipment and software for surface chemical analysis, and new technologies and algorithms are continually being developed. Furthermore, research and development of new materials require the same material to be measured and analysed using multiple measurement and analysis equipment and software, i.e., multi-modal analysis. For this reason, data-driven research and development that fully uses AI and other technologies, integrating diverse, cutting-edge measurement and analysis, is required.

Standardisation Needs

There is a need for a standardized data format that can comprehensively handle data/metadata, including pre-processing for measuring, correlation analysis by multiple instruments, etc. This will enable the extraction and comparison of measurement conditions, sample information, and analysis results using the same application, which will benefit data-driven research. The data format must be capable of describing a variety of workflows in response to complex measurements. Even if the observed data of complex measurements are placed in cyberspace, they must be usable as data independent of the device, and the data uniqueness must be guaranteed. Furthermore, to deal with issues such as data tampering, there must be information that ensures reproducibility and traceability.

Work Programme

Participants are asked to use the measurement examples described in this comprehensive data format to evaluate their independent usability, usefulness, and readability.

Inter-laboratory comparisons are expected to be conducted in triplicate.

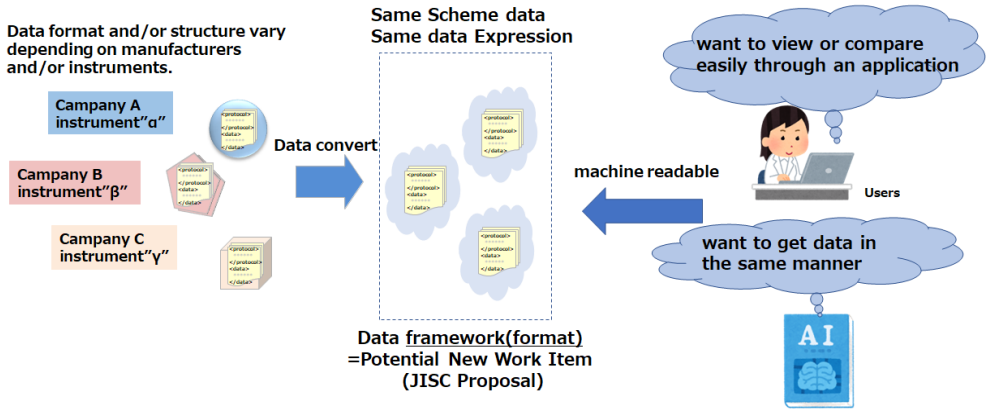
1. It will be evaluated whether it has a mechanism for expressing metadata and data from various measurements and expressing them within the analysis process.
2. It will be assessed whether it has a mechanism for recording results obtained according to the method described in the format, including metadata/data related to the process.
3. It will evaluate whether it has a mechanism for recording the analysis results, processes, and logs that guarantee traceability by performing correlation analysis using multi-modal measurements from the same sample.

The prototype tools will be available for participants to use freely for verification.

Deliverables and Dissemination

The results of the inter-laboratory comparison evaluating this format, undertaken according to the proposed procedure, will be documented in a VAMAS/ TWA 2 technical report and also in a scientific journal paper and will then be used to draft a generic data format for future proposals of relevant ISO standards.

Call for Participation



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September 2024

Polymer Nanocomposites

Technical Working Area 33

Call for Participation

Project 1

Determination of the shape, size and size distribution of nanofiller particles

Objectives

The project aims to develop pre-standardized experimental procedures for determination of size, shape and size distribution of clay nanoplatelets. These include protocols on clay exfoliation into individual platelets, platelets deposition on suitable substrate, image acquisition by scanning electron microscopy (SEM) and atomic force microscopy (AFM) techniques, image processing and statistical analysis.

Proposed activities include the preparation and realization of an inter-laboratory comparison with the objective to obtain a value for laboratory reproducibility of the dimensional measurements of clay nanoparticles using the developed protocols.

Background

The worldwide use of Polymer Nanocomposites (PNC) for industrial and research purposes is already large and it continues to grow rapidly. The design of nanoparticles is critical for nanocomposite structure. In particular, a careful characterization of nanoparticles morphology is required for both an interface design in composite systems and design of a whole product.

Standardization needs

There are no internationally recognized standards for PNC testing. Particularly, there are no detailed procedures on reproducible dimensional analysis of 2D nanofiller particles. The main goal of the project is to standardize the essential experimental test methods for nanofillers.

Work Programme

Development of the method of clay characterization will proceed in following stages:

1. Discussion of a protocol on clay nanoplatelets exfoliation and deposition.
2. Discussion of a protocol on acquisition of SEM and AFM images.
3. Discussion of a protocol on image processing.
4. Discussion of a protocol on statistical treatments of processed images.
5. Distribution of deposited clay nanoplatelets between participants for interlaboratory comparison of dimensional analyses of the samples.

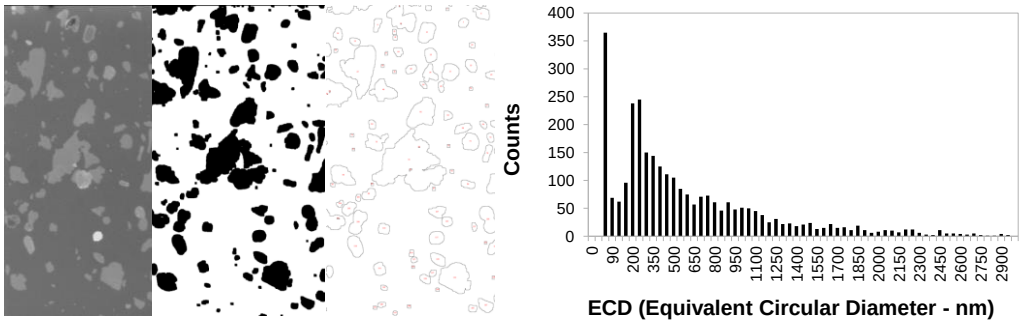

Figure 1: SEM micrograph and image processing of Somasif ME-100.

Deliverables and Dissemination

- Development of experimental methods for the nanofiller characterization
- VAMAS Technical Report
- Publications in scientific journals
- ISO TC 229 links aimed at development of an ISO standard

Funding

Participants fund their own involvement in the project.

Status

Protocol for clay nanoplatelets deposition is under preparation for the distribution between participants. Protocols on images acquisition and processing are currently under preparation.

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December 2024

Polymer Nanocomposites

Technical Working Area 33

Call for Participation

Project 3

Cross-validation of Young's modulus measurements of graphene PNCs between DMA and stress-strain tests

Objectives

This project aims to develop reproducible test methods and validated cross method relationships for determining the Young´s modulus of polyamide-graphene oxide (GO) nanocomposites *by* dynamic mechanical analysis (DMA) and conventional stress-strain tests.
The envisaged procedures will include protocols for specimen preparation, covering parameters of nanocomposite melt extrusion processing as well as necessary basic characterization of GO fillers (chemical-physical state and homogeneity of dispersion in the matrix).

Background

The mechanical properties of polymer nanocomposites (PNCs) are relevant to many industrial sectors, especially the construction, packaging, solar energy, and automotive-related structural parts industries. A general application of PNCs is substitution for conventional metal components, due to advantageous combination of strength, rigidity and light weight. In this context, introduction of new acceptance test methods connecting achievable properties to readily determined measurands becomes highly relevant.

Standardization needs

There are no internationally recognized procedures connecting the DMA measurement results for Young's modulus with the values obtained in conventional stress-strain tests.
The establishment of factors defining a correlation between these two measurement techniques requires demonstration of reproducible precise and accurate measurement of Young's modulus for both techniques. As a relevant material, nanocomposites of polypropylene, ethylene-propylene-diene-methylene rubber reinforced with graphene nanoplatelets are proposed.

Work Programme

1. Participants will receive protocols for Young's modulus measurements by DMA and stress-strain tests to review and comment on with regards to their usability and technical details.
2. An interlaboratory comparison (ILC) will be performed using the detailed methods and PNCs batches with different nanofiller contents centrally produced by melt extrusion and evaluated for homogeneity of nanofiller dispersion.
3. Data collected from the ILC will be used to evaluate the reproducibility of the measurement methods and the need for additional refinement.
4. Data from successfully reproducible methods will be used to propose inter-relations for measurands by the two methods.

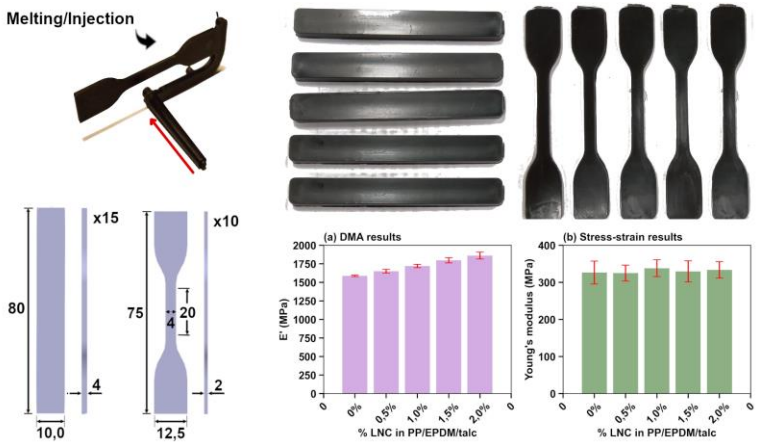

Figure 1: Specimens of PP/EPDM/LNC for mechanical tests.

Deliverables and Dissemination

- A VAMAS technical report and scientific publication describing the developed experimental methods for PNCs characterization by DMA and stress –strain tests are expected.
- ISO TC 229 links aimed at development of an ISO standard.

Funding

Participants fund their own involvement in the project.

Status

Production of a batch of LNC PNCs is currently in progress.

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December 2024

Nanoparticle Populations

Technical Working Area 34

Project 17

Line notation and unique identifiers for nanomaterials and groups of nanomaterials

Objectives

Clear, unambiguous reporting of the identity of a nanomaterial is a complex and not completely solved task. A standardized line notation encoding important physicochemical characteristics will improve this situation. It could replace other suboptimal unique identifiers and provide better machine readability. Specific objectives are:

Identify and agree on a set of characteristics needed to be encoded in the line notation.

Generate a technical specification and software implementation compatible with the chemical line notation InChI and its extensions endorsed by IUPAC.

Test the line notation on a set of diverse nanomaterial classes to guarantee broad applicability but also to define the applicability domain of the identifier.

Background

VAMAS and CODATA jointly developed the Uniform Description System (UDS) for materials at the nanoscale to define minimal reporting guidelines for physicochemical characterizations of nanomaterials. This can be used as the basis for a line notation, which encodes all this information (or parts of it) in a compact form that is easy to extract from different documents, enabling comparisons, supporting searches for specific nanomaterials and corresponding data, and identifying similar materials. A first prototype was published recently as an extension of the InChI.

Standardization needs

The new line notation (NInChI) will improve the UDS by providing a unique identifier for a material or group of materials and, at the same time, a summary of the major characteristics of the material and its provenance. Standards based on the UDS should be updated accordingly.

Work Programme

- Dataset curation to develop sets of real-world nanomaterials libraries to challenge the implementation and coding of the NInChI as much as possible.
- Monthly virtual hackathons with nanomaterials experts and IUPAC NInChI working group experts to develop workable suggestions for how to encode different aspects of nanomaterials descriptors.
- Face-to-face workshops.

Call for Participation

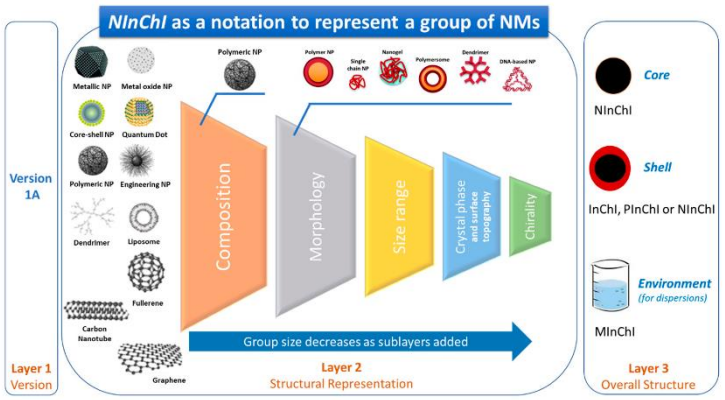

Illustration of the *NInChI*a notation to represent a particular group of NMs (reproduced from Lynch at al., https://doi.org/10.3390/nano10122493)

Deliverables and Dissemination

- Specification of a line notation for nanomaterials (NInChI, multiple development cycles) as extension to the IUPAC International Chemical Identifier (InChI).
- Standard implementation to be used in data management and reporting tools
- Update(s) of the UDS to integrate the NInChI and additional reporting requirements identified during the development of the NInChI.
- Update of ASTM E3144-19 and other standards based on UDS to include NInChIs and other reporting requirements.

International Participation

Current participants include volunteers from countries on all continents. Anyone with expertise in specific nanomaterial classes, standards for nanomaterial characterization, and machine-readable identifiers and representations is welcome.

Funding: Participants fund their own involvement in the project. Organization of workshops can be supported.

Status: The project started in 2022. Additional participants are welcome.

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December 2024

VAMAS

Nanoparticle Populations

Technical Working Area 34

Project 18

Thermogravimetric analysis of carbon nanotube materials

Objectives

1. Evaluate a proposed protocol for thermogravimetric analysis (TGA) of carbon nanotube populations, including for usability, reproducibility, and uncertainty.

2. Demonstrate use of the protocol to establish homogeneity of three different carbon nanotube samples, and determination of the primary oxidation temperature, residual mass, oxidation temperature of each peak(s).

Background

Thermogravimetric analysis (TGA) is an important measurement technique for carbon nanotube (CNT) containing samples, providing a qualitative assessment of thermal stability and homogeneity. TGA can give quantitative measure of the types of carbon species present as well as non-carbon impurities in a material.
This effort will support transition of the current documentary standard for TGA, ISO/TS 11308:2020 "Nanotechnologies - Characterization of carbon nanotube samples using thermogravimetric analysis", from a technical specification to an international standard (IS) in ISO. This standard specifies how to prepare carbon nanotube materials for thermogravimetric analysis, how to perform the measurement, and steps in the data interpretation. An interlaboratory comparison (ILC) study is needed to validate the measurement protocol as presented in this document.

Standardization needs

There is a need for standardized measurements of CNTs by TGA. Validation of published international standards has not been established; this measurement is an essential measurement in the manufacturing environment.

Work Programme

Initial work will be development and evolution of a specific measurement protocol based on ISO/TS 11308 for TGA of CNT materials applicable to multiple sample formats. Experimental ILC round(s) of TGA on provided CNT materials using the supplied measurement protocol will follow.
Provision of three (3) samples, including single-wall and multi-wall carbon nanotube samples is anticipated, along with several material format types, e.g., powder or fibers, to represent a variety of real-world materials. Measurands will include primary oxidation temperature, residual mass, oxidation temperature of other peaks, along with collection of instrumental details, and any variances from the specified procedure. Reported values will be returned for central analysis in a provided spreadsheet. Statistical analysis of the results from participants will be conducted as consistent with ISO/TS 11308.

Call for Participation

Figure: Picture of a TGA instrument

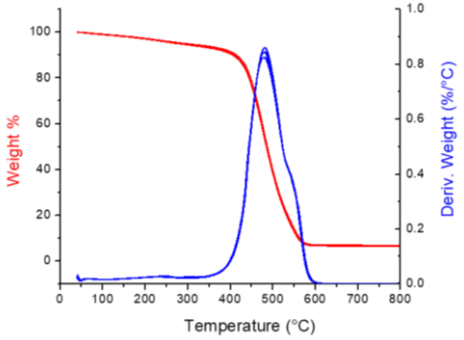

Figure: Example TGA of a single-wall carbon nanotube population

Deliverables and Dissemination

This ILC study will support revision of ISO/TS 11308 to an IS level document. A Peer Reviewed publication of ILC results with statistical analysis of the sample results will also be pursued. Summary of ILC results will also be included in ISO/IS 11308 revision (in progress as of 11/2023) and VAMAS and NIST reports.

Funding: Participants fund their own involvement in the project.

Reference: ISO/TS 11308:2020 "Nanotechnologies— Characterization of carbon nanotube samples using thermogravimetric analysis"

Status: Distribution of methods for comments, samples in Feb. 2025, additional participants welcomed.

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December 2024

VAMAS

Quantitative Microstructural Analysis

Technical Working Area 37

Call for Participation

Project 4

Measurement of dislocation density in metallic materials by Transmission Electron Microscope (TEM)

Objectives

The aim of this international interlaboratory comparison (ILC) is to determine the dislocation density in thin metals by using Transmission Electron Microscope (TEM).

The results will be used directly for further development of the ISO/PWI 13139 “Microbeam analysis - Analytical electron microscopy - Measurement of the dislocation density in thin metals by TEM” with a validated measurement procedure, which will be optimized based on the results gained in this ILC.

Background

The dislocation is the physical nature of the deformation, and the dislocation density in metals is closely related to their strength and toughness. The dislocation density can be measured by TEM *via* an intersections-counting method, which can solve the 3D measurement by a 2D model.

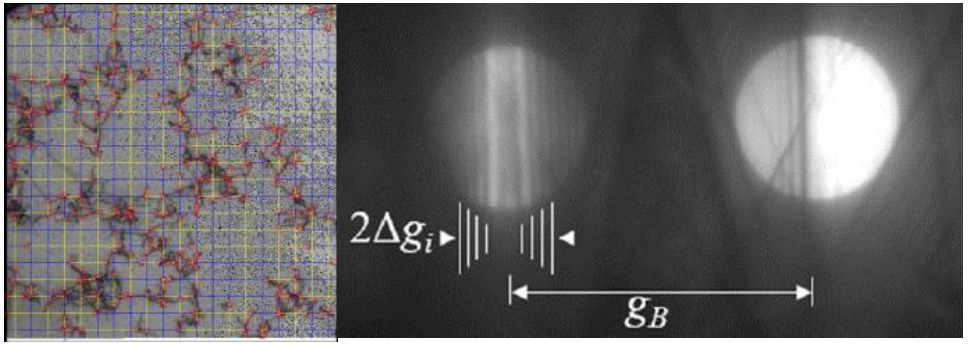
Taking STEM images of a dislocation, counting intersections of a grid on the dislocation image and determining the thickness of the foil are the three key points of this method.

Standardization needs

The TEM method to measure the dislocation is the most direct measurement procedure, and its result can be used as a reference for other indirect methods. A good dislocation image, which shall display as many visible dislocations as possible and have a homogenous background, is the basics of the intersections-counting method and can greatly facilitate the automation of the process of accurate counting. There are many parameters, such as sample condition, imaging method, etc., which will significantly affect the quantitative analysis results of the dislocation density. Manual counting of dislocation intersections is time-consuming and troublesome. An automated counting method is recommended and also specified in the prepared measurement protocols in order to obtain reproducible results. Based on the results gained in this ILC, the standardized measurement of the dislocation density in metals by TEM will be developed and included into the ISO 13139 project.

Relevant Standards Committee:

ISO/TC 202/SC 3 Analytical Electron Microscopy”



Left: Intersections-counting on the dislocation image

Right: Determination of foil thickness by CBED method

Work Programme

The dislocation density in a designated grain in the delivered foil sample will be measured by S/TEM, according to a protocol including the dislocation image acquisition and the foil thickness determination.
Sample will be prepared by the project leadership and provided to each participant.

Deliverables and Dissemination

This interlaboratory comparison will be used to develop ISO 13139 “Microbeam analysis - Analytical electron microscopy - Measurement of the dislocation density in thin metals”.

Funding: Participants fund their own involvement in the project. Estimated at approximately 1 day of effort.

Status: The project started in May 2024, but additional participants are requested and welcomed.

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December 2024

VAMAS

Quantitative Microstructural Analysis

Technical Working Area 37

Project 5

Repeatability of high angular resolution electron backscatter diffraction (HR-EBSD) analysis for elastic strain measurements

Objectives

Compare different analysis methods and software packages on identical experimental data from a Si-SiGe semiconductor candidate reference material with known elastic tetragonal strain.

Background

High angular resolution electron backscatter diffraction (HR-EBSD) is a scanning electron microscopy (SEM) based technique to measure elastic strains and lattice rotations in crystalline materials using image analysis of diffraction patterns. Multiple image analysis methods are available through commercial, open-source and in-house software packages.

HR-EBSD application areas include residual stress and geometrically necessary dislocation density measurements in semiconductor devices and structural materials such as metallic polycrystals where these properties influence the device of materials behaviour.

Standardisation needs

This study will provide pre-standardisation information for a potential new HR-EBSD standard in ISO/TC 202:

- Reproducibility of HR-EBSD analysis using different image analysis methods and software packages;
- Sensitivity of the measurand to tunable calculation parameters within each method.

Measurement methods

Participants will use their own analysis methods on the same set of experimental data - EBSD line-scans acquired from a set of candidate reference materials (RM 8191) for HR-EBSD strain measurement developed by NIST.

Work Programme

HR-EBSD data acquired by NPL and NIST will be sent to participants along with a measurement reporting template.

Participants will measure elastic strain using their own HR-EBSD analysis software and in-house protocol and return the completed measurement report.

Deliverables and Dissemination

We expect the following deliverables:

- Report published by the National Physical Laboratory, and paper submitted to a relevant peer-reviewed journal. Participants and software methods will be anonymised where possible.
- Findings presented at the ISO/TC 202 Plenary meeting in 2025 and other relevant scientific meetings.
- Submission of a draft Technical Specification to ISO/TC 202 Microbeam Analysis.
- A follow-on study based on the results of the current ILC, potentially on an industrially relevant system, such as residual stress measurement of metallic polycrystalline materials.

Call for Participation

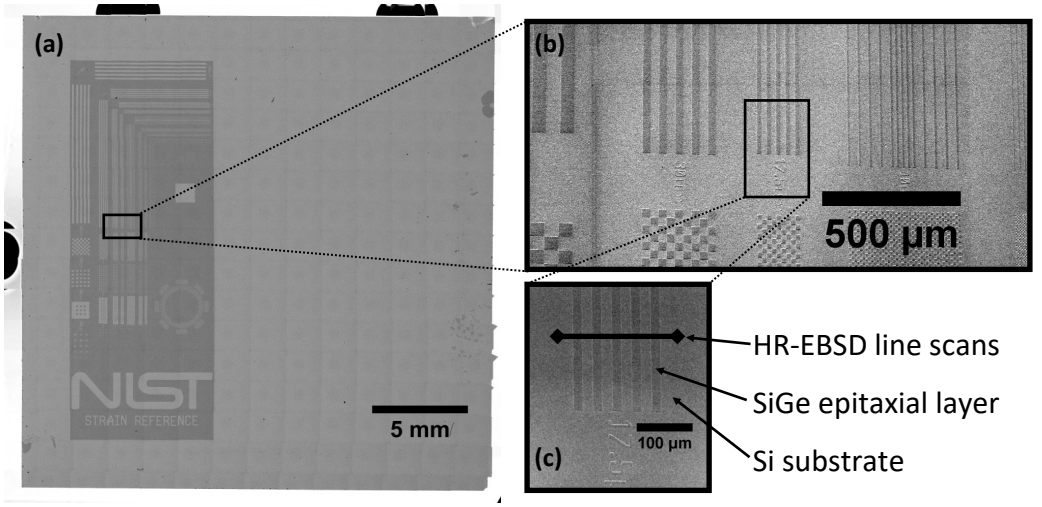

Figure 1 (a) RM8191 Si-SiGe candidate reference material developed at NIST; **(b)** Si-SiGe lines with tetragonal elastic strains; **(c)** HR-EBSD line scan region of interest on 17.5 µm lines.

International Participation

Current participation includes volunteers from countries from multiple continents. Additional participants are welcome to join the ILC.

Participants need to be skilled operators with access to their own HR-EBSD analysis software.

Funding

Participants fund their own involvement in the project.

Project Status

The project is due to start in August 2024 for a duration of 9 months.

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July 2024

VAMAS

Quantitative Microstructural Analysis
Technical Working Area 37

Project 6

Measurement of grain size and distribution of nano-scale metal using Transmission Kikuchi Diffraction (TKD) technique in SEM

Objectives

- Validate the performance of Transmission Kikuchi Diffraction (TKD) technique in a SEM to measure the grain and distribution of 304 austenite steel and pure nickel
- Validate the performance of TKD method in on-axis mode, off-axis mode and other modes.

Background

TKD is a widely used technique for charactering crystallographic orientation and grain size of nanocrystalline and ultra-fine grain materials because of its significantly higher spatial resolution than that of conventional EBSD.
This project will consider spatial resolution of TKD method by measuring grain size of nanocrystalline steel and pure nickel. A unique feature of these samples is that the grain distribution and average grain size can be easily and uniquely identified, which enables traceable characterization with TKD.

Standardisation needs

TKD has received significant interest in recent years, already being applied to a wide range of fields, including nanocrystalline and ultra-fine grain materials, corrosion studies, geological samples, nanostructures and functional materials.
In order to make best use of the TKD technique and the data produced, it is desirable that a common procedure of orientation measurement by TKD method is established worldwide.

Relevant Standards

ISO 13067 Microbeam analysis - Electron backscatter diffraction - Measurement of average grain size.

Deliverables and dissemination

This interlaboratory study will be disseminated at scientific conferences and in a peer-reviewed scientific journal. Further, the key guidelines for orientation measurement by TKD method will be established by ISO TC 202.

Relevant Committees

- ISO/TC 202 Microbeam analysis
- ISO/TC 229 Nanotechnologies

Call for Participation

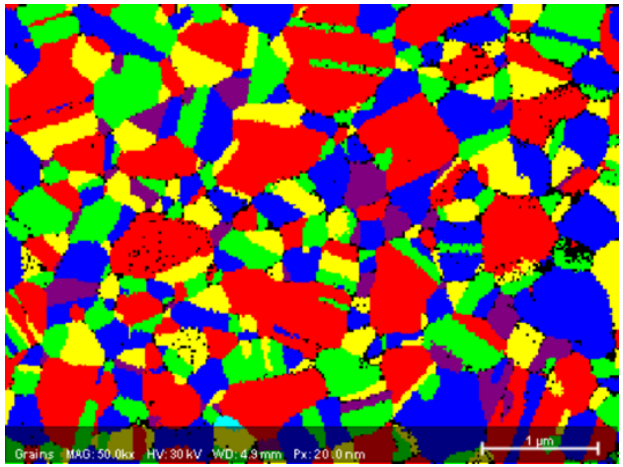

Figure 1: Grain map of pure Ni by on-axis TKD

International Participation

Current participation includes volunteers from countries from multiple continents. Additional participants are welcome.

Work Programme

- Nov 2024: Prepare samples for the interlaboratory comparison (ILC) exercise
- Mar 2025: Measurements by the ILC participants
- Sep 2025: Data analysis, evaluation and reporting
- Mar 2026: Publication in a peer-review journal.

Funding

Participants fund their own involvement in the project.

Project Status

The project is due to start in November 2024 for a duration of 12 months.

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October 2024

Graphene and Related 2D Materials

Technical Working Area 41

VAMAS

Project 6

Specific surface area of a powder containing graphene flakes using Brunauer-Emmett-Teller (BET) method

Objectives

- This project aims to validate the methodology for measuring the specific surface area (SSA) of a powder containing graphene flakes using the Brunauer-Emmett-Teller (BET) method.
- The uncertainties associated with the sample, preparation, measurement and data analysis will be explored.

Background

Recently, graphene has attracted enormous attention due to its unique properties. The SSA of a powder containing graphene flakes is a significant parameter, especially for applications such as energy storage.

However, reliable SSA values can be difficult to obtain for a powder containing graphene flakes using the BET measurement method. This can be because commercially-available graphene can be fabricated through different production routes and therefore exhibit different forms, which have distinct differences in microstructure, including defects and porosity. Other issues are around the adsorption gas, selection of the BET curve, and analysis of measurement data.

Standardization needs

As industry uptake of this material increases, international standardization is critical to enable commercialization. Reliable, accurate and reproducible measurements are important in order to maintain quality, considering that there are multiple production routes and producers of the material.

Several standards are under development within ISO TC 229 and IEC TC 113, focusing on the measurement of key physicochemical or electrical properties of graphene. Measurement of the SSA of graphene is an urgent need as a key physical property.

Work Programme

The sample will be sourced from the industrial collaborators. The specific surface area and pore size distribution will be measured. Different kinds of graphene flakes with different microstructure will be used. The samples will be prepared and delivered to each participating laboratory by the project leader.

Call for Participation

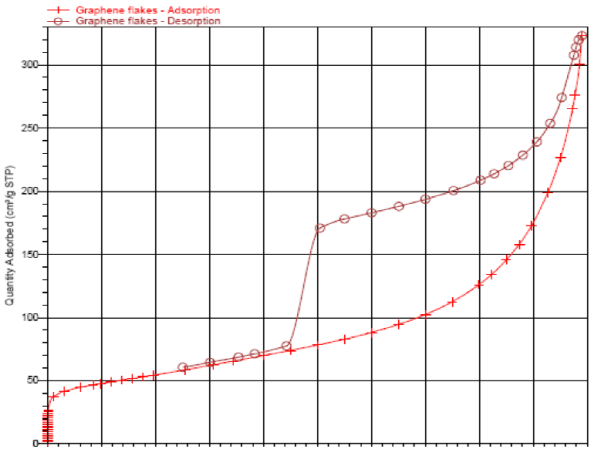

Figure: An example BET isotherm measured for a test graphene flake sample

International Participation

Current participation includes volunteers from Australia, Brazil, China, UK, Korea, Japan and USA. More participants are welcome.

Deliverables and Dissemination

- VAMAS Technical Report
- Publications in peer-reviewed scientific journals
- This study will be used to aid development of standards within ISO TC229 'Nanotechnologies'.

Funding

Participants fund their own involvement in the project.

Status

Call for additional participants

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December 2024

VAMAS

Graphene and Related 2D Materials

Technical Working Area 41

Project 15

The sheet resistivity measurement of the Flexible Fabric Composited by GR2M

Objectives

This project aims to ensure accuracy and consistency of sheet resistivity measurements on flexible fabrics comprised of graphene and related 2D materials (GR2M) across different laboratories. Interlaboratory comparisons will seek to validate measurement methods, assess uncertainties, and develop new measurement standards to promote international trade and enhanced reliability of scientific data.

Background

Flexible fabrics comprised of GR2M are often composite material. Due to the unique physical, chemical, and electrical properties of GR2M, such fabrics are widely applied across fields such as flexible electronics, smart textiles, and wearable devices. Sheet resistivity, i.e., the electrical resistance of a material per unit area, is a key material property for these applications, which has led to a need for accurate and comparable measurement methods specific for these materials.

Standardization needs

There are currently no internationally recognized standards for sheet resistivity measurements on flexible fabrics composited with GR2M. Confidence in determined values and intercomparability of measurements are crucial for achieving commercialization.

Due to the multiple production routes and producers of graphene similar materials, e.g., GaN wafers, reliable, accurate, and repeatable measurement methods are essential to maintain manufacturing quality and promote international business. If methods are determined to produce consistent and repeatable data, they can be considered for future international standardization.

Work Programme

Composite flexible fabrics containing GR2M and measurement methods will be provided for determining sheet resistivity through a combination of electrical measurements.

Homogenous samples will be centrally prepared and distributed to each participant. Data analysis will be conducted by participants, as well as providing raw data for central analysis.

Call for Participation

International Participation

Current participation includes volunteers from Australia, Brazil, China, UK, Korea, Japan and USA. More participants are welcome.

Deliverables and Dissemination

Planned dissemination of developed experimental methods includes production of a VAMAS technical report and publication of results in a scientific journal. An additional intention is to inform potential international standardisation efforts (via ISO TC 229)

Funding

Participants fund their own involvement in the project.

Status

Call for additional participants

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December 2024

VAMAS

Graphene and Related 2D Materials

Technical Work Area 41

Project 16

In-plane Thermal Diffusivity Measurements of Graphene and Related 2D Materials Films

Objectives

This project aims to test a protocol for flash method measurement of the in-plane thermal diffusivity of graphene and related 2D materials (GR2M) films. Results will be used to validate interlaboratory reproducibility and to determine uncertainties associated with the measurement and data analysis.

Background

Due to their ultra-high thermal conductivity, flexibility, light weight, and low-cost, GR2M films are under development or applied worldwide for thermal management in various applications including micro-electronics, integrated circuits, communications, and new energy vehicles. The primary material property for performance evaluation towards such applications is the in-plane thermal diffusivity.

The flash method is considered to be effective for thermal diffusivity measurements of GR2M films, and, as such, is a widespread technique. However, no validated method is available as an international standard, and the degree of interlaboratory variation using a single measurement protocol, arising from sample preparation, test conditions and/or instrument calibration, has not been evaluated.

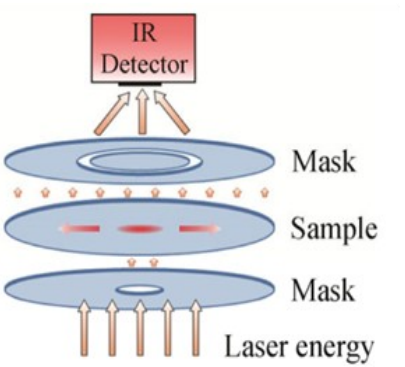
Standardization Needs

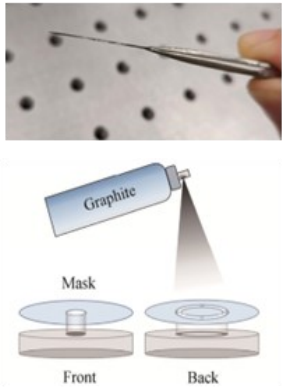
There are no internationally recognized standards at present for in-plane thermal diffusivity measurement of GR2M films using the flash method. Accurate and reproducible measurement methods are important to maintain quality in manufacturing and promote international commerce as there are multiple production routes and suppliers of GR2M materials. Data from this effort will be used to inform potential international standardization.

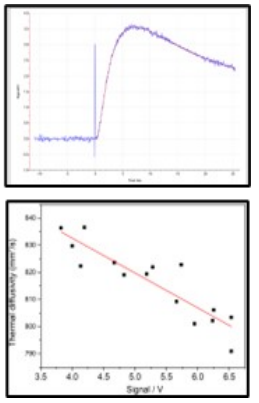
Work Programme

Participants are expected to conduct thermal diffusivity measurements of GR2M films by the flash method using a provided data analysis method. Flexible GR2M films will be centrally prepared and screened for flatness before shipping to participants. Several samples may be provided.

Call for Participation







Measurements are to be based on the protocol, and both analyzed and raw data will be collected.

Deliverables and Dissemination

Results will be included in a VAMAS technical report and in an anticipated scientific journal publication. They may also inform international standardization efforts in ISO TC 229.

International Participation

Current participation includes institutes from Australia, Asia and Europe. More participants are welcome.

Funding

Participants fund their own involvement in the project.

Project Status

The project started in March 2020, but is seeking additional participants. Completion is expected in late 2025.

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December 2024

Thermal Properties

Technical Working Area 43

Project 4

Reliable material characterisation methods for Thermal Protection System (TPS) critical ablative materials

Objectives

Carbon-phenolic materials require very specific methods to determine their specific heat capacity (c_p). Due to their low density, it is difficult to maintain sufficient mass of the sample and contact with the carrier. They also respond with strong internal decomposition by pyrolysis to thermal heating, making their analysis complex, and requiring multiple experiments to investigate the virgin and charred status. A test approach is proposed.

Measurement Techniques

- Differential Scanning Calorimetry (DSC)
- Simultaneous Thermal Analysis (STA / TG-DSC)

Measurement Property

- Specific heat capacity (Cp) from ambient to 1000 °C

Background

Carbon-phenolic ablators are one of the most chosen material candidates for the heat shields of spacecraft. The wide required temperature range and the strong internal pyrolysis processes make a temperature-dependent characterization a difficult task. Specific heat capacity and pyrolysis enthalpies especially are tightly coupled parameters, resulting on low reliability of material-property related data.

A recent Technology Readiness Program (TRP) between the European Space Agency (ESA) and von Karmen Institute (VKI) had the goal to “Test the capabilities, analyse interference sources and propose improvements of carbon-phenolic material characterization techniques for definition of an updated standardized test protocol allowing reliable and repeatable material properties extraction.”

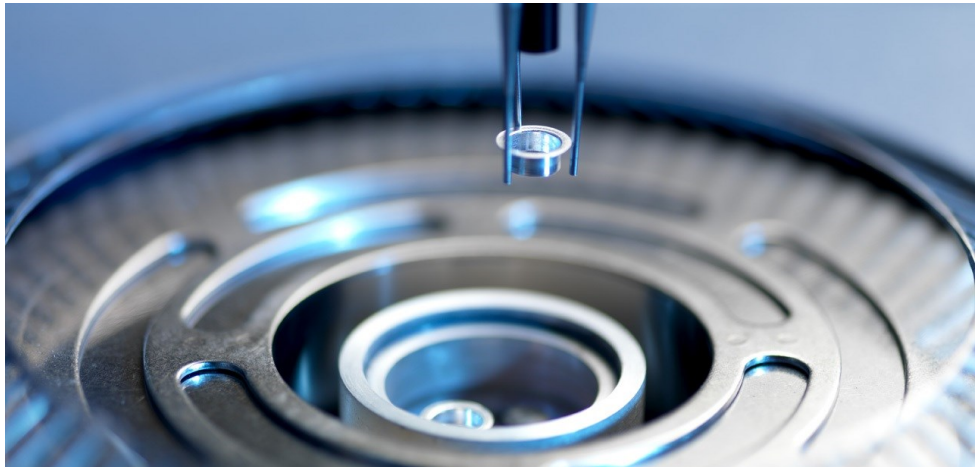
Standardisation Needs

A pre-standardisation method for both DSC and STA measurements for analysis of specific heat capacity c_p and pyrolysis enthalpy h_{pyro} was an outcome of this study. A validation study was conducted involving limited collaborators and now being extended globally.

Work Programme

- Material procurement and sample preparation by VKI

Call for Participation



- Distribution of disc geometry samples (5 mm diameter / 1 mm thickness).
- Data management

Deliverables and Dissemination

- VAMAS Technical Report with refined measurement protocol, and codes of practice for determination of specific heat capacity c_p and enthalpy of pyrolysis h_{pyro} of pyrolyzing carbon-phenolic materials.
- Dissemination through publications, conferences, and presentations at the space agencies.
- Several international research groups exist for detailed simulations of the material response of the materials, with great interest in the research outcome.

Funding

Participants fund their own involvement in the project. Materials for the interlaboratory comparison will be supplied by VKI.

Register your interest

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February 2024

VAMAS

Micro and Nano Plastics in the Environment

Technical Working Area 45

Project 3

An interlaboratory comparison on candidate reference materials to support the standardization of nanoplastic analysis

Objectives

To validate method performance for targeted techniques that measure the size distribution, number concentration and mass fraction of nanoplastic particles.

Measurement Techniques

Size distribution:
- Light Scattering methods including versions hyphenated to fractionation (DLS, MADLS, FFF-MALS)

Number Concentration:
- Particle Tracking Analysis (PTA)

Mass Fraction:
- Thermoanalytical techniques (Py-GC/MS and TED-GC/MS)

Results will support harmonization and pre-standardization, providing data for assessing method repeatability and reproducibility.

Background

Several research needs have been identified by International Organizations as the European Food Safety Agency (EFSA), the Science Advice for Policy by European Academies (SAPEA), and the World Health Organization (WHO). Among these is the development of representative reference materials for plastic particles across various size ranges.

As part of the EURAMET Plastics Trace Project (https://plastictrace.eu/) sub-micron plastic particles and nanoplastics, reflective of common industrial polymers, have been produced and characterized. However, standardized measurement protocols and methodologies for the reliable analysis of the physico-chemical properties of nanoplastics are still absent. For the validation of methods, instrumentation and parameters for nanoplastics, this interlaboratory comparison (ILC) is being organized to evaluate the performance of targeted techniques to measure the size distribution, number concentration and mass fraction of nanoplastic particles.

Standardization needs

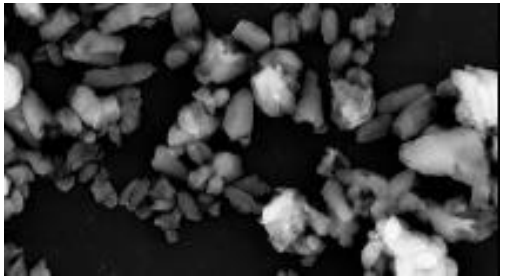
- There is a critical need for validated and standardized protocols for nanoplastics characterization.
- Results from this ILC aim to contribute to new documentary standards in ISO.

Work Programme

- Surfactant-free aqueous suspensions of nano-polypropylene test materials will be distributed in February 2025.
- A minimum of five measurement replicates is demanded.

Call for Participation

PlasticTrace
Tracing Micro and NanoPlastics in Food and Environment



Figures: Morphology of nano-polypropylene test material captured via electron microscopy imaging (left); (Right) a photograph of the surfactant-free aqueous suspension sample.

- Participants will receive SOPs for sample preparation, measurement, and data analysis.
- The time required to carry out the measurements may depend on the technique; 1 week is a rough estimate.
- All participants are asked to return data and other requested information (e.g., quality control details, technical issues encountered, etc.) using the data reporting templates provided by the study organizers.
- Measurement results will be statistically evaluated for repeatability and reproducibility using ISO 5725-2.

Deliverables and Dissemination

This interlaboratory study will be summarized in a VAMAS report, and disseminated at scientific conferences, and in a peer-reviewed scientific journal. Data produced in this ILC may contribute to assignment of reference values to the test material.

Funding: Participants fund their own involvement in the project.

Status: The project will start in February 2025 for a duration of 6 months.

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Register your interest

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January 2025