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**AGREEMENT**

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## Materials characterisation - Terminology, metadata and classification

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## European foreword

This CEN Workshop Agreement (CWA 17815:2021) has been developed in accordance with the CEN-CENELEC Guide 29 “CEN/CENELEC Workshop Agreements – A rapid prototyping to standardization” and with the relevant provisions of CEN/CENELEC Internal Regulations - Part 2. It was approved by a Workshop of representatives of interested parties on 2021-10-01, the constitution of which was supported by CEN following the public call for participation made on 2020-11-05. However, this CEN Workshop Agreement does not necessarily include all relevant stakeholders.

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## Introduction

Materials characterisation involves the identification and measurement of properties that are either intrinsic or manifest in a material. These properties identify the type, manufacturing/process history, and the state of the material. Characterisation allows us to handle, transport, process, engineer and use the material in the intended application. Characterisation methods can be divided into two broad categories; a) those used to identify the nature (structure, chemistry, microstructure, etc) of the material and b) those evaluating material behaviour and/or performance.<sup>1</sup>

The production of materials presents many challenges, particularly in the industrial environment. Real time monitoring of materials during synthesis and processing is desired for obtaining e.g. high precision, small specialty batches, processing monitoring of nucleation/growth at different scales (lab, pilot, production) and potential for feedback loops (adapt T, pH, etcetera). Such latest developments have widened the application domains for materials characterisation and increased its potential impacts to economy and society.

Specific recent domains for advanced characterisation include:

- (a) Off-line characterisation of nanostructures/nanoparticles, where the focus is on detailed analyses of nanostructure/nanoparticle properties that can influence a wide range of material behaviour.
- (b) Real-time material characterisation, where the focus is on obtaining real-time information on (nano)materials properties, delivering multiple benefits (e.g. better process knowledge, and control; reduced development time and waste) in materials manufacturing processes by providing real-time information.
- (c) In-line material characterisation, where the characterisation is performed in the production line, and the focus can be for example on:
  - detailed analyses of (nano) particle properties directly in the flowing media and/or process flows
  - analysis of the optical, electronic, structural properties and/or thickness of vacuum deposited or printed nanolayers, for long timescales and/or for large sample areas.
- (d) Multi-technique and/or multimodal characterisation, where multiple advanced methods are combined to achieve complex information on material's behaviour and performance.
- (e) Operando characterisation, where characterisation of the material is performed during its operation, and its performance is simultaneously evaluated.

As a consequence, communication becomes critical of the characterization tool to the process control of the material fabrication equipment, to enable improved process control and defect detection.

It has been demonstrated in many individual cases that materials characterisation is a key enabler of research & development efficiency and innovation and that the use of this technology can generate a huge economic impact.

Due to the huge variety and complexity of materials and the wide range of applications, the materials characterisation field consists of many communities. These communities have established different terminologies, which typically focus on specific application domains and on types of characterisation methods. As a result, a wide range of domain specific characterisation methods have evolved. However, applications to industrial problems in advanced materials and nanotechnology require a strong interdisciplinary approach among these fields and communities, especially when profound interaction

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<sup>1</sup> From 2017 EMCC roadmap, [www.characterisation.eu](http://www.characterisation.eu)

with material modelling is needed. There is therefore a need to establish a common terminology (definition of concepts and vocabulary) in materials characterisation, to arrive at a common structure of materials characterisation metadata.

A standardised terminology will improve future exchanges among experts in the entire area of materials characterisation, facilitate the exchange with industrial end-users, experimentalists, as well as computational modellers, and reduce the barrier utilising advanced materials characterisation. The common language is expected to foster dialogue and mutual understanding between industrial end-users, equipment manufacturers, scientists, and theoreticians. Standardization of terminology and classification has been identified as critical to collaboration and dissemination of European research projects. In particular, standards will facilitate interoperability between methods and databases. The standardization is relevant for an integrated technological development and brings benefits for industrial end-users due to simplified and much more efficient communication in the field of materials characterisation.

The terminology, metadata and standardised documentation helps data interpreters by translating industrial problems into problems that can be analysed with characterisation methods. It assists workflow development where several methods can interoperate in addressing a specific end-user question.

In the future, this standardized terminology and metadata classification can be formalised into a taxonomy and an ontology of materials characterisation. Such an ontology will form the basis for formal metadata development with which methods and databases can be linked. These developments will further support efficient solutions for materials characterisation and the communication, dissemination, storage, retrieval, and mining of data about materials characterisation and increases the interoperability.

## 1 Scope

The main purpose of this CWA is to propose a widely agreed and common basic architecture for materials characterization data (CHADA), which can be used as a building block for the most complex characterization case studies, also comprising interactions with modelling<sup>2</sup> and process workflows.

The materials characterization field consists of a merge combination of process from multiple scientific communities, which have established different terminologies that focus on various application domains and types of characterization methods.

In addition to that, characterisation is an integral part of materials and product development, processing, and application. It involves stakeholders from:

- Industrial end-users of characterisation from processing and manufacturing industry.
- Scientists in both academia and industry developing new materials, properties, and applications;
- Technology integrators providing materials testing, multiscale analysis, characterisation, and consultancy services.
- Scientists from Academia, Research Institutes and instrument manufacturers who develop characterisation methods and methodologies.
- Manufacturers and developers of analytical instruments from both academia and industry.
- Standardisation Bodies and Metrology institutes.

For such reasons, the development of a common terminology for materials characterisation is mandatory and should include (a) Standardization of terminology and method classification, and (b) a guideline to translate industrial problems into problems that can be analysed with characterization methods.

On this basis, standardized terminology metadata and classification will be formalized into taxonomies and ontologies of materials characterization.

This CWA includes definitions of fundamental terms for the field of materials characterisation, followed by a detailed description of a new concept for data documentation in materials characterization (CHADA).

The definitions also enable a classification of materials characterisation methods.

Using the concepts of “user case” (which includes the sampling process, specimen, testing environment and characteristic length scale), “experiment” (which include the details of the measurement chain), “raw data” and “data processing”, we propose a more straightforward classification of materials characterisation methods, replacing the current situation of opacity that makes the field hard to access for industry and outsiders.

This CWA also provides a systematic description and documentation of methods including the user case, method, raw data generation and analysis and post-processing of data: the “materials CHAracterisation DAta” (CHADA). This document seeks to organize the information so that even complex characterisation workflows can be conveyed more easily and key data about the methods, raw data generation and analysis, and post-processing of data and their implementation can be captured. A template CHADA for the methods is described to guide users towards a complete documentation of material characterisation.

## 2 Normative references

There are no normative references in this document.

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<sup>2</sup> [www.emmc.eu](http://www.emmc.eu), MODA

### 3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <http://www.electropedia.org/>

#### 3.1

##### **Reference material and Certified Reference Material**

Material, sufficiently homogeneous and stable with respect to one or more specified properties, which has been established to be fit for its intended use in a measurement process”.

Another definition of a CRM is “Quality control sample used to determine accuracy and precision of method”.

[ISO 17858:2007].

#### 3.2

##### **Material characterization**

Broad and general process of collecting existing information about a material’s chemistry, structure, and other properties, and if appropriate, new data, to facilitate the evaluation of these properties

[EN ISO 10993-1].

#### 3.3

##### **Stage**

Temporal part of a Process that typically operates independently from other process stages.

EXAMPLE 1 to entry: Characterisation Experiment, Data Processing are stages of the Materials characterization process.

[SOURCE: adapted from ISA-88 (S88) Batch Control Explained]

#### 3.4

##### **Operation**

Temporal part of a Stage that represents major processing activities

EXAMPLE 1 to entry: Selection of test location

[SOURCE: adapted from ISA-88 (S88) Batch Control Explained]

#### 3.5

##### **Action**

Lowest level of the Process specifying individual activities needed to complete the operations.

EXAMPLE 1 to entry: Moving probe to test location

[SOURCE: adapted from ISA-88 (S88) Batch Control Explained]

### **3.6**

#### **Nanomaterial**

Material with any external dimension in the nanoscale (2.1) or having internal structure or surface structure in the nanoscale

Note 1 to entry: This generic term is inclusive of nano-object (2.5) and nanostructured material (2.7).

Note 2 to entry: See also definitions 2.8 to 2.10.

[ISO/TS 80004-1:2015, term 2.4.]

### **3.7**

#### **Quantity**

Property of a phenomenon, body, or substance, where the property has a magnitude that can be expressed as a number and a reference [SOURCE: ISO/IEC Guide 99:2007, 1.1.].

Note 1 to entry: A physics quantity is a physical property of a phenomenon, body, or substance that can be quantified by measurement.

Note 2 to entry: The behaviour of an entity material can be described by more than one physics quantity (e.g., mass, charge, velocity, and temperature).

### **3.8**

#### **Production batch (LOT)**

Definite amount of material produced during a single manufacturing cycle and intended to have uniform character and quality.

Note 1 to entry: The uniform conditions of manufacture or production of the batch or LOT must be such as to ensure a homogeneous product (or at least representative of the existing scatter in the materials properties). In statistics, an entire batch may be considered a finite population (totality of items under consideration).

### **3.9**

#### **Sampling process**

Act of extracting a portion (amount) of material from a larger quantity of material. This operation results in obtaining a sample representative of the batch with respect to the property or properties being investigated.

Note 1 to entry: The term can be used to cover either a unit of supply or a portion for analysis. The portion taken may consist of one or more sub-samples and the batch may be the population from which the sample is taken.

### **3.10**

#### **Sample**

Portion of material selected from a larger quantity of material. The term needs to be qualified, e.g., bulk sample, representative sample, primary sample, bulked sample, test sample, etc. The term 'sample' implies the existence of a sampling error, i.e., the results obtained on the portions taken are only estimates of the concentration of a constituent or the quantity of a property present in the parent material. If there is no or negligible sampling error, the portion removed is a test portion, aliquot, or specimen.

[Sources: PAC, 1989, 61, 1657. (Nomenclature for automated and mechanised analysis (Recommendations 1989)) on page 1660]

Note 1 to entry: The concept of “sample” also includes the structural and microstructural features that will be present in the interaction volume.

Note 2 to entry: In some cases, the sample volume can be smaller than the volume of the physical specimen being investigated.

### 3.11

#### **Specimen**

Portion taken under conditions such that the sampling variability cannot be assessed (usually because the population is changing), and is assumed, for convenience, to be zero.

[PAC, 1990, 62, 1193. (Nomenclature for sampling in analytical chemistry (Recommendations 1990)) on page 1202].

Note 1 to entry: The manner of selection of the sample should be prescribed in a sampling plan.

Note 2 to entry: Specimen can also be the result of a series of sample preparation processes (e.g., machining, polishing, cutting to size, etc.) before actual observation and measurement.

### 3.12

#### **Hazard**

Set of inherent properties of a substance, mixture of substances, or a process involving substances that, under production, usage, or disposal conditions, make it capable of causing adverse effects to organisms or the environment, depending on the degree of exposure; in other words, it is a source of danger.

[PAC, 2004, 76, 1033. (Glossary of terms used in toxicokinetics (IUPAC Recommendations 2003)) on page 1054].

### 3.13

#### **Characterisation environment**

Medium of the characterisation experiment defined by the set of environmental conditions that are controlled and measured over time during the experiment.

Note 1 to entry: A test described as “conducted at ambient conditions” was performed at whatever conditions were prevailing at that time on that day.

[ISO/IEC 29197:2015]

EXAMPLE 2 to entry: Characterisation can either be made in air (ambient conditions, without specific controls on environmental parameters), or at different temperatures, different pressures (or in vacuum), or using different types of working gases (inert or reactive with respect to sample), different levels of humidity, etc.

### 3.14

#### **Experimental plan**

Higher level planning activity for the experimental activity, including the selection of the most appropriate technique to assess the desired property or performance in the material.

### 3.15

#### Test plan

General testing methodology established to ensure that a system meets requirements.

Note 1 to entry: Test Plan can also refer to the collection of protocols or qualifications used to test and document that a system meets requirements.

### 3.16

#### Validation Protocol

A collection of Test plans, used to document the validation of a system

### 3.17

#### Probe

Probe is the physical tool (i.e., a disturbance, primary solicitation, or a gadget), controlled over time, that generate measurable fields that interact with the sample to acquire information on sample's behaviour and properties.

EXAMPLE 1 to entry: In x-ray diffraction, the probe is a beam of x-rays with known energy that is properly focused on the sample's surface with a well-defined geometry.

EXAMPLE 2 to entry: In electron microscopy (SEM or TEM), the probe is a beam of electrons with known energy that is focused (and scanned) on the sample's surface with a well-defined beam-size and scanning algorithm.

EXAMPLE 3 to entry: In mechanical testing, the probe is a force actuator that is designed to apply a force over-time on a sample. Many variants can be defined depending on way the force is applied (tensile/compressive uniaxial tests, bending test, indentation test) and its variation with time (static tests, dynamic/cyclic tests, impact tests, etc...)

EXAMPLE 4 to entry: In dynamic light scattering, temporal fluctuations of backscattered light due to Brownian motion and flow of nanoparticles are the probe, resolved as function of pathlength in the sample. From fluctuation analysis (intensity correlations) and the wavelength of light in the medium, the (distribution of) diffusion coefficient(s) can be measured during flow. The Stokes-Einstein relation yields the particle size characteristics.

EXAMPLE 5 to entry: In spectroscopic methods, the probe is a beam of light with pre-defined energy (for example in the case of laser beam for Raman measurements) or pre-defined polarization (for example in the case of light beam for Spectroscopic Ellipsometry methods), that will be properly focused on the sample's surface with a well-defined geometry (specific angle of incidence).

### 3.18

#### Physics of interaction

Set of physics principles (and associated governing equations) that describes the interaction between the sample and the probe.

Note 1 to entry: A characteristic "interaction" volume is usually associated to the process of sample/probe interaction.

EXAMPLE 1 to entry: In x-ray diffraction, this is represented by the set of physics equations that describe the relation between the incident x-ray beam and the diffracted beam (the most simple form for this being the Bragg's law).

### 3.19

#### Interaction Volume

The volume of material, and the surrounding environment, that interacts with the probe and generate a detectable (measurable) signal (information).

Note 1 to entry: It is important to note that, in some cases, the **volume of interaction** could be different from the **volume of detectable signal emission**. Example: in Scanning Electron Microscopy (SEM), the volume of interaction between the electron probe and the material is different from the volumes that generate the captured signal.

Note 2 to entry: In some cases, (like tribological characterisations) the “sample” can also be the “probe”. When analysing a system of samples that interact each other, finding a clear definition can become a complex problem.

EXAMPLE 1 to entry: In x-ray diffraction, the interaction volume is the volume of material that interacts directly with the x-ray beam and is usually smaller than the volume of the entire specimen. Depending on sample’s structure and microstructure, the interaction between the sample and the x-ray incident beam generates a secondary (reflected) beam that is measured by a detector and contains information on certain sample’s properties (e.g., crystallographic structure, phase composition, grain size, residual stress, ...).

EXAMPLE 2 to entry: In Scanning Electron Microscopy (SEM), the interaction volume is the volume of material that interact directly with the incident electron beam, is usually much smaller than the entire specimen’s volume, and can be computed by using proper models. The interaction between the scanning probe and the sample generates a series of detectable signals (back scattered electrons, secondary electrons, x-rays, specimen current, etc.) which contain information on sample morphology, microstructure, composition, ...

### 3.20

#### **Representative Volume Element (RVE)**

RVE is the smallest material volume over which a measurement can be made, to achieve a representative (meaningful) value of a desired property for the material under investigation.

Note 1 to entry: The quantification of RVE can be different depending on the nature of the material, its microstructure, and the specific application.

Note 2 to entry: The selection of a specific characterisation technique for a material should be done by considering that sample volume (see 3.1) should be similar to the selected RVE for that material.

EXAMPLE 1 to entry: in case of thin film testing, the RVE usually (but not always) should include the entire film thickness. This makes unsuitable those techniques whose characteristic volume of interaction is much larger than film thickness.

### 3.21

#### **Signal (Probe-Material interaction effect - physical process for signal emission)**

result (effect) of the interaction between the sample and the probe, which usually is a measurable and quantifiable quantity.

According to UPAC Compendium of Chemical Terminology, a “signal” is “A representation of a quantity within an analytical instrument” (<https://goldbook.iupac.org/terms/view/S05661> ).

Note 1 to entry: Signal is usually emitted from a characteristic “emission” volume, which can be different from the sample/probe “interaction” volume and can be usually quantified using proper physics equations and/or modelling of the interaction mechanisms.

### 3.22

#### **Detector (physical process for signal detection)**

Physical device (or the chain of devices) that is used to measure, quantify and store the signal after its interaction with the sample.

EXAMPLE 1 to entry: Back Scattered Electrons (BSE) and Secondary Electrons (SE) detectors for SEM

EXAMPLE 2 to entry: Displacement and force sensors for mechanical testing

### 3.23

#### Noise

The components associated to the measured signal, which are not generated by the physical interaction induced by the probe inherently with the desired characterization process.

The set of all unwanted modifications of the signal (or even additional undesired signals) by external environmental conditions (for example, light, vibrations, acoustic noise, electrical signals, atmospheric, chemical, etc), that are present during the experiment.

According to UPAC Compendium of Chemical Terminology, noise is the “The random fluctuations occurring in a signal that are inherent in the combination of instrument and method” (<https://goldbook.iupac.org/terms/view/N04175>).

Note 1 to entry: noise may be generated during all steps of signal acquisition and processing.

Note 2 to entry: a possible way to evaluate noise(s) is to perform a Fast Fourier Transform (FFT) on the raw data signal(s) Reference would be required

### 3.24

#### Data levels

It is possible to use the following classification for data associated with the data processing level:

#### 3.24.1

##### Raw data

what we get from the equipment with the manufacturer’s software including automatic pre-processing that is not modified by the user once the acquisition method is defined and the equipment calibrated. The raw data is a set of (unprocessed) data that is given directly as output from the detector, usually expressed as a function of time or position, or photon energy.

#### 3.24.2

##### Primary data

after pre-processing for corrections to normalize/harmonize (i.e., x and y axes correction, baseline subtraction, noise reduction, etc.)

#### 3.24.3

##### Secondary or elaborated data

after (post)processing or model generation (i.e., intensity maps, deconvoluted curves, etc.)

#### 3.24.4

##### Property

after classification, quantification, (manually or by a model) etc.

Note 1 to entry: According to ISO 20397-2:2021(en), 3.21, raw is the primary sequencing data produced by a sequencer without involving any software-based pre-filtering for analysis purpose.

EXAMPLE 1 to entry: In some cases, raw data can be considered to have already some level of data processing, e.g., in electron microscopy a “raw image” that is formed on the screen is already result from multiple processing after the signal is acquired by the detector.

EXAMPLE 2 to entry: In mechanical testing, the raw data are the raw-force and raw-displacement, as function of time. In spectroscopic testing, the raw data are light intensity, or refractive index, or optical absorption as a function of the energy (or wavelength) of the incident light beam.

**3.25****Calibration workflow (process)**

Sequence of operations/actions that are needed to convert the initial signal (primary data, as produced by the detector) into a meaningful and useable raw data.

Note 1 to entry: Within this workflow, a possible way is to use a reference sample (with pre-defined, specific, and stable physical characteristics and known properties) for the interaction with the measurement probe, in order to extract characterization data that will be compared to predefined values. In this way, the accuracy of the measurement tool and its components (for example the probe) will be evaluated and confirmed.

EXAMPLE 1 to entry: In nanoindentation, the electrical signal coming from capacitive displacement gauge is converted into a real raw-displacement signal after using a proper calibration function (as obtained by the equipment manufacturer). Then, additional calibration procedures are applied to define the point of initial contact and to correct for instrument compliance, thermal drift, and indenter area function to obtain the real useable displacement data.

**3.26****Data normalisation**

Data normalization involves adjusting raw data to a notionally common scale.

NOTE 1 to entry: It involves the creation of shifted and/or scaled versions of the values to allow post-processing in a way that eliminates the effects of influences on subsequent properties extraction.

**3.27****Data post-processing**

Analysis, that allows one to calculate the final material property from the calibrated primary data.

EXAMPLE 1 to entry: In nanoindentation testing, this is the Oliver-Pharr method, which allows calculating the elastic modulus and hardness of the sample by using the load and depth measured signals.

EXAMPLE 2 to entry: Analysis of SEM (or optical) images to gain additional information (image filtering/integration/averaging, microstructural analysis, grain size evaluation, Digital Image Correlation procedures, etc.)

**3.28****User (or End User)**

User concept includes all the information required to track the people who have physically made the experiment (operator name, level of expertise, ...), and those steps of the experiment which are more sensitive to human errors.

Note 1 to entry: more than one operator (user) could be associated to a single experiment, e.g., for those cases where the measurement of raw data is made by a person (technician, student, etc.) and the analysis of data is made by another person.

**3.29****System manufacturer**

Company that is responsible for manufacturing of a measurement device or characterisation instrument.

**3.30****System owner**

Individual who is ultimately responsible for the analytical system.

### **3.31**

#### **Measurement**

A specific operation that results in the determination of values (e.g. coordinate values, length values, false/true values)

[SOURCE: ISO 10360-1:2000, 2.7 — modified.]

### **3.32**

#### **Characterisation experiment**

Single and complete set of operations and actions, performed using instrumentation and defined in the metadata structure, to arrive at the raw data. It can be considered as a more complex form of measurement.

### **3.33**

#### **Characterisation method**

Characterisation method is the combination of the experiment and the associated experimental plan, pre-processing and post-processing activities, to arrive at a measured material structure/property.

### **3.34**

#### **Characterisation workflow**

Combination of more than one characterisation method to form the entire workflow that is needed to describe the desired materials behaviour and/or performance.

Example 1: mechanical testing can be combined “in-situ” with microscopy and spectroscopy tools to develop multi-technique characterisation workflow.

### **3.35**

#### **Survey sampling**

Process of selecting a sample (3.1.8) of sampling units (3.1.5) from a target population (3.1.1) to obtain information regarding the characteristics of the items in the population

Note 1 to entry: Survey sampling in every day usage often implicitly suggests the methodology and can refer to the general field of expertise associated with investigating finite populations (3.1.2) via questionnaires, opinion surveys (3.1.23), political polls, and customer satisfaction surveys.

Note 2 to entry: This definition differs from the definition (sampling used in enumerative or analytic studies to estimate the values of one or more characteristics in a population or for estimating how those characteristics are distributed across the population) given in ISO 3534-2:2006, 1.2.18, which emphasizes the objective of survey sampling rather than the process itself as is given here.

[SOURCE ISO 3534-4:2014(en) Statistics — Vocabulary and symbols — Part 4]

### **3.36**

#### **Chada**

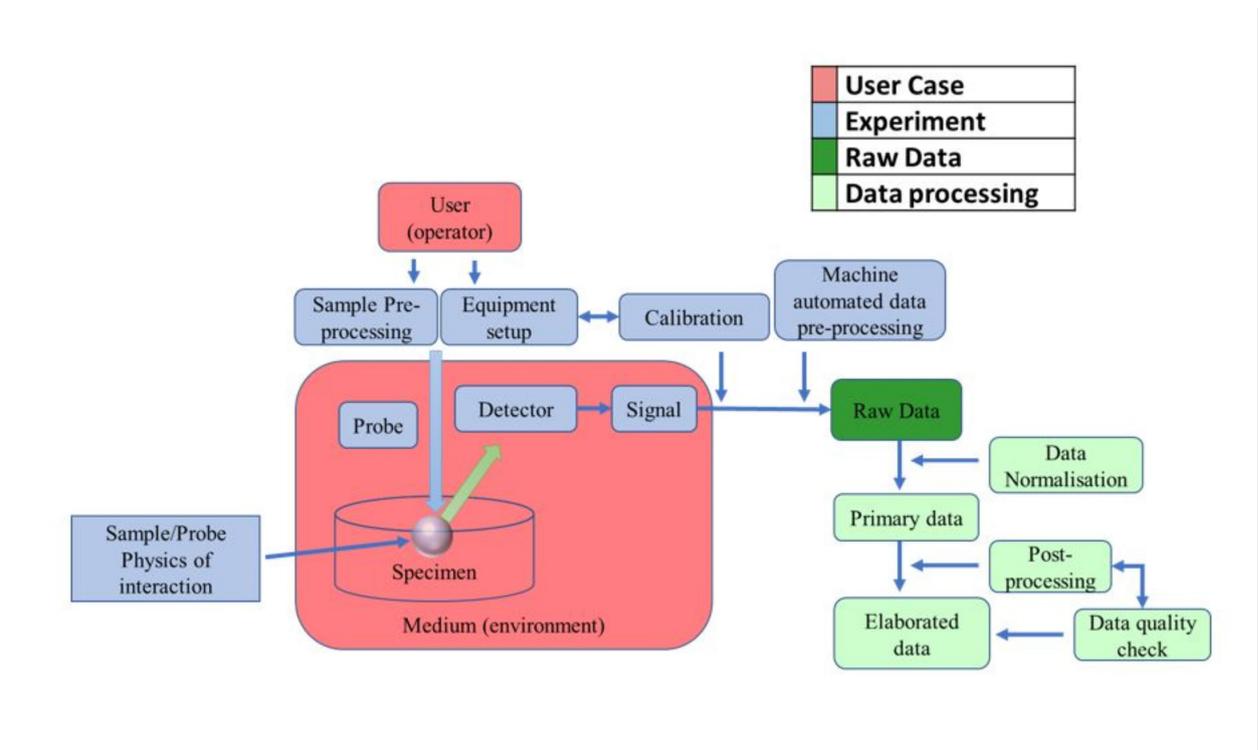
Materials CHAracterisation DAta documentation, according to description in the following chapters.

## **4 Materials characterisation data (chada)**

### **4.1 Documentation of a characterisation method**

The general scheme in figure 1 represents all essential elements for a characterisation method (in some cases, multiple methods could be connected to achieve a more complex evaluation of material's behaviour). This scheme also represents the main structure of CHADA.

Note: The CHADA relates to one experiment for a particular user case (and does not necessarily describe the wide capabilities of the method).



**Figure 1 — Visual representation of a general characterisation method with keywords (and colours)**

The first step towards this goal is the definition of the terminology associated to materials' characterization methods. We propose that only four types of concepts are used for the classifications of the different steps of an entire characterisation workflow (which can be simply called "characterisation"):

1. **User case** (which also include the sample and the information on environment of testing), which represents *volume of probed material*, and the information on the surrounding *environment*, which interacts with the probe and generate a detectable (measurable) *signal* (information);
2. **Experiment**, which represents the process stage (**sequence of operation/actions**) by which the metrological chain is defined; within a single experiment, the following fundamental elements are identified: probe, signal, detector, noise (according to previous definitions);
3. **Raw data**, is the set of data that is given directly as output from the metrological chain, usually expressed as a function of time, position, or photon energy; the different data levels as defined before should be considered here.
4. **Data processing**, which represents the process stage (sequence of operations/actions) by which the data are analysed to arrive to the final shape.

By using this simplified approach, a generic characterization method can be documented by the schemes reported in the presentation, which can be used for the construction of the metadata structure of any generic material's characterisation process.

Clearly, modifications of such flow are possible, depending on the specific application and/or user needs. Similarly, different calibration steps and/or different data processing steps can be defined.

On this basis, the following templates for CHADA characterisation methods can be adopted:

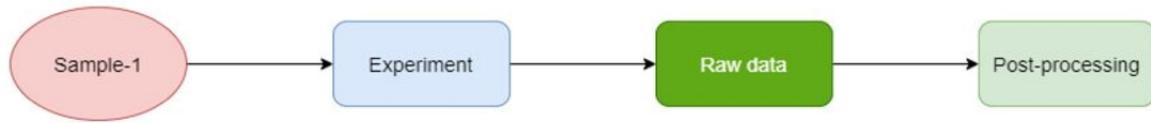


Figure 2a — User case

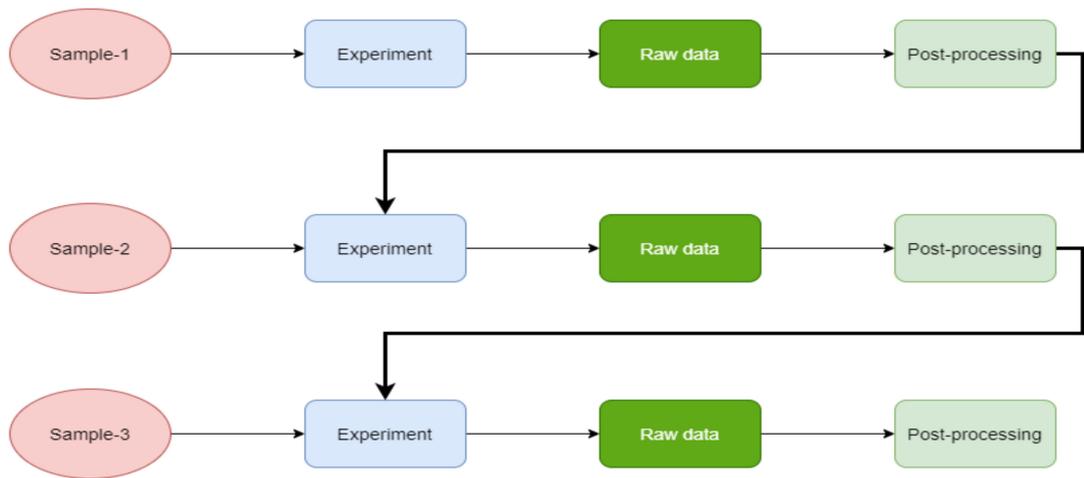


Figure 2b — Experiment



Figure 2c — Raw data

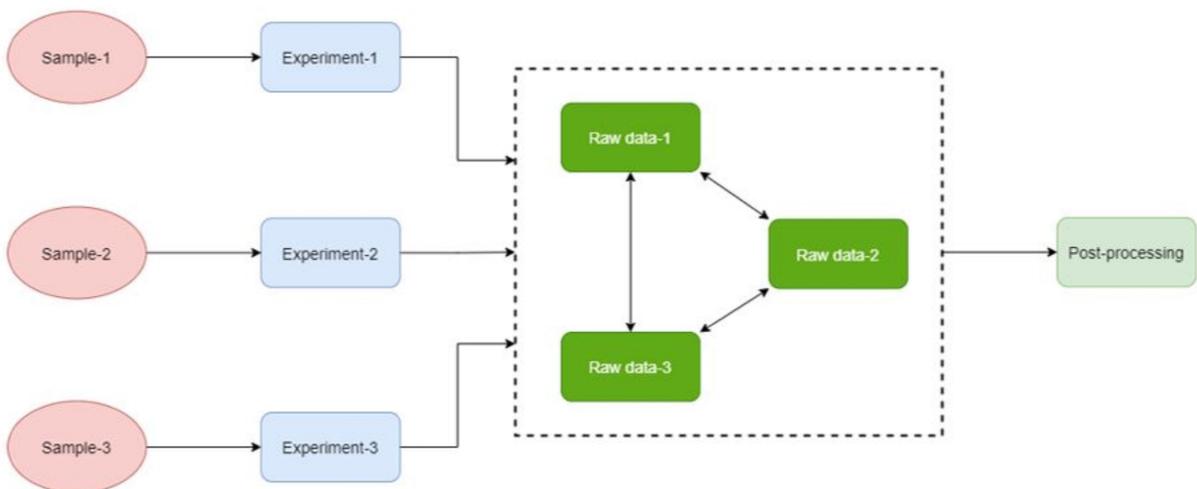


Figure 2d — Data processing

## 4.2 Possible approach for classification of characterisation experiments

### 4.2.1 General

Based on the terms and definition, materials characterisation methods can be classified, at the top level, by the combination of (a) the nature of the **signal** that is analysed , and (b) the nature of the **probe** that is used for interacting with the sample.

After defining this, the nature of (a) the emitted signal and (b) the raw data that are generated are consequently (and uniquely) defined.

### 4.2.2 Examples

1. In conventional x-ray **diffraction**, the **probe** is a beam of x-rays with known energy that is properly focused on the sample's surface with a well-defined geometry, **AND** the resulting **signal** (after interaction with the sample) is a scattered x-ray beam that is detected as a function of the diffraction angle.

2. In scanning electron **microscopy** with back-scattered electrons (SEM-BSE), the probe is a beam of electrons with known energy that is focused (and scanned) on the sample's surface with a well-defined beam-size and scanning algorithm, **AND** the resulting **signal** (after interaction with the sample) is an image that is formed after collection of the emitted back-scattered electrons from the sample.

3. In RAMAN **spectroscopy**, the **probe** is a beam of light with pre-defined energy, which is properly focused on the sample's surface with a well-defined geometry (specific angle of incidence), **AND** the resulting **signal** (after interaction with the sample) is the measured intensity of scattered light plotted vs. energy.

4. In mechanical testing, the **probe** is a force actuator that is designed to apply a force over-time on a sample, and the **AND** the resulting **signal** (after interaction with the sample) is the measured displacement (with many possible variants depending on the specific geometry of testing and nature of the displacement gages).

Using this approach, and having the new CHADA scheme as a reference, an example of simple (not yet exhaustive) classification can be proposed as follows:

- Mechanical testing methods
  - o Macroscopic scale mechanical testing (tensile, compressive, torsional, creep, fatigue, toughness and hardness)
  - o Micro and nano-indentation
  - o In-situ micro/nano-mechanical testing
- Microscopy based methods
  - o Optical microscopy
  - o Electron microscopy
  - o Scanning probe microscopy
  - o Focused Ion Beam microscopy
  - o Scanning probe microscopy
- Spectroscopy based methods
  - o Raman spectroscopy
  - o Infrared and near-infrared spectroscopy
  - o UV and visible spectroscopy
  - o Spectroscopic Ellipsometry
  - o Mass spectroscopy
  - o Nuclear magnetic resonance
  - o X-ray spectroscopy
  - o Atomic emission spectroscopy

- o Ultrasound spectroscopy
- o Energy dispersive spectroscopy
- Diffraction based methods
  - o X-ray diffraction
  - o Neutron diffraction
  - o Electron diffraction
- Light scattering techniques for nanoparticle analysis
  - o Laser diffraction
  - o Dynamic light scattering
  - o Nanoparticle Tracking Analysis
  - o Static Light Scattering
- Tribological characterisation
- Thermal analysis methods
  - o Differential thermal analysis (DTA)
  - o Dielectric thermal analysis (DEA, DETA)
  - o Thermogravimetric analysis (TGA)
  - o Differential scanning calorimetry (DSC)
- Electrical characterisation methods
- Magnetic characterisation methods

#### Documentation of characterisation

According to the CHADA defined in previous chapters, we can propose more detailed documentation forms that can be used to build the metadata describing a general characterisation experiment. Detailed documentation forms should be structured as described in the following point (4.3)

## 4.3 Overview of the characterisation

1	User Case	<i>General description of the sample AND the testing environment,...</i>
2	<b>Characterisation method</b>	<p><i>Experiment/Operation 1. Please identify the first method used. Most characterisation processes should consist of only one method. However, the user has the possibility to describe a characterisation that includes multiple chained methods (e.g. mass spectrometry followed by gas chromatography)</i></p> <p><i>Experiment/Operation 2</i></p> <p>...</p>
3	<b>Validation of Characterisation</b>	<i>Describe why the characterization method was chosen and deemed to be the most useful for the sample. Evidence this with a peer reviewed paper/article.</i>
4	<b>Access conditions (what is needed to repeat the experiment)</b>	<p><i>Was the access to your sample preparation an in-house routine or required a 3<sup>rd</sup> party service?</i></p> <p><i>Was the access to your characterisation tool an in-house routine or required a 3<sup>rd</sup> party service? In case of national or international facilities such as synchrotrons describe the programme that enabled you to access these.</i></p> <p><i>If applicable: Is your post-processing software open-source or commercial?</i></p>

## 4.4 Main body of the Chada

1. USER CASE		
1.1	<b>USER</b>	<i>Describe the user (level of expertise) and the level of automation of the test</i>
1.2	<b>Sample</b>	<i>Describe the sample specifications: dimensions, surface conditions, nature bulk material, coating, heterogeneous material, biomaterial, etc.).</i>
1.3	<b>Sample material properties</b>	<i>Main properties of the sample material under investigation – chemical composition – metal/ceramic/polymer/natural/composite – microstructure.</i>
1.4	<b>Sampling process</b>	<i>How the sample was extracted from the batch.</i>
1.5	<b>Sample preparation</b>	<i>Describe the sample preparation procedures, sample holder, if the sample amount and/or quality was within or below the threshold for the characterisation method.</i>
1.6	<b>Hazard</b>	<i>Describe the inherent properties of the sample that can cause adverse effects during handling</i>
1.7	<b>Characterisation environment</b>	<i>Describe the environment of the experiment (temperature, pressure, working environment – in air, controlled pressure, or vacuum – humidity, noise, vibrations)</i>

<b>2. EXPERIMENT</b>		
<b>Interaction nature and character (destructive or non-destructive) of the probe with the sample</b>		
<b>2.1</b>	<b>Probe/Physics of interaction</b>	<i>Describe the NATURE of the probe used to test the material, as well as the physics of interaction between the sample volume and the probe. Provide physical equations that describe your method, e.g., Lambert-Beer for UV.</i>
<b>2.2</b>	<b>Interaction Volume</b>	<i>Describe the characteristic volume of interaction between the sample</i>
<b>2.3</b>	<b>Calibration process</b>	<i>Describe the calibration process needed to acquire the data. State if your equipment is professionally calibrated and the frequency thereof.</i>
<b>2.4</b>	<b>Detector</b>	<i>Describe the nature and main functions of the used detector including the brand.</i>
<b>2.5</b>	<b>Signal</b>	<i>Describe the signals that are acquired.</i>
<b>2.6</b>	<b>Measurement time</b>	<i>Quantify the time needed for the acquisition</i>
<b>2.7</b>	<b>Measurement parameters</b>	<i>Describe the main input parameters that are needed to acquire the signal</i>

<b>3. RAW DATA</b>		
<b>3.1</b>	<b>Raw Data</b>	<i>Describe the nature and data format of the acquired raw data</i>
<b>3.2</b>	<b>Unit</b>	<i>Describe the units of your data, e.g. km/mol for IR intensities, etc. or state unitless.</i>
<b>3.3</b>	<b>Data acquisition rate</b>	<i>Quantify the raw data acquisition rate, if applicable.</i>

4. DATA PROCESSING		
4.1	<b>Level of expertise</b>	<p><i>Little expertise: Person can read out results directly</i></p> <p><i>Medium expertise: Person needs to use a simple computer program to fetch data and interpret them (e.g., Win-NMR)</i></p> <p><i>Domain expertise: Person needs to be able to retrieve data and fit data.</i></p> <p><i>High expertise: Person cannot rely on any computer algorithm and requires years of expertise to make ad hoc decisions how to process data</i></p>
	<b>Data normalisation</b>	<i>Description of any processes that are adopted to normalise the raw data (e.g. set of the zero points of the measurements, subtraction of noise, etc.)</i>
4.2.	<b>Processing reproducibility</b>	<p><i>Description of performed statistical analysis to check for data reproducibility.</i></p> <p><i>Easily reproducible for everyone</i></p> <p><i>Reproducible for a domain expert</i></p> <p><i>Reproducible only for Data processing Expert</i></p>
4.3	<b>Data processes filtering</b>	<i>Describe the main raw data filtering processes</i>
4.4	<b>Data analysis procedures</b>	<i>Describe the main raw data analysis workflow and describe the software used.</i>
4.5	<b>Main signals processed</b>	<i>Describe the main processed channels</i>
4.6	<b>Data processing through calibrations</b>	<i>Describe how raw data are corrected and/or modified through calibrations.</i>
4.7	<b>Properties (elaborated data)</b>	<i>Describe how the elaborated data are converted into properties.</i>
	<b>Quality of the data</b>	<i>Example evaluation of S/N ratio, or other quality indicators (limits of detection/quantification, statistical analysis of data, data robustness analysis)</i>
4.8.	<b>Data management</b>	<i>Describe if the data will be entered into a repository, e.g. CCDC, etc. and thus can be made accessible to a wider audience</i>

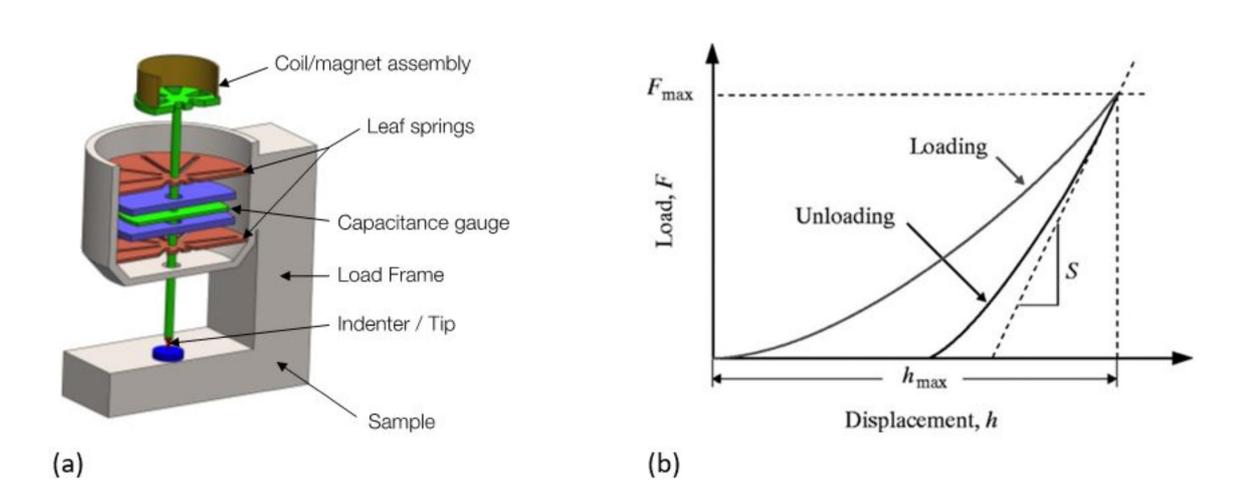
## Annex A (informative)

### Example of a table, a figure and a formula

#### A.1 Nanoindentation

Nanoindentation is a standard and commonly used technique for the measurement of hardness and elastic modulus at the micro and nano scales, which has become the reference characterization method for surface mechanical characterization<sup>3</sup>.

The Oliver-Pharr method represents the standardized approach describing the characterization workflow and has been in 1992 The main features of this method, including hardware description and a typical load-displacement curve, are shown in the following figure<sup>4</sup>.



**Figure A.1. Basic instrumentation and output of a quasi-static nanoindentation technique.**

More recently, the full experiments and guidelines have been formalized into an international standard (ISO 14577) that should be followed by the user to complete the experiment.

By using the concepts and basic structure of CHADA described in the previous chapter, the following high-detail scheme can be developed for this specific technique, according to ISO 14577 standard. In fact, each of the block in the workflow represents a set of information that is stored in the metadata and will be required for reproduction and reuse of the information.

The benefits of using such a novel structure for the metadata associated to nanoindentation experiments can be summarized as follows:

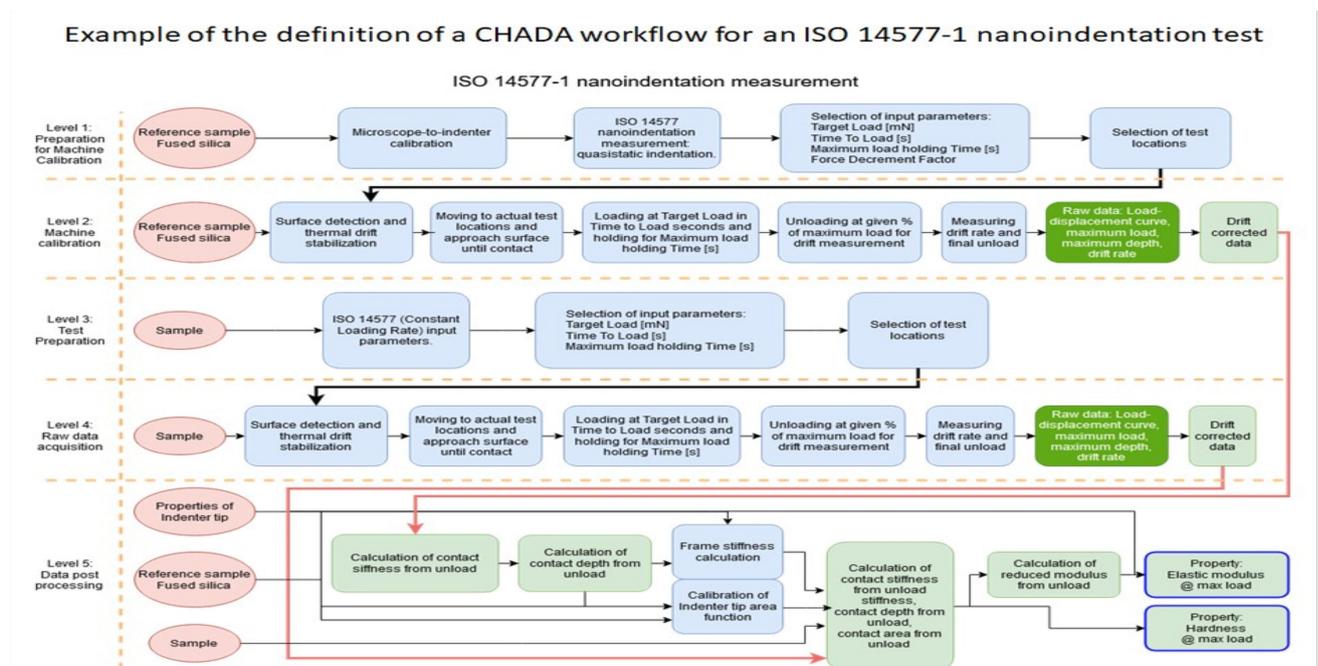
1. First of all, the calibration procedure associated to the experiment is stored in the metadata, thus enabling the user to identify any calibration issue even in the future;

<sup>3</sup> Oliver, W. C., and Pharr, G. M., J., An improved technique for determining hardness and elastic modulus using load and displacement sensing indentation experiments, 1992, Mater. Res., 7, 1564-1583

<sup>4</sup> Oliver, W. C., and Pharr, G. M., Measurement of hardness and elastic modulus by instrumented indentation: Advances in understanding and refinements to methodology, 2004, J. Mater. Res.,19, 3-20

2. Then, all information about the test sequence, environmental conditions and test conditions are stored in the metadata;
3. Finally, all the data analysis steps leading to property evaluation are stored, thus enabling future users to re-evaluate the quality of the data analysis process.

The relevance and potential impact of the adopted approach becomes even more relevant for high-speed nanoindentation, where a very large amount of experimental data is created.

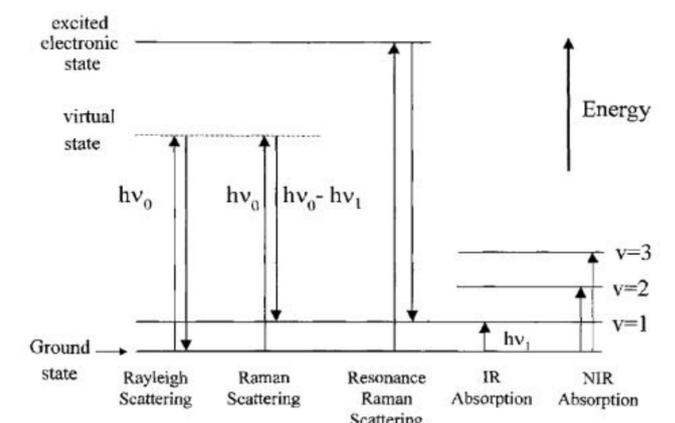


**Figure A.2. CHADA workflow for an ISO 14577-1 nanoindentation test**

## A.2 Raman spectroscopy

### A.2.1 General

Raman Spectroscopy is a spectroscopic technique typically used to determine vibrational modes of molecules (or crystal lattices), although rotational and other low-frequency modes of systems may also be observed. Raman spectroscopy is commonly used in chemistry to provide a structural fingerprint by which molecules can be identified. Raman spectroscopy relies upon inelastic scattering of photons, known as Raman scattering. When monochromatic light of energy  $h\nu_0$ , where  $h$  is the Planck constant and  $\nu_0$  is the frequency of light, encounters matter (gas, solid, or liquid), there is some probability that it will be scattered. If the object in question (e.g., a molecule) is much smaller than the wavelength of the light, the dominant scattering process is Rayleigh scattering, as shown in Figure 1, where the scattered light has the same frequency as the incident light. The “virtual state” is not necessarily a true quantum state of the molecule but can be considered a very short-lived distortion of the electron cloud caused by the oscillating electric field of the light. The electron cloud of the molecule is also perturbed by molecular vibrations, and it is possible for the optical and vibrational oscillations to interact, leading to Raman scattering. Raman scattering is shown in Figure 1 in which the scattered photon is lower in energy (Stokes scattering) by an amount equal to a vibrational transition,  $h\nu_1$ .



**Figure A.3. Energy-level diagram showing the states involved in Raman scattering.**

A Raman spectrum consists of measured intensity of scattered light plotted vs. energy. Each peak corresponds to a given Raman shift with respect to the incident light energy. If the system is in an excited vibrational state when an incident photon is scattered, the photon may gain (rather than lose) energy when scattered, leading to anti-Stokes Raman scattering. The Stokes and anti-Stokes Raman peaks are symmetrically positioned about the Rayleigh peak, but their intensities are very different except for at very low vibrational energies. The magnitude of the Raman effect correlates with polarizability of the electrons in a molecule. [2]

### A.2.2 Calibration

Raman spectrometers require both spectral and intensity response calibration, though most users rely on factory calibration for the latter. In day-to-day use, the most used approach for calibration of Raman shift involves the 520.7 cm<sup>-1</sup> Raman peak of crystal silicon as a reference. Also, low-pressure calibration lamps can be used (e.g., Hg, Ar, Xe, Ne, etc.) which produce sharp and narrow spectral lines with well-known emission wavelengths. Finally, the emission wavelength of the excitation laser itself is also used as a reference measurement and should appear at a Raman shift of 0 cm<sup>-1</sup> however this may be difficult to observe in spectrometers equipped with a highly effective filter for the Rayleigh scattering.

### A.2.3 Acquisition routine and Parameters

Raman spectroscopy is commonly performed in a back-scattering geometry (like reflectance measurements) however, measuring the absolute intensity of scattered light is challenging and usually impractical.

The first step for a RS measurement is the proper alignment of the sample on the Raman stage (height, tilt etc). The correct position can be identified by the highest value of the scattered light beam intensity. Generally, the collection efficiency of Raman scattered photons, or the Intensity of Raman signal is correlated with the following parameters:

$$I_{\text{Raman}} \sim \lambda^{-4} * \alpha^2 * IE$$

Where  $\lambda$  is the excitation wavelength,  $\alpha$  the polarizability tensor for the Raman mode of interest, which is an intrinsic property of the sample material and IE the laser Energy density on the sample which is the product of power density on sample and acquisition time.

The appropriate selection of acquisition parameters strongly affects the measured spectrum characteristics. In summary these are the following (synthetic list):

- Excitation wavelength
- Microscope objective

- Polarized laser excitation
- Diffraction Grating and slit width
- Laser Power and Acquisition Time
- CCD Cooling Temperature
- Spectra Accumulation
- Ambient Conditions

#### **A.2.4 Post-processing and Analysis of RS data**

In many applications the information can be deduced directly from the measured RS which is the spectral fingerprint of the material e.g. material identification for chemical analysis. However quantitative data can only be obtained by numerical fitting with specific mathematical functions to the measured SE spectra. This procedure includes the subtraction of the background photoluminescence effects and the implementation of mathematical functions which approximate the Raman peak characteristics of the studied material. Fitting the measured spectrum with these functions provides quantitative values for the desired parameters. The functions most commonly involve high-order polynomials, splines and even straight lines for the baseline subtraction and Lorentzian, Gaussian and Voigt equations to fit the peak characteristics. However, there are no definite rules for the use and implementation of these functions on the Raman mode peak properties. Detailed discussion on this topic is beyond the scope of this document. The quality of the fit is usually evaluated using a regression coefficient as the figure of merit to guide the numerical algorithm which searches for the best-fit values of the equation parameters. The figure of merit behaves like a multivariate mathematical function which depends on a given number of fitting parameters. The principal difficulty that arises is that the figure of merit may have either multiple minima with the same value, or multiple partial minima with different values. It is important to note that at the end of a minimization process, review of the results is necessary to check pertinence, accuracy and efficiency.

Finally, in many cases, the Raman modes characteristics can be translated and interpreted into material properties. This typically requires examination of various samples with distinct reference properties and complementary characterization techniques along with theoretical calculations on the phonon dispersion modes by DFT.

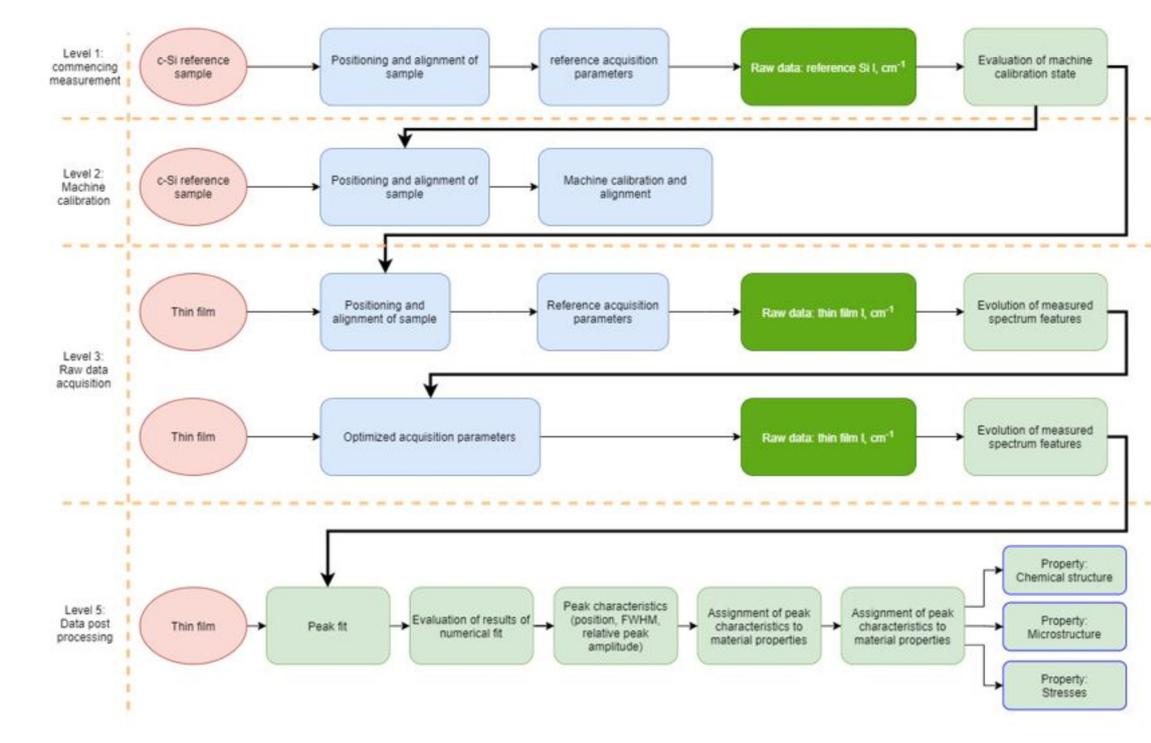


Figure A.4. RAMAN spectroscopy