



## Deliverable D1.3

### Description of the Characterization workflow according to CWA 17851

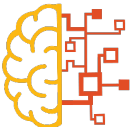
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Version:	1.0

<sup>1</sup> PU = PUBLIC fully open ((warning) automatically posted online on the Project Results platforms)

SEN = Sensitive — limited under the conditions of the Grant Agreement

EUCl = EU classified under Decision 2015/444



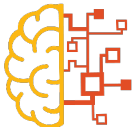


## Project Profile

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<b>Topic</b>	HORIZON-CL4-2022-RESILIENCE-01-19 - Advanced materials modelling and characterisation (RIA)
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<b>Acronym</b>	CoBRAIN
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<b>Duration</b>	48 months

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<b>0.1</b>	23-06-2023	UR3	First draft
<b>1.0</b>	29-06-2023	UR3, UMR	Final version



## Executive Summary<sup>2</sup>

The deliverable shows all the workflow diagrams of the characterization techniques that have been developed. For the main characterization techniques, the workflow diagrams, called CHADA, have been integrated by a descriptive documentation. The development of the CHADAs and related documentation contributes to the definition of the ontology of the processes, helps in the correct management of product data and allows an easier and faster optimization of the characterization processes. The CHADAs and related documentation have been drawn up based on what is reported in the CWA 17851. The techniques taken into consideration were identified through the table of *Experimental Facilities* present on the CoBRAIN SharePoint site. In this deliverable the CHADAs of the following characterization techniques are reported:

- SEM - Scanning Electron Microscopy;
- XRD - X Ray Diffraction;
- TEM - Transmission Electron Microscopy;
- Micro Raman Spectroscopy;
- XPS - X-Ray Photoelectron Spectroscopy;
- AES - Auger Electron Spectroscopy;
- FIB-based microscale residual stress;
- Profilometry;
- Vickers Microhardness;
- Standard Nanoindentation;
- Nanoindentation with High Speed Mapping;
- Nanoindentation with pillar splitting;
- Pin-on-Disk test;
- Jet erosion test;
- Abrasion test;
- Scratch Test;
- Electrochemical Polarization Test;
- Electrochemical Impedance Spectroscopy;
- Potentiostatic chronoamperometry;
- Combustion analysis.

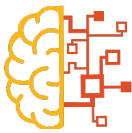
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<sup>2</sup> This summary will be used for public dissemination of CoBRAIN's activities

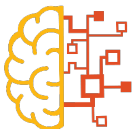


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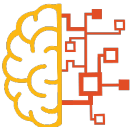


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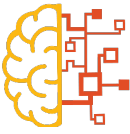
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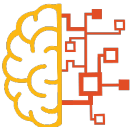
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## Abbreviations

Abbreviation	Definition
CHADA	Characterization Data
TS	Thermal Spray
CWA	CEN Workshop Agreement
HVOF	High Velocity Oxy-Fuel
HVAF	High Velocity Air-Fuel
SEM	Scanning Electron Microscope
EDX	Energy-Dispersive X-ray Spectroscopy
FIB	Focused ion Beam
XRD	X-Ray Diffraction
AES	Auger Electron Spectroscopy
DIC	Digital Image Correlation
CCD	Charge-Coupled Device

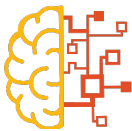


# 1 Introduction

In this deliverable, all the main characterization techniques that will be used within the project are reported. The specific list of these techniques is also available on the CoBRAIN SharePoint site in the *Experimental Facilities* table. These techniques have been identified based on the main properties of interest, at different scales, that a thermal-sprayed coating can offer. For each of them, a descriptive diagram integrated with descriptive documentation has been developed.

The diagrams of the characterization techniques have been developed in the perspective of generating a descriptive ontology of the TS process: indeed, the experimental characterization of the produced samples is a relevant activity of every process. Having a shared representative structure of the characterization workflows within the project contributes to better storage and management of the large amount of data produced. Their accessibility and interoperability are guaranteed by using a common terminology and by the description of the extant relationships: the latter always allow the product data to be contextualized, linking them back to the step of the TS process that generated the data themselves. It should also be noted that the correlation between the different characterization techniques and their integration with modeling can lead to the identification of new relationships between properties and composition-microstructure.

Furthermore, establishing a clear characterization workflow helps in the consistency of sample management, sharing and timing. Indeed, all the workflow diagrams of the characterization techniques that have been developed and reported in this deliverable will be tested and optimized in WP3, to be utilized in WP4 for the main experimental campaign. How the diagrams and associated documentation were developed is described in the CWA 17851 *Materials characterization - Terminology, metadata and classification*, illustrated below.



## 2 The CWA 17851

Due to the huge variety and complexity of materials and the wide range of applications, the materials characterization field consists of many communities. These communities have established different terminologies, which typically focus on specific application domains and on types of characterization methods. However, applications to industrial problems in advanced materials and nanotechnology require a strong interdisciplinary approach among these fields and communities. There is, therefore, a need to establish a common terminology (definition of concepts and vocabulary) in materials characterization, to arrive at a common structure of materials characterization metadata. A standardized terminology will improve future exchanges among experts in the entire area of materials characterization, facilitate the exchange with industrial end-users, experimentalists, as well as computational modelers, and reduce the barrier utilizing advanced materials characterization. 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.

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.

CWA 17851 was created with the aim of proposing a widely agreed and common basic architecture for materials characterization data (CHADA). This CWA includes therefore definitions of fundamental terms for the field of materials characterisation. Furthermore, the creation of descriptive diagrams of the characterization methods is illustrated, based on the concepts of “user case”, “experiment”, “raw data” and “data processing”.

1. **User case** (which also includes 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 generates 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 independent variables like 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 analyzed to arrive at the final shape.

A template CHADA for the methods is described to guide users to build the diagrams (Figure 1).

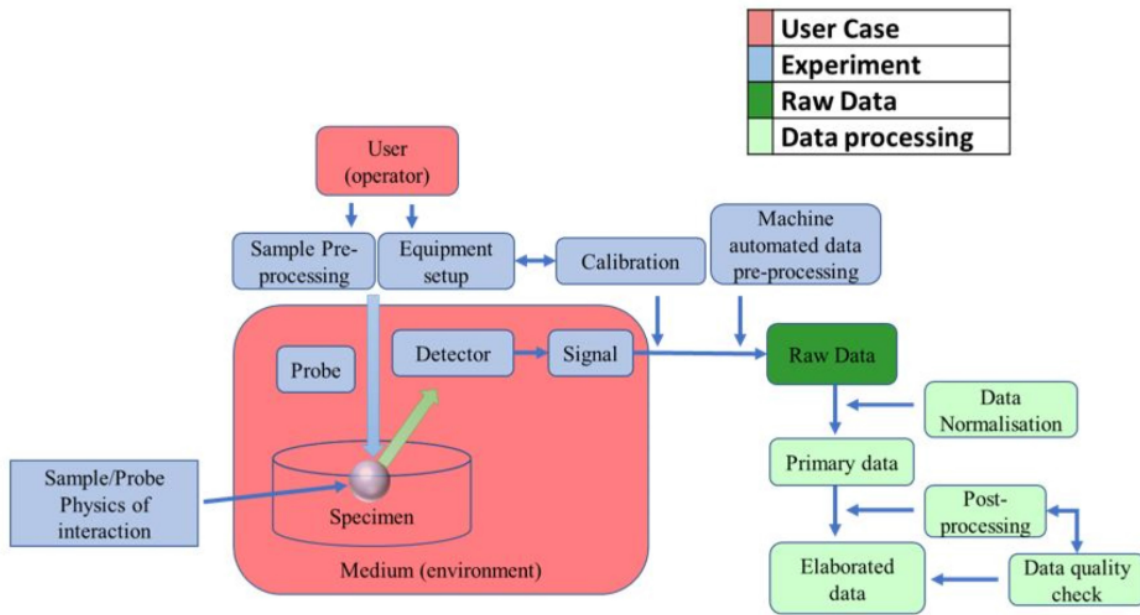
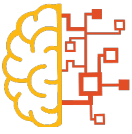


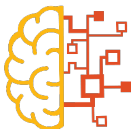
Figure 1. CHADA template

This CWA also provides a systematic description and documentation of characterization methods. This document seeks to organize the information so that even complex characterisation workflows can be conveyed more easily and key data can be captured. At first, an overview of the characterization is needed (Figure 2).



1	User Case	<i>General description of the sample AND the testing environment,...</i>
2	Characterisation method	<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>
		<i>Experiment/Operation 2</i>
		<i>...</i>
3	Validation of Characterisation	<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	Access conditions (what is needed to repeat the experiment)	<i>Was the access to your sample preparation an in-house routine or required a 3<sup>rd</sup> party service? 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. If applicable: Is your post-processing software open-source or commercial?</i>

Figure 2. Guidelines for CHADA documentation - overview of the characterization



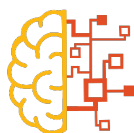
Then, specific descriptions of the four main concepts are required (Figure 4, Figure 3, Figure 6, Figure 5).

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

Figure 4. Guidelines for CHADA documentation – user case details

2. EXPERIMENT		
Interaction nature and character (destructive or non-destructive) of the probe with the sample		
2.1	Probe/Physics of interaction	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.
2.2	Interaction Volume	Describe the characteristic volume of interaction between the sample
2.3	Calibration process	Describe the calibration process needed to acquire the data. State if your equipment is professionally calibrated and the frequency thereof.
2.4	Detector	Describe the nature and main functions of the used detector including the brand.
2.5	Signal	Describe the signals that are acquired.
2.6	Measurement time	Quantify the time needed for the acquisition
2.7	Measurement parameters	Describe the main input parameters that are needed to acquire the signal

Figure 3. Guidelines for CHADA documentation – interaction details

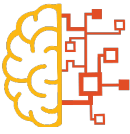


3. RAW DATA		
3.1	Raw Data	Describe the nature and data format of the acquired raw data
3.2	Unit	Describe the units of your data, e.g. km/mol for IR intensities, etc. or state unitless.
3.3	Data acquisition rate	Quantify the raw data acquisition rate, if applicable.

Figure 5. Guidelines for CHADA documentation – interaction details

4. DATA PROCESSING		
4.1	Level of expertise	<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>
	Data normalisation	<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.	Processing reproducibility	<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	Data filtering processes	<i>Describe the main raw data filtering processes</i>
4.4	Data analysis procedures	<i>Describe the main raw data analysis workflow and describe the software used.</i>
4.5	Main processed signals	<i>Describe the main processed channels</i>
4.6	Data processing through calibrations	<i>Describe how raw data are corrected and/or modified through calibrations.</i>
4.7	Properties (elaborated data)	<i>Describe how the elaborated data are converted into properties.</i>
	Quality of the data	<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.	Data management	<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>

Figure 6. Guidelines for CHADA documentation – data processing details



### 3 CHADAs of the characterization techniques

All the characterization techniques used in the project have been listed in the table *Experimental Facilities*, created for this purpose on the CoBRAIN SharePoint sites. The partners were asked to develop the CHADAs of the techniques in which, according to the table, they are most involved. The developed CHADAs are given below. The techniques have been grouped according to the classification suggested in the CWA.

#### 3.1 Microstructure characterization methods

##### 3.1.1 Optical Microscopy

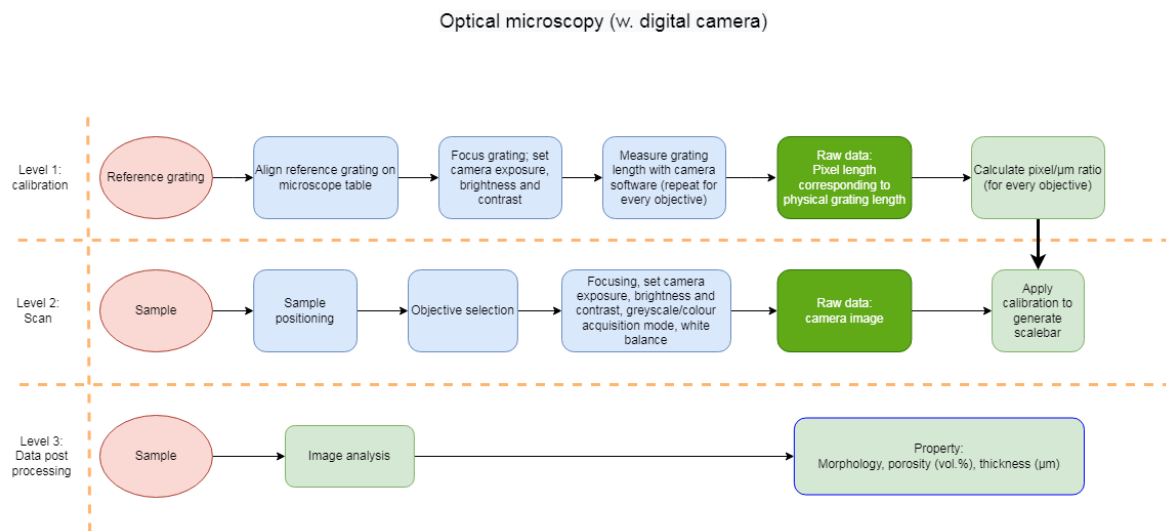
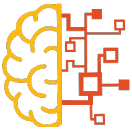


Figure 7. Optical microscopy CHADA



### 3.1.2 Optical Profilometry

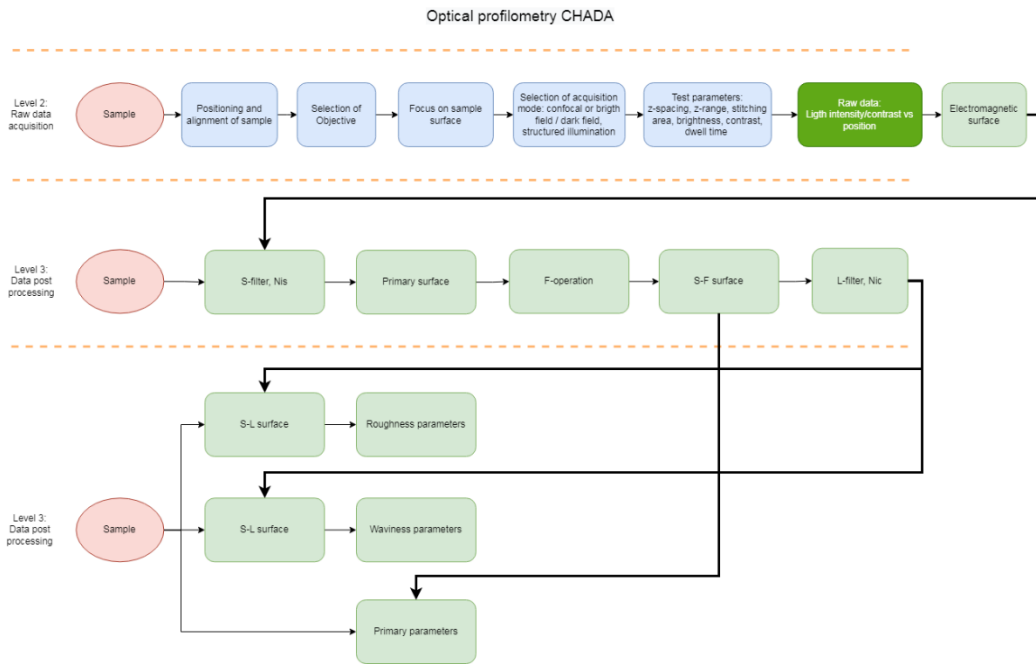


Figure 8. Optical profilometry CHADA

### 3.1.3 SEM – Scanning Electron Microscopy

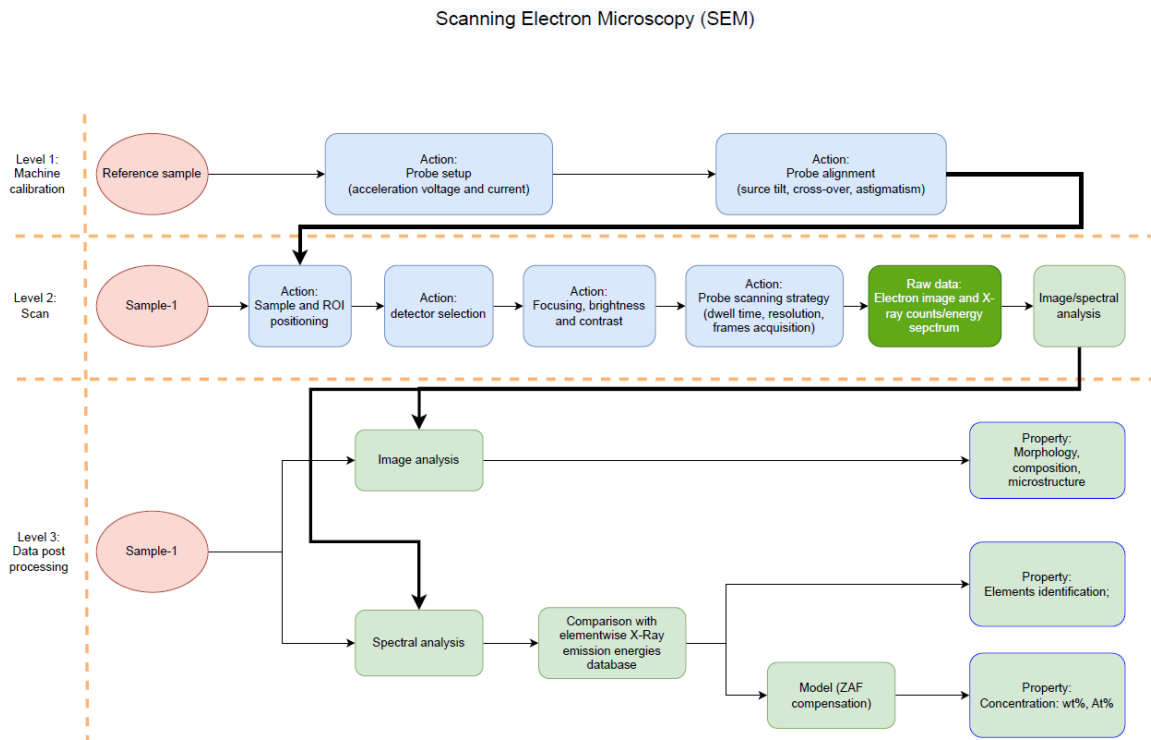
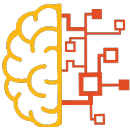


Figure 9. SEM CHADA



### 3.1.4 X-Ray Diffraction (XRD)

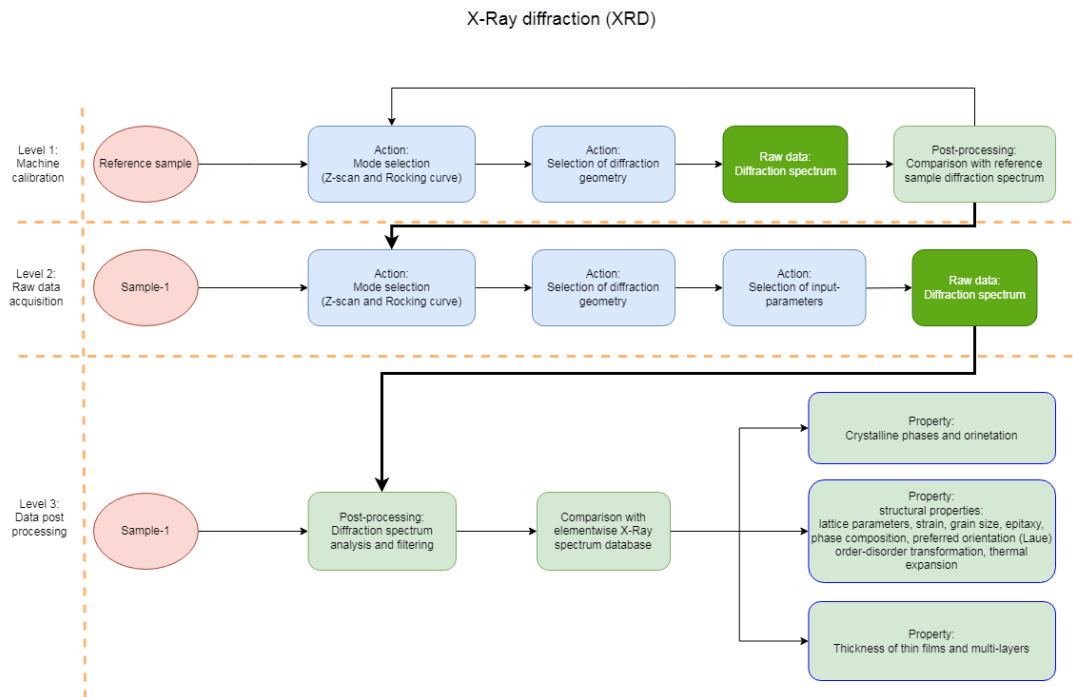


Figure 10. XRD CHADA

### 3.1.5 TEM – Transmission Electron Microscopy

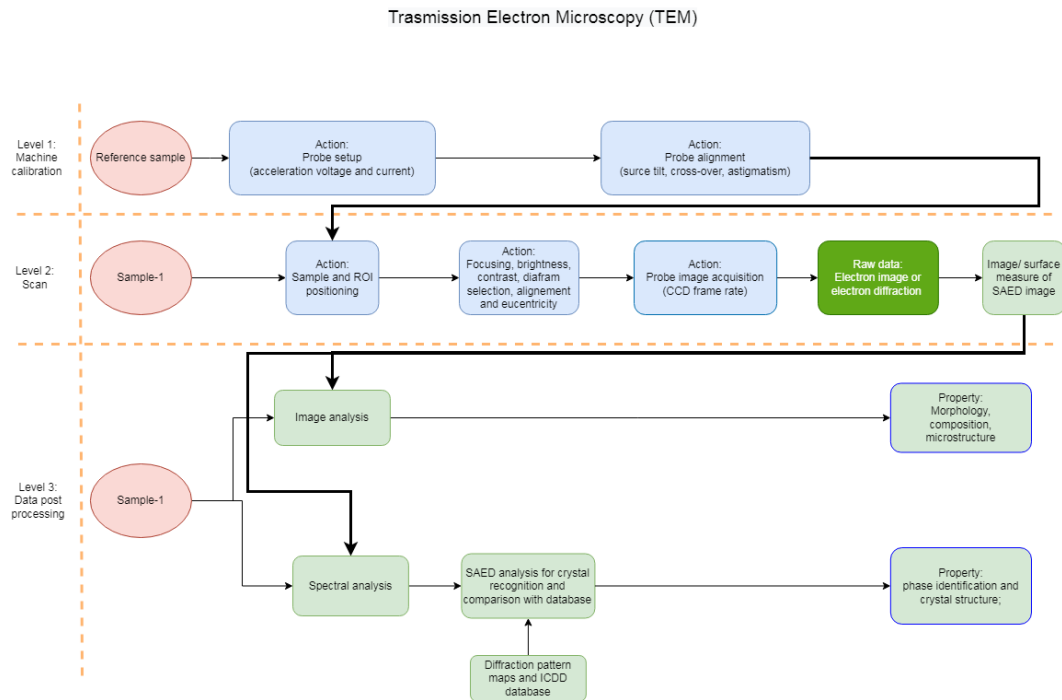
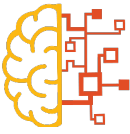


Figure 11. TEM CHADA



### 3.1.6 Micro Raman Spectroscopy

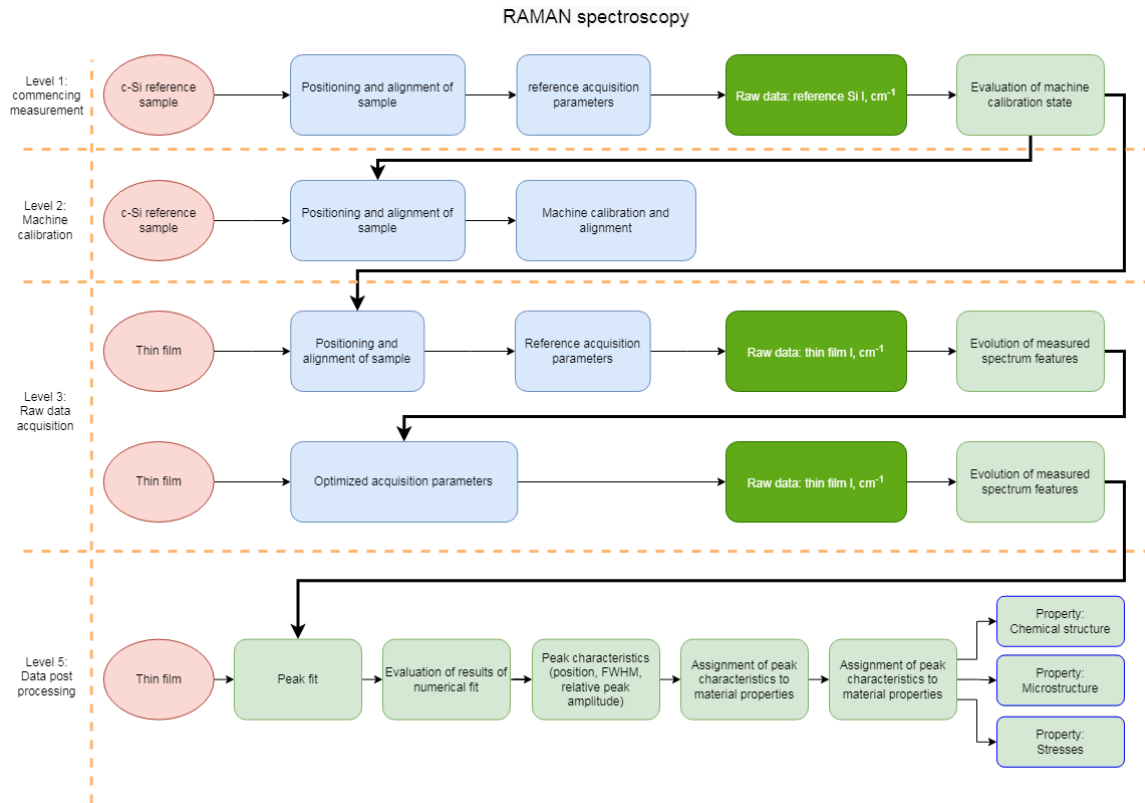
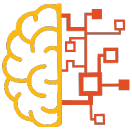


Figure 12. Raman spectroscopy CHADA



### 3.1.7 XPS - X-Ray Photoelectron Spectroscopy

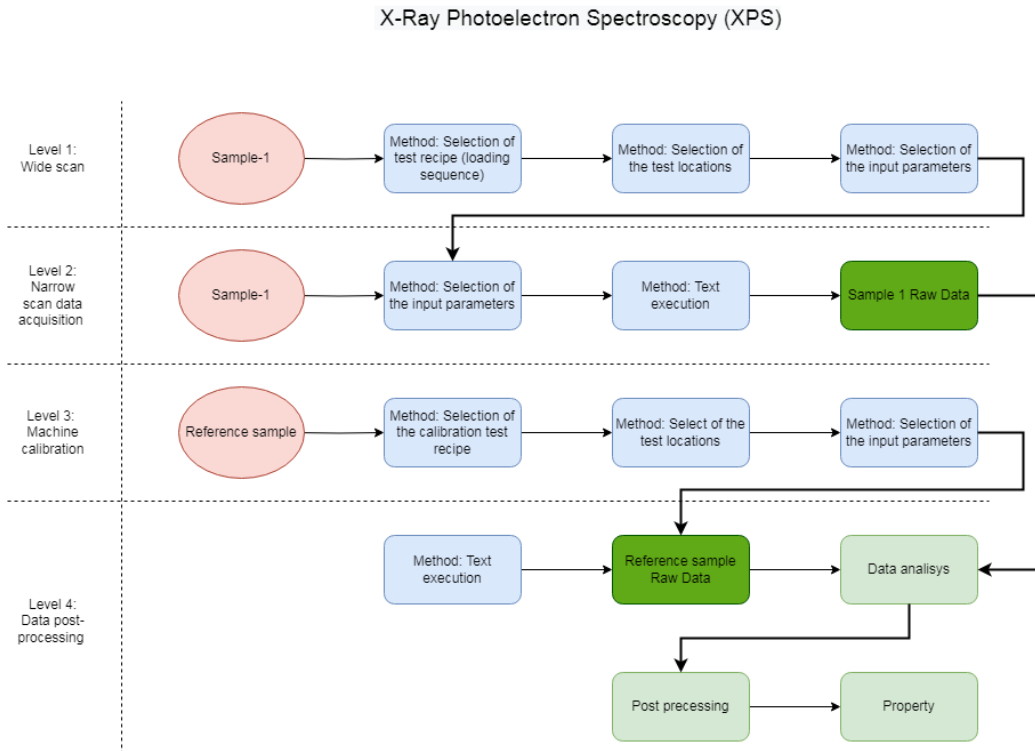


Figure 13. XPS CHADA

### 3.1.8 FIB-DIC based microscale residual stress measurement

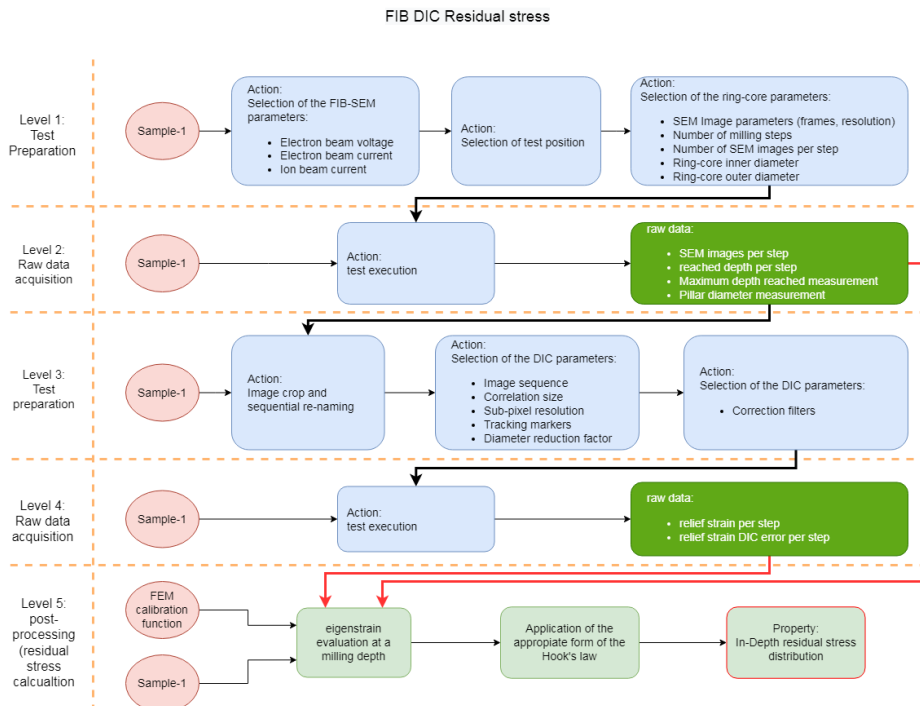
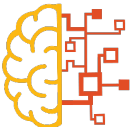


Figure 14. FIB + DIC for residual stress measurement - CHADA



### 3.1.9 AES – Auger Electron Spectroscopy

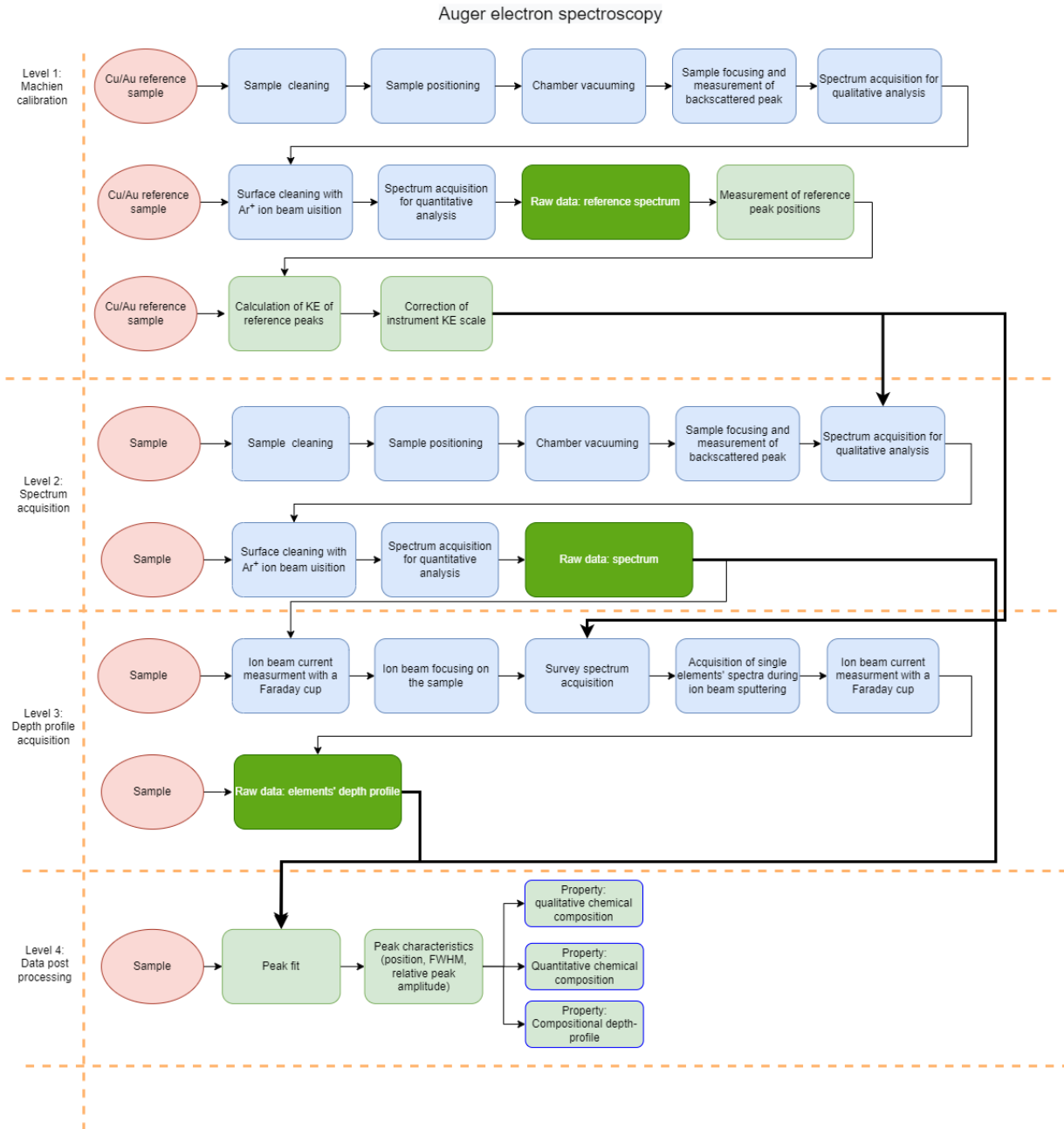
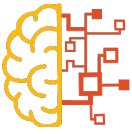


Figure 15. Auger Electron Spectroscopy CHADA



## 3.2 Mechanical characterization methods

### 3.2.1 Vickers Microhardness

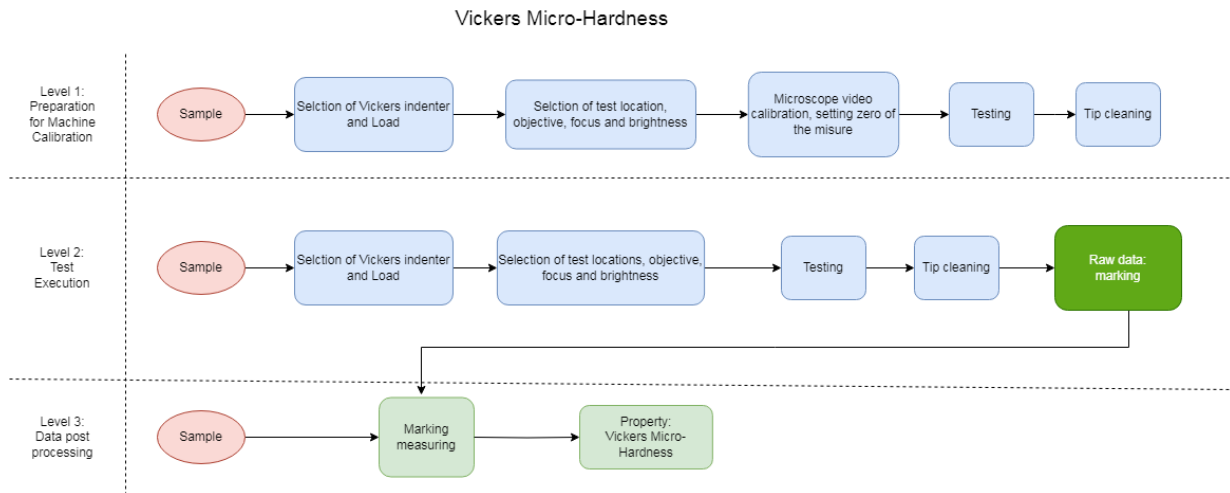
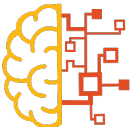


Figure 16. Vickers microhardness measurement CHADA



### 3.2.2 Nanoindentation

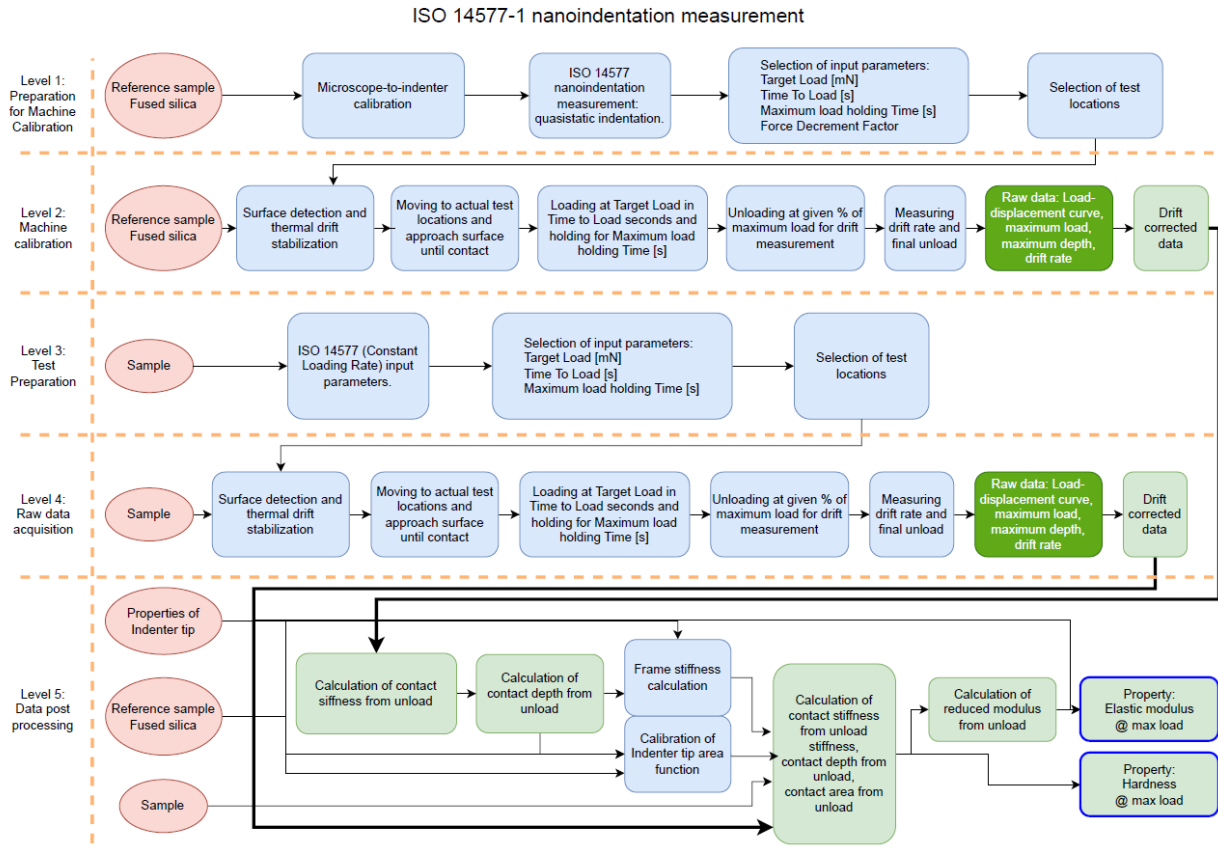


Figure 17. Nanoindentation CHADA

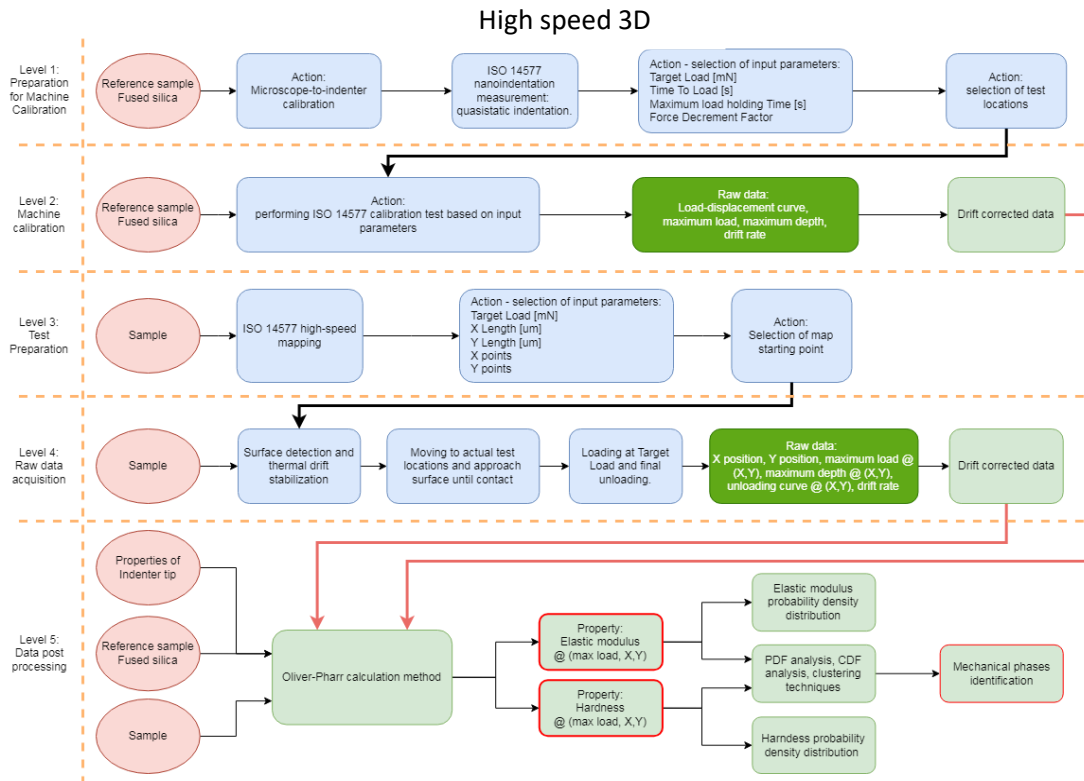
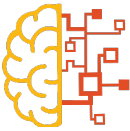


Figure 19. High-speed nanoindentation CHADA

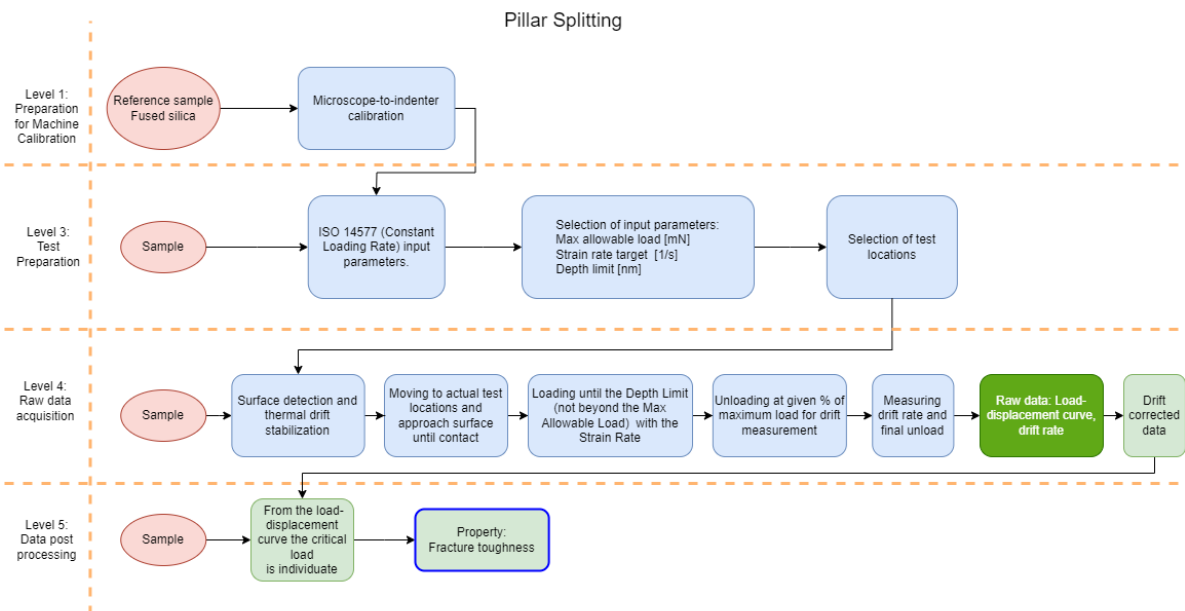
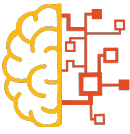


Figure 18. Nanoindenter-based pillar splitting CHADA



### 3.3 Wear characterization methods

#### 3.3.1 Sliding wear – Pin on disk

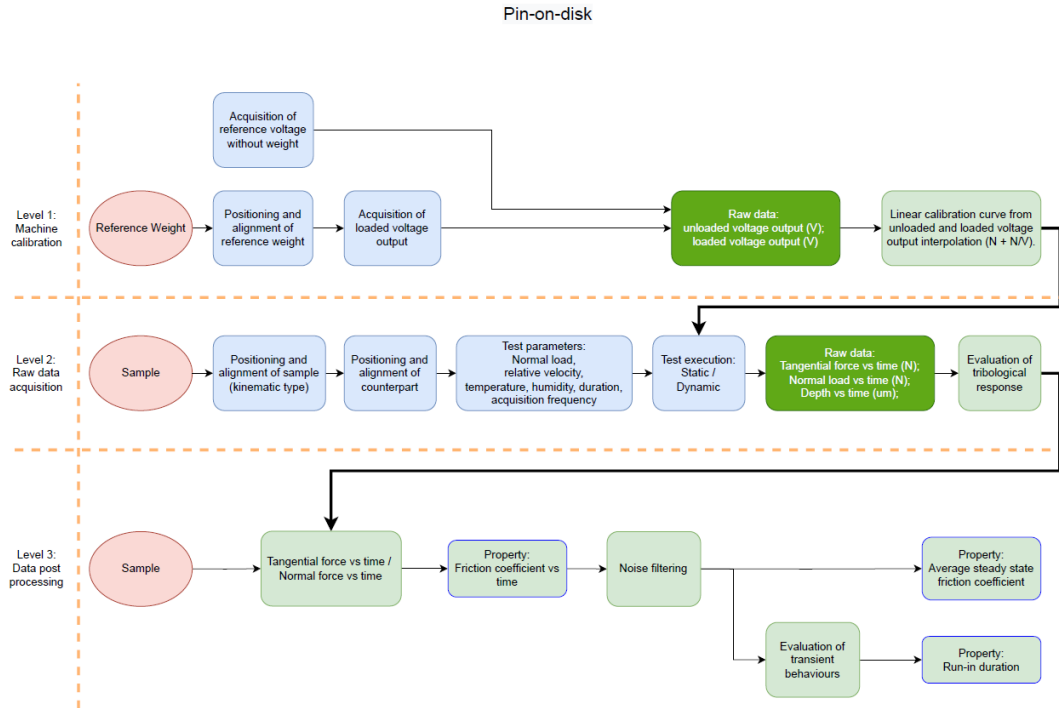
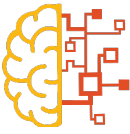


Figure 20. Pin-on-disk sliding wear test CHADA



### 3.3.2 Jet erosion test

Jet erosion test CHADA

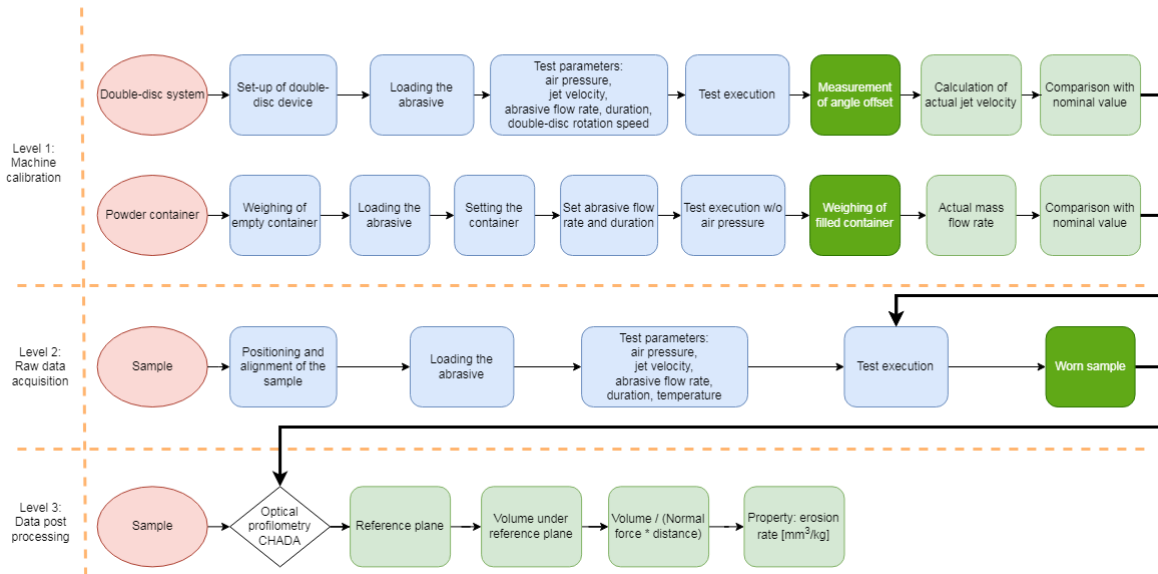


Figure 21. Jet erosion test CHADA

### 3.3.3 Abrasion Test

Abrasion test CHADA

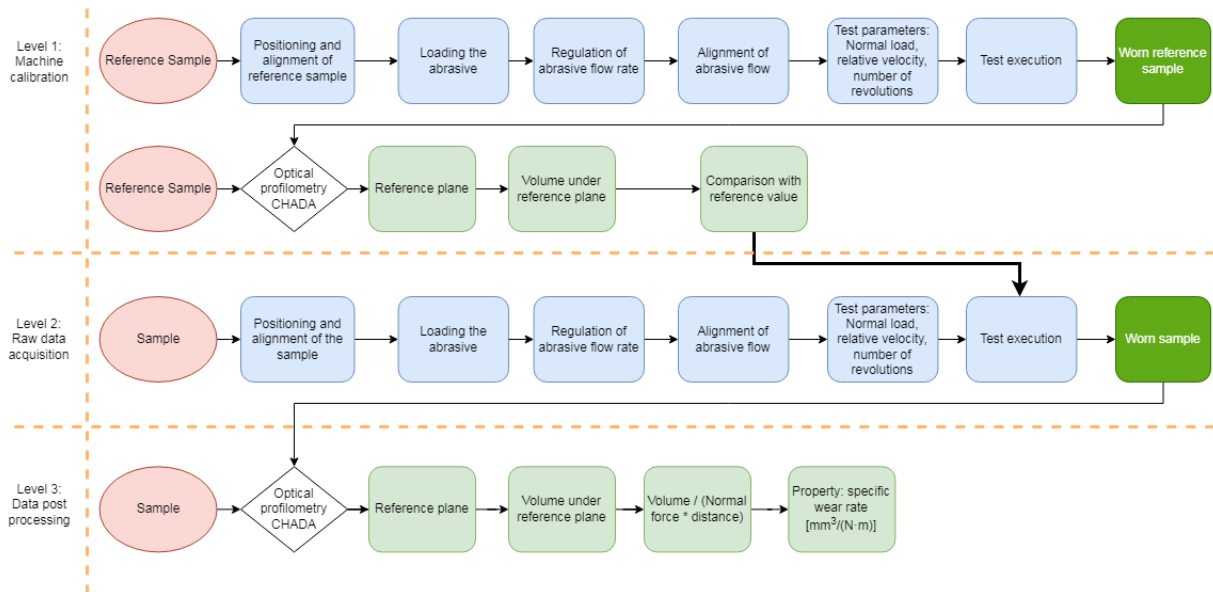
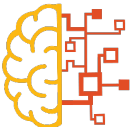


Figure 22. Dry particles abrasion test CHADA



### 3.3.4 Scratch Test

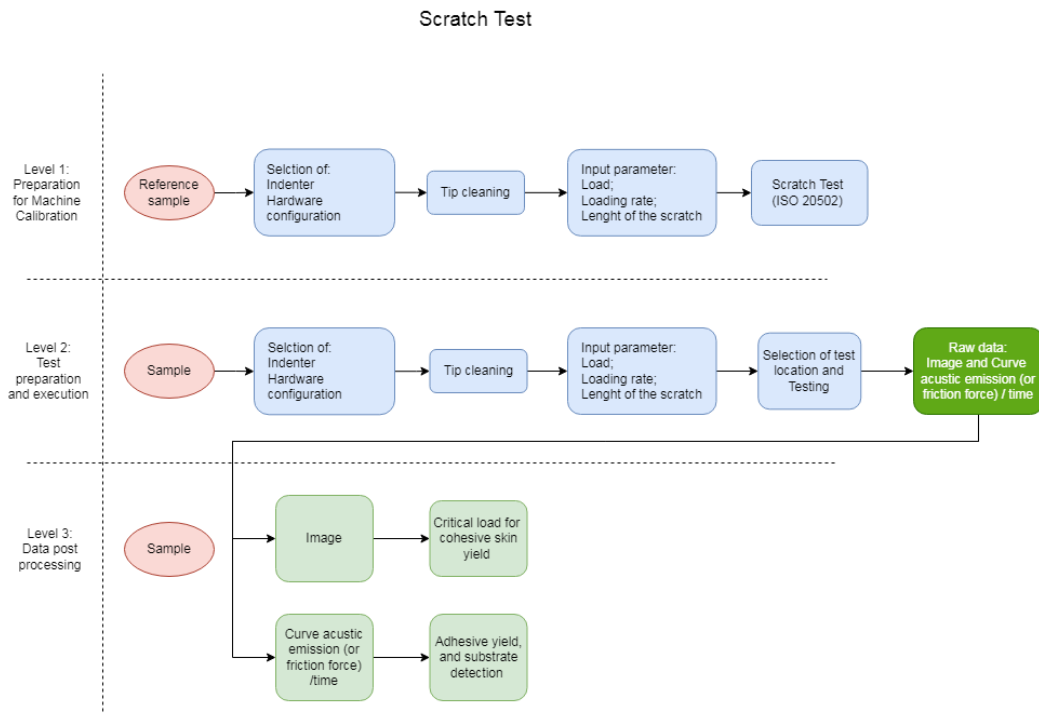
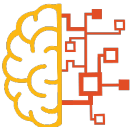


Figure 23. Scratch test CHADA



## 3.4 Corrosion characterization methods

### 3.4.1 Electrochemical polarization test

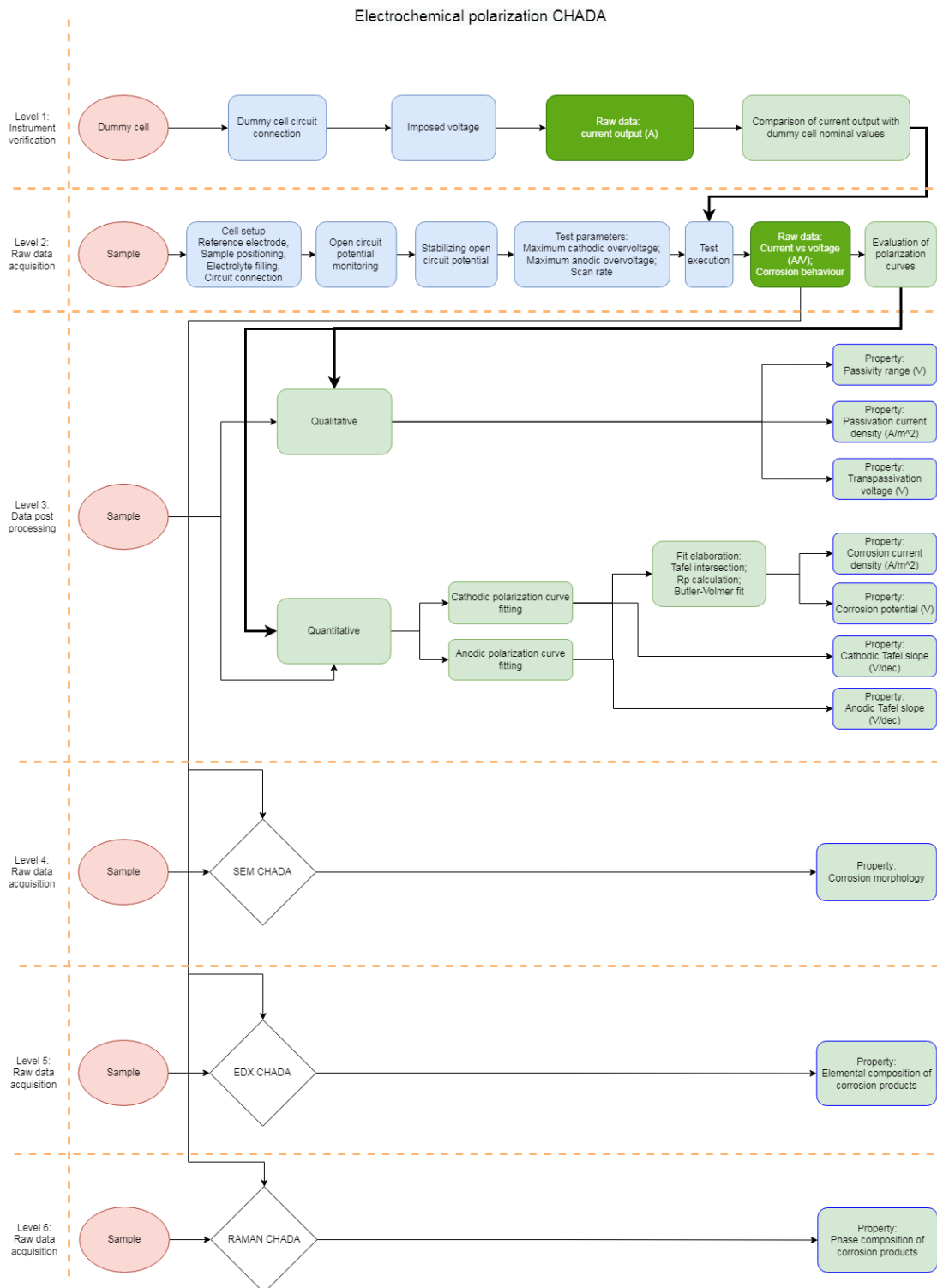
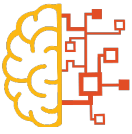


Figure 24. Electrochemical polarization test CHADA



### 3.4.2 Potentiostatic Chronoamperometry

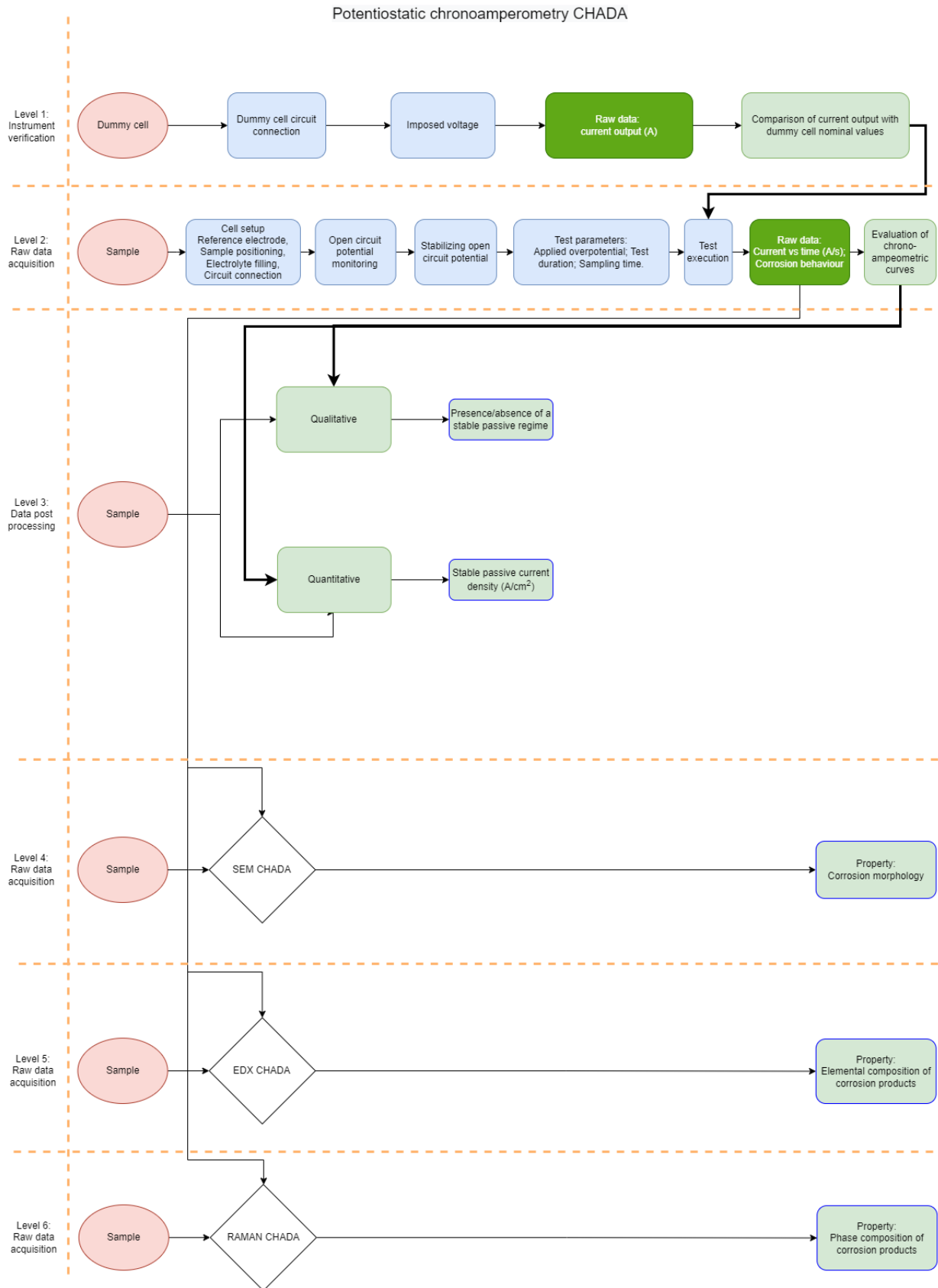
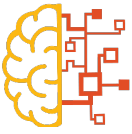


Figure 25. Potentiostatic chronoamperometry CHADS



### 3.4.3 Electrochemical Impedance Spectroscopy

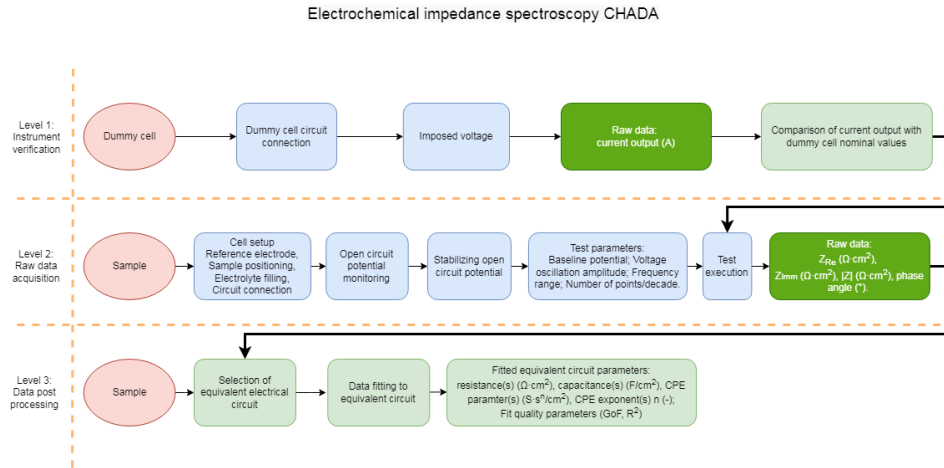
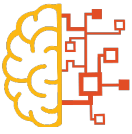


Figure 26. Electrochemical impedance spectroscopy CHADA



## 3.5 Chemical analysis methods

### 3.5.1 Combustion analysis

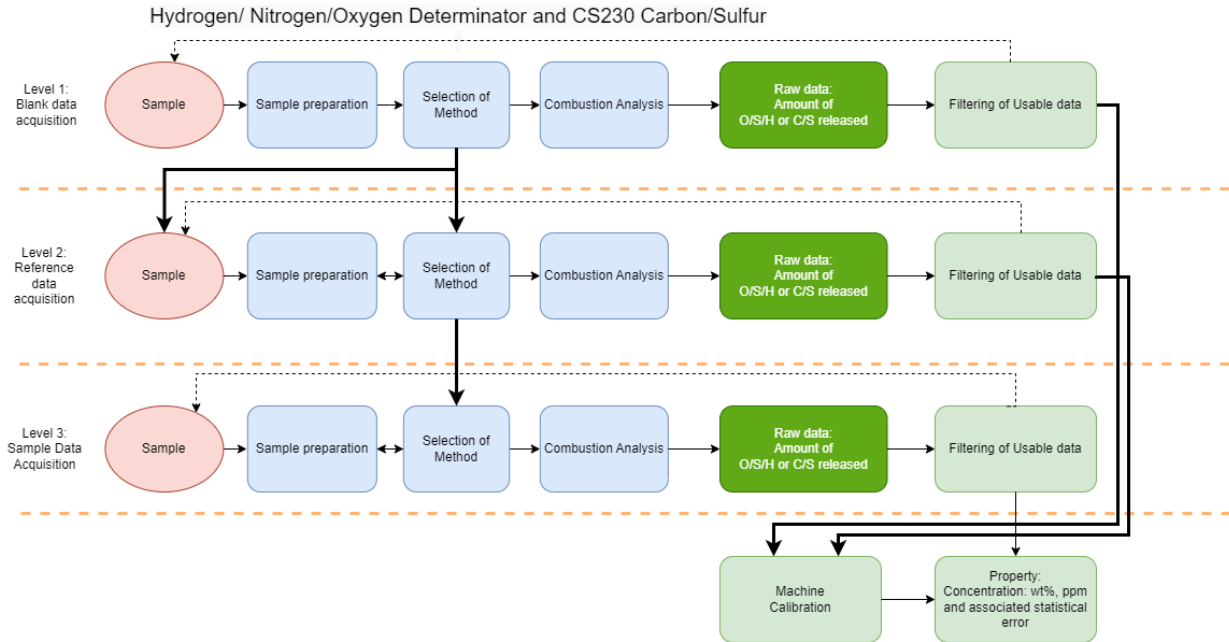
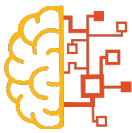


Figure 27. Combustion analysis (Oxygen/Sulphur/Carbon quantification) CHADA



## 4 Documentation forms

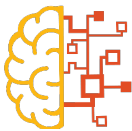
For the main characterization methods, associated descriptive documentation was developed. The accuracy and depth of this documentation is related to the level of detail that users seek to represent. The documentation below follows the same classification adopted for the CHADAs.

### 4.1 Microstructure characterization methods

#### 4.1.1 SEM – Scanning Electron Microscopy

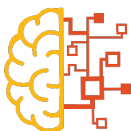
##### OVERVIEW OF THE CHARACTERISATION SEM

1	<b>User Case</b>	<p><i>Human operator with different level of automation.</i></p> <p><i>Sample: solid and dry materials with good electron conductivity.</i></p> <p><i>Sample dimensions: Typically 30mm x 30mm with thickness less than 10mm.</i></p> <p><i>Environment: Vacuum Chamber, Electron Source, Detectors.</i></p>
2	<b>Characterisation method</b>	<p><i>Imaging and Topography Analysis. SEM generates detailed, topographic images by scanning a focused electron beam across the sample surface and detecting various signals emitted or scattered by the sample. The two main imaging techniques in SEM are: Secondary Electron Imaging (SEI), that provides high-resolution images, highlighting surface features, topography, and details of the sample; Backscattered Electron Imaging (BEI), that are useful for distinguishing different materials or phases within a sample.</i></p> <p><i>Energy-Dispersive X-ray Spectroscopy (EDS): The emitted X-rays are unique to each element and can be detected and analyzed to determine the sample's elemental composition, quantitatively and qualitatively.</i></p>
3	<b>Validation of Characterisation</b>	<p><i>The SEM offers:</i></p> <ul style="list-style-type: none"><li><i>High resolution, for a detailed view of morphology and structural features;</i></li><li><i>Extended depth of field;</i></li><li><i>Chemical analysis capability;</i></li></ul>
4	<b>Access conditions (what is needed to repeat the experiment)</b>	<p><i>The sample preparation require an in-house routine.</i></p> <p><i>The characterisation tool require an in-house routine.</i></p>



1. USER CASE		
1.1	<b>USER</b>	<i>Human operator (different levels of automation are available). High level of expertise.</i>
1.2	<b>Sample</b>	<i>Sample dimensions (Typically 30mm x 30mm with thickness less than 10mm). Sample have to be flat, clean and dry.</i>
1.3	<b>Sample properties material</b>	<i>Solid and dry materials with good electron conductivity (Metals, ceramics, biological and polymers). If electron conductivity is not enough, sample have to be metallized or carbon coated.</i>
1.4	<b>Sampling process</b>	<i>The method of extraction will depend on the nature of the sample and the desired characteristics to be observed under the SEM. It may involve cutting, drilling, grinding, or other suitable techniques.</i>
1.5	<b>Sample preparation</b>	<i>The sample needs to be mounted on a sample holder or stub for SEM observation: sample holder provides a stable platform to secure and position the sample during analysis.  The sample needs to be clean to remove any contaminants or residues that could affect the imaging quality or interfere with the analysis.  In some cases, samples may require a thin conductive coating to enhance their conductivity.</i>
1.6	<b>Hazard</b>	<i>Some of the cleaning techniques can induce risks.</i>
1.7	<b>Characterisation environment</b>	<i>High vacuum (typically) or low vacuum (environmental SEM).</i>

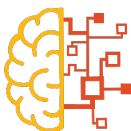
2. EXPERIMENT		
Interaction nature and character (destructive or non-destructive) of the probe with the sample		
2.1	<b>Probe/Physics of interaction</b>	<i>Energetic electrons in the microscope strike the sample and various reactions can occur in a restricted volume with a consequent generation of secondary electrons, backscattered electrons and X ray photons (for elastic and anaelastic interactions). The probe rasters the sample pixel by pixel.</i>
2.2	<b>Interaction Volume</b>	<i>The interaction volume depends on several factors, including the energy of the primary electrons, the density of the sample, and the characteristics of the material.  The depth of penetration of the primary electrons into the sample determines the upper limit of the interaction</i>



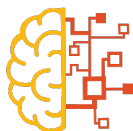
		<i>volume. At high electron energies, the interaction occurs deeper in the material, while at lower energies the interaction occurs more superficially.</i>
<b>2.3</b>	<b>Calibration process</b>	<i>Generally only during instrument maintenance.</i>
<b>2.4</b>	<b>Detector</b>	<i>Solid state or photomultiplier to detect secondary signal emitted by the sample</i>
<b>2.5</b>	<b>Signal</b>	<i>Electrons and X-ray</i>
<b>2.6</b>	<b>Measurement time</b>	<i>Typical acquisition times can range from a few minutes to several hours, depending on the complexity of the sample, desired resolution, and other experimental parameters.</i>
<b>2.7</b>	<b>Measurement parameters</b>	<p><i>The main input parameters are:</i></p> <ul style="list-style-type: none"> <li>- <i>The accelerating voltage;</i></li> <li>- <i>Working Distance;</i></li> <li>- <i>Magnification;</i></li> </ul>

### 3. RAW DATA

<b>3.1</b>	<b>Raw Data</b>	<i>Morphological or compositional images. X-ray counts/energy spectrum.</i>
<b>3.2</b>	<b>Unit</b>	<p><i>SEM images are often represented as grayscale images, where different shades of gray indicate variations in signal intensity.</i></p> <p><i>For the EDS the unit is X-ray counts or counts per second.</i></p>
<b>3.3</b>	<b>Data rate acquisition</b>	<i>Depends on the measurement time and the type of measurement.</i>



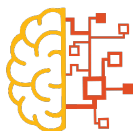
<b>4. DATA PROCESSING</b>		
<b>4.1</b>	<b>Level of expertise</b>	<i>Medium expertise</i>
	<b>Data normalisation</b>	<i>Normalization methods can include dividing the image by a reference signal, such as a reference sample or a flat-field image, to normalize the overall signal intensity.</i>
<b>4.2.</b>	<b>Processing reproducibility</b>	<i>Easily reproducible for everyone</i>
<b>4.3</b>	<b>Data filtering processes</b>	<i>Specific techniques, such as beam drift correction, charge compensation, or geometric distortion correction, can be employed to remove or minimize these artifacts and improve the accuracy and reliability of the data. Also Contrast enhancement techniques are used to improve the visibility and distinguishability of features within an SEM imag.</i>
<b>4.4</b>	<b>Data analysis procedures</b>	<i>Image analysis is dictated by the experience of the operator. X-ray energy data is quantified using the ZAF model.</i>
<b>4.5</b>	<b>Main processed signals</b>	<i>Electrons and X-ray</i>
<b>4.6</b>	<b>Data processing through calibrations</b>	<i>No correction of raw data.</i>
<b>4.7</b>	<b>Properties (elaborated data)</b>	<i>Topography, morphology, particles metrology (dimensions and shape).</i>
<b>4.8.</b>	<b>Data management</b>	<i>Values will be stored locally.</i>



## 4.1.2 Micro Raman Spectroscopy

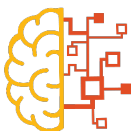
## OVERVIEW OF THE CHARACTERISATION

1	<b>User Case</b>	<p><i>Human operator (different levels of automation are available)</i></p> <p><i>Sample and sampling environment (flow cell, heating, etc.)</i></p> <p><i>Standards for calibration and verification</i></p>
2	<b>Characterisation method</b>	<p><i>Raman unit verification, calibration (x- and y- axes) and normalisation (resolution)</i></p> <p><i>Raman spectra acquisition</i></p> <p><i>Processing and analysis based on chemometrics and/or database search</i></p>
3	<b>Validation of Characterisation</b>	<p><i>Raman Spectroscopy is typically used to determine vibrational modes of molecules (or crystal lattices), although rotational and other low-frequency modes of systems may also be observed. It provides a structural fingerprint by which molecules can be identified.</i></p> <p><i>Raman spectroscopy is fast and non-destructive, offers high resolution, gives structural and electronic information, and is applicable at both laboratory and industrial scale.</i></p> <p><i>The position of any Raman band represents the energy of a molecular vibration, while its intensity (I) is proportional to the number of the corresponding vibrations in the sample. Generally, the intensity is correlated with the following parameters:</i></p> $I \sim \lambda^{-4} \cdot \alpha^2 \cdot IE$ <p><i>Where <math>\lambda</math> is the excitation wavelength, <math>\alpha</math> the polarizability tensor for the Raman mode of interest (an intrinsic property of the sample) and IE the incident energy density, which is the product of the laser power density on sample and acquisition time.</i></p>
4	<b>Access conditions (what is needed to repeat the experiment)</b>	<p><i>Standards are commercially available and in our lab. Samples do not require preparation for simple measurements. Flow cells with heating are available in our labs.</i></p> <p><i>Several Raman units are available in our lab to perform or repeat measurements for specific samples and check data reliability and reproducibility.</i></p> <p><i>Post processing can be done by using the manufacturer's software as well as other programs (OMNIC, Peaxact, Spectragrph, Excel, Origin, etc.). Harmonisation tools are under development in CHARISMA project (GA 952921).</i></p>



1. USER CASE		
1.1	<b>USER</b>	<i>An expert human operator (different levels of automation are available)</i>
1.2	<b>Sample</b>	<i>Solid or liquid sample (with specific equipment, gas samples can also be analysed)</i>
1.3	<b>Sample material properties</b>	<i>Chemical composition and structure. Raman is used to determine the number and orientation of layers, the quality and types of edge, the effects of perturbations such as electric and magnetic fields, strain, doping, disorder and functional groups</i>
1.4	<b>Sampling process</b>	<i>Samples received from the partners. Each sample and Raman spectrum is given an internal code.</i>
1.5	<b>Sample preparation</b>	<i>No preparation required. The correct position can be identified by the highest value of the scattered light beam intensity.</i>
1.6	<b>Hazard</b>	<i>Exposure to the laser</i>
1.7	<b>Characterisation environment</b>	<i>Cell with controlled environment available when appropriate: gas flow, temperature control and ramp.</i>

2. EXPERIMENT		
Interaction nature and character (destructive or non-destructive) of the probe with the sample		
2.1	<b>Probe/Physics of interaction</b>	<i>Laser beam, can be focused in a microscope. Raman spectroscopy is a photonic technique based on vibrational inelastic scattering. Raman scattered photons are only a tiny proportion (&lt;1 in 10<sup>6</sup>) of the photons scattered by a molecule, and have a different frequency to that of the incident photons of the laser used for excitation due to an energy gain or loss that depends on the energy states of the molecule.</i>
2.2	<b>Interaction Volume</b>	<i>Area of 100-200 μm<sup>2</sup> for probes, &lt;1 μm<sup>2</sup> for microscope. Confocal micro-Raman can spatially filter the analysis volume of the sample in the XY (lateral) and Z (depth) axes. Typical spatial resolution is in the order of 0.5-1 μm.</i>
2.3	<b>Calibration process</b>	<i>Raman units should be verified and often recalibrated, ideally per mode of measurement, for both spectral and intensity response. Besides manufacturer's indications, there exist several standard protocols and guides for this purpose, though most users rely on factory calibration for the y-axis.  <i>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. Low-pressure calibration lamps (e.g., Hg, Ar, Xe, Ne, etc.) that produce sharp and narrow spectral lines with well-known emission wavelengths can also be used. Finally, the emission</i></i>



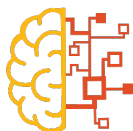
		<p>wavelength of the excitation laser itself is also used as a reference, as it should appear at a Raman shift of <math>0\text{ cm}^{-1}</math>, however this may be difficult to observe in spectrometers equipped with a highly effective filter for the Rayleigh scattering.</p> <p>For intensity calibration (y-axis) standard reference materials (NIST) or white light are used. Calcite and polystyrene may be used for resolution and alignment assessment.</p>
2.4	<b>Detector</b>	charge-coupled device (CCD)
2.5	<b>Signal</b>	The x axis shows the frequency differences (i.e., the energy change) between the incident and the Raman scattered photons, which are represented as wavenumbers in the unit of $\text{cm}^{-1}$ . The y axis denotes the intensity of the Raman scattered light at different wavenumber positions.
2.6	<b>Measurement time</b>	Variable, depends on the measurement parameters. Typically 1-100 s (if 2-10 acquisitions of 0.5-10 s are accumulated for a single spectrum)
2.7	<b>Measurement parameters</b>	<ul style="list-style-type: none"> <li>• excitation (wavelength, polarization, power)</li> <li>• optical path (optics, grating, slit width)</li> <li>• acquisition time and averaged accumulations</li> </ul>

### 3. RAW DATA

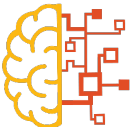
3.1	<b>Raw Data</b>	The CCD array data is converted into spectral data using the calibration to assign Raman shift to pixel position and normalise the intensity.
3.2	<b>Unit</b>	Intensity (a.u.) vs. Raman shift
3.3	<b>Data acquisition rate</b>	Depends on the measurement time and the type of measurement

### 4. DATA PROCESSING

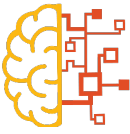
4.1	<b>Level of expertise</b>	Domain expertise: Person needs to be able to retrieve data and fit data. Expertise is needed for identification.
-----	---------------------------	--



	<b>Data normalisation</b>	<i>Data should be normalized to zero Raman shift for the laser peak. Sometimes, noise should be reduced and peak integration is needed.</i>
4.2.	<b>Processing reproducibility</b>	<i>Reproducible for a domain expert. Data analysis and data processing are reproducible by an expert in the Raman analysis (accumulations, integration time, laser focus, etc)</i>
4.3	<b>Data filtering processes</b>	<i>Data will be processing or filtered using: peak integration, bands selection, masks, background subtraction, raman shift mapping and analysis, etc.</i>
4.4	<b>Data analysis procedures</b>	<i>Acquired data is in form of Raman spectra, and it should be analysed by means of peak integration, spectra averages, scanning of phases distribution based on the corresponding Raman bands, mapping of the Raman shifts. This analysis is carried out by means the Witec commercial software.</i>
4.5	<b>Main processed signals</b>	<i>Raman spectra (bonding vibrations) in plane and in depth. In single points, lines or surfaces.</i>
4.6	<b>Data processing through calibrations</b>	<i>Raman spectra should be calibrated to the zero raman shift given by the laser signal.</i>



4.7	<b>Properties (elaborated data)</b>	<i>Raman shift is directly correlated with the bonding strain (stress and compression), so stress mapping can be done. Moreover, phase distribution and phases changes can be measured.</i>
	<b>Quality of the data</b>	<i>Quality of data is checked by statistical analysis and robustness analysis. The quality of the data is reliable.</i>
4.8.	<b>Data management</b>	<i>Data will be converted to csv and stored on share point and can be shared with a 3rd party. Moreover, data is usually published in peer reviewed journal.</i>



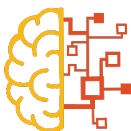
### 4.1.3 Optical Profilometry

#### OVERVIEW OF THE CHARACTERISATION

1	<b>User Case</b>	<i>Sample: Coated or uncoated flat or curved sample</i>
2	<b>Characterisation method</b>	<i>Execution of a profile measurement to identify the volume of a wear track or of a portion of a wear track</i>
		<i>Execution of a profile measurement to quantify the roughness and/or waviness of a surface</i>
3	<b>Validation of Characterisation</b>	<p><i>Optical profilometry techniques have quickly been gaining widespread acceptance because of their advantages, including high sensitivity, ability to operate with soft samples that would be altered by contact with a stylus, ability to acquire large areal scans over relatively short times, and reliability (minimal amount of mechanical or moving parts).</i></p> <p>See:</p> <p><i>J.R. Leach (ed.), Optical Measurement of Surface Topography, Springer, 2011</i></p>
4	<b>Access conditions (what is needed to repeat the experiment)</b>	<p><i>Sample preparation: in-house routine</i></p> <p><i>Access to characterization tools: in-house routine</i></p> <p><i>Commercial post-processing software (MountainsMap).</i></p>

#### 1. USER CASE

1.1	<b>USER</b>	<i>The user must be properly trained in the execution of the experiment. Sample setup and instrument focusing involve some manual operations that need to be performed with accuracy</i>
1.2	<b>Sample</b>	<i>Bulk or coated sample of max. 35 mm height × 100 mm side</i>
1.3	<b>Sample material properties</b>	<i>Any solid sample can be tested unless it is fully transparent.</i>
1.4	<b>Sampling process</b>	<i>The sample is usually obtained by metallographic cutting from the part to be characterized. Alternatively, parts can be manufactured at the required size (and coated).</i>

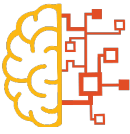


1.5	<b>Sample preparation</b>	<i>Samples must be cleaned in an ultrasonic bath using an organic solvent (e.g. ethanol or acetone) for degreasing prior to profile measurement.</i>
1.6	<b>Hazard</b>	<i>Organic solvents for cleaning can be hazardous (e.g. ethanol – flammable liquid, causes serious eye irritation; acetone – flammable liquid, causes serious eye irritation, causes drowsiness or dizziness by inhalation, repeated exposure may cause skin dryness or cracking).</i>
1.7	<b>Characterisation environment</b>	<i>Laboratory air.</i>

## 2. EXPERIMENT

### Interaction nature and character (destructive or non-destructive) of the probe with the sample

2.1	<b>Probe/Physics of interaction</b>	<p><i>The probe is a visible light (electromagnetic radiation) beam, either monochromatic (laser or LED source) or polychromatic (white light). The beam is focused on the sample surface through an optical system ending with a focusing objective of known numerical aperture. The beam is reflected back into the optical system and is directed to a detector, typically using a semi-reflecting mirror.</i></p> <p><i>Different kinds of physical interactions can be exploited, including:</i></p> <ul style="list-style-type: none"> <li>- <i>Phase-shifting interferometry: a monochromatic light source and an objective containing an interferometric system are employed in order to produce an interference pattern based on the different height of every point on the surface area imaged by the objective. The shifting interference patterns obtained through a vertical scan of the sample area imaged by the objective are then interpreted with a software to reconstruct the point-by-point height of the surface.</i></li> <li>- <i>Structured illumination microscopy: two identical, monochromatic light sources are employed to project two complementary light patterns onto the sample area imaged by the objective. When a point is in focus, the reflected images of the patterns remain complementary; increasingly large distances from the focal plane cause increasing degrees of overlapping between the reflected patterns. Through a stepwise movement of the sample in a direction parallel to the optical axis of the system, it is possible to reconstruct the trend of pattern contrast as a function of sample position at each point, thus identifying the exact focus height at each point.</i></li> <li>- <i>Focus variation microscopy: images of the sample surface under white-light illumination are acquired during a stepwise movement in a direction parallel to the optical axis of the system. The contrast between each point on the image and the adjacent ones is calculated at every step. Because such contrast is maximum when that point is in the focal plane, it is possible to reconstruct a trend of contrast as a function of sample position at each point, thus identifying the exact focus height at each point.</i></li> </ul>
2.2	<b>Interaction Volume</b>	<i>The lateral resolution is limited by Abbe's law for a diffraction-limited optical system and it is usually of the order of some hundreds of nanometres. The vertical resolution depends on the numerical aperture of the objective, the step size, and the method, and it can</i>



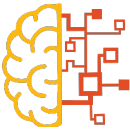
		<i>be around 10 nm using an objective with NA = 0.90 with a phase-shifting interferometry or a structured illumination microscopy system.</i>
2.3	<b>Calibration process</b>	<i>No periodic calibration needed.</i>
2.4	<b>Detector</b>	<i>CCD camera</i>
2.5	<b>Signal</b>	<i>Light intensity</i>
2.6	<b>Measurement time</b>	<i>A typical profilometric acquisition lasts between 2 min and 1 h depending on the needed lateral and vertical resolution and the size of the acquisition area.</i>
2.7	<b>Measurement parameters</b>	<i>z-spacing (<math>\mu\text{m}</math>), z-range (<math>\mu\text{m}</math>), stitching area (mm), brightness, contrast, dwell time (s)</i>

### 3. RAW DATA

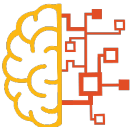
3.1	<b>Raw Data</b>	<i>Text file with x, y, z coordinates at each location.</i>
3.2	<b>Unit</b>	<i>X coordinate – mm Y coordinate - mm Z coordinate - <math>\mu\text{m}</math></i>
3.3	<b>Data acquisition rate</b>	<i>Automatically set by the instrument</i>

### 4. DATA PROCESSING

4.1	<b>Level expertise of</b>	<i>Domain expertise: Person needs to be able to retrieve data and fit data.</i>
	<b>Data normalisation</b>	<i>No data normalization</i>
4.2.	<b>Processing reproducibility</b>	<i>Easily reproducible for everyone</i>
4.3	<b>Data filtering processes</b>	<i>Lowpass filter with short wavelength (<math>\sim 10^0 \mu\text{m}</math>) Form filtering (fitting and subtraction of polynomial, spherical, or cylindrical surface) Roughness/waviness filter (5 times the lateral dimension of the largest roughness structure of interest)</i>



4.4	<b>Data analysis procedures</b>	<ul style="list-style-type: none"> <li>- Removal of non-measured points</li> <li>- Lowpass noise filtering (S filter)</li> <li>- Edge trimming</li> <li>- Form removal (F-filter)</li> <li>- Primary parameters' calculation</li> <li>- Roughness/waviness filter (L-filter)</li> <li>- Calculation of roughness parameters (S-L surface) and/or of waviness parameters (L-F surface)</li> </ul> <ul style="list-style-type: none"> <li>- Removal of non-measured points</li> <li>- Lowpass noise filtering (S filter)</li> <li>- Edge trimming</li> <li>- Form removal (F-filter)</li> <li>- Identification of the boundaries of the wear scar or wear scar section</li> <li>- Choice of method for defining the reference surface</li> <li>- Calculation of wear scar volume or wear scar section volume below the reference surface</li> <li>- For a wear scar section: division of the volume by the section length to calculate the average cross-sectional wear scar area</li> <li>- For a wear scar section: multiplication of the average cross-sectional wear scar area to calculate the overall wear scar volume</li> </ul>
4.5	<b>Main processed signals</b>	<ul style="list-style-type: none"> <li>- S-F surface</li> <li>- S-L surface</li> <li>- L-F surface</li> </ul>
4.6	<b>Data processing through calibrations</b>	No correction of raw data
4.7	<b>Properties (elaborated data)</b>	<p>Primary surface parameters</p> <p>Roughness parameters</p> <p>Waviness parameters</p> <p>Wear scar volume/wear scar section volume</p>
	<b>Quality of the data</b>	<ul style="list-style-type: none"> <li>- Statistical analysis of roughness/waveiness/volume (from multiple repeat tests): half-difference between maximum and minimum value (if up to 4 repeats), standard deviation (if 5 or more repeats).</li> </ul>
4.8.	<b>Data management</b>	<p>Raw profilometry data stored locally (on machine or in a Cloud storage facility, e.g. MS OneDrive) for at least 2 years [note extremely large file size].</p> <p>Computed roughness/waviness/volume data stored locally and in the project's SharePoint folder.</p>



## 4.2 Mechanical characterization methods

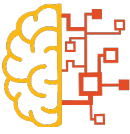
### 4.2.1 Vickers MicroHardness

#### OVERVIEW OF THE CHARACTERISATION

<p><b>1 User Case</b></p>	<p><i>Any solid sample can be tested included bulk material, coating, heterogeneous material, biomaterial and no specific Sample dimensions is required.</i></p> <p><i>Environment: Laboratory air, room temperature.</i></p>
<p><b>2 Characterisation method</b></p>	<p><i>Hardness Value Determination. Additionally, the Vickers microhardness test can be combined with other characterization techniques to gain further insights into the material's properties:</i></p> <ul style="list-style-type: none"> <li>• <i>combined with microstructural analysis using microscopy techniques can provide a correlation between the material's microstructure and its hardness properties;</i></li> <li>• <i>Phase Identification is possible identifying variations in hardness;</i></li> <li>• <i>Depth Profiling, combined with indentation depth measurements to assess the hardness as a function of depth below the sample surface.</i></li> </ul>
<p><b>3 Validation of Characterisation</b></p>	<p><i>Vickers microhardness not only provides information about the hardness, but can also be used to assess other mechanical properties such as cohesion, and adhesion of the coating to the substrate. Hardness measurement can be correlated with other coating properties and help evaluate its suitability for specific applications.</i></p>
<p><b>4 Access conditions (what is needed to repeat the experiment)</b></p>	<p><i>The sample preparation require an in-house routine.</i></p> <p><i>The characterisation tool require an in-house routine.</i></p>

#### 1. USER CASE

<p>1.1</p>	<p><b>USER</b></p>	<p><i>The user must be properly trained in the execution of the experiment, including the simple manual operations that need to be performed with accuracy.</i></p>
<p>1.2</p>	<p><b>User case (sample specifications)</b></p>	<p><i>No specific Sample dimensions is required for the test, however, it is important that the sample's surface must be flat and polished.</i></p>

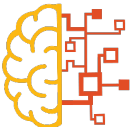


1.3	<b>Specimen</b>	<i>Any solid sample can be tested included bulk material, coating, heterogeneous material, biomaterial, etc.</i>
1.4	<b>Sampling process</b>	<i>The sample is usually obtained by metallographic cutting from a (coated) plate. Alternatively, plates/discs can be manufactured at the required size (and coated).</i>
1.5	<b>Sample preparation</b>	<i>Samples can be employed "as-is" or can be surface ground and polished (preferably to <math>Ra/Sa \leq 0.1 \mu m</math>). Surface roughness is verified by profilometry on at least one sample per batch.</i>
1.6	<b>Hazard</b>	<i>No specific hazard occur</i>
1.7	<b>Characterisation environment</b>	<i>Laboratory air, room temperature.</i>

## 2. EXPERIMENT

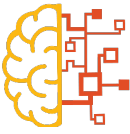
### Interaction nature and character (destructive or non-destructive) of the probe with the sample

2.1	<b>Probe/Physics of interaction</b>	<p><i>Elastic Deformation: As the diamond indenter contacts the material's surface, it exerts a force, causing elastic deformation in both the diamond indenter and the material. Elastic deformation occurs when the atoms in the material are displaced from their equilibrium positions but return to their original positions once the force is removed.</i></p> <p><i>Plastic Deformation: If the applied force exceeds the material's elastic limit, plastic deformation occurs. Plastic deformation involves the permanent displacement of atoms or dislocations in the material. The material undergoes plastic flow, and its structure is altered due to the rearrangement of atoms.</i></p>
2.2	<b>Interaction Volume</b>	<i>The dimensions of the interaction volume are typically determined by the contact area between the indenter and the material and the depth of the indentation.</i>
2.3	<b>Calibration process</b>	<i>No periodic calibration needed.</i>
2.4	<b>Detector</b>	<i>An internal force measurement system is used to measure the force applied during indentation.</i>
2.5	<b>Signal</b>	<i>An optical microscopy is used to view the residual imprint.</i>
2.6	<b>Measurement time</b>	<i>A typical test duration is 5 – 10 min</i>
2.7	<b>Measurement parameters</b>	<i>The main input parameters to acquire the signal are the load and the location of the test.</i>



3. RAW DATA		
3.1	<b>Raw Data</b>	<i>Vickers tip residual imprint</i>
3.2	<b>Unit</b>	<i>X coordinate – <math>\mu\text{m}</math> Y coordinate - <math>\mu\text{m}</math></i>
3.3	<b>Data acquisition rate</b>	<i>Automatically set by the instrument</i>

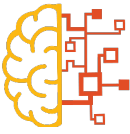
4. DATA PROCESSING		
4.1	<b>Level of expertise</b>	<i>Little expertise: Person can read out results directly</i>
	<b>Data normalisation</b>	<i>None</i>
4.2.	<b>Processing reproducibility</b>	<i>Easily reproducible for everyone</i>
4.3	<b>Data filtering processes</b>	<i>No data filtering</i>
4.4	<b>Data analysis procedures</b>	<i>Through visualization with the optical microscope, the length of the diagonal of the residual imprint is measured and automatically sent to the instrument's own software.</i>
4.5	<b>Main processed signals</b>	<i>The image of the residual imprint.</i>
4.6	<b>Data processing through calibrations</b>	<i>No correction of raw data</i>
4.7	<b>Properties (elaborated data)</b>	<i>The internal software of the instrument associates the value of the residual imprint length with that of the load applied during the test and generates the hardness value.</i>
	<b>Quality of the data</b>	<i>Each test produces only one imprint and therefore only one hardness value: to have a reliable value it is therefore necessary to evaluate the sufficient number of tests to be carried out through a basic statistical analysis.</i>
4.8.	<b>Data management</b>	<i>Microhardness value will be stored locally.</i>



## 4.2.2 Nanoindentation

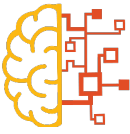
### OVERVIEW OF THE CHARACTERISATION

<p><b>1 User Case</b></p>	<p><i>Useful to Bulk material, coatings, heterogeneous materials, bio-material.</i></p> <p><i>Equipment Box: Air, Temperature, Pressure, Humidity, Noise, Vibrations (Acoustic or mechanical).</i></p>
<p><b>2 Characterisation method</b></p>	<p><i>Hardness and Elastic Modulus Measurement: The nanoindenter is primarily used to measure the hardness and elastic modulus of materials on a nanoscale. It applies a controlled load to the surface of the sample and measures the resulting indentation depth. From these measurements, the hardness and elastic modulus of the material can be calculated using the Oliver-Pharr method.</i></p> <p><i>Three acquisition methods can be distinguished</i></p> <ul style="list-style-type: none"> <li>• <i>Static nanoindentation: A constant load is applied to the sample: only single value of H and E is calculated, related to the overall depth set.</i></li> <li>• <i>Dynamic nanoindentation: The load is applied oscillatory: the acquisition is continuous, as the oscillation makes it possible to calculate the values of H and E for several depths of sinking, up to the overall one set.</i></li> <li>• <i>3D-4D high speed maps: by making nanoindentation tests extended to a large area (thanks to the high speed of the test), it is possible to create an H and E map that is 3D (i.e. <math>H(x,y)</math>) or 4D (i.e. <math>H(x,y,z)</math>).</i></li> </ul>
<p><b>3 Validation of Characterisation</b></p>	<p><i>The nanoindenter provides the necessary resolution to assess the mechanical behavior and properties at the nanoscale.</i></p> <p><i>The nanoindenter offers a wide range of mechanical property measurements. Nanoindentation is also a non-destructive testing technique: it requires minimal sample preparation and leaves small indents on the surface.</i></p>
<p><b>4 Access conditions (what is needed to repeat the experiment)</b></p>	<p><i>The sample preparation require an in-house routine.</i></p> <p><i>The characterisation tool require an in-house routine.</i></p>



1. USER CASE		
1.1	<b>USER</b>	<i>Human Operator (different levels of automation are available)</i>
1.2	<b>Sample</b>	<i>Sample dimensions (1 inch diam. – 5 mm thick.). Surface flat and polished. Sample embedding on sample-holder (hot glue or acrylic glue). Optical sample surface alignment with reference sample (SiO<sub>2</sub>) surface.</i>
1.3	<b>Sample material properties</b>	<i>Bulk material, coatings, heterogeneous materials, bio-material.</i>
1.4	<b>Sampling process</b>	<i>The sample is usually obtained by metallographic cutting from the part to be characterized. Alternatively, parts can be manufactured at the required size (and coated).</i>
1.5	<b>Sample preparation</b>	<i>No specific sample preparation is requested</i>
1.6	<b>Hazard</b>	<i>No hazard occurred</i>
1.7	<b>Characterisation environment</b>	<i>Equipment Box: Air, Temperature, Pressure, Humidity, Noise, Vibrations (Acoustic or mechanical).</i>

2. EXPERIMENT		
Interaction nature and character (destructive or non-destructive) of the probe with the sample		
2.1	<b>Probe/Physics of interaction</b>	<ul style="list-style-type: none"> <li>- <i>Elastic Deformation: As the diamond indenter contacts the material's surface, it exerts a force, causing elastic deformation in both the diamond indenter and the material. Elastic deformation occurs when the atoms in the material are displaced from their equilibrium positions but return to their original positions once the force is removed.</i></li> <li>- <i>Plastic Deformation: If the applied force exceeds the material's elastic limit, plastic deformation occurs. Plastic deformation involves the permanent displacement of atoms or dislocations in the material. The material undergoes plastic flow, and its structure is altered due to the rearrangement of atoms.</i></li> </ul>
2.2	<b>Interaction Volume</b>	<i>The interaction volume can be approximated using analytical or numerical models. One commonly used model is the Oliver-Pharr model, which assumes that the interaction volume is approximately conical in shape. The dimensions of the interaction volume are typically determined by the contact area between the indenter and the material and the depth of the indentation.</i>
2.3	<b>Calibration process</b>	<i>Standard CSM tests on reference sample.</i>
2.4	<b>Detector</b>	<i>Electronic controllers and capacitive gauges.</i>
2.5	<b>Signal</b>	<i>Electrical current in a coil -&gt; Force (Load)</i>
2.6	<b>Measurement time</b>	<i>A typical test lasts between 3 and 8 h depending on the input parameters selected</i>



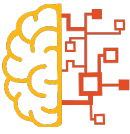
2.7	<b>Measurement parameters</b>	<i>Optical alignment of the sample. Method selection and Input parameters for the test (Sample Poisson's Ratio, Prescribed Depth or Load, number of tests, locations of the tests, Engage options).</i>
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### 3. RAW DATA

3.1	<b>Raw Data</b>	<i>Hardware and software channels graphs (Raw Load, Raw displacement, Stiffness, Load, Depth...).</i>
3.2	<b>Unit</b>	<ul style="list-style-type: none"> <li>• Displacement Into Surface (nm)</li> <li>• Hardness (GPa)</li> <li>• Harmonic Contact Stiffness (N/m)</li> <li>• Load (mN)</li> <li>• Stiffness Squared Over Load(GPa)</li> <li>• Time (s)</li> </ul>
3.3	<b>Data rate acquisition</b>	<i>Defined by the user as a parameter; it specifies the force variation with time required to achieve the movement of the indenter tip toward the sample surface with a specified velocity .</i>

### 4. DATA PROCESSING

4.1	<b>Level of expertise</b>	<i>Domain expertise: Person needs to be able to retrieve data and fit data.</i>
	<b>Data normalisation</b>	<i>No normalisation</i>
4.2.	<b>Processing reproducibility</b>	<i>Reproducible for a domain expert</i>
4.3	<b>Data filtering processes</b>	<i>No filtering process</i>
4.4	<b>Data analysis procedures</b>	<ul style="list-style-type: none"> <li>-Check of the surface detection</li> <li>-Check of the Load vs depth quadratic curve trend,</li> <li>-Check of the slope of the unloading curve,</li> <li>-Removal of the not significative tests.</li> <li>- Application of the Oliver-Pharr method (or other data analysis methods)</li> </ul>
4.5	<b>Main processed signals</b>	<ul style="list-style-type: none"> <li>• Load</li> <li>• Displacement</li> </ul>
4.6	<b>Data processing through calibrations</b>	<i>Calibration helps to obtain more accurate and reliable measurements of mechanical properties minimizing the systematic errors arising from the imperfect indenter geometry. Raw data are corrected applying the area tip function correction. The area tip function describes the contact area between the indenter and the sample as a function of the recorded depth. To extract the area tip function Oliver-Pharr method is used.</i>



4.7	<b>Properties (elaborated data)</b>	<ul style="list-style-type: none"><li>• <i>Raw data calibration using tests on reference sample,</i></li><li>• <i>Check of the results (see data analysis),</i></li><li>• <i>Selection of the load (or depth) range to evaluate the mechanical properties,</i></li><li>• <i>Graphs or hystograms of interest.</i></li></ul>
	<b>Quality of the data</b>	<ul style="list-style-type: none"><li>• <i>Statistical analysis of Young's modulus and hardness (from multiple repeat tests)</i></li></ul>
4.8.	<b>Data management</b>	<i>All data are stored locally and in the project's SharePoint folder.</i>

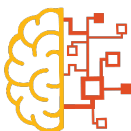


## 4.3 Wear characterization methods

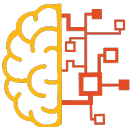
### 4.3.1 Sliding wear – Pin on disk

#### OVERVIEW OF THE CHARACTERISATION

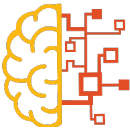
1	<b>User Case</b>	<p><i>Sample: Coated or uncoated plate, ground/polished (<math>Ra/Sa \leq 0.1 \mu m</math>)</i></p> <p><i>Test environment: laboratory air</i></p>
2	<b>Characterisation method</b>	<p><i>Execution of a pin(ball)-on-disc experiment on a flat sample, usually obtained from a coated or uncoated plate through metallographic preparation methods (abrasive cutting, grinding with SiC papers of diamond discs, polishing with diamond suspensions, optional polishing with colloidal silica), against a static counterbody with flat or spherical geometry made of a standard material (typically, <math>Al_2O_3</math>, yttria-stabilized zirconia, WC-6%Co, or 100Cr6 steel)</i></p> <hr/> <p><i>Optical inspection and measurement of pin(ball) wear</i></p> <hr/> <p><i>SEM+EDX analysis of worn sample (surface and optionally cross-section)</i></p> <hr/> <p><i>Micro-Raman spectroscopy analysis of worn sample surface and/or wear debris</i></p>
3	<b>Validation of Characterisation</b>	<p><i>The pin(ball)-on-disc test is a simple but widely accepted and recognized method to simulate sliding wear processes. Suitable selection of test conditions allows to reproduce a variety of wear mechanisms and to match those expected in actual applications.</i></p> <p><i>See:</i></p> <p><i>N. Axén, S. Hogmark, S. Jacobson, in: Modern Tribology Handbook, CRC Press, USA, 2001, Ch. 13</i></p> <p><i>ASTM G99-17: Standard Test Method for Wear Testing with a Pin-on-Disk Apparatus</i></p>
4	<b>Access conditions (what is needed to repeat the experiment)</b>	<p><i>Sample preparation: in-house routine</i></p> <p><i>Access to characterization tools: in-house routine</i></p> <p><i>Commercial post-processing software (MS Excel, Matlab).</i></p>



1. USER CASE		
1.1	<b>USER</b>	<p><i>The user must be properly trained in the execution of the experiment.</i></p> <p><i>Sample setup involves several manual operations that need to be performed with accuracy</i></p>
1.2	<b>Sample</b>	<p><i>Flat: discs (<math>\varnothing = 30, 32</math> or <math>50</math> mm) or corresponding inscribed squares, <math>3 - 5</math> or <math>8 - 10</math> mm thickness. Samples can be bulk or coated.</i></p> <p><i>Samples should have a planarity error on the test face and a parallelism error between the test face and the opposite face both lower than <math>0.01</math> mm although less precise samples can be tested. Noise in the friction and vertical counterbody position signals must be expected in the latter case.</i></p> <p><i>Counterbody: usually, commercial balls with mirror-like surface finish, <math>\varnothing = 3</math> or <math>6</math> mm, bulk (uncoated). Other ball sizes of flat samples can be procured if needed. The ball must be cleaned in an ultrasonic bath using an organic solvent (e.g. ethanol or acetone) for degreasing prior to testing.</i></p>
1.3	<b>Sample material properties</b>	<p><i>Any solid sample can be tested.</i></p>
1.4	<b>Sampling process</b>	<p><i>The sample is usually obtained by metallographic cutting from a (coated) plate. Alternatively, plates/discs can be manufactured at the required size (and coated).</i></p>
1.5	<b>Sample preparation</b>	<p><i>Samples should be surface ground and polished to <math>Ra/Sa \leq 0.1 \mu\text{m}</math> and cleaned in an ultrasonic bath using an organic solvent (e.g. ethanol or acetone) for degreasing prior to testing. Roughness is verified by profilometry on at least one sample per batch.</i></p> <p><i>The ball must be cleaned in an ultrasonic bath using an organic solvent (e.g. ethanol or acetone) for degreasing prior to testing.</i></p> <p><i>The sample is mounted into the disc holder using a flange fixed with screws. A support disc can be used for thin samples.</i></p> <p><i>The ball is mounted on a ball holder and fixed with a screw. Different ball holders must be used for room-temperature or high-temperature testing. The holder is fixed onto the swinging arm with a grain.</i></p>
1.6	<b>Hazard</b>	<p><i>Organic solvents for cleaning can be hazardous (e.g. ethanol – flammable liquid, causes serious eye irritation; acetone – flammable liquid, causes serious eye irritation, causes drowsiness or dizziness by inhalation, repeated exposure may cause skin dryness or cracking).</i></p> <p><i>The tribometer must not be touched while hot (risk of severe skin burns).</i></p>
1.7	<b>Characterisation environment</b>	<p><i>Laboratory air, optional induction heating of the disc.</i></p>



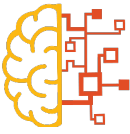
2. EXPERIMENT		
Interaction nature and character (destructive or non-destructive) of the probe with the sample		
2.1	<b>Probe/Physics of interaction</b>	<p>The probe is a stationary flat or rounded (e.g. spherical) body (“counterbody”) pressed against the flat moving sample by a known normal load applied either via dead weight using a swinging arm or via an actuator (electromechanical, pneumatic or hydraulic) equipped with a normal load sensor and a feedback control system. The sample is rotated unidirectionally at a fixed, user-defined revolution speed. The sample and counterbody can optionally be heated to a specified temperature.</p> <p>The relative motion between the sample and the counterbody causes wear, defined as a volumetric material loss from the sample and/or the counterbody, and friction, defined as the resistance against the relative motion. Wear can result from one or more of the following mechanisms: adhesion, abrasion, tribochemical interactions, surface fatigue. Friction can result from one or more of the following phenomena: adhesion, abrasion, viscoelastic hysteresis, third-body effects.</p> <p>The progression of the volumetric wear (<math>V</math>) of the sample and the probe (counterbody) is correlated to the applied normal load (<math>F_N</math>) and the total sliding distance (<math>s</math>) by Archard's law:  <math display="block">V = (K_{ad}/H) \cdot F_N \cdot s</math></p> <p>The resistance against relative motion is quantified by measuring the tangential force between the bodies (frictional force, <math>F_T</math>) and the friction coefficient is defined as:  <math display="block">\mu = F_T/F_N</math></p>
2.2	<b>Interaction Volume</b>	<p>The interaction volume, i.e. the extension of the contact stress distribution and the location of the equivalent stress maximum within the sample and the counterbody, depends on the counterbody geometry (e.g. flat or ball) and size (e.g. ball radius), on the applied normal load, and on the magnitude of the friction coefficient.</p> <p>In a frictionless non-conformal sphere-on-plane contact, the radius (<math>a</math>) of the circular contact area due to elastic deformation between the mating bodies is:</p> $a = \left( \frac{3F_N R'}{4E'} \right)^{\frac{1}{3}}$ <p>Where:</p> $E' = \left( \frac{1-\nu_1^2}{E_1} + \frac{1-\nu_2^2}{E_2} \right)^{-1}$ <p><math>E_i, \nu_i</math> (<math>i=1,2</math>) = elastic modulus and Poisson's ratio of the sample and the counterbody</p> $R' = \left( \frac{1}{R_1} + \frac{1}{R_2} \right)^{-1}$ <p><math>R_i</math> (<math>i=1,2</math>) = curvature radius of the sample and the counterbody (for a flat sample, <math>R_1 = \infty</math>)</p> <p>The depth (<math>z</math>) of the shear stress maximum is <math>z \approx 0.48 \cdot a</math>.</p>



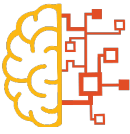
		<i>In a conformal contact, the stress maximum is located on the contact surface and the extension of the contact area is coincident with the extension of the smallest of the mating bodies.</i>
<b>2.3</b>	<b>Calibration process</b>	<i>The calibration of the frictional force sensor (with approximately monthly frequency) is based on measuring the sensor output voltage under no applied load and under a known tangential load applied via a dead weight and a pulley system, building a linear force/voltage response curve.</i>
<b>2.4</b>	<b>Detector</b>	<ol style="list-style-type: none"> <li>1) <i>Frictional force detector: load cell attached to the static counterbody holder. It contains a deformable element (thin metal sheet) attached to a displacement transducer that generates an electric voltage in response to a deformation.</i></li> <li>2) <i>Static counterbody position monitor: a displacement transducer sensing the displacement of the static counterbody holder in the direction perpendicular to the sample surface.</i></li> <li>3) <i>Thermocouple in contact with the rear face of the flat sample or with the support disc.</i></li> </ol>
<b>2.5</b>	<b>Signal</b>	<i>Electric voltage</i>
<b>2.6</b>	<b>Measurement time</b>	<i>A typical sliding wear test duration lasts from approx. 30 min to several hours.</i>
<b>2.7</b>	<b>Measurement parameters</b>	<i>Normal force (N), relative sliding speed (m/s), total sliding distance (m) or test duration (s), wear track radius (m), output data sampling frequency (Hz), testing temperature (°C).</i>

### 3. RAW DATA

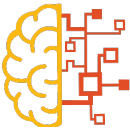
<b>3.1</b>	<b>Raw Data</b>	<p><i>Text file with tangential force (<math>F_T</math>), vertical position of the static counterbody, furnace temperature and sample temperature as a function of test time and elapsed distance.</i></p> <p><i>Physically worn sample and counterbody.</i></p>
<b>3.2</b>	<b>Unit</b>	<p><math>F_T</math> – N</p> <p>Vertical position – m</p> <p>Test time – s</p> <p>Distance – m</p> <p>Sample temperature – °C</p> <p>Furnace temperature – °C</p>
<b>3.3</b>	<b>Data acquisition rate</b>	<i>Set by the user, typically at least 3 times the disc rotation speed, therefore usually in the range of <math>10^0</math> – <math>10^2</math> Hz.</i>



4. DATA PROCESSING		
4.1	<b>Level of expertise</b>	<i>Domain expertise: Person needs to be able to retrieve data and fit data.</i>
	<b>Data normalisation</b>	<i>Calculation of friction coefficient <math>\mu = F_T/F_N</math></i>
4.2.	<b>Processing reproducibility</b>	<i>Reproducible for a domain expert (see the round-robin test results in ASTM G99 as well as E. Rabinowicz, J. Lubrication Technol. 103(2) (1981) 188-193)</i>
4.3	<b>Data filtering processes</b>	<i>Optional FFT or adjacent-average filter to remove instrumental noise and vibrations (planarity errors, misalignment, etc.)</i>
4.4	<b>Data analysis procedures</b>	<ul style="list-style-type: none"> <li>- <i>Calculation of friction coefficient <math>\mu = F_T/F_N</math></i></li> <li>- <i>Identification of the transition point from run-in stage to steady-state stage</i></li> <li>- <i>Averaging of the friction coefficient over the steady-state stage</i></li>   <li>- <i>Weighing of flat pin before and after the wear test</i></li> <li>- <i>Calculation of mass loss</i></li> <li>- <i>Calculation of volume loss = mass loss/density</i></li> <li>- <i>Calculation of specific wear rate</i></li>   <li>- <i>Observation of worn ball under an optical microscope</i></li> <li>- <i>Measurement of the diameter of the worn cap (if any)</i></li> <li>- <i>Calculation of wear cap volume</i></li> <li>- <i>Calculation of specific wear rate</i></li>   <li>- <i>Profilometric analysis of the worn flat sample</i></li> <li>- <i>Calculation of wear track volume</i></li> <li>- <i>Calculation of specific wear rate</i></li> </ul>
4.5	<b>Main signals processed</b>	<ul style="list-style-type: none"> <li>- <i>Friction force (output from load cell)</i></li> <li>- <i>Vertical displacement of static counterbody holder (output from position sensor)</i></li> <li>- <i>Worn sample</i></li> <li>- <i>Worn counterbody</i></li> </ul>
4.6	<b>Data processing through calibrations</b>	<i>No correction of raw data</i>
4.7	<b>Properties (elaborated data)</b>	<p><i>Averaging of the friction coefficient over the steady-state stage.</i></p> <p><i>Specific wear rate of the static counterpart</i></p> <p><i>Specific wear rate of the flat sample</i></p>



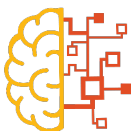
	<b>Quality of the data</b>	<ul style="list-style-type: none"><li>- <i>Statistical analysis of friction data (single friction curve): standard deviation, 95% confidence interval associated with the average steady-state friction coefficient of a single test.</i></li><li>- <i>Statistical analysis of friction data (from multiple repeat tests): half-difference between maximum and minimum value (if up to 4 repeats), standard deviation (if 5 or more repeats).</i></li><li>- <i>Statistical analysis of specific wear rates (from multiple repeat tests): half-difference between maximum and minimum value (if up to 4 repeats), standard deviation (if 5 or more repeats).</i></li></ul>
4.8.	<b>Data management</b>	<p><i>Friction data stored locally and in the project's SharePoint folder.</i></p> <p><i>Optical microscopy images stored locally and in the project's SharePoint folder.</i></p> <p><i>Raw profilometry data stored locally (on machine or in a Cloud storage facility, e.g. MS OneDrive) for at least 2 years [note extremely large file size].</i></p> <p><i>Computed pin(ball) and sample volume data stored locally and in the project's SharePoint folder.</i></p>



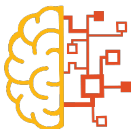
### 4.3.2 Jet erosion test

#### OVERVIEW OF THE CHARACTERISATION

<b>1 User Case</b>	<p><i>Sample: Coated or uncoated plate, ground/polished (<math>Ra/Sa \leq 0.1 \mu m</math>) or as-supplied</i></p> <p><i>Test environment: laboratory air</i></p>
<b>2 Characterisation method</b>	<p><i>Execution of a jet erosion experiment on a flat sample, usually obtained from a coated or uncoated plate through metallographic preparation methods (abrasive cutting), either in the as-supplied condition or after grinding with SiC papers of diamond discs and polishing with diamond suspensions (optional polishing with colloidal silica), using abrasive particles (usually <math>Al_2O_3</math>, <math>50 \mu m</math> average size) accelerated through a calibrated orifice (usually with <math>\varnothing = 1.5 mm</math>) by compressed air.</i></p> <p><i>SEM+EDX analysis of worn sample (surface and optionally cross-section)</i></p>
<b>3 Validation of Characterisation</b>	<p><i>The jet erosion test is the most appropriate method to simulate a dry particle erosion process. Suitable selection of test conditions allows to simulate erosion at different impingement energies and different impact angles.</i></p> <p><i>See:</i></p> <p><i>ASTM G76-18: Standard Test Method for Conducting Erosion Tests by Solid Particle Impingement Using Gas Jets</i></p> <p><i>ASTM G211-14(2020): Standard Test Method for Conducting Elevated Temperature Erosion Tests by Solid Particle Impingement Using Gas Jets</i></p>
<b>4 Access conditions (what is needed to repeat the experiment)</b>	<p><i>Sample preparation: in-house routine</i></p> <p><i>Access to characterization tools: in-house routine</i></p>

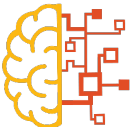


1. USER CASE		
1.1	<b>USER</b>	<p><i>The user must be properly trained in the execution of the experiment.</i></p> <p><i>Sample setup involves manual operations that need to be performed with accuracy.</i></p>
1.2	<b>Sample</b>	<p><i>Flat sample: 19.5 mm squares or 25 mm discs, 3 – 5 mm thick. Samples can be bulk or coated.</i></p>
1.3	<b>Sample material properties</b>	<p><i>Any solid sample can be tested.</i></p>
1.4	<b>Sampling process</b>	<p><i>The sample is usually obtained by metallographic cutting from a (coated) plate. Alternatively, plates/discs can be manufactured at the required size (and coated).</i></p>
1.5	<b>Sample preparation</b>	<p><i>Samples can be employed “as-is” or can be surface ground and polished (preferably to <math>Ra/Sa \leq 0.1 \mu m</math>), and in any case must be cleaned in an ultrasonic bath using an organic solvent (e.g. ethanol or acetone) for degreasing prior to testing. Surface roughness is verified by profilometry on at least one sample per batch.</i></p> <p><i>The sample is laid into the holder using a suitable fixture. The holder is hanged directly below the jet nozzle using the provided frame.</i></p>
1.6	<b>Hazard</b>	<p><i>Whilst the abrasive material (<math>Al_2O_3</math>) is inert, the user must wear a protective dust mask to avoid inhaling the powder during every stage of the execution of the test.</i></p> <p><i>The tribometer must not be touched while hot (risk of severe skin burns) and removal of hot samples must be performed with the provided clamps and wearing heat-resistant gloves.</i></p> <p><i>Organic solvents for cleaning can be hazardous (e.g. ethanol – flammable liquid, causes serious eye irritation; acetone – flammable liquid, causes serious eye irritation, causes drowsiness or dizziness by inhalation, repeated exposure may cause skin dryness or cracking).</i></p>
1.7	<b>Characterisation environment</b>	<p><i>Laboratory air, optional resistance heating of the sample and the air jet.</i></p>



2. EXPERIMENT		
Interaction nature and character (destructive or non-destructive) of the probe with the sample		
<b>2.1</b>	<b>Probe/Physics of interaction</b>	<p><i>The probe is a jet of solid (dry) particles carried by compressed air. The particles typically exhibit angular shape and are harder than the tested sample.</i></p> <p><i>The impingement of the particles onto the sample surface causes wear, mostly by abrasion and secondarily by surface fatigue.</i></p> <p><i>Ductile materials, which are prone to ductile abrasive wear by plastic grooving mechanisms including cutting, ploughing, wedging and indentation by the impinging particles, often exhibit maximum erosion rates at impact angles of 30°-45°.</i></p> <p><i>Brittle materials, which are prone to abrasion by brittle fracture and to surface fatigue, often exhibit maximum erosion rates at impact angles approaching 90° and display a marked transition from mild to severe wear with increasing kinetic energy of the impinging particles.</i></p> <p><i>Because the exact kinetic energy of each impinging particle, the amount of energy it transfers to the eroded surface, and the duration of each individual contact are hardly definable, no universally accepted relation exists to relate the progression of volumetric wear to the erosion conditions.</i></p>
<b>2.2</b>	<b>Interaction Volume</b>	<p><i>The sample is affected by the impinging solid particle jet down to depths that are usually of the order of some tens or few hundreds of micrometres.</i></p>
<b>2.3</b>	<b>Calibration process</b>	<p><i>The calibration of particle velocity as a function of air pressure is done with a double-disc apparatus.</i></p> <p><i>The validation of the powder feed rate is done by weighing the mass of powder fed through the nozzle under pressure-less conditions for a defined period of time.</i></p>
<b>2.4</b>	<b>Detector</b>	<p><i>No detector directly coupled to the sample to measure wear-related parameters</i></p> <p><i>(Optional) thermocouple to monitor the sample temperature</i></p>
<b>2.5</b>	<b>Signal</b>	<p><i>No direct signal related to wear parameters</i></p> <p><i>(Optional) Electric voltage from the thermocouple</i></p>
<b>2.6</b>	<b>Measurement time</b>	<p><i>A typical air jet erosion test lasts for approx. 1 – 10 min.</i></p> <p><i>Heating and cooling times must be considered when performing high-temperature tests, In this case, the duration of a single test can be of several hours.</i></p>
<b>2.7</b>	<b>Measurement parameters</b>	<p><i>Air pressure (bar), jet velocity (m/s), abrasive flow rate (g/s), duration (s), temperature (°C).</i></p>

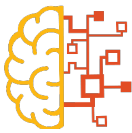
3. RAW DATA		
<b>3.1</b>	<b>Raw Data</b>	<p><i>Text file with sample temperature as a function of test time.</i></p> <p><i>Physically worn sample.</i></p>



3.2	<b>Unit</b>	Sample temperature – °C
3.3	<b>Data acquisition rate</b>	Usually in the range of $10^0 - 10^2$ Hz.

#### 4. DATA PROCESSING

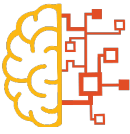
4.1	<b>Level of expertise</b>	Domain expertise: Person needs to be able to retrieve data and fit data.
	<b>Data normalisation</b>	Calculation of volumetric erosion rate = wear volume / mass of erosive particles impinging on the sample during the entire test = $V / (\dot{m} \cdot t)$ where $V$ = wear volume, $\dot{m}$ = mass flow rate of erosive particles, $t$ = erosion time.
4.2.	<b>Processing reproducibility</b>	Reproducible for a domain expert.
4.3	<b>Data filtering processes</b>	No data filtering
4.4	<b>Data analysis procedures</b>	<ul style="list-style-type: none"> <li>- Profilometric analysis of the worn sample</li> <li>- Calculation of wear track volume</li> <li>- Calculation of volumetric erosion rate</li> </ul>
4.5	<b>Main signals processed</b>	<ul style="list-style-type: none"> <li>- Worn sample</li> </ul>
4.6	<b>Data processing through calibrations</b>	No correction of raw data
4.7	<b>Properties (elaborated data)</b>	Volumetric erosion rate of the flat sample
	<b>Quality of the data</b>	Statistical analysis of specific wear rates (from multiple repeat tests): half-difference between maximum and minimum value (if up to 4 repeats), standard deviation (if 5 or more repeats).
4.8.	<b>Data management</b>	<p>Raw profilometry data stored locally (on machine or in a Cloud storage facility, e.g. MS OneDrive) for at least 2 years [note extremely large file size].</p> <p>Computed sample volume data stored locally and in the project's SharePoint folder.</p>



### 4.3.3 Abrasion Test

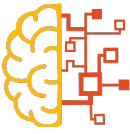
#### OVERVIEW OF THE CHARACTERISATION

1	<b>User Case</b>	<p><i>Sample: Coated or uncoated plate, ground/polished (<math>Ra/Sa \leq 0.1 \mu m</math>) or as-supplied</i></p> <p><i>Test environment: laboratory air</i></p>
2	<b>Characterisation method</b>	<p><i>Execution of a dry particle abrasion test on a flat sample, usually consisting of a coated or uncoated plate, either in the as-supplied condition or after grinding with SiC papers or diamond discs and polishing with diamond suspensions (optional polishing with colloidal silica). The sample is abraded by a flow of hard particles (usually <math>Al_2O_3</math>, <math>185 \mu m</math> average size) dragged onto its surface by the rotating motion of a steel disc pressed by a fixed normal force.</i></p> <p><i>SEM+EDX analysis of worn sample (surface and optionally cross-section)</i></p>
3	<b>Validation of Characterisation</b>	<p><i>The dry particle abrasion test with a steel wheel produces a high-stress, three-body abrasive wear process and it is particularly suitable for hard materials (e.g. hardmetal coatings) where it can readily reveal issues like brittleness or poor cohesion. See:</i></p> <p><i>V. Testa, S. Morelli, G. Bolelli, F. Bosi, P. Puddu, A. Colella, T. Manfredini, L. Lusvarghi, Corrosion and wear performances of alternative TiC-based thermal spray coatings, Surf. Coat. Technol. 438 (2022) 128400</i></p>
4	<b>Access conditions (what is needed to repeat the experiment)</b>	<p><i>Sample preparation: in-house routine</i></p> <p><i>Access to characterization tools: in-house routine</i></p>



1. USER CASE		
1.1	<b>USER</b>	<p>The user must be properly trained in the execution of the experiment.</p> <p>Sample and equipment setup involve manual operations that need to be performed with accuracy.</p>
1.2	<b>Sample</b>	Flat sample, at least 50×20 mm.
1.3	<b>Sample material properties</b>	Any solid sample can be tested.
1.4	<b>Sampling process</b>	The sample can be obtained by metallographic cutting from a (coated) plate. Alternatively, plates/discs can be manufactured at the required size (and coated).
1.5	<b>Sample preparation</b>	Samples can be employed “as-is” or can be surface ground and polished (preferably to $Ra/Sa \leq 0.1 \mu m$ ), and in any case must be cleaned in an ultrasonic bath using an organic solvent (e.g. ethanol or acetone) for degreasing prior to testing. Surface roughness is verified by profilometry on at least one sample per batch.
1.6	<b>Hazard</b>	<p>Whilst the abrasive material (<math>Al_2O_3</math>) is inert, the user must wear a protective dust mask to avoid dust inhalation (including fine debris produced by fragmentation of the abrasive and by wear of the sample) during every stage of the the test.</p> <p>Organic solvents for cleaning can be hazardous (e.g. ethanol – flammable liquid, causes serious eye irritation; acetone – flammable liquid, causes serious eye irritation, causes drowsiness or dizziness by inhalation, repeated exposure may cause skin dryness or cracking).</p>
1.7	<b>Characterisation environment</b>	Laboratory air.

2. EXPERIMENT		
Interaction nature and character (destructive or non-destructive) of the probe with the sample		
2.1	<b>Probe/Physics of interaction</b>	<p>The probe is a flow of free-falling, solid (dry) particles. The flow is tangential to the contact point between the vertically-oriented flat sample and the steel disc. The disc, which is loaded by a dead-weight against the sample surface and rotates at fixed speed, drags and presses the particles against the sample itself.</p> <p>The particles typically exhibit angular shape and are harder than the tested sample; thus, they cause abrasive grooving of the sample itself. The main form of wear is high-stress, three-body abrasion.</p>



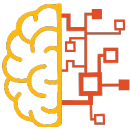
		The progression of the volumetric wear ( $V$ ) of the sample is correlated to the applied normal load ( $F_N$ ) and the total sliding distance of the disc relative to the sample surface ( $s$ ) by Archard's law: $V = (K_{ad}/H) \cdot F_N \cdot s$
2.2	<b>Interaction Volume</b>	The sample is affected by the impinging solid particle jet down to depths that are usually of the order of few hundreds of micrometres.
2.3	<b>Calibration process</b>	The calibration of the powder feed rate is done by weighing the mass of powder fed through an adjustable orifice for a defined period of time. The verification of the instrument is made by abrading a known reference material, e.g. HVOF-sprayed WC-10%Co-4%Cr
2.4	<b>Detector</b>	No detector.
2.5	<b>Signal</b>	No measured signal during the test.
2.6	<b>Measurement time</b>	A typical dry particle abrasion test lasts for approx. 1 – 5 min.
2.7	<b>Measurement parameters</b>	Abrasive flow rate (g/min), Normal load (N), Disc speed (rpm), Number of revolutions (-)

### 3. RAW DATA

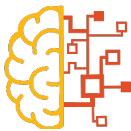
3.1	<b>Raw Data</b>	Physically worn sample.
3.2	<b>Unit</b>	$Mm^3$
3.3	<b>Data acquisition rate</b>	No direct data acquisition during the test.

### 4. DATA PROCESSING

4.1	<b>Level of expertise</b>	Domain expertise.
	<b>Data normalisation</b>	Calculation of specific wear rate = $V/(F_N \cdot s)$
4.2.	<b>Processing reproducibility</b>	Reproducible for a domain expert.
4.3	<b>Data filtering processes</b>	No data filtering



4.4	<b>Data analysis procedures</b>	<ul style="list-style-type: none"><li>- Profilometric analysis of the worn sample</li><li>- Calculation of wear track volume</li><li>- Calculation of specific wear rate</li></ul>
4.5	<b>Main processed signals</b>	<ul style="list-style-type: none"><li>- Worn sample</li></ul>
4.6	<b>Data processing through calibrations</b>	<i>No correction of raw data</i>
4.7	<b>Properties (elaborated data)</b>	<i>Specific wear rate of the flat sample</i>
	<b>Quality of the data</b>	<i>Statistical analysis of specific wear rates (from multiple repeat tests): half-difference between maximum and minimum value (if up to 4 repeats), standard deviation (if 5 or more repeats).</i>
4.8.	<b>Data management</b>	<i>Raw profilometry data stored locally (on machine or in a Cloud storage facility, e.g. MS OneDrive) for at least 2 years [note extremely large file size]. Computed sample volume data stored locally and in the project's SharePoint folder.</i>

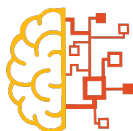


## 4.4 Corrosion characterization methods

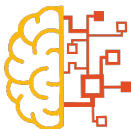
### 4.4.1 Electrochemical polarization test

#### OVERVIEW OF THE CHARACTERISATION

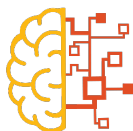
<p><b>1 User Case</b></p>	<p><i>Sample: Coated or uncoated metal plate, usually ground/polished (Ra/Sa ≤ 0.1 μm)</i></p> <p><i>Test environment: water-based electrolyte, aerated, room temperature</i></p>
<p><b>2 Characterisation method</b></p>	<p><i>Execution of an electrochemical polarization test on a flat sample, usually obtained from a coated or uncoated metal plate through metallographic preparation methods (abrasive cutting, grinding with SiC papers or diamond discs, polishing with diamond suspensions, optional polishing with colloidal silica), in contact with a liquid electrolyte, usually a water-based solution (e.g. 3.5 wt./vol.% NaCl), using a three-electrode cell where the sample is the working electrode, the counterelectrode is an inert material, e.g. a platinum plate, wire or mesh, and the reference electrode consists of a standard redox couple with known and stable electrochemical potential, e.g. Ag/AgCl/KCl<sub>(sat.)</sub></i></p> <p><i>SEM+EDX analysis of corroded sample surface and/or cross-section</i></p> <p><i>Micro-Raman spectroscopy analysis of corroded sample surface and/or cross-section</i></p>
<p><b>3 Validation of Characterisation</b></p>	<p><i>The electrochemical polarization test is a simple but widely accepted and recognized method to simulate an aqueous corrosion process, inferring both qualitative and quantitative information on the corrosion mechanisms and the corrosion rate</i></p> <p><i>See:</i></p> <p><i>J.R. Scully, D.W. Taylor, Electrochemical Methods of Corrosion Testing, in: S.D. Cramer, B.S. Covino, Jr. (Eds.), ASM Handbook Vol. 13A – Corrosion: Fundamentals, Testing, and Protection, ASM International, Materials Park, OH, USA, 2003</i></p>
<p><b>4 Access conditions (what is needed to repeat the experiment)</b></p>	<p><i>Sample preparation: in-house routine</i></p> <p><i>Access to characterization tools: in-house routine</i></p> <p><i>Commercial post-processing software (MS Excel, Matlab).</i></p>



1. USER CASE		
1.1	<b>USER</b>	<i>The user must be properly trained in the execution of the experiment, including the manual operations that need to be performed with accuracy, and in safe handling, storage, and disposal of chemicals.</i>
1.2	<b>Sample</b>	<p><i>Flat sample: discs or plates of 15 – 50 mm size and 3 – 5 mm thickness. Samples can be bulk or coated.</i></p> <p><i>The backside of the sample must be electrically conductive, i.e. it is important to ensure it is free from oxides or other insulating coverages.</i></p> <p><i>The sample must be cleaned in an ultrasonic bath using an organic solvent (e.g. ethanol or acetone) for degreasing prior to testing.</i></p>
1.3	<b>Sample material properties</b>	<i>Any electrically conductive solid sample can be tested.</i>
1.4	<b>Sampling process</b>	<i>The sample is usually obtained by metallographic cutting from a (coated) plate. Alternatively, plates/discs can be manufactured at the required size (and coated if needed).</i>
1.5	<b>Sample preparation</b>	<p><i>Samples must be surface ground and polished to <math>Ra/Sa \leq 0.1 \mu m</math>. The backside must be freed from oxides by grinding, if needed. The ground and polished sample must be cleaned in an ultrasonic bath using an organic solvent (e.g. ethanol or acetone) for degreasing prior to testing. Surface roughness is verified by profilometry on at least one sample per batch.</i></p> <p><i>The sample is mounted into the support of the electrochemical cell.</i></p>
1.6	<b>Hazard</b>	<p><i>Many electrolytes and the chemicals used in their preparation can be hazardous for human health and the environment. The user must refer to the SDSs of the relevant products and to lab practices for waste disposal. Execution of the experiment under a ventilation hood is usually required.</i></p> <p><i>Organic solvents for cleaning can be hazardous (e.g. ethanol – flammable liquid, causes serious eye irritation; acetone – flammable liquid, causes serious eye irritation, causes drowsiness or dizziness by inhalation, repeated exposure may cause skin dryness or cracking).</i></p>
1.7	<b>Characterisation environment</b>	<i>Laboratory air, room temperature.</i>



2. EXPERIMENT	
Interaction nature and character (destructive or non-destructive) of the probe with the sample	
<p><b>2.1</b></p> <p><b>Probe/Physics of interaction</b></p>	<p><i>Interaction of a metal with a water-based medium involves different stages of active corrosion, passivation, or immunity, depending on the electrochemical potential, pH, and nature of dissolved species, as inferable from a Pourbaix diagram for the relevant material and environment, which can also be obtained by theoretical computations.</i></p> <p><i>The thermodynamic driving force for an aqueous corrosion process is the difference between the equilibrium electrochemical potentials of the metal/metal cation couple and of the oxidizing species according to the Nernst equation:</i></p> $E = \left( E_{M/M^{n+}}^0 - E_{ox./red.}^0 \right) - 2.303 \frac{RT}{nF} \log \left( \frac{c_{M^{n+}} \cdot c_{red.}}{c_{ox.}} \right)$ <p><i>Where <math>E_{M/M^{n+}}^0</math> and <math>E_{ox./red.}^0</math> are the standard electrochemical potentials of the <math>M^0 \rightleftharpoons M^{n+}</math> and <math>ox. \rightleftharpoons red.</math> reactions; <math>ox.</math> and <math>red.</math> are the oxidized and reduced forms of the oxidizing species, e.g. <math>2H^+ \rightleftharpoons H_2</math> or <math>2H_2O + O_2 \rightleftharpoons 4OH^-</math>; <math>T</math> is the temperature; <math>R</math> is the ideal gas constant; <math>F</math> is Faraday's constant; <math>n</math> is the number of electrons exchanged in the metal oxidation process; <math>c_{M^{n+}}</math>, <math>c_{red.}</math>, <math>c_{ox.}</math> are the concentrations of the oxidized metal cations, the reduced and the oxidized form of the oxidizing agent.</i></p> <p><i>The kinetics of active corrosion are controlled by the electrochemical reaction rate according to the Butler-Volmer equation:</i></p> $i = i_0 \left\{ \exp \left( \frac{\alpha F}{RT} \eta \right) - \exp \left( \frac{(1 - \alpha) F}{RT} \eta \right) \right\}$ <p><i>Where <math>i</math> is the overall current in an electrical circuit connecting an actively dissolving anode and a cathode under an externally applied overpotential <math>\eta</math>; <math>i_0</math> is the exchange current, i.e. the absolute value of the anodic and cathodic currents under no applied overpotential (where the overall current is zero because the anodic and cathodic currents are identical in absolute value and with opposite sign); <math>\alpha</math> is a symmetry coefficient associated with the shape of the activation energy barrier and usually <math>\approx 0.5</math>; and all other terms have the meaning defined above.</i></p> <p><i>The kinetics of passive corrosion are controlled by the mass transport (diffusion) rate of oxidized metal cations and/or reduced oxidizer across the passive film covering the metal surface.</i></p>
<p><b>2.2</b></p> <p><b>Interaction Volume</b></p>	<p><i>The interaction volume is highly dependent on the type of response of the metal sample. A fully dense metal surface with defect-less passive film only interacts down to a depth equivalent to the passive film thickness, usually <math>\sim 10^0 - 10^1</math> nm. A porous surface which does not passivate and is infiltrated by the electrolyte reacts down to the same depth as the penetration depth of the electrolyte. Usually, a thermal spray coating containing some defects deposited onto a dense</i></p>



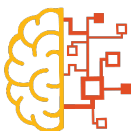
		<i>substrate reacts down to a depth comparable with its thickness, i.e. <math>\sim 10^2 \mu\text{m}</math>.</i>
2.3	<b>Calibration process</b>	<i>The verification of the potentiostat/galvanostat is performed yearly using a “dummy cell” consisting of an electrical circuit with known electrical resistances, which produces a known response when subjected to potential scans.</i>
2.4	<b>Detector</b>	<i>Potentiostat/galvanostat</i>
2.5	<b>Signal</b>	<i>Electric voltage and electric current</i>
2.6	<b>Measurement time</b>	<i>A typical electrochemical polarization test duration is 1 – 2 h</i>
2.7	<b>Measurement parameters</b>	<i>Stabilization time (s), Maximum cathodic overpotential (V), Maximum anodic overpotential (V), Potential step (V), Step time (s)</i>

### 3. RAW DATA

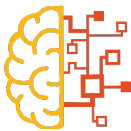
3.1	<b>Raw Data</b>	<i>Text file with current, potential, measurement time. Physically corroded sample.</i>
3.2	<b>Unit</b>	<i>Current – A Potential – V Measurement time – s</i>
3.3	<b>Data rate acquisition</b>	<i>Typically 1 measurement point per potential step. Typical potential step: 1 mV; Typical step time: 2 s <math>\Rightarrow</math> Typical potential scan rate mV/s</i>

### 4. DATA PROCESSING

4.1	<b>Level of expertise</b>	<i>Domain expertise: Person needs to be able to retrieve data and fit data.</i>
	<b>Data normalisation</b>	<i>Calculation of current density (<math>I</math>) by dividing the current (<math>i</math>) values over the exposed working electrode surface area (<math>A</math>): <math>I = i / A</math> [<math>\text{A}/\text{cm}^2</math>].</i>
4.2.	<b>Processing reproducibility</b>	<i>Reproducible for a domain expert.</i>
4.3	<b>Data filtering processes</b>	<i>No filtering</i>
4.4	<b>Data analysis procedures</b>	<ul style="list-style-type: none"> <li>- <i>Plotting of polarization curve: <math>\log(I)</math> vs. potential</i></li> <li>- <i>Identification of the rest potential and of the anodic and cathodic curves</i></li> <li>- <i>Identification of active, passive, and transpassive regions on the anodic curve</i></li> </ul>



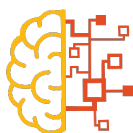
		<ul style="list-style-type: none"> <li>- Identification of the passivity potential range (V) and the transpassivation potential (V)</li> <li>- Averaging of the current density over the passive potential range to identify the average passive current density (A/cm<sup>2</sup>)</li> <li>- Identification of the first linear portions on the anodic and cathodic polarization curves at an overpotential <math> \eta  \geq 50</math> mV</li> <li>- Linear (Tafel) fit: identification of anodic and cathodic Tafel slopes (V/decade)</li> <li>- Calculation of the intersection point of the anodic and cathodic Tafel fits: corrosion potential (V), corrosion current density (A/cm<sup>2</sup>)</li> <li>- Usually done with commercial software packages (MS Excel, Matlab)</li> <li>- SEM+EDX analysis of corroded sample (surface and/or cross-section): identification of corrosion mechanisms</li> <li>- Micro-Raman spectroscopic analysis of corroded sample (surface and/or cross-section): identification of corrosion product compounds.</li> </ul>
4.5	<b>Main processed signals</b>	<ul style="list-style-type: none"> <li>- Current (A)</li> <li>- Voltage (V)</li> <li>- Corroded sample</li> </ul>
4.6	<b>Data processing through calibrations</b>	No correction of raw data
4.7	<b>Properties (elaborated data)</b>	<p>Passivity range (V)</p> <p>Transpassivation potential (V)</p> <p>Passive current density (A/cm<sup>2</sup>)</p> <p>Corrosion potential (V)</p> <p>Corrosion current density (A/cm<sup>2</sup>)</p> <p>Cathodic Tafel slope (V/decade)</p> <p>Anodic Tafel slope (V/decade)</p>
	<b>Quality of the data</b>	<ul style="list-style-type: none"> <li>- Evaluation of S/N ratio of anodic and cathodic polarization curves</li> <li>- Statistical analysis of elaborated data (from multiple repeat tests): half-difference between maximum and minimum value (if up to 4 repeats), standard deviation (if 5 or more repeats).</li> </ul>
4.8.	<b>Data management</b>	Raw data files (polarization curves) and elaborations stored locally and on the project's SharePoint folder



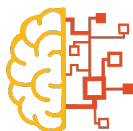
## 4.4.2 Electrochemical Impedance Spectroscopy

### OVERVIEW OF THE CHARACTERISATION

1	<b>User Case</b>	<p><i>Sample: Coated or uncoated metal plate, usually ground/polished (<math>Ra/Sa \leq 0.1 \mu m</math>)</i></p> <p><i>Test environment: water-based electrolyte, aerated, room temperature</i></p>
2	<b>Characterisation method</b>	<p><i>Execution of an electrochemical impedance spectroscopy test on a flat sample, usually obtained from a coated or uncoated metal plate through metallographic preparation methods (abrasive cutting, grinding with SiC papers or diamond discs, polishing with diamond suspensions, optional polishing with colloidal silica), in contact with a liquid electrolyte, usually a water-based solution (e.g. 3.5 wt./vol.% NaCl), using a three-electrode cell where the sample is the working electrode, the counterelectrode is an inert material, e.g. a platinum plate, wire or mesh, and the reference electrode consists of a standard redox couple with known and stable electrochemical potential, e.g. <math>Ag/AgCl/KCl_{(sat.)}</math></i></p>
3	<b>Validation of Characterisation</b>	<p><i>The electrochemical impedance spectroscopy (EIS) test is a recognized method to draw detailed information on the corrosion mechanisms of an uncoated or coated metallic sample, with the ability to break down the overall corrosion process into its constitutive stages, and to operate non-destructively as the applied voltage oscillation amplitude is minimum and does not accelerate the spontaneous corrosion rate.</i></p> <p><i>See:</i></p> <p><i>J.R. Scully, D.W. Taylor, Electrochemical Methods of Corrosion Testing, in: S.D. Cramer, B.S. Covino, Jr. (Eds.), ASM Handbook Vol. 13A – Corrosion: Fundamentals, Testing, and Protection, ASM International, Materials Park, OH, USA, 2003</i></p>
4	<b>Access conditions (what is needed to repeat the experiment)</b>	<p><i>Sample preparation: in-house routine</i></p> <p><i>Access to characterization tools: in-house routine</i></p> <p><i>Commercial post-processing software (Matlab).</i></p>



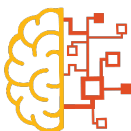
1. USER CASE		
1.1	<b>USER</b>	<i>The user must be properly trained in the execution of the experiment, including the manual operations that need to be performed with accuracy, and in safe handling, storage, and disposal of chemicals.</i>
1.2	<b>Sample</b>	<i>Flat sample: discs or plates of 15 – 50 mm size and 3 – 5 mm thickness. Samples can be bulk or coated.</i> <i>The backside of the sample must be electrically conductive, i.e. it is important to ensure it is free from oxides or other insulating coverages.</i> <i>The sample must be cleaned in an ultrasonic bath using an organic solvent (e.g. ethanol or acetone) for degreasing prior to testing.</i>
1.3	<b>Sample material properties</b>	<i>Any electrically conductive solid sample can be tested.</i>
1.4	<b>Sampling process</b>	<i>The sample is usually obtained by metallographic cutting from a (coated) plate. Alternatively, plates/discs can be manufactured at the required size (and coated if needed).</i>
1.5	<b>Sample preparation</b>	<i>Samples must be surface ground and polished to <math>Ra/Sa \leq 0.1 \mu\text{m}</math>. The backside must be freed from oxides by grinding, if needed. The ground and polished sample must be cleaned in an ultrasonic bath using an organic solvent (e.g. ethanol or acetone) for degreasing prior to testing. Surface roughness is verified by profilometry on at least one sample per batch.</i> <i>The sample is mounted into the support of the electrochemical cell.</i>
1.6	<b>Hazard</b>	<i>Many electrolytes and the chemicals used in their preparation can be hazardous for human health and the environment. The user must refer to the SDSs of the relevant products and to lab practices for waste disposal. Execution of the experiment under a ventilation hood is usually required.</i> <i>Organic solvents for cleaning can be hazardous (e.g. ethanol – flammable liquid, causes serious eye irritation; acetone – flammable liquid, causes serious eye irritation, causes drowsiness or dizziness by inhalation, repeated exposure may cause skin dryness or cracking).</i>
1.7	<b>Characterisation environment</b>	<i>Laboratory air, room temperature.</i>



## 2. EXPERIMENT

### Interaction nature and character (destructive or non-destructive) of the probe with the sample

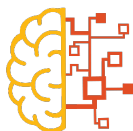
<b>2.1</b>	<b>Probe/Physics of interaction</b>	<p><i>Interaction of a metal with a water-based medium involves different stages of active corrosion, passivation, or immunity, depending on the electrochemical potential, pH, and nature of dissolved species, as inferable from a Pourbaix diagram for the relevant material and environment, which can also be obtained by theoretical computations.</i></p> <p><i>Each of these interaction types often consists of multiple electrochemical phenomena acting over different time scales. Examples can be the charge transfer across a (dense or porous) surface layer or coating, and the associated capacitive effects; the charge transfer across the electrical double-layer formed in contact with a polarized electrode, and the associated capacitive effects; etc.</i></p> <p><i>Each phenomenon thus controls the system's response to an externally applied AC electrical signal having a frequency within the range of its characteristic timescale.</i></p> <p><i>Specifically, each charge-transfer and capacitive effect can be viewed as equivalent to an electrical element (e.g. a resistor or a capacitor, respectively) in an electrical circuit that simulates the electrical response of the system.</i></p> <p><i>By probing the system's impedance when subjected to a small-amplitude AC voltage oscillation spanning a wide range of frequencies and fitting the impedance response to a suitable equivalent circuit model, it is therefore possible to measure the magnitudes of the corresponding electrical elements, which represent the quantification of physical phenomena occurring in the electrochemical system.</i></p>
<b>2.2</b>	<b>Interaction Volume</b>	<p><i>The interaction volume is highly dependent on the type of response of the metal sample. A fully dense metal surface with defect-less passive film only interacts down to a depth equivalent to the passive film thickness, usually <math>\sim 10^0 - 10^1</math> nm. A porous surface which does not passivate and is infiltrated by the electrolyte reacts down to the same depth as the penetration depth of the electrolyte. Usually, a thermal spray coating containing some defects deposited onto a dense substrate reacts down to a depth comparable with its thickness, i.e. <math>\sim 10^2</math> <math>\mu</math>m.</i></p>
<b>2.3</b>	<b>Calibration process</b>	<p><i>The verification of the potentiostat/galvanostat is performed yearly using a "dummy cell" consisting of an electrical circuit with known electrical resistances, which produces a known impedance spectrum when subjected to an AC frequency sweep.</i></p>
<b>2.4</b>	<b>Detector</b>	<p><i>Potentiostat/galvanostat</i></p>
<b>2.5</b>	<b>Signal</b>	<p><i>Electric voltage and electric current</i></p>
<b>2.6</b>	<b>Measurement time</b>	<p><i>A typical electrochemical impedance spectroscopy test duration is 1 – 3 h</i></p>



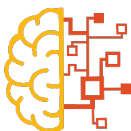
2.7	<b>Measurement parameters</b>	<i>Stabilization time (s), Baseline overpotential (V), Potential oscillation amplitude (V), Start frequency (Hz), End frequency (Hz), Number of frequency values per logarithmic decade (-), Data acquisition quality (-).</i>
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3. RAW DATA		
3.1	<b>Raw Data</b>	<i>Text file with measurement time, applied voltage, frequency, real impedance (or admittance) component, imaginary impedance (or admittance) component, impedance (or admittance) modulus, phase angle.</i>
3.2	<b>Unit</b>	<i>Impedance – <math>\Omega</math> Potential – V Measurement time – s Phase angle – deg or rad</i>
3.3	<b>Data rate acquisition</b>	<i>One point per frequency value. Typical frequency range <math>\sim 10^5</math> Hz – <math>10^{-3}</math> Hz.</i>

4. DATA PROCESSING		
4.1	<b>Level of expertise</b>	<i>High expertise: Person cannot rely solely on computer algorithms for data fitting to an equivalent circuit model and requires expertise to make ad hoc decisions how to choose the circuit model and process the data</i>
	<b>Data normalisation</b>	<i>Normalization of admittance values over the exposed electrode surface area to convert them to S/cm<sup>2</sup>. Correspondingly, impedance values are converted to <math>\Omega \cdot \text{cm}^2</math></i>
4.2.	<b>Processing reproducibility</b>	<i>Reproducible only for Data processing Expert</i>
4.3	<b>Data filtering processes</b>	<i>No filtering</i>
4.4	<b>Data analysis procedures</b>	<ul style="list-style-type: none"> <li>- <i>Plotting of the Nyquist (-Z<sub>imm</sub> vs. Z<sub>re</sub>) and/or Bode ( Z  vs. frequency, phase angle vs. frequency)</i></li> <li>- <i>Selection of suitable equivalent circuit model based on the literature and on the number of time constants identifiable from the Nyquist and/or Bode plots</i></li> <li>- <i>(Stepwise) fitting of the equivalent circuit model to the experimental data</i></li> <li>- <i>(Optional) computation of equivalent capacitances from constant phase element (CPE) parameters</i></li> <li>- <i>(Optional) extraction of additional pertinent information such as the thickness (d) of a passive film and of the electrical double layer from the corresponding capacitances (C)</i></li> </ul>



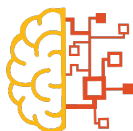
		<p>according to <math>C = \varepsilon \cdot \varepsilon_0 \cdot A/d</math> where <math>\varepsilon</math> = dielectric constant of the film or double layer, <math>\varepsilon_0</math> = dielectric constant of vacuum, <math>A</math> = exposed electrode area.</p>
4.5	<b>Main processed signals</b>	<ul style="list-style-type: none"> <li>- Impedance per unit area (<math>\Omega \cdot \text{cm}^2</math>)</li> <li>- Capacitance (<math>\text{F}/\text{cm}^2</math>)</li> <li>- CPE parameter (<math>\text{S} \cdot \text{cm}^{-2} \cdot \text{s}^{-n}</math>)</li> <li>- CPE exponent (-)</li> </ul>
4.6	<b>Data processing through calibrations</b>	No correction of raw data
4.7	<b>Properties (elaborated data)</b>	<p>Charge transfer resistance (<math>\Omega \cdot \text{cm}^2</math>)</p> <p>Double-layer capacitance (<math>\text{F}/\text{cm}^2</math>)</p> <p>Coating/Surface layer resistance (<math>\Omega \cdot \text{cm}^2</math>)</p> <p>Coating/surface layer capacitance (<math>\text{F}/\text{cm}^2</math>)</p>
	<b>Quality of the data</b>	<ul style="list-style-type: none"> <li>- Evaluation of S/N ratio of anodic and cathodic polarizaiton curves</li> <li>- Statistical analysis of elaborated data (from multiple repeat tests): half-difference between maximum and minimum value (if up to 4 repeats), standard deviation (if 5 or more repeats).</li> </ul>
4.8.	<b>Data management</b>	Raw data files (electrochemical impedance spectroscopy data) and elaborations stored locally and on the project's SharePoint folder



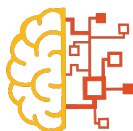
### 4.4.3 Potentiostatic Chronoamperometry

#### OVERVIEW OF THE CHARACTERISATION

1	<b>User Case</b>	<p><i>Sample: Coated or uncoated metal plate, usually ground/polished (<math>Ra/Sa \leq 0.1 \mu m</math>)</i></p> <p><i>Test environment: water-based electrolyte, aerated, room temperature</i></p>
2	<b>Characterisation method</b>	<p><i>Execution of a potentiostatic chronoamperometry test on a flat sample, usually obtained from a coated or uncoated metal plate through metallographic preparation methods (abrasive cutting, grinding with SiC papers or diamond discs, polishing with diamond suspensions, optional polishing with colloidal silica), in contact with a liquid electrolyte, usually a water-based solution (e.g. 3.5 wt./vol.% NaCl), using a three-electrode cell where the sample is the working electrode, the counterelectrode is an inert material, e.g. a platinum plate, wire or mesh, and the reference electrode consists of a standard redox couple with known and stable electrochemical potential, e.g. <math>Ag/AgCl/KCl_{(sat.)}</math></i></p> <p><i>SEM+EDX analysis of corroded sample surface and/or cross-section</i></p> <p><i>Micro-Raman spectroscopy analysis of corroded sample surface and/or cross-section</i></p>
3	<b>Validation of Characterisation</b>	<p><i>The potentiostatic chronoamperometry test is a simple method to probe the long-term stability of a passivation regime and to promote the growth of the passive film up to a thickness that makes it more easily detectable by surface spectroscopic techniques like micro-Raman spectroscopy.</i></p> <p><i>See:</i></p> <p><i>R.K. Franklin, S.M. Martin, T.D. Strong, R.B. Brown, Chemical and Biological Systems: Chemical Sensing Systems for Liquids, in: Reference Module in Materials Science and Materials Engineering, Elsevier, 2016</i></p> <p><i>A. Lekatou, E. Regoutas, A.E. Karantzalis, Corrosion behaviour of cermet-based coatings with a bond coat in 0.5M H<sub>2</sub>SO<sub>4</sub>, Corrosion Science 50(12) (2008) 3389-3400.</i></p>
4	<b>Access conditions (what is needed to repeat the experiment)</b>	<p><i>Sample preparation: in-house routine</i></p> <p><i>Access to characterization tools: in-house routine</i></p> <p><i>Commercial post-processing software (MS Excel, Matlab).</i></p>

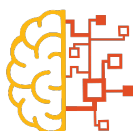


1. USER CASE		
1.1	<b>USER</b>	<i>The user must be properly trained in the execution of the experiment, including the manual operations that need to be performed with accuracy, and in safe handling, storage, and disposal of chemicals.</i>
1.2	<b>Sample</b>	<p><i>Flat sample: discs or plates of 15 – 50 mm size and 3 – 5 mm thickness. Samples can be bulk or coated.</i></p> <p><i>The backside of the sample must be electrically conductive, i.e. it is important to ensure it is free from oxides or other insulating coverages.</i></p> <p><i>The sample must be cleaned in an ultrasonic bath using an organic solvent (e.g. ethanol or acetone) for degreasing prior to testing.</i></p>
1.3	<b>Sample material properties</b>	<i>Any electrically conductive solid sample can be tested.</i>
1.4	<b>Sampling process</b>	<i>The sample is usually obtained by metallographic cutting from a (coated) plate. Alternatively, plates/discs can be manufactured at the required size (and coated if needed).</i>
1.5	<b>Sample preparation</b>	<p><i>Samples must be surface ground and polished to <math>Ra/Sa \leq 0.1 \mu m</math>. The backside must be freed from oxides by grinding, if needed. The ground and polished sample must be cleaned in an ultrasonic bath using an organic solvent (e.g. ethanol or acetone) for degreasing prior to testing. Surface roughness is verified by profilometry on at least one sample per batch.</i></p> <p><i>The sample is mounted into the support of the electrochemical cell.</i></p>
1.6	<b>Hazard</b>	<p><i>Many electrolytes and the chemicals used in their preparation can be hazardous for human health and the environment. The user must refer to the SDSs of the relevant products and to lab practices for waste disposal. Execution of the experiment under a ventilation hood is usually required.</i></p> <p><i>Organic solvents for cleaning can be hazardous (e.g. ethanol – flammable liquid, causes serious eye irritation; acetone – flammable liquid, causes serious eye irritation, causes drowsiness or dizziness by inhalation, repeated exposure may cause skin dryness or cracking).</i></p>
1.7	<b>Characterisation environment</b>	<i>Laboratory air, room temperature.</i>



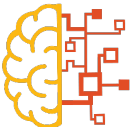
2. EXPERIMENT		
Interaction nature and character (destructive or non-destructive) of the probe with the sample		
2.1	<b>Probe/Physics of interaction</b>	<p><i>Interaction of a metal with a water-based medium involves different stages of active corrosion, passivation, or immunity, depending on the electrochemical potential, pH, and nature of dissolved species, as inferable from a Pourbaix diagram for the relevant material and environment, which can also be obtained by theoretical computations.</i></p> <p><i>Especially within a passive regime, long-term stability can be probed in an accelerate manner by applying a constant anodic overpotential within the passivity range for a predefined duration and measuring the current flowing through the system.</i></p> <p><i>Abrupt increases in current signal a passivity breakdown, whilst a continuous decrease with asymptotic stabilization signal a stable, slow-growing passive film. Under this diffusion-controlled kinetics, the current response (<i>i</i>) should be independent of the exact potential value, and it can be modelled with Cottrell's equation:</i></p> $i = \frac{nFAC_0^*\sqrt{D}}{\sqrt{\pi t}}$ <p><i>Where <math>n</math> = number of exchanged electrons, <math>F</math> = Faraday constant, <math>C_0^*</math> = original concentration of the diffusing species, <math>D</math> = diffusion coefficient, <math>t</math> = time.</i></p>
2.2	<b>Interaction Volume</b>	<p><i>The interaction volume is highly dependent on the type of response of the metal sample. A fully dense metal surface with defect-less passive film only interacts down to a depth equivalent to the passive film thickness, usually <math>\sim 10^0 - 10^1</math> nm. A porous surface which does not passivate and is infiltrated by the electrolyte reacts down to the same depth as the penetration depth of the electrolyte. Usually, a thermal spray coating containing some defects deposited onto a dense substrate reacts down to a depth comparable with its thickness, i.e. <math>\sim 10^2</math> <math>\mu\text{m}</math>.</i></p>
2.3	<b>Calibration process</b>	<p><i>The verification of the potentiostat/galvanostat is performed yearly using a "dummy cell" consisting of an electrical circuit with known electrical resistances, which produces a known response when subjected to potential scans.</i></p>
2.4	<b>Detector</b>	<i>Potentiostat/galvanostat</i>
2.5	<b>Signal</b>	<i>Electric voltage and electric current</i>
2.6	<b>Measurement time</b>	<i>A typical chronoamperometric test duration is 1 – 24 h</i>
2.7	<b>Measurement parameters</b>	<i>Stabilization time (s), Applied overpotential (V), Duration (s), Sampling time (s).</i>

3. RAW DATA		
3.1	<b>Raw Data</b>	<p><i>Text file with current, potential, measurement time.</i></p> <p><i>Physically corroded sample.</i></p>



3.2	<b>Unit</b>	Current – A Potential – V Measurement time – s
3.3	<b>Data acquisition rate</b>	Typically 1 data point / s for short tests (up to a few hours), 1 data point / 10 s (up to 24 h).

4. DATA PROCESSING		
4.1	<b>Level of expertise</b>	Domain exp.: Person needs to be able to retrieve and fit data.
	<b>Data normalisation</b>	Calculation of current density ( $I$ ) by dividing the current ( $i$ ) values over the exposed working electrode surface area ( $A$ ): $I = i / A$ [ $A/cm^2$ ].
4.2.	<b>Processing reproducibility</b>	Reproducible for a domain expert.
4.3	<b>Data filtering processes</b>	Optional noise filtering (FFT or adjacent-averaging)
4.4	<b>Data analysis procedures</b>	<ul style="list-style-type: none"> <li>- Plotting of chronoamperometric curve: <math>I</math> vs. time</li> <li>- Identification of a steady-state passivity regime</li> <li>- Calculation of average steady-state passive current density</li> <li>- Usually done with commercial software packages (MS Excel, Matlab)</li> <li>- SEM+EDX analysis of corroded sample (surface and/or cross-section): identification of corrosion mechanisms</li> <li>- Micro-Raman spectroscopic analysis of corroded sample (surface and/or cross-section): identification of corrosion product compounds.</li> </ul>
4.5	<b>Main processed signals</b>	<ul style="list-style-type: none"> <li>- Current (A)</li> <li>- Voltage (V)</li> <li>- Corroded sample</li> </ul>
4.6	<b>Data processing through calibrations</b>	No correction of raw data
4.7	<b>Properties (elaborated data)</b>	Passive current density ( $A/cm^2$ )
	<b>Quality of the data</b>	<ul style="list-style-type: none"> <li>- Evaluation of S/N ratio of anodic and cathodic polarization curves</li> <li>- Statistical analysis of elaborated data (from multiple repeat tests): half-difference between maximum and minimum value (if up to 4 repeats), standard deviation (if 5 or more repeats).</li> </ul>
4.8.	<b>Data management</b>	Raw data files (chronoamperometric) and elaborations stored locally and on the project's SharePoint folder



## 5 Integrated Characterization Workflow

The workflow shown below is the overall one, which links all the characterization techniques mentioned above. Within this workflow, each technique mentioned refers to a specific CHADA. The diagram presents several levels as a function of the dimensional scale (nano-micro-meso) of the identified property and each of these levels is accompanied by correlative microscopy and spectroscopy methods to gain the necessary understanding of the underlying mechanisms and the links to composition and microstructure. It also includes the distinction between mechanical and corrosion properties.

The integration between the different characterization techniques can lead to the identification of new relationships between properties and composition-microstructure. Indeed, the diagram highlights the innovative process for characterizing the corrosion resistance as it is proposed in this project. This novel, inverse approach consists of correlating the interlamellar fracture toughness, as a measure of lamellar bonding tightness, and the corrosion protection offered by a thermal spray coating to its substrate, as a consequence of the presence/absence and amount of interconnected (interlamellar) porosity.

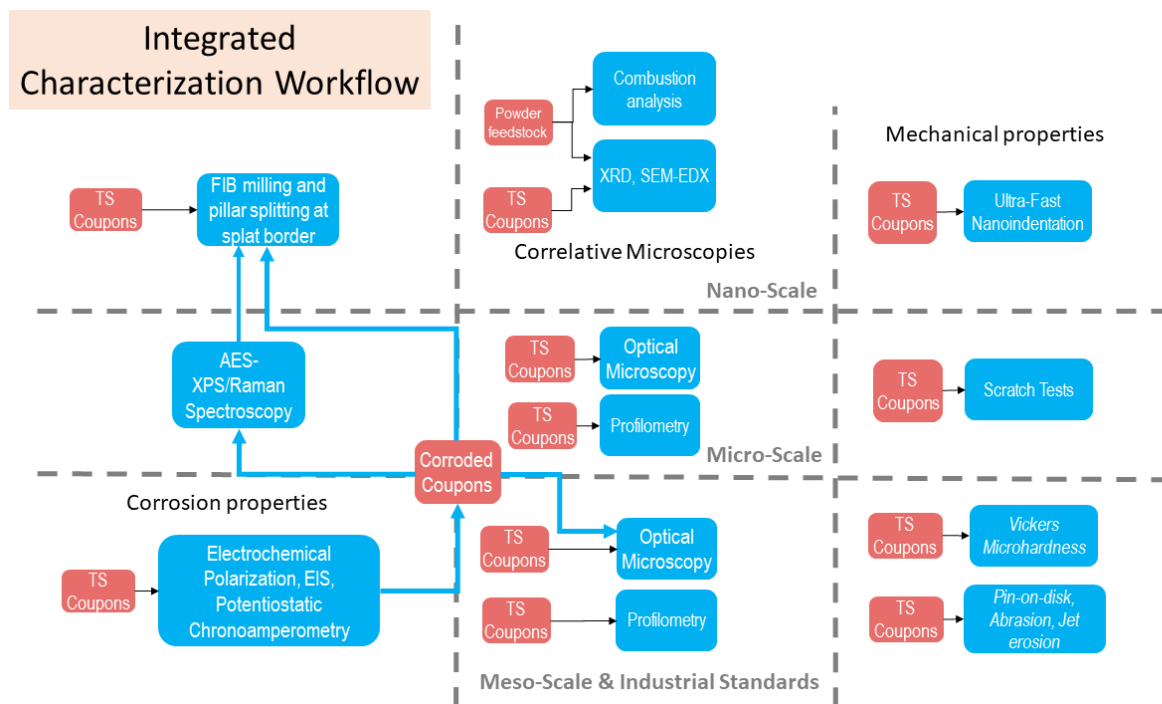
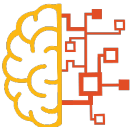
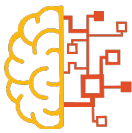


Figure 28. Integrated characterization workflow



## 6 Conclusions

CHADAs have been developed for the main characterization techniques expected to be used in the project. These diagrams will be applied in WP3 in order to be optimized and subsequently used in WP4. The diagrams will also be exploited to ensure the accessibility and interoperability of the large amount of data produced. Furthermore, by integrating the various characterization techniques with the modelling part, it is possible to discover new relationships between the properties: this type of advanced characterization can produce new knowledge and, in addition, it can generate business opportunities, complementing industrial standards.



## 7 Attainment of Objectives

Table 1. Contribution to the attainment of the general objectives of CoBRAIN

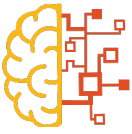
Ref. <sup>3</sup>	General Objective	Status
O2	To validate an Integrated Computational-Experimental Material Engineering workflow	The descriptive documentation of the experimental part, reported in these deliverable, is exploited to establish correlation between properties: this implies a facilitation in material design and the creation of new knowledge.

Table 2. Contribution to the attainment of specific workpackage objectives

Ref. <sup>4</sup>	Work Package Objective	Status
WP1.3	To create ontological representation that describe Characterization Techniques	All the main Characterization techniques, used in the project, are described in this deliverable. These have been created following the indications and the terminology reported in CWA 17851.

<sup>3</sup> Reference to the general project objectives stated from page 4 of Part B of the Annex 1 – Description of Action

<sup>4</sup> Reference to the specific objectives of the workpackage as listed in Part A of Annex 2 – Description of Action



## 8 References

CWA 17815:2021 – CEN Workshop Agreement “Materials characterisation - Terminology, metadata and classification”