Integrated Computational-Experimental Material Engineering of Thermal Spray Coatings

CoBRAIN

Deliverable D3.3

Guidelines for the realization of the experimental dataset and CHADA update

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Document History

Version	Date	Entity	Remarks
0.1	09/12/2024	UR3	First draft
0.2	18/12/2024	UMR	Revisions to the document; addition of the electrochemical polarization testing CHADA.
0.3	23/12/2024	UR3	Images update and general revision.
1.0	28/12/2024	UR3, UMR	Final version
2.0	13/05/2026	UMR	Revised version

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Publishable Summary[2]

This document contains the guidelines for the realisation of the experimental dataset expected within the project. These include:

- Detailed description of the experimental campaign to be performed: the characterization workflow is defined and a conceptual map has also been developed that illustrates the connections between the different characterisation techniques and the other different scientific domains, such as physics-based and data-driven modeling.
- Guidelines for managing the characterizations process's reporting and documentation: the most representative characterisation techniques used in the project were described according to CWA 17815 and its last updates.
- Guidelines for sample management: they outline how many samples must be produced, how they must be distributed among the many partners, and what unique nomenclature must be used.

These guidelines are the outcome of a continuous and optimised review process that ensures their direct application in the following WP4, where the last refinements will be added.

[2] This summary will be used for public dissemination of CoBRAIN's activities

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Abbreviations

Abbreviation	Definition
BPMN	Business Process Model and Notation
CGS	Cold Gas Spray
CHADA	Material characterization data
CWA	CEN Workshop Agreement
FIB	Focused Ion Beam
GMM	Gaussian Mixture Model
HSNM	High-Speed Nanoindentation Mapping
HVOF	High Velocity Oxy-Fuel
HVAF	High Velocity Air-Fuel
ML/DL	Machine Learning/Deep Learning
RT/HT	Room Temperature/High Temperature
TEM	Transmission Electron Microscope
XPS	X-Ray Photoelectron Spectroscopy
WP	Work Package

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1 The experimental campaign in the COBRAIN project

This deliverable describes the experimental campaign that is performed within the COBRAIN project on wear-resistant and anti-corrosion coatings, produced through three different thermal spray techniques. The materials under investigation are high entropy alloy and high entropy hard metal, and the deposition techniques considered are HVOF, HVAF, and CGS.

In this field, true innovation cannot rely only on experimental tests or physical modelling, because these cannot provide reliable results on a timescale compatible with industrial responses to fluctuating markets, supply chains, and regulations. CoBRAIN aims to offer a solution to this need by exploiting the integration of computational and experimental data through semantic interoperability and developing an artificial intelligence algorithm that will be capable of proposing new high-entropy hard metal class materials for direct deposition using the previously mentioned Thermal Spray techniques.

Regarding the experimental data a highly refined characterization process was used in the project to obtain the most complete and meaningful description of the materials under examination: COBRAIN wants to suggest an exhaustive integrated workflow that can be applied to most material development needs in the field of Thermal Spray coatings. Thus, the multiple techniques adopted for the characterization not only belong to different fields but also refer to distinct dimensional scales. Overall, the experimental workflow has been designed to have general validity, avoiding biases related to personal preferences, specific expertise, and instrumental availability. While, the strong interconnection between the physics-based and data-driven modeling part was clearly defined: the data flow between the different scientific domains allows for a more in-depth and wider understanding of the material, improving its development.

Then, since accessibility, interoperability and repeatability are crucial for an experimental workflow that wants to be able to integrate with modelling and be used outside the specific project, the European Materials Characterization Council's guidelines are carried out, by applying CWA17851. The adoption of specific architecture for the description of the characterisation techniques employed is suggested by this CEN standard: this makes it possible to establish a common language and a direct connection between the generated data and its source, which opens the way for the creation of an ontology that can effectively handle the data.

1.1 Integrated characterization concept map

An overall concept map (*Figure 1,* updated with respect to proposal, DoA, and deliverable 1.3), that shows the relationships between the outputs of the several experimental techniques, is used to explain the characterisation workflow performed in this study. It divides the procedures vertically according to their thematic area (corrosion resistance, microstructure/composition, and mechanical qualities) and horizontally according to their dimensional scale (nano, micro, meso).

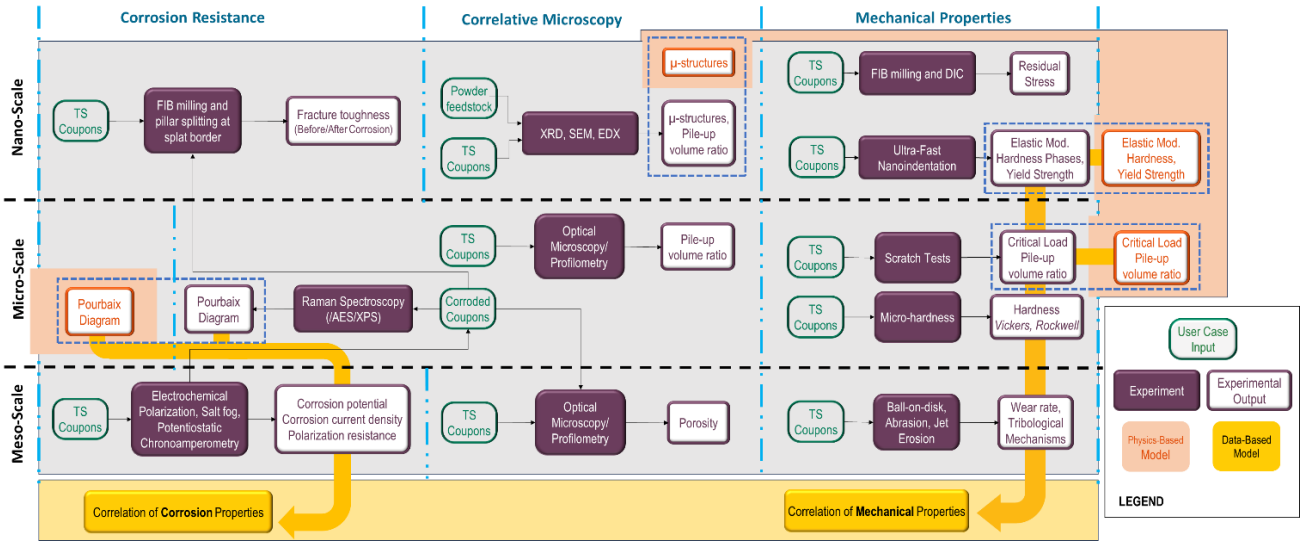

Figure 1. Integrated characterization concept map

Specifically, two methods/test sequences are provided to estimate the two primary responses expected from this kind of coating: (a) wear resistance and (b) corrosion protection.

1. **Wear resistance:** the ambition is to be able to correlate the microstructures and nano-micro mechanical properties of materials with their tribological response during specific tribological processes and under given environmental conditions.

   At the nanoscale, nanoindentation (high-speed maps and spherical indentation) is used to determine hardness, modulus and yield strength. Specifically, nanoindentation maps will make it possible to determine the hardness and modulus of the current mechanical phases, which can then be linked to the material's microstructure via correlative microscopy. Furthermore, the FIB milling and digital image correlation (DIC) technique can be applied to identify any residual stress states.

   At the microscale scratch tests are used to estimate critical loads, groove volume and pile-up size. The tribological processes are then simulated by performing the relevant wear tests on a macro scale: ball-on-disc for sliding wear, steel-wheel abrasion for grooving wear.

   Each test performed is always supported by correlative microscopic/spectroscopic analyses to assess composition, microstructure and, in general, the wear mechanisms induced.

   In addition, the information obtained at the nanoscale is used to validate atomistic models and as input for continuum models (FEM of the single-multi-asperity contact process), which in turn are validated by the results of scratch tests.

2. **Corrosion resistance:** The aim is to study both the corrosion resistance due to the chemical nature of the coating and that due to its microstructure, which should be non-porous in order to avoid exposing the substrate to direct contact with the corrosive environment.

   In order to evaluate the former, corrosion tests are carried out with different environmental conditions and different levels of applied potential (Potentiostatic chronoamperometry/Electrochemical polarisation): the corrosion products formed as a result of the tests are evaluated with microscopic/spectroscopic analyses. In addition, the results of these analyses are compared with the thermodynamic calculations of the Pourbaix diagram.

In order to assess the validity of the microstructure, the fracture toughness between the lamellae is estimated using the column splitting technique: the cohesion between the lamellae, which essentially depends on the presence of voids, is thus related to the ability of a coating to screen the substrate and thus protect it from corrosion.

In the **integrated workflow**, the relationships with the artificial intelligence domain are also shown: all output data from characterisation tests, whether related to wear resistance or corrosion protection estimation, are provided as input to an ML model with the dual purpose of identifying possible correlations between material properties and training artificial intelligence on homogeneous data.

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1.2 CWA 17851 update

As previously stated, CWA 17851 was implemented within the project with the aim of standardizing terminology and structuring the documentation related to material characterization methods. The increased communication and improved understanding between all those involved in materials characterisation, both direct characterisation experts and experts from other domains, such as modelling, both within and outside the project. In addition, the structured documentation proposed in this CWA will improve the traceability and reproducibility of materials characterisation procedures and thus the interpretation and reliability of the resulting materials data. The information structured in this way will be formalised in an ontology (WP1) in order to develop formal metadata to link methods and databases: archiving data in such a way that it can always be contextualised, keeping track of the process step that generated it, allows for greater accessibility, extraction and correlation.

The previous version of CWA 17815:2021 presented standardised schemas to describe the workflow of a given materials characterisation method by using the Materials CHAracterisation DAta (CHADA) documentation concept. Following this version, the CHADA of the main characterisation techniques used within the project were developed and reported in D1.3.

Following the guidance and recommendations gathered during the implementation of the CWA and the development of CHADA, an updated version of the standard was developed. The present version describes a more general workflow that is understandable by humans and supports machine-processable implementations. The CHADA starts with documenting the context and the rationale for the specific characterisation to be undertaken. The CHADA then documents the materials characterisation workflow, including a description of the sample and of all possible experimental and data processing workflow stages (with modules for each stage) until the output. The output of a characterisation method can be either qualitative, semi-quantitative or quantitative. Finally, the outcome, which is the digestion and/or interpretation of the characterisation output(s), is presented. The adoption of a standard graphical notation, the Business Process Model and Notation" (BPMN), is new in this version.

1.2.1 Updated CWA-17815 application

More specifically, **in the updated CWA-17815** the CHADA is divided into four sections: (1) User Story, (2) Rationale, (3) Characterisation Workflow Documentation including (a) diagram and (b) workflow tables, (4) Result. Each of these sections is meant to be of general validity, i.e. the CHADAs described in this section are not specifically addressed only to the particular needs of the CoBRAIN project but are applicable to any characterization workflow done for any purpose.

1. The **User Story** documents the overall context; the question or issue to be addressed and the desired information that the characterisation should provide. This can be the property of a material in a certain operational environment or the behaviour of a material during production.

2. The **Rationale** describes why the characterisation approach was chosen to address the User Story. If possible, evidence the choice by means of e.g. a peer reviewed paper/article or reference to an industry standard. The Rationale informs the choices made in the characterisation workflow, including the material(s) sampling, and the measurements, measurement conditions and data processing. Hence the Rationale can be understood as providing the basis for the characterisation experiments or test cases documented in the characterisation workflow tables.

3. The **Characterisation Workflow** can consist of parallel experiments or tests for different materials, different external loads or time series. Each characterisation experiment or test is a process which consists of a series of subprocesses and tasks. Each task is documented in the tables with an input and

an output. The documentation of the characterisation workflow starts with the description of the sample material, including general information, a presentation of the processing history experienced by the sample material before the characterization procedure, and any already established knowledge on the properties of the material – e.g. consolidated knowledge from textbooks and the literature, from openly available databases, from prior tests or simulations on the same material, etc. The documentation then describes in detail the sample (e.g. its dimensions, its description, etc.), its extraction procedure and any subsequent operation (e.g. grinding/polishing), including a detailed description of the processing equipment and methods to be employed in these steps. Typically, a sample is extracted from the batch material. In non-destructive testing and in on-line monitoring the volume that is to be tested is identified but not isolated. In each case, the sample is submitted to a measurement process using a device, which is described in detail including its calibration procedures and operation sequence. The resulting raw data are then post-processed, leading to the output of the characterisation experiment. The output of a characterisation experiment can be either qualitative, semi-quantitative or quantitative.

This section includes:

a A **BPMN diagram** (*Figure 2)* of the characterisation workflow depicting the specific tasks, inputs and outputs documented in the Workflow tables. A standard process modelling notation should be used. Here we adopt the Business Process Model Notation BPMN.

b For detailed documentation of all the experimental procedures or test cases in a Characterisation Workflow, the CHADA Section on Characterisation Experiment/Test Case Modules provides **template tables for documenting all processes** and related objects and data of the materials characterisation/test case. As stated above, the entries can represent arrays, and multiple tables can be used to represent for example a series of samples.

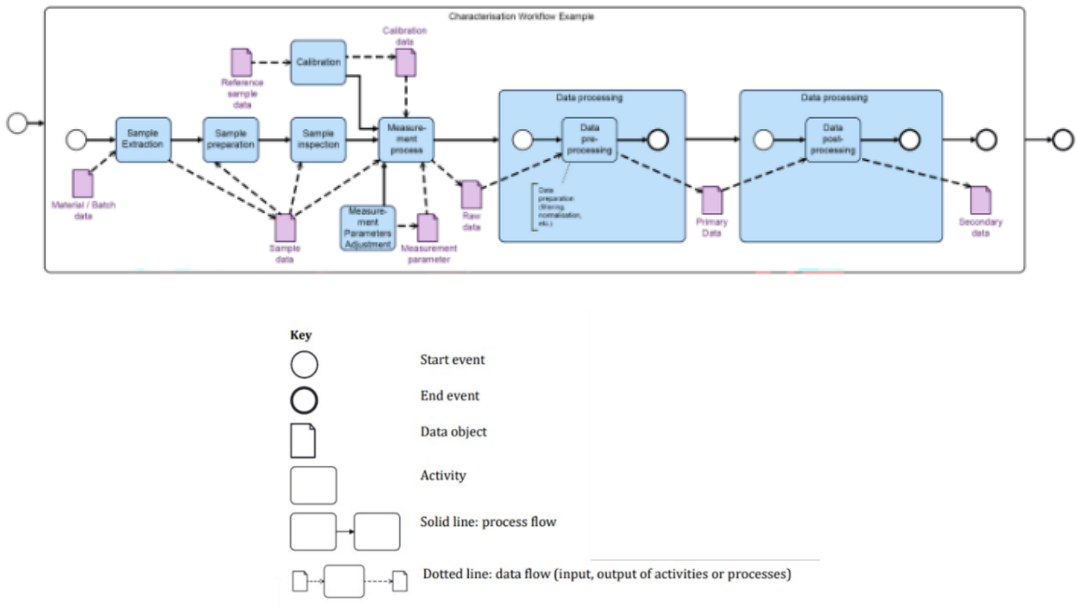


Figure 2. Example of a BPMN diagram for a generic characterisation workflow

4. The final section reports on the digested **Results** of the characterisation workflow carried out. It can be understood as the response to the desired information described in the User Story. If there is a series of test cases, then the answer to the User Story will be found by considering all results together.

This updated, general version of the CWA was applied to three case studies pertinent to the CoBRAIN project, covering the most representative characterisation techniques of the project itself. One of these cases has been shared with the EMMC/EMCC communities as one of the annexes of the updated CWA-17815, to be published in the Zenodo platform of EMMC.

High-speed nanoindentation maps on hardmetals coatings

1.1. User story

User story	
Name	High-speed nanoindentation maps on hardmetals coatings
Description	We want to identify the material's mechanical phases (hardness and modulus) and we want to evaluate possible correlations between these phases and the structural ones. Replicating the analysis on hardmetal with varying compositions will yield information able to improve our understanding of this wide and complex range of materials and contribute to their development.
Client/requester	Anyone who wants to identify a correspondence between mechanical phases and microstructural ones on hardmetals coatings.

1.2. Material System

Material System	
Name	High entropy hardmetal coating
Description including any useful information about structure, composition, performance and properties	The coating is a thermal-sprayed hardmetal with a high entropy alloy matrix, while the substrate is a metal or alloy, e.g. AISI304 stainless steel. Different compositions are evaluated for the high entropy alloy matrices, while a 60 vol.% titanium carbide is primarily used as ceramic reinforcement. Replacing traditional binders with high-entropy alloys shows a good wettability toward the carbides, high toughness, prominent wear resistance, and high temperature stability.
Reference to standard *(Add reference to standard/datasheet, if any)*	
Further documentation *(Link to a relevant document/web resource)*	

1.3. Environment and operating conditions

Environment and operating conditions	
Description	Room temperature 25 °C and controlled humidity 40%

Further documentation *(Link to a relevant document/web resource)*	

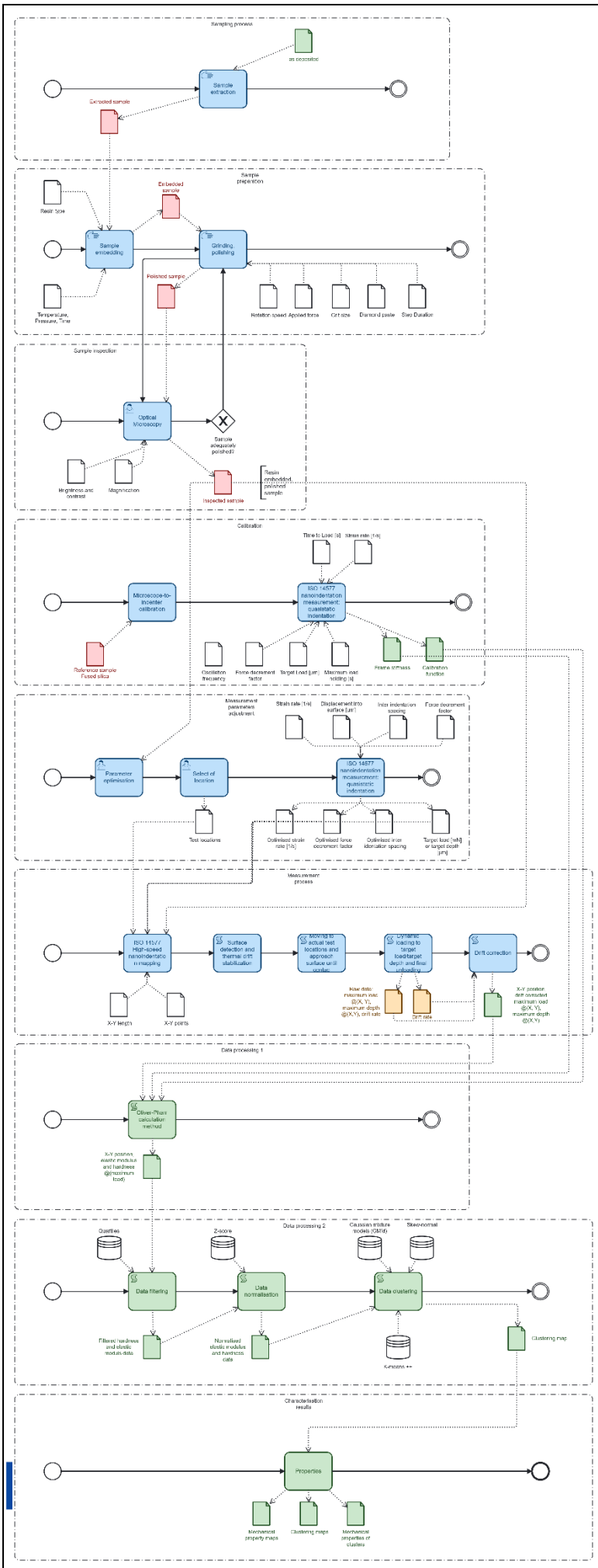
2. Rationale

Rationale	
Description	The high-speed nanoindentation mapping was the selected method for characterising hardmetals coatings due to its ability to obtain maps of the mechanical properties, elastic modulus and hardness of extensive surfaces in a relatively short period of time. The high spatial resolution of this method enabled the discernment of the mechanical properties of the Titanium Carbide particles from those of the matrix. This allowed to be established a correlation between microstructure phases and the mechanical phase.
Further documentation *(Link to a relevant document/web resource)*	

3. Characterisation Workflow Overview

Characterisation Workflow Metadata	
Name	Characterisation procedure for high-speed nanoindentation mapping
Description	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. The high-speed 3D map method was used in this characterisation workflow by performing nanoindentation tests over a large area (due to the high speed of the test), it is possible to create a three-dimensional (H(x,y) and E(x,y)) map.
All Sample(s) to be tested	1 Al_{14}(CrMnFeNi)+60TiC
All Test Methods and Environment(s)	High-speed nanoindentation mapping Room temperature 25 °C and controlled humidity 40%
Reference to standard *(Add reference to standard, if any)*	ISO 14577
Laboratory	Roma Tre University
Operator	
Further documentation *(Link to a relevant document/web resource)*	

Characterisation Workflow Diagram


Sampling process
as received
Sample extraction
Extracted sample
Sample preparation
Resin type
Embedded sample
Sample embedding
Grinding, polishing
Polished sample
Temperature, Pressure, Time
Rotation speed
Applied force
Grit size
Diamond paste
Step Duration
Sample inspection
Optical Microscopy
Sample adequately polished?
Brightness and contrast
Magnification
Inspected sample
Resin embedded polished sample
Calibration
Time to Load [s]
Strain rate [1/s]
Microscope-to-indenter calibration
ISO 14577 nanoindentation measurement: quasistatic indentation
Reference sample Fused silica
Oscillation frecuency
Force decrement factor
Target Load [µN]
Maximum load holding [s]
Frame stiffness
Calibration function
Measurement parameters adjustment
Strain rate [1/s]
Displacement into surface [nm]
Inter indentation spacing
Force decrement factor
Parameter optimisation
Select of location
ISO 14577 nanoindentation measurement: quasistatic indentation
Test locations
Optimised strain rate [1/s]
Optimised force decrement factor
Optimised inter idendation spacing
Target load [mN] or target depth [nm]
Measurement process
ISO 14577 High-speed nanoindentation mapping
Surface detection and thermal drift stabilization
Moving to actual test locations and approach surface until contact
Dynamic loading to target load/target depth and final unloading
Drift correction
X-Y length
X-Y points
Raw data: maximum load @(X, Y); maximum depth @(X,Y); drift rate
Drift rate
X-Y position, drift corrected maximum load @(X, Y); maximum depth @(X,Y)
Data processing 1
Oliver-Pharr calculation method
X-Y position, elastic modulus and hardness @(maximum load)
Data processing 2
Quantiles
Z-score
Gaussian Mixture models (GMM)
Skew-normal
Data filtering
Data normalisation
Data clustering
Filtered hardness and elastic modulus data
Normalised elastic modulus and hardness data
K-means ++
Clustering map
Characterisation results
Properties
Mechanical property maps
Clustering maps
Mechanical properties of clusters

Link to diagram *(In case there is a publicly available version of the diagram)*	https://modeler.camunda.io/diagrams/cde52050-16ff-4753-bea1-a9db2ce772bb--hsnm_bpmn-cobrain/

4. Characterisation Experiment/Test Case Documentation

4.1. Material

Material	
Name	High entropy hardmetal thermal-spray coatings
Description	The coating is a high entropy hardmetal thermal-sprayed, while the substrate is a metal or alloy, e.g. AIS304 stainless steel. Different compositions are evaluated for the high entropy alloy matrices, while a 60 vol.% titanium carbide is primarily used as ceramic reinforcement. Replacing traditional binders with high-entropy alloys shows a good wettability toward the carbides, high toughness, prominent wear resistance, and high temperature stability.
Identifier (s) *(e.g. CAS number, batch identifier)*	The identification code used is like: Al_{14}(CrMnFeNi)+60TiC (simplified identifier of matrix composition + vol.% of hard phase + composition of hard phase)
Process history *(including manufacturing steps or other prior processing information that describes the state of the material)*	Powders of the composition to be evaluated are produced by a mechanical alloying process thorugh high-energy ball mills. A heat treatment process is performed on some powders to facilitate their deposition. Finally, the powders are deposited by thermal-spray techniques like High Velocity Oxygen Fuel (HVOF) spraying.
Physical structure *(if known prior to the characterisation)*	Coating attached to a Substrate.
Material composition / Chemical structure *(if known prior to the characterisation)*	Coating: ceramic titanium carbide particles, constituting 60% by volume, embedded in a high-entropy alloy matrix of variable composition. Substrate: steel (e.g. AISI 304).
Material properties known prior to the characterisation	Good wettability toward the carbides, high hardness, prominent wear resistance, and high temperature stability.

5.2 Sample

Sample	
Name This can be a list of names denoting each step of sample preparation	Al_{14}(CrMnFeNi)+60TiC Raw sample, extracted sample, embedded sample, polished sample, inspected sample
Description	The sample is the coated component: • the substrate is AISI 304 stainless steel; the coating is a high entropy hardmetal.
Reference to standard *(Add reference to standard, if any)*	
Further documentation *(Link to a relevant document/web resource)*	
Identifier	Al_{14}(CrMnFeNi)+60TiC
Dimensions	Flat sample, at least 50×20 mm.
Physical structure	Coating. Substrate.
Material composition / Chemical structure	$Al_{14}(Cr_{20}Mn_{25}Fe_{40}Ni_{15})_{86}$+ 60 vol.% TiC
Other sample properties	Coating thickness ≥100 µm
Hazard *(Link to a relevant document/web resource)*	

4.3 Sample extraction

Sample Extraction	
Name (unique)	Sample Extraction
Description	The sample can be obtained by metallographic cutting from a larger (coated) plate.
Laboratory	University of Modena and Reggio Emilia.

Operator		
Level of expertise of the operator and/or laboratory capabilities *(with reference to relevant standards)*	Medium. The operator must be familiar with metallographic/materialographic abrasive cutting machines and the specificities of cutting thermal spray coated samples.	
Level of automation (*Describe what can be automated in the process*)	Medium. Cutting machine setup is manual. Relative movement between sample and cutting disc and regulation of the cutting feed velocity can be automatic (numerical control machines), semi-automatic (hydraulic control), or manual. Control of the disc revolution speed can be manual (through a knob) or automatic (numerically controlled).	
Reference to standard *(Add reference to standard, if any)*	ASTM E1920-03(2021) – section 6	
Further documentation *(Link to a relevant document/web resource)*	https://www.buehler.com/uk/solutions/buehler-solutions/solutions-by-material/solutions-by-material-thermal-spray-coatings/	
Input: Batch	Material	As-deposited sample. The coating is a thermal-spray high entropy hardmetal coating, while the substrates is AISI 304 stainless steel.
	Provider	University of Modena and Reggio Emilia.
	Identifier	As-deposited.
	Description	Coating. Substrate.
Output: Sample *(Unique name or ID of sample, see also Sample section for describing the sample in detail*)	Extracted sample	
Instrument	Name	Metallographic cutting machine
	Manufacturer	Remet Sas
	Model	Micromet (samples < 100 × 100 x 4 mm^3) / TR100 (larger samples)
	Unique ID	
Follows (*Unique name of the preceding step*)	HVOF spraying	

4.4 Sample preparation

Sample preparation		
Name (unique)	Sample embedding	
Description	The sample is put vertically in a plastic cup specific for cold mounting or in a hot mounting press, both with a diameter of 30 mm. An epoxy resin for cold mounting is poured in the cup. After 24h of drying, the sample is taken out from the cup in a form of a transparent cylinder. A phenolic or transparent acrylic resin in powder form is filled into the hot mounting press and consolidated according to a time-temperature-pressure program recommended by the supplier of each resin.	
Laboratory	University of Modena and Reggio Emilia.	
Operator		
Level of expertise of the operator and/or laboratory capabilities *(with reference to relevant standards)*	Medium	
Level of automation	Low	
Reference to standard *(Add reference to standard, if any)*	ASTM E1920-03(2021)	
Further documentation *(Link to a relevant document/web resource)*		
Input: Sample *(Unique name or ID of sample, see also Sample section for a detailed description of the sample)*	Extracted sample	
Holder *(in case the preparation involves mounting on a holder)*		
Output: prepared sample	Embedded sample	
Instrument	Name	Hot mounting press

	Manufacturer	Remet Sas
	Model	IPA 30
	Unique ID	
Parameters *(settings for the preparation)*	Epofix resin from Struers / Hardrock resin from Remet / Phenol resin from Remet / Acrylic resin from Remet	
Input *(from other steps of this characterisation procedure, other procedures or external data)*	Pressure (bar) / Temperature (°C) / time (min) for hot mounting press.	
Environment	Description	Laboratory room environmental condition.
	Properties	RT
Follows (*Unique name of the preceding step*)	Sample extraction	

4.5 Sample preparation

Sample preparation	
Name (unique)	Grinding and polishing.
Description	The sample is ground and mirror-polished with a manual or automatic polishing machine step by step, initially with different sandpaper grits, and then with polishing cloths with two different suspensions
Laboratory	University of Modena and Reggio Emilia.
Operator	
Level of expertise of the operator and/or laboratory capabilities *(with reference to relevant standards)*	Medium. The operator must be trained on metallographic grinding/polishing procedures for different types of materials and be aware of the possible artefacts that can be caused by improper procedures.
Level of automation	High/Low
Reference to standard	ASTM E1920-03(2021)

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(Add reference to standard, if any)		
Further documentation *(Link to a relevant document/web resource)*		
Input: Sample *(Unique name or ID of sample, see also Sample section for a detailed description of the sample)*	Sample embedded	
Holder *(in case the preparation involves mounting on a holder)*	Clamp	
Output: prepared sample	Ground and polished sample	
Instrument	Name	Metallographic polishing machine
	Manufacturer	Remet Sas / Struers SAS
	Model	LS2 / LaboPol20
	Unique ID	
Parameters *(settings for the preparation)*	Grit sizes (SiC sandpapers P400, P800, P1200, P2500, and P4000) Polishing cloth with diamond paste (3 µm) Polishing cloth with colloidal silica (<100 nm) in water-based or organic medium Rotation speed Applied force Step duration	
Input *(from other steps of this characterisation procedure, other procedures or external data)*		
Environment	Description	Laboratory room environmental condition.
	Properties	RT
Follows (*Unique name of the preceding step*)	Sample extraction	

4.6 Sample inspection

Sample inspection		
Name (unique)	Optical microscopy	
Description	Optical microscopy inspection of the polished surface to verify its cleanliness, and the absence of polishing scratches and other artefacts (e.g. pull-out porosity, cracks, etc.).	
Laboratory	University of Modena and Reggio Emilia.	
Operator		
Level of expertise of the operator and/or laboratory capabilities *(with reference to relevant standards)*	Medium. The operator must be trained to identify possible artefacts induced by polishing.	
Level of automation	Low	
Reference to standard *(Add reference to standard, if any)*	ASTM E1920-03(2021)	
Further documentation *(Link to a relevant document/web resource)*		
Input: Sample *(unique name of the sample)*	Polished sample	
Output: Sample validation report	Inspected sample	
Instrument	Name	Optical microscope
	Manufacturer	Olympus
	Model	GS30
	Unique ID	
Parameters *(Settings for the inspection)*	Magnification Brightness and contrast	

Input: Information from other resources relevant for the validation (not described in this CHADA)	
Follows (*Unique name of the preceding step*)	Grinding and polishing

4.7 Calibration

Calibration		
Name	Machine calibration	
Description	The calibration process in nanoindentation is a crucial step; however, before calibrating the tip, it is essential to verify that it is free from debris and in optimal condition. To calibrate the tip, a standard reference sample is utilised, on which an array of 25 indents is performed using the CSM technique. The nanoindentations curves are analysed to determine the calibration function of the contact area and to confirm that the tip is intact.	
Laboratory	Roma Tre University	
Operator		
Level of expertise of the operator and/or laboratory capabilities *(with reference to relevant standards)*	High	
Level of automation	High	
Reference to standard *(Add reference to standard, if any)*	ISO 14577	
Further documentation *(Link to a relevant document/web resource)*		
Input: Reference sample *(Specify in case of a Reference Sample, Reference Material or a Certified Reference Material)*	Fused silica	
Output: Calibration data	Frame stiffness Calibration function	
Instrument	Name	Nanoindenter

	Manufacturer	KLA
	Model	G200
	Unique ID	
	Date of last calibration	
Parameters (settings)	Target Load [mN] Time To Load [s] Maximum load holding Time [s] Force Decrement Factor Oscillation frequency Strain rate [1/s]	
Input (from other steps of this characterisation procedure, other procedures or external data)		
Follows (*Unique name of the preceding step*)	Sample inspection	

4.8 Measurement parameters adjustment

Measurement parameters adjustment	
Name	Parameter optimisation
Description	Identification of the nanoindentation displacement into the surface, and related inter-indentation spacing criteria. To avoid surface roughness effects, the displacement into surface must be approximately 10 times the average roughness. Identification of the target load corresponding to the target depth.
Laboratory	Roma Tre University
Operator	
Level of expertise of the operator and/or laboratory capabilities *(with reference to relevant standards)*	High

Level of automation	Medium
Reference to standard *(Add reference to standard, if any)*	
Further documentation *(Link to a relevant document/web resource)*	
Input: Sample *(unique name of the sample, see also Sample section)*	Inspected sample
Output: Adjusted settings for the instruments	Optimised inter-indentation spacing Target load [mN] or to target depth [µm] Optimised force decrement factor Optimised strain rate [1/s] Test locations
Input *(from other steps of this characterisation procedure, other procedures or external data)*	Displacement into surface [µm] Inter-indentation spacing Force decrement factor Strain rate [1/s]
Follows (*Unique name of the preceding step*)	Machine calibration

4.9 Measurement Process

Measurement Process	
Name	High speed nanoindentation mapping
Description	The high-speed nanoindentation mapping (HSNM) allows for one complete indentation cycle per second, including approach, contact detection, load, unload, and movement to the n^{th} indent location, thus enabling high-resolution, spatially resolved hardness (H) and elastic modulus (E) mapping.
Laboratory	Roma Tre University
Operator	

Level of expertise of the operator and/or laboratory capabilities *(with reference to relevant standards)*	High	
Level of automation	High	
Reference to standard *(Add reference to standard, if any)*		
Further documentation *(Link to a relevant document/web resource)*		
Input: Sample *(unique name of the sample, see also Sample section)*	Inspected sample	
Output *Adjusted settings for the instruments*	X-Y position, drift corrected maximum load @(X, Y), maximum depth @(X,Y)	
Input *(from other steps of this characterisation procedure, other procedures or external data)*	Target load [mN] or target depth [µm] Optimised force decrement factor Optimised strain rate [1/s] Optimised inter-indentation spacing Test locations X-Y length and X-Y points	
Instrument	Name	Nanoindenter
	Manufacturer	KLA
	Model	G200
	Unique ID	
Parameters	X-Y length X-Y points	
Probe	Diamond Berkovich tip	
Interaction volume	Ten times the displacement into the surface	
Detector	Capacitive sensor	
Environment	Description	Ex-situ nanoindenter

	Properties	RT
Characterisation Signal	Electrical signal for displacement	
Follows (*Unique name of the preceding step*)	Parameter optimisation	

4.10 Data processing

Data processing	
Name	Oliver-Pharr calculation method
Type of data processing *(e.g. data normalisation, data filtering, post-processing, measurement uncertainty calculation)*	Data post-processing
Description	The Oliver-Pharr method establishes a methodology for the analysis of nanoindentation data to obtain hardness and elastic modulus. This method is based on the precise measurement of the load applied during the test, from which the stiffness and subsequently the contact depth h_c can be derived. Then, thanks to the tip calibration function, previously calculated on a reference sample, the contact area can be calculated from the contact depth h_c. Finally, the hardness and elastic modulus can be determined thanks to the Sneddon theory.
Laboratory	Roma Tre University
Operator	
Level of expertise of the operator and/or laboratory capabilities *(with reference to relevant standards)*	Low
Level of automation	High
Reference to standard *(Add reference to standard, if any)*	
Further documentation *(Link to a relevant document/web resource)*	W.C. Oliver, G.M. Pharr, *An improved technique for determining hardness and elastic modulus using load and displacement sensing indentation experiments*, J. Mater. Res., 7 (6) (1992), pp. 1564-1583, 10.1557/jmr.1992.1564
Input: <name>	Raw data: X-Y position, maximum load @(X, Y), maximum depth @(X,Y), drift rate

from other steps of this characterisation procedure, other procedures or external data	
Output: <name>	X-Y position, elastic modulus and hardness @(maximum load)
Data Processing Model	Oliver–Pharr method
Data Processing Software	NanoSuite
Follows (*Unique name of the preceding step*)	High speed nanoindentation mapping

4.11 Data processing

Data processing	
Name	Outlier removal
Type of data processing *(e.g. data normalisation, data filtering, post-processing, measurement uncertainty calculation)*	Outlier removal
Description	Removal of outlier, due to measurement errors occurring during test execution as a result of software, hardware or noise problems.
Laboratory	Roma Tre University
Operator	
Level of expertise of the operator and/or laboratory capabilities *(with reference to relevant standards)*	The operator must be able to select the correct threshold factor value so that only outliers will be removed from the acquisition data.
Level of automation	High
Reference to standard *(Add reference to standard, if any)*	
Further documentation *(Link to a relevant document/web resource)*	
Input: <name>	Elastic modulus and hardness @(maximum load)

from other steps of this characterisation procedure, other procedures or external data	
Output: <name>	Filtered elastic modulus and hardness data @(maximum load)
Data Processing Model	Detection method: Quartiles
Data Processing Software	Matlab
Follows (*Unique name of the preceding step*)	Oliver-Pharr calculation method

4.12 Data processing

Data processing	
Name	Data normalisation
Type of data processing *(e.g. data normalisation, data filtering, post-processing, measurement uncertainty calculation)*	Data normalisation
Description	Data normalisation is a process used to transform data to a common scale to avoid data bias towards the variable with the largest scale. This is fundamental to improving the performance of machine learning algorithms or removing duplication and anomalies in relational databases.
Laboratory	Roma Tre University
Operator	
Level of expertise of the operator and/or laboratory capabilities *(with reference to relevant standards)*	High
Level of automation	High
Reference to standard *(Add reference to standard, if any)*	
Further documentation	

(Link to a relevant document/web resource)	
Input: <name> *from other steps of this characterisation procedure, other procedures or external data*	Filtered elastic modulus and hardness data
Output: <name>	Normalised elastic modulus and hardness data
Data Processing Model	Normalization method: Z-score
Data Processing Software	Matlab
Follows (*Unique name of the preceding step*)	Outlier removal

4.13 Data processing

Data processing	
Name	Data deconvolution
Type of data processing *(e.g. data normalisation, data filtering, post-processing, measurement uncertainty calculation)*	Data deconvolution
Description	Statistical models such as GMM (Gaussian Mixture Model) that assumes data are generated by a combination of different gaussian distributions, or skewed normal distributions for asymmetrical data, are applied to the hardness and modulus of elasticity datasets. This allows to identify the number of phases, their mean value and the standard deviation associated with their mean value.
Laboratory	Roma Tre University
Operator	
Level of expertise of the operator and/or laboratory capabilities *(with reference to relevant standards)*	Users should be experts to evaluate the correct number of the identified phases.
Level of automation	High
Reference to standard	

(Add reference to standard, if any)	
Further documentation *(Link to a relevant document/web resource)*	
Input: <name> *from other steps of this characterisation procedure, other procedures or external data*	X-Y position Filtered and normalised hardness and elastic modulus
Output: <name>	Mechanical phase: number and value.
Data Processing Model	Gaussian mixture models (GMM)
Data Processing Software	Matlab
Follows (*Unique name of the preceding step*)	Data normalisation

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5. Characterisation Outcome

Characterisation Outcome	
Input which is the output of the above documented experiments or tests	The objective of this characterisation is to identify and quantify the mechanical phases (hardness and modulus) in hardmetal coatings with varying compositions.
Method: Description including method by which results were obtained across several characterisation procedures or test series	The high-speed nanoindentation mapping allowed large indentation arrays (consisting of 3000 indentations) to be performed, enabling the construction of elastic modulus and hardness maps.
Output: Interpretation and explanation of final results	The elastic modulus and hardness data obtained from the high-speed nanoindentation mapping (HSNM) were subsequently subjected to several post-processing steps to identify the different mechanical phases. In the case of the samples under investigation, different hardness phases with their respective values were identified: based on the knowledge of the material obtained from previous structural-compositional analyses, a correlation with the microstructural phases can be identified.
Further documentation *(Link to a relevant document/web resource)*	

Funded by the European Union

Abrasion wear test on hardmetal coatings

1.1 User story

User story	
Name	Abrasion wear rate measurement on hard-metal coatings
Description	The abrasion test is adopted to determine the dry particle abrasion resistance of hard-metal thermal-spray coatings used in high-stress abrasion applications, e.g. components of mineral grinding or earthmoving systems. The data produced will make it possible to reproducibly classify coatings produced with different compositions according to their resistance under a given set of conditions, but also to assess the surface morphology resulting from wear mechanisms.
Client/requester	Anyone who wants to assess the abrasive wear resistance of hardmetal coatings.

1.2 Material/Material System

Material/Material System	
Name	High entropy hardmetal coating in abrasive wear conditions
Description including any useful information about structure, composition, performance and properties	Wear testing involves analysis of SYSTEM properties, where the system is defined by three main components: (a) material to be tested, (b) counterpart, (c) third body. a. **Material:** steel coated with a high entropy alloy reinforced with ceramic particles, where coating thickness is known. Different compositions are evaluated for high entropy alloys, while titanium carbide is primarily used as ceramic reinforcement. Replacing traditional binders with high-entropy alloys shows a good wettability toward the carbides, high toughness, prominent wear resistance, and high temperature stability. b. **Third body:** The abrasive particles used for wear might be silicates or oxides, such as magnetite c. **Counterpart**, in this case, is a steel wheel.
Reference to standard *(Add reference to standard/datasheet, if any)*	ISO 10545-6:2012
Further documentation *(Link to a relevant document/web resource)*	https://doi.org/10.1002/adem.202302064

1.3 Environment and operating conditions

Environment and operating conditions	
Description	Room temperature, pressure and humidity. Operating conditions of dry particle abrasion.
Further documentation *(Link to a relevant document/web resource)*	-

2.1 Rationale

Rationale	
Description	The dry particle abrasion test with a steel wheel differs from the more established ASTM G65 rubber-wheel test. The latter causes a two-body abrasion condition, since the abrasive particles become embedded in the rubber wheel. The contact pressures are also relatively mild, i.e. they correspond to a low-stress condition. On the other hand, the steel wheel produces a high-stress condition, whereby the abrasive particles are pushed forcefully into the tested surface. It is also a partly three-body configuration, since some particles remain free to move between the steel wheel and the sample surface. A high-stress, three-body abrasion condition gives a better approximation of real-world conditions in severe applications, such as mineral grinding and mixing and earthmoving machinery, in terms of wear mechanism, contact pressure and relative motion. In particular, it is well suited to hard materials (e.g. hardmetal coatings), where it can readily reveal issues like brittleness or poor cohesion (e.g. pull-out).
Further documentation *(Link to a relevant document/web resource)*	10.1016/j.surfcoat.2022.128400

3.1 Characterisation Workflow Overview

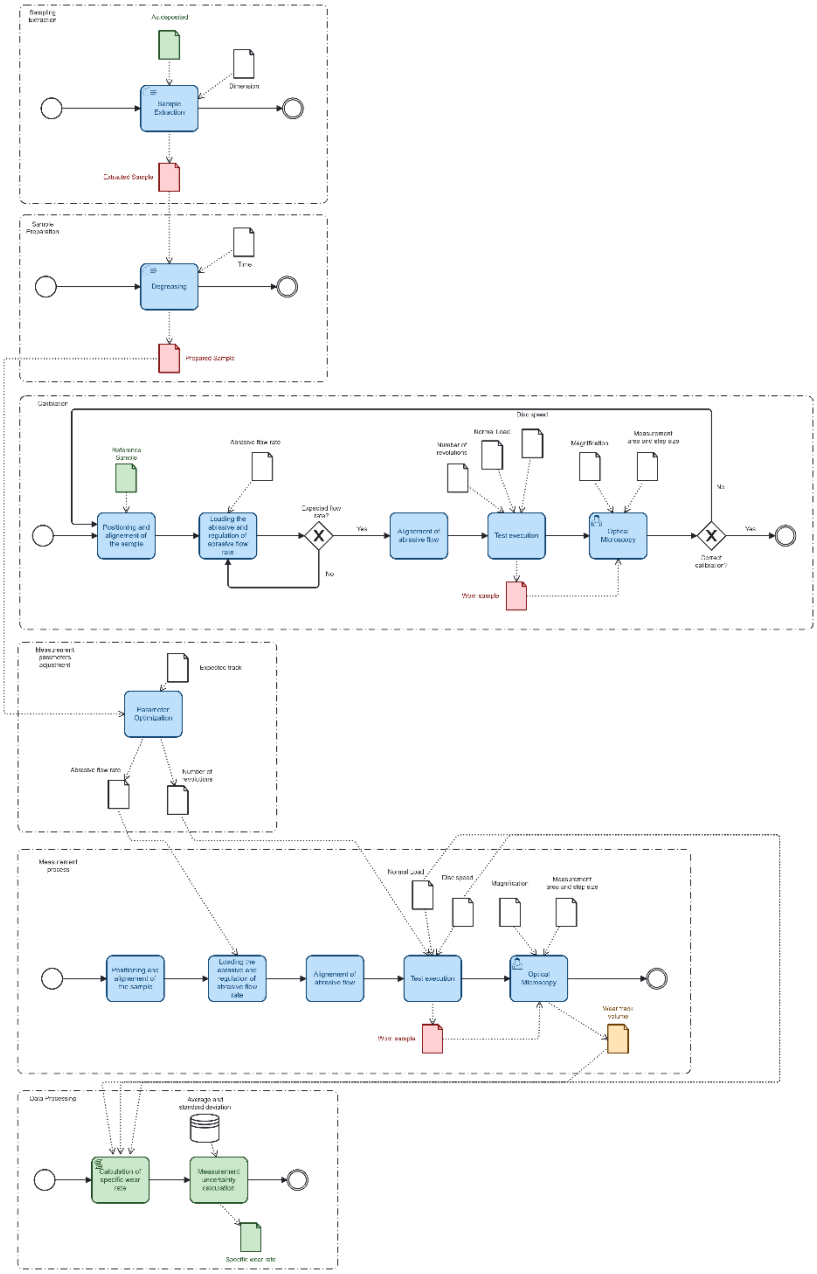
The colours coding in the tables highlights the following

- process [cyan],
- input[green],
- output [red]

Characterisation Workflow Metadata

Name	Characterization procedure for the determination of dry particle abrasion resistance.
Description	After a preparation process, the sample is subjected to a dry abrasion test with a flow of abrasive sand dragged by a steel wheel. Subsequently, optical profilometry is performed on the worn sample: from the topographical analysis, the volume loss is computed, and the material's resistance is determined.
All Sample(s) to be tested	1 Al_{14}(CrMnFeNi)+60TiC
All Test Methods and Environment(s)	• Dry abrasion test with an abrasive flow and a steel wheel. • Optical profilometry. Room temperature, pressure and humidity in the whole workflow.
Reference to standard *(Add reference to standard, if any)*	ISO 10545-6:2012
Laboratory	University of Modena and Reggio Emilia.
Operator	
Further documentation *(Link to a relevant document/web resource)*	https://doi.org/10.1016/j.surfcoat.2005.04.057

Characterisation Workflow Diagram



Link to diagram *(In case there is a publicly available version of the diagram)*	https://modeler.camunda.io/share/2c3d51b4-86be-4a67-9240-e6565f3b1b11

4. Characterisation Experiment/Test Case Documentation

3.4 Material

Material	
Name	High entropy hardmetal thermal-spray coatings
Description	From the material system, only the coated component is accounted for. The coating is a high entropy hardmetal thermal-sprayed, while the substrate is a metal or alloy, e.g. AIS304 stainless steel. Different compositions are evaluated for the high entropy alloy matrices, while a 60 vol.% titanium carbide is primarily used as ceramic reinforcement. Replacing traditional binders with high-entropy alloys shows a good wettability toward the carbides, high toughness, prominent wear resistance, and high temperature stability.
Identifier (s) *(e.g. CAS number, batch identifier)*	The identification code used is like: Al_{14}(CrMnFeNi)+60TiC (simplified identifier of matrix composition + vol.% of hard phase + composition of hard phase)
Process history *(including manufacturing steps or other prior processing information that describes the state of the material)*	Powders of the composition to be evaluated are produced by a mechanical alloying process through high-energy ball mills. A heat treatment process is performed on some powders to facilitate their deposition. Finally, the powders are deposited by thermal-spray techniques like High Velocity Oxygen Fuel (HVOF) spraying.
Physical structure *(if known prior to the characterisation)*	Coating attached to a Substrate.
Material composition / Chemical structure *(if known prior to the characterisation)*	Coating: ceramic titanium carbide particles, constituting 60% by volume, embedded in a high-entropy alloy matrix of variable composition. Substrate: steel (e.g. AISI 304).
Properties known prior to the characterisation	Good wettability toward the carbides, high hardness, prominent wear resistance, and high temperature stability.

4.2.1 Sample

Sample	
Name	Al_{14}(CrMnFeNi)+60TiC:

This can be a list of names denoting each step of sample preparation	• As-deposited • Extracted sample • Prepared sample • Inspected sample • Worn sample.
Description	The sample is the coated component: • the substrate is AISI 304 stainless steel; • the coating is a high entropy hardmetal.
Reference to standard *(Add reference to standard, if any)*	
Further documentation *(Link to a relevant document/web resource)*	
Identifier	Al_{14}(CrMnFeNi)+60TiC
Dimensions	Flat sample, at least 50×20 mm.
Physical structure	Coating. Substrate.
Material composition / Chemical structure	$Al_{14}(Cr_{20}Mn_{25}Fe_{40}Ni_{15})_{86}$+ 60 vol.% TiC
Other sample properties	Coating thickness ≥100 µm
Hazard *(Link to a relevant document/web resource)*	

4.3 Sample extraction

Sample Extraction	
Name (unique)	Sample Extraction
Description	The sample can be employed as-is or obtained by metallographic cutting from a larger (coated) plate.
Laboratory	University of Modena and Reggio Emilia.

Operator		
Level of expertise of the operator and/or laboratory capabilities *(with reference to relevant standards)*	Medium. The operator must be familiar with metallographic/materialographic abrasive cutting machines and the specificities of cutting thermal spray coated samples.	
Level of automation (*Describe what can be automated in the process*)	Medium. Cutting machine setup is manual. Relative movement between sample and cutting disc and regulation of the cutting feed velocity can be automatic (numerical control machines), semi-automatic (hydraulic control), or manual. Control of the disc revolution speed can be manual (through a knob) or automatic (numerically controlled).	
Reference to standard *(Add reference to standard, if any)*	ASTM E1920-03(2021) – section 6	
Further documentation *(Link to a relevant document/web resource)*	https://www.buehler.com/uk/solutions/buehler-solutions/solutions-by-material/solutions-by-material-thermal-spray-coatings/	
Input: Batch	Material	As-deposited sample. The coating is a thermal-spray high entropy hardmetal coating, while the substrates is AISI 304 stainless steel.
	Provider	University of Modena and Reggio Emilia.
	Identifier	As-deposited.
	Description	Coating. Substrate.
Output: Sample *(Unique name or ID of sample, see also Sample section for describing the sample in detail*)	Extracted sample	
Instrument	Name	Metallographic cutting machine
	Manufacturer	Remet Sas
	Model	Micromet (samples < 100 × 100 x 4 mm^3) / TR100 (larger samples)
	Unique ID	
Follows (*Unique name of the preceding step*)	HVOF spraying	

4.4 Sample preparation

Sample preparation		
Name (unique)	Degreasing	
Description	Samples must be cleaned in an ultrasonic bath using an organic solvent (e.g. ethanol, acetone, or isopropanol) for degreasing prior to testing.	
Laboratory	University of Modena and Reggio Emilia.	
Operator		
Level of expertise of the operator and/or laboratory capabilities *(with reference to relevant standards)*	Low. The operator must adjust the filling of the solvent suitable for cleaning.	
Level of automation	Medium. The operator needs to insert the sample into the properly prepared instrument, set the cleaning time, extract the sample and dry it using clean compressed air.	
Reference to standard *(Add reference to standard, if any)*		
Further documentation *(Link to a relevant document/web resource)*		
Input: Sample *(Unique name or ID of sample, see also Sample section for a detailed description of the sample*)	Extracted Sample	
Holder *(in case the preparation involves mounting on a holder)*	The sample is placed in a beaker with the solvent, submerged in the main tank filled with water.	
Output: prepared sample	Prepared Sample	
Instrument	Name	SONICA
	Manufacturer	SOLTEC
	Model	Ultrasonic cleaner 1200 M S3
	Unique ID	

Parameters *(settings for the preparation)*	Time (min).	
Input *(from other steps of this characterisation procedure, other procedures or external data)*	-	
Environment	Description	
	Properties	
Follows (*Unique name of the preceding step*)	Sample Extraction	

4.5 Calibration

Calibration	
Name	Calibration on reference sample
Description	The calibration of the powder feed rate is done by weighing the mass of powder fed through an adjustable orifice for a defined period. The verification of the instrument is periodically made by abrading a known reference material, e.g. HVOF-sprayed WC-10%Co-4%Cr.
Laboratory	University of Modena and Reggio Emilia.
Operator	
Level of expertise of the operator and/or laboratory capabilities *(with reference to relevant standards)*	Medium
Level of automation	Low. The operator sets up the machine by fixing the sample with clamps and filling the powder tank, adjusts manually the powder flow gate, manually opens and closes the gate to collect the powder in a beaker while recording the time with a stopwatch, sets the test duration (n° of revolutions), starts the test and opens the powder flow. At the end of the test, the operator stops the powder flow, unmounts the sample, and removes excess loose powder with dry, clean compressed air.
Reference to standard *(Add reference to standard, if any)*	ISO 10545-6:2012
Further documentation *(Link to a relevant document/web resource)*	https://doi.org/10.1016/j.surfcoat.2005.04.057

Input: Reference sample *(Specify in case of a Reference Sample, Reference Material or a Certified Reference Material)*	Worn Reference Sample	
Output: Calibration data	Checking the correct performance of the instrument.	
Instrument	Name	• Dry particle abrasion tester • Optical profilometer, mounted on a Nikon Eclipse LV150N optical microscope
	Manufacturer	• Ceramic Instruments. • Confovis GmbH
	Model	• AP/87 • ConfoSurf
	Unique ID	
	Date of last calibration	
Parameters (settings)	Powder flow rate N° of disc revolutions Magnification Measurement area (mm^2) and step size (µm)	
Input (from other steps of this characterisation procedure, other procedures or external data)	Normal Load (N), Disc speed (rpm).	
Follows (*Unique name of the preceding step*)	Sample preparation	

4.6 Measurement parameters adjustment

Measurement parameters adjustment	
Name	Parameter Optimization
Description	The operator sets the desired number of disc revolutions.

Laboratory	University of Modena and Reggio Emilia.
Operator	
Level of expertise of the operator and/or laboratory capabilities *(with reference to relevant standards)*	Medium.
Level of automation	Low
Reference to standard *(Add reference to standard, if any)*	ISO 10545-6:2012
Further documentation *(Link to a relevant document/web resource)*	https://doi.org/10.1016/j.surfcoat.2005.04.057
Input: Sample *(unique name of the sample, see also Sample section)*	Inspected sample
Output: Adjusted settings for the instruments	Abrasive flow rate (g/min), number of revolutions
Input *(from other steps of this characterisation procedure, other procedures or external data)*	Expected worn track
Follows (*Unique name of the preceding step*)	Calibration on reference sample

4.7 Measurement Process

Measurement Process	
Name	Dry abrasion test with a steel wheel.
Description	In the dry abrasion test with grit/steel wheel the abrasive, FEPA80 (≈180 µm average size) Al_2O_3, is dragged by a rotating steel disc against the surface of a flat sample. The specimen is pressed against a rotating wheel (Fe360A) of 200 mm diameter by a fixed normal force (40.2 N) using a dead weight and a rope-and-pulleys system, while a controlled flow of grit abrades the test surface.

	The rotation of the wheel is such that its contact surface moves in the direction of the grit flow. Note that the sample holder moves along linear guides along a direction perpendicular to the wheel axis to allow the adjustment of the position of the flat specimen according to its thickness, and the application of the normal load. The powder hopper and orifice are on a movable system to allow the alignment of the abrasive flow with the sample and the wheel. The particles typically exhibit angular shape and are harder than the tested sample: they cause abrasive grooving of the sample itself. The main form of wear is high-stress, three-body abrasion. Then the volumetric wear loss is measured by acquiring the profile of the wear track through profilometry. In the first instance, the surface morphology resulting from wear mechanisms is also observed.	
Laboratory	University of Modena and Reggio Emilia.	
Operator		
Level of expertise of the operator and/or laboratory capabilities *(with reference to relevant standards)*	Medium	
Level of automation	Low	
Reference to standard *(Add reference to standard, if any)*	ISO 10545-6:2012	
Further documentation *(Link to a relevant document/web resource)*		
Input: Sample *(unique name of the sample, see also Sample section)*	Inspected sample	
Output	Wear track volume (mm^3).	
Input *(from other steps of this characterisation procedure, other procedures or external data)*	Adjusted parameter, Normal load (N), Disc speed (rpm).	
Instrument	Name	• Dry particle abrasion tester • Optical profilometer, mounted on a Nikon Eclipse LV150N optical microscope
	Manufacturer	• Ceramic Instruments. • Confovis GmbH
	Model	• AP/87 • ConfoSurf
	Unique ID	

Parameters	Abrasive flow rate (g/min), Number of revolutions (-), Magnification, Measurement area and step size.	
Probe	Flow of free-falling, solid (dry) particles dragged and pressed by the steel disk.	
Interaction volume	The sample is affected by the impinging solid particle jet down to depths that are usually of the order of few hundreds of micrometres.	
Detector	No detector.	
Environment	Description	Room temperature, pressure and humidity.
	Properties	
Characterisation Signal	No measured signal during the test.	
Follows (*Unique name of the preceding step*)	Parameter Optimization	

4.8 Data processing

Data processing	
Name	Calculation of specific wear rate
Type of data processing *(e.g. data normalisation, data filtering, post-processing, measurement uncertainty calculation)*	Calculation of wear rate value by combining the outputs from the two applied methods. Then the measurement uncertainty is calculated, due to the repetitions of the test done on the same sample.
Description	• Calculation of specific wear rate: the volumetric wear (V) of the sample is correlated to the applied normal load (F_N) and the total sliding distance of the wheel surface relative to the sample surface (s) by Archard's law: $V = (K_{ad}/H)\cdot F_N \cdot s$. • Measurement related uncertainty.
Laboratory	University of Modena and Reggio Emilia.
Operator	
Level of expertise of the operator and/or laboratory capabilities *(with reference to relevant standards)*	Medium. The operator must be familiar with the basics of tribology to understand the values being entered and obtained and detect possible inconsistencies that might reflect measurement errors.
Level of automation	Medium. Data is entered by the operator in a spreadsheet for the calculation of specific wear rates and related uncertainties.
Reference to standard *(Add reference to standard, if any)*	
Further documentation *(Link to a relevant document/web resource)*	https://doi.org/10.1016/C2011-0-07515-4
Input: <name> *from other steps of this characterisation procedure, other procedures or external data*	• Normal load (N), Disc speed (rpm) • Wear track volume (mm^3).
Output: <name>	**Specific wear rate** (mm^3/(N*m)), surface morphology resulting from wear mechanisms.
Data Processing Model	Average and standard deviation
Data Processing Software	MS Excel or similar

Follows (*Unique name of the preceding step*)	Measurement process

5. Characterisation Outcome

Characterisation Outcome	
Input which is the output of the above documented experiments or tests	**Specific wear rate of the sample material**
Method: Description including method by which results were obtained across several characterisation procedures or test series	The sample is subjected to a dry abrasion test with a steel wheel to simulate the tribological operating system. Then, using optical profilometry on the worn specimen, the volume loss is measured and, through this, the wear resistance of the material is determined. The worn surface of the sample is usually observed by optical or scanning electron microscopy (SEM) to identify the wear mechanisms. The cross-section of the worn sample can optionally be observed as well to investigate abrasion-induced sub-surface alterations.
Output: Interpretation and explanation of final results	The specific wear rate of each sample, according to which the abrasion resistance of the different coatings can be ranked. In addition, comparison of the observed morphologies with known expected morphologies due to the various known wear mechanisms and their sub-categories (e.g. abrasive wear mechanism / sub-categories: ductile wear by cutting, ductile wear by ploughing, ductile wear by wedging, ductile wear by indentation, brittle wear by fracture) can be carried out.
Further documentation *(Link to a relevant document/web resource)*	https://doi.org/10.1016/C2011-0-07515-4

Electrochemical polarization on hardmetals coatings

1.1. User story

User story	
Name	Electrochemical polarization testing on hardmetal coated samples
Description	The test characterizes the electrochemical response of a coated system exposed to an electrolyte (e.g. an aqueous solution), providing a simple and accelerated corrosion testing method from which qualitative and quantitative information can be extracted concerning the electrochemical nobility, corrosion rate, passivation ability, and transpassivation onset of the system.
Client/requester	Anyone who wants to characterize the corrosion resistance of metallic substrates with a hardmetal coating in a specified environment.

1.2. Material System

Material System	
Name	Metal substrate with high entropy hardmetal coating
Description including any useful information about structure, composition, performance and properties	The system comprises a coated sample and a test solution: • The coating is a thermal-sprayed hardmetal with high entropy alloy matrix, while the substrate is a metal or alloy, e.g. AISI304 stainless steel. Different compositions are evaluated for the high entropy alloy matrices, while a 60 vol.% titanium carbide is primarily used as ceramic reinforcement. Replacing traditional binders with high-entropy alloys shows a good wettability toward the carbides, high toughness, prominent wear resistance, and high temperature stability. • The test solution is any electrically conductive electrolyte that is relevant to the intended application. A frequent choice that provides a simple simulant of many conditions of practical relevant is a solution of 3.5% (wt./vol.) NaCl in deionized water, which can be employed to test the resistance to corrosion in chlorine-containing media like seawater.
Reference to standard *(Add reference to standard/datasheet, if any)*	
Further documentation *(Link to a relevant document/web resource)*	

1.3. Environment and operating conditions

Environment and operating conditions	
Description	Room temperature 25 °C. Operating conditions of wet corrosion.
Further documentation *(Link to a relevant document/web resource)*	

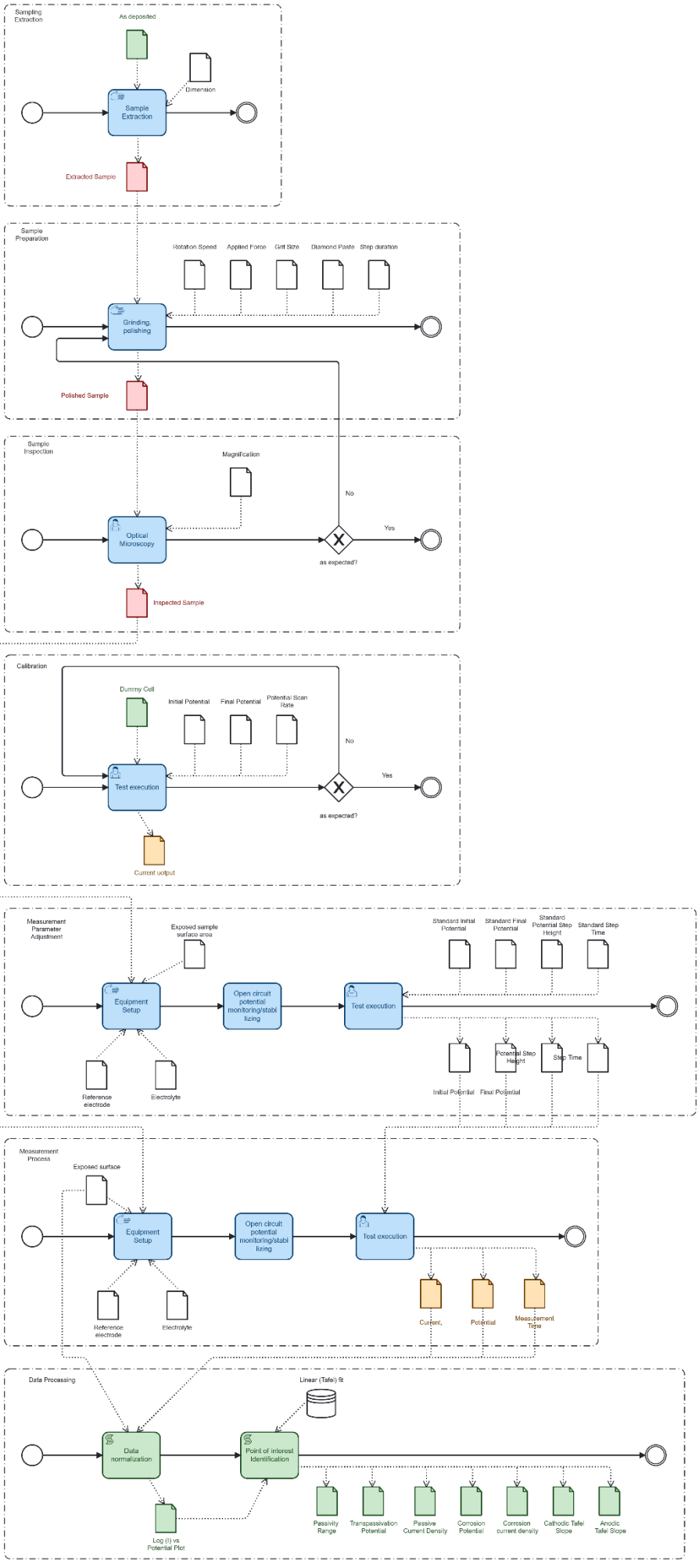
2. Rationale

Rationale	
Description	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.
Further documentation *(Link to a relevant document/web resource)*	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

3. Characterisation Workflow Overview

Characterisation Workflow Metadata	
Name	Characterisation procedure for electrochemical polarization testing
Description	Execution of an electrochemical polarization test on a flat sample, usually obtained from a coated metal plate through metallographic preparation methods including cutting and surface grinding/polishing, 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 counter-electrode 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/$KCl_{(sat.)}$
All Sample(s) to be tested	1 Al_{14}(CrMnFeNi)+60TiC
All Test Methods and Environment(s)	Electrochemical polarization testing Room temperature 25 °C
Reference to standard *(Add reference to standard, if any)*	ASTM G3-14
Laboratory	University of Modena and Reggio Emilia
Operator	
Further documentation *(Link to a relevant document/web resource)*	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

Characterisation Workflow Diagram


Sampling Extraction
As deposited
Dimension
Sample Extraction
Extracted Sample
Sample Preparation
Rotation Speed
Applied Force
Grit Size
Diamond Paste
Step duration
Grinding, polishing
Polished Sample
Sample Inspection
Magnification
No
Yes
Optical Microscopy
as expected?
Inspected Sample
Calibration
Dummy Cell
Initial Potential
Final Potential
Potential Scan Rate
No
Yes
Test execution
as expected?
Current output
Measurement Parameter Adjustment
Exposed sample surface area
Standard Initial Potential
Standard Final Potential
Standard Potential Step Height
Standard Step Time
Equipment Setup
Open circuit potential monitoring/stabilizing
Test execution
Reference electrode
Electrolyte
Potential Step Height
Step Time
Initial Potential
Final Potential
Measurement Process
Exposed surface
Equipment Setup
Open circuit potential monitoring/stabilizing
Test execution
Reference electrode
Electrolyte
Current
Potential
Measurement Time
Data Processing
Linear (Tafel) fit
Data normalization
Point of interest Identification
Log (i) vs Potential Plot
Passivity Range
Transpassivation Potential
Passive Current Density
Corrosion Potential
Corrosion current density
Cathodic Tafel Slope
Anodic Tafel Slope

Link to diagram *(In case there is a publicly available version of the diagram)*	https://modeler.camunda.io/diagrams/54657c1a-d532-48a3-a47f-5abb5fadd7d5--electrochemical-polarization/

4. Characterisation Experiment/Test Case Documentation

4.1. Material

Material	
Name	High entropy hardmetal thermal-spray coatings
Description	The coating is a high entropy hardmetal thermal-sprayed, while the substrate is a metal or alloy, e.g. AIS304 stainless steel. Different compositions are evaluated for the high entropy alloy matrices, while a 60 vol.% titanium carbide is primarily used as ceramic reinforcement. Replacing traditional binders with high-entropy alloys shows a good wettability toward the carbides, high toughness, prominent wear resistance, and high temperature stability.
Identifier (s) *(e.g. CAS number, batch identifier)*	The identification code used is like: Al_{14}(CrMnFeNi)+60TiC (simplified identifier of matrix composition + vol.% of hard phase + composition of hard phase)
Process history *(including manufacturing steps or other prior processing information that describes the state of the material)*	Powders of the composition to be evaluated are produced by a mechanical alloying process through high-energy ball mills. A heat treatment process is performed on some powders to facilitate their deposition. Finally, the powders are deposited by thermal-spray techniques like High Velocity Oxygen Fuel (HVOF) spraying.
Physical structure *(if known prior to the characterisation)*	Coating attached to a substrate.
Material composition / Chemical structure *(if known prior to the characterisation)*	Coating: ceramic titanium carbide particles, constituting 60% by volume, embedded in a high-entropy alloy matrix of variable composition. Substrate: steel (e.g. AISI 304).
Material properties known prior to the characterisation	Good wettability toward the carbides, high hardness, prominent wear resistance, and high temperature stability.

4.2 Sample

Sample	
Name This can be a list of names denoting each step of sample preparation	Al_{14}(CrMnFeNi)+60TiC Raw sample, extracted sample, polished sample, inspected sample
Description	The sample is the coated component: • the substrate is AISI 304 stainless steel; the coating is a high entropy hardmetal.
Reference to standard *(Add reference to standard, if any)*	
Further documentation *(Link to a relevant document/web resource)*	
Identifier	Al_{14}(CrMnFeNi)+60TiC
Dimensions	Flat sample, at least 50×20 mm.
Physical structure	Coating. Substrate.
Material composition / Chemical structure	$Al_{14}(Cr_{20}Mn_{25}Fe_{40}Ni_{15})_{86}$+ 60 vol.% TiC
Other sample properties	Coating thickness ≥100 µm
Hazard *(Link to a relevant document/web resource)*	

4.3 Sample extraction

Sample Extraction	
Name (unique)	Sample Extraction
Description	The sample can be obtained by metallographic cutting from a larger (coated) plate.
Laboratory	University of Modena and Reggio Emilia.

Operator		
Level of expertise of the operator and/or laboratory capabilities *(with reference to relevant standards)*	Medium. The operator must be familiar with metallographic/materialographic abrasive cutting machines and the specificities of cutting thermal spray coated samples.	
Level of automation (*Describe what can be automated in the process*)	Medium. Cutting machine setup is manual. Relative movement between sample and cutting disc and regulation of the cutting feed velocity can be automatic (numerical control machines), semi-automatic (hydraulic control), or manual. Control of the disc revolution speed can be manual (through a knob) or automatic (numerically controlled).	
Reference to standard *(Add reference to standard, if any)*	ASTM E1920-03(2021) – section 6	
Further documentation *(Link to a relevant document/web resource)*	https://www.buehler.com/uk/solutions/buehler-solutions/solutions-by-material/solutions-by-material-thermal-spray-coatings/	
Input: Batch	Material	As-deposited sample. The coating is a thermal-spray high entropy hardmetal coating, while the substrates is AISI 304 stainless steel.
	Provider	University of Modena and Reggio Emilia.
	Identifier	As-deposited.
	Description	Coating. Substrate.
Output: Sample *(Unique name or ID of sample, see also Sample section for describing the sample in detail*)	Extracted sample	
Instrument	Name	Metallographic cutting machine
	Manufacturer	Remet Sas
	Model	Micromet (samples < 100 × 100 x 4 mm^3) / TR100 (larger samples)
	Unique ID	
Follows (*Unique name of the preceding step*)	HVOF spraying	

4.4 Sample preparation

Sample preparation		
Name (unique)	Grinding and polishing	
Description	The sample surface is ground and polished with a manual or automatic polishing machine step by step, initially with different sandpaper grits, and then with polishing cloths with a diamond suspension.	
Laboratory	University of Modena and Reggio Emilia.	
Operator		
Level of expertise of the operator and/or laboratory capabilities *(with reference to relevant standards)*	Medium. The operator must be trained on metallographic grinding/polishing procedures for different types of materials and be aware of the possible artefacts that can be caused by improper procedures.	
Level of automation	High/Low	
Reference to standard *(Add reference to standard, if any)*	ASTM E1920-03(2021)	
Further documentation *(Link to a relevant document/web resource)*		
Input: Sample *(Unique name or ID of sample, see also Sample section for a detailed description of the sample)*	Extracted sample	
Holder *(in case the preparation involves mounting on a holder)*	None / clamp	
Output: prepared sample	Ground and polished sample	
Instrument	Name	Metallographic polishing machine
	Manufacturer	Remet Sas / Struers SAS
	Model	LS2 / LaboPol20

	Unique ID	
Parameters *(settings for the preparation)*	Grit sizes (SiC sandpapers P400, P800, P1200, P2500, and P4000) Polishing cloth with diamond paste (3 µm) Rotation speed Applied force Step duration	
Input *(from other steps of this characterisation procedure, other procedures or external data)*		
Environment	Description	Laboratory room environmental condition.
	Properties	RT
Follows *(Unique name of the preceding step)*	Sample extraction	

4.5 Sample inspection

Sample inspection	
Name (unique)	Optical profilometry
Description	Optical profilometry inspection of the polished surface to verify its roughness
Laboratory	University of Modena and Reggio Emilia.
Operator	
Level of expertise of the operator and/or laboratory capabilities *(with reference to relevant standards)*	Medium.
Level of automation	Medium
Reference to standard *(Add reference to standard, if any)*	ISO 25178-2:2022 ISO 25178-606
Further documentation	

(Link to a relevant document/web resource)		
Input: Sample *(unique name of the sample)*	Polished sample	
Output: Sample validation report	Inspected sample	
Instrument	Name	Optical profilometer Optical microscope
	Manufacturer	Confovis GmbH Nikon
	Model	ConfoSurf Eclipse LV150N
	Unique ID	
Parameters *(Settings for the inspection)*	Magnification Acquisition area [µm × µm] Step size [µm]	
Input: Information from other resources relevant for the validation (not described in this CHADA)		
Follows (*Unique name of the preceding step*)	Grinding and polishing	

4.6 Calibration

Calibration	
Name	Instrument verification on dummy cell
Description	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.
Laboratory	University of Modena and Reggio Emilia.
Operator	
Level of expertise of the operator and/or laboratory capabilities	Medium

(with reference to relevant standards)		
Level of automation	Medium	
Reference to standard *(Add reference to standard, if any)*		
Further documentation *(Link to a relevant document/web resource)*		
Input: Reference sample *(Specify in case of a Reference Sample, Reference Material or a Certified Reference Material)*	Dummy cell	
Output: Calibration data	Checking the correct performance of the potentiostat / galvanostat	
Instrument	Name	• Potentiostat / galvanostat
	Manufacturer	• Ametek
	Model	• Versastat 3
	Unique ID	
	Date of last calibration	
Parameters (settings)	Initial potential (V) Final potential (V) Potential scan rate (mV/s)	
Input (from other steps of this characterisation procedure, other procedures or external data)	-	
Follows (*Unique name of the preceding step*)	None (verification is done periodically and is not part of a measurement workflow)	

4.6 Measurement parameters adjustment

Measurement parameters adjustment

Name	Parameter optimisation
Description	Identification of initial and final potential, potential scan step height and potential scan step time. The potential range (initial – final) must be large enough to observe all electrochemical phenomena of interest, moving from the cathodic range into the anodic range and covering all possible anodic stages of activation, passivation, and transpassivation. Continuing the test long after transpassivation is unnecessary. The scan rate (step height / step time) must be slow enough that each point reflects a steady-state electrochemical condition.
Laboratory	University of Modena and Reggio Emilia
Operator	
Level of expertise of the operator and/or laboratory capabilities *(with reference to relevant standards)*	High. The operator must be familiar with the basics of electrochemistry.
Level of automation	Medium.
Reference to standard *(Add reference to standard, if any)*	
Further documentation *(Link to a relevant document/web resource)*	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
Input: Sample *(unique name of the sample, see also Sample section)*	Inspected sample
Output: Adjusted settings for the instruments	Standard Initial potential (V) Standard Final potential (V) Standard Potential step height (V) Standard Step time (s)
Input *(from other steps of this characterisation procedure, other procedures or external data)*	Initial potential (V) Final potential (V) Potential step height (V) Step time (s)
Follows	Sample inspection

(*Unique name of the preceding step*)	

4.7 Measurement Process

Measurement Process	
Name	Electrochemical polarization testing
Description	The interaction of a metallic surface 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. 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: $$E = \left(E^0_{M/M^{n+}} - E^0_{ox./red.}\right) - 2.303\frac{RT}{nF}\log\left(\frac{c_{M^{n+}} \cdot c_{red.}}{c_{ox.}}\right)$$ Where $E^0_{M/M^{n+}}$ and $E^0_{ox./red.}$ are the standard electrochemical potentials of the $M^0 \rightleftarrows M^{n+}$ and ox. $\rightleftarrows$ red. reactions; ox. and red. are the oxidized and reduced forms of the oxidizing species, e.g. $2H^+ \rightleftarrows H_2$ or $2H_2O + O_2 \rightleftarrows 4OH^-$; T is the temperature; R is the ideal gas constant; F is Faraday's constant; n is the number of electrons exchanged in the metal oxidation process; $c_{M^{n+}}$, $c_{red.}$, $c_{ox.}$ are the concentrations of the oxidized metal cations, the reduced and the oxidized form of the oxidizing agent. The kinetics of active corrosion are controlled by the electrochemical reaction rate according to the Butler-Volmer equation: $$i = i_0\left\{\exp\left(\frac{\alpha F}{RT}\eta\right) - \exp\left(\frac{(1-\alpha)F}{RT}\eta\right)\right\}$$ Where i is the overall current in an electrical circuit connecting an actively dissolving anode and a cathode under an externally applied overpotential η; i_0 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); α is a symmetry coefficient associated with the shape of the activation energy barrier and usually ≈0.5; and all other terms have the meaning defined above. 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. By performing a potential scan over a known surface area of the tested sample exposed to a given electrolyte of practical interest, it is possible to identify the current-voltage response characteristic of both the cathodic and anodic reactions and, by suitable data analysis based on the above equations, extract the equilibrium potential of the sample, also known as the corrosion potential, and the equilibrium value of the exchange current density describing the spontaneous dissolution rate of the sample, i.e. the corrosion current density.

	The sample is therefore mounted in an electrochemical cell leaving a known exposed surface area, typically by pressing it against an inert polymer (e.g. Teflon) gasket. An inert counter-electrode (Pt mesh) and a reference electrode (Ag/AgCl/$KCl_{(sat.)}$) are also inserted into the cell. The cell is then filled with a known amount of test solution, e.g. 300 mL of NaCl 3.5 % (wt./vol.) in deionized water. The sample is electrically connected to the working electrode of a potentiostat/galvanostat. The other electrodes are likewise suitably connected to the corresponding connections of the potentiostat/galvanostat. A potential scan is then applied by polarizing the sample (working electrode) with respect to the reference electrode, and the current flowing between the sample (working electrode) and the counter-electrode through the test solution is measured at each applied potential value.	
Laboratory	University of Modena and Reggio Emilia	
Operator		
Level of expertise of the operator and/or laboratory capabilities *(with reference to relevant standards)*	High. The operator must be familiar with the basics of electrochemistry and, in case hazardous chemical are employed as the test solution and/or in its preparation, must be well trained in safe laboratory operation.	
Level of automation	Medium. Test setup is manual. The test is then executed automatically by the potentiostat/galvanostat.	
Reference to standard *(Add reference to standard, if any)*		
Further documentation *(Link to a relevant document/web resource)*	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	
Input: Sample *(unique name of the sample, see also Sample section)*	Inspected sample	
Output *Adjusted settings for the instruments*	Raw data: Current – A, Potential – V, Measurement time – s	
Input *(from other steps of this characterisation procedure, other procedures or external data)*	Initial potential (V) Final potential (V) Potential step height (V) Step time (s)	
Instrument	Name	Potentiostat / galvanostat

	Manufacturer	Ametek
	Model	Versastat 3
	Unique ID	
Instrument	Name	Flat cell
	Manufacturer	Princeton Applied Research
	Model	K0235
	Unique ID	
Parameters	Exposed sample surface area (cm^2)	
Probe	Reference electrode: Ag/AgCl/$KCl_{(sat.)}$ Counter-electrode: platinum mesh	
Interaction volume	The interaction volume is highly dependent on the type of response of the coated sample. A fully dense surface with defect-less passive film only interacts down to a depth equivalent to the passive film thickness, usually ~10^0 – 10^1 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. ~10^2 µm.	
Detector	Potentiostat/galvanostat	
Environment	Description	NaCl 3.5% (wt./vol.) solution in deionized water
	Properties	RT
Characterisation Signal	Current	
Follows (*Unique name of the preceding step*)	Parameter optimisation	

4.8 Data processing

Data processing	
Name	Tafel extrapolation

Type of data processing *(e.g. data normalisation, data filtering, post-processing, measurement uncertainty calculation)*	Data post-processing
Description	• Normalization of the current raw data output (i.e. current flowing between sample and counter-electrode [A]) is normalized with respect to the sample surface area exposed to the counter-electrode, thus obtaining a current density I [A/cm^2]. • Plotting of the polarization curve: log(I) vs. potential of the sample against the reference electrode [V] • Identification of the rest potential and the anodic and cathodic branches • Identification of active, passive, and transpassive regions on the anodic branch • Identification of the passivity potential range [V] and the transpassivation potential [V] • Identification of the first linear portions on the anodic and cathodic polarization curves at overpotentials $\|\eta\| \geq 50$ mV from the rest potential • Linear (Tafel) fit: identification of anodic and cathodic Tafel slopes [V/decade] • Calculation of the intersection point of the anodic and cathodic Tafel fits: corrosion potential [V], corrosion current density [A/cm^2]
Laboratory	University of Modena and Reggio Emilia
Operator	
Level of expertise of the operator and/or laboratory capabilities *(with reference to relevant standards)*	High
Level of automation	Medium
Reference to standard *(Add reference to standard, if any)*	
Further documentation *(Link to a relevant document/web resource)*	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
Input: <name> *from other steps of this characterisation procedure, other procedures or external data*	Raw data: Current – A, Potential – V, Measurement time – s
Output: <name>	Passivity range (V)

	Transpassivation potential (V) Passive current density (A/cm²) Corrosion potential (V) Corrosion current density (A/cm²) Cathodic Tafel slope (V/decade) Anodic Tafel slope (V/decade)
Data Processing Model	Tafel analysis
Data Processing Software	MS Excel / Matlab
Follows (*Unique name of the preceding step*)	Measurement process

5 Characterisation Outcome

Characterisation Outcome	
Input which is the output of the above documented experiments or tests	The objective of this characterisation is to identify and quantify the electrochemical corrosion response of a hardmetal-coated metal sample exposed to a specified electrolyte solution.
Method: Description including method by which results were obtained across several characterisation procedures or test series	The electrochemical polarization curve allows exacting qualitative and quantitative information on the electrochemical response of the sample exposed to the test solution, including the presence/absence of a passivity range, its extension and the corresponding passive current density, and, most of all, the corrosion current density and corrosion potential, which provide a direct measurement of the corrosion rate and practical electrochemical nobility of the sample, respective.
Output: Interpretation and explanation of final results	By comparing the corrosion current densities, passive current densities, and passivity ranges of different coated samples, a ranking of their corrosion resistance can be obtained in the chosen test environment. Further, the tested samples can be inspected by optical or scanning electron microscopy with energy-dispersive X-ray microanalysis on the top surface and/or (preferably) on their polished cross-section to identify the through-thickness damage mechanisms of the coating and the presence/absence of substrate corrosion. The conspicuous acceleration of the corrosion process that occurs especially after transpassivation allows to identify the presence of open, interconnected porosity allowing sub-surface damage to the coating and to the coating/substrate interface with a relatively short and accelerated testing procedure, compared to the often months- or years-long spontaneous corrosion under natural exposure to the relevant environment.
Further documentation *(Link to a relevant document/web resource)*	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

2 Characterisation workflow

The experimental campaign within the project is ambitious. We want to analyse 30 different material compositions: the first 10 will be identified through an in-depth literature review and characterized during WP3, while the subsequent 20 will be characterized during WP4 and will be suggested by modelling. For each identified composition three different thermal-spray deposition techniques will be considered (HVOF, HVAF, CGS) and for each of them a maximum of 3-4 sets of deposition parameters will be evaluated. About 300–400 samples will be examined in total, yielding over 15,000 single characterisation data points: these will constitute the extensive and complete experimental dataset on which the ML/DL algorithm will be trained.

The characterization workflow, developed through an iterative optimization process over the first 10 material compositions in WP3 and ready for a direct follow-up in WP4, is described below (*Figure 3)*. Only high entropy hard metals will be analysed in WP4, unlike what happened during WP3, where high entropy alloys were also characterized to validate the processes. In order to carry out such a large experimental program in the most efficient manner, a progressive selection of the samples to be analysed is foreseen, moving to more advanced and time-consuming characterization techniques only with the most significant samples after having performed a more basic set of tests on all samples.

2.1 Characterization tiers

The workflow is divided in two main levels of characterisation, tier 1 and tier 2. It is necessary to remember that, based on the outcomes of the analyses carried out during WP3, among the sets of deposition parameters identified in task 3.2, only the most promising set for each deposition technique has been selected and will be applied in the production of samples throughout WP4: so this, which could be defined as a tier 0, will not be repeated in WP4 and is not included in the overall workflow, but it is still considered proper to mention it here.

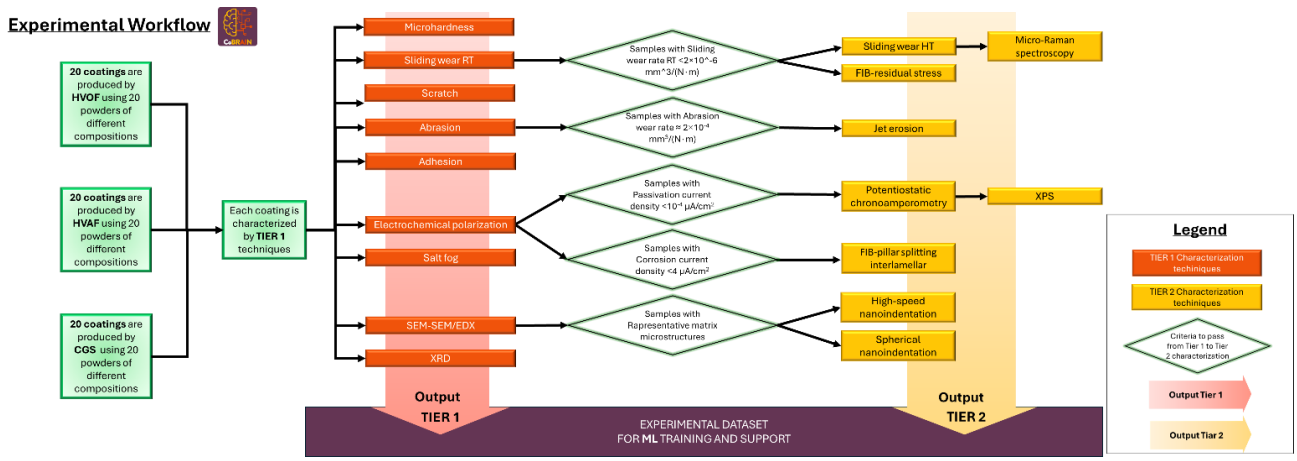


Figure 3. Experimental workflow

In **tier 1** of the workflow, the primary and most significant characteristics based on the coating purposes must be determined. With this aim, both microstructural/compositional analyses and mechanical, wear, and corrosion tests will be performed. The techniques chosen for determining the values of the properties mentioned depend on how quickly they can be applied (short execution time and wide applicability) and how clear the results are. They are:

1. SEM-EDX (with image analysis)
2. XRD
3. Microhardness
4. Scratch Test
5. Sliding wear test (RT)
6. Abrasion test
7. Electrochemical polarisation test
8. Salt fog
9. Adhesion test

The outcomes of tier 1 will determine which samples are chosen for **tier 2** characterisation, which will involve more in-depth analysis to further understand their properties, as more extensively described in the previous chapter. The selection criteria, used to select the subset samples of tier 2, are specific to each characterization technique based on the thematic area of belonging; for example, only those samples that show promising wear rate values after the abrasion test will be characterized with the erosion test; or nanoindentations will be performed only on those samples that exhibit interesting microstructures for which it would be appropriate to know the hardness of the phases: should be noted that the purpose of the experimental part carried out in WP4 is not so much to identify the most performant composition but to gather as much useful information to identify the correlations between the properties and augment the machine learning model. Obviously, in case that all samples exceed the threshold values, a maximum number of samples to be analysed per characterization technique is identified; likewise, if none of the samples were to exceed the threshold value, the samples with values closest to the selection criteria would be considered. In the *Table 1*, the selection criteria applied are explicitly stated, together with the techniques used within tier 2.

Selection Criteria	Characterization method
Max* 5 samples among those subjected to potentiostatic chronoamperometry (see below) – identification of passive film composition	XPS
Wear tracks of samples selected for high temperature sliding wear testing (see below), after sliding wear testing at both RT and HT (analysis of tribo-oxidation compounds), and outer surfaces of samples tested at HT (analysis of thermally grown oxide scale)	Micro-Raman spectroscopy
Max* 10 samples representative of the main types of matrix structures: one with FCC matrix, one with BCC matrix, one with two-phase matrix, and one with intermetallics-containing matrix (if any)	High-speed nanoindentation

Funded by the European Union

Max* 10 samples representative of the main types of matrix structures: one with FCC matrix, one with BCC matrix, one with two-phase matrix, and one with intermetallics-containing matrix (if any)	Spherical nanoindentation
Samples selected for high-temperature wear testing	FIB-residual stress
Max* 5 samples with corrosion current density <4 $\mu A/cm^2$ (comparison between interlamellar toughness below and away from the electrochemical polarization testing area)	FIB-pillar splitting interlamellar
Max* 5 samples with passivation current density $<10^{-4}$ $\mu A/cm^2$	Potentiostatic chronoamperometry
Max* 10 samples with sliding wear rate @RT $<2\times10^{-6}$ $mm^3/(N\cdot m)$	Sliding wear test (HT)
Max* 10 samples with abrasion wear rate $\approx 2\times10^{-4}$ $mm^3/(N\cdot m)$	Jet erosion test

* Including all deposition techniques

Table 1. Criteria for tier 2 sample selection and applied techniques.

2.2 Sample Management

Given the large amount of samples that need to be produced and shared across the many partners, it appears necessary to include a section with some considerations on sample management.

In task 3.3 it was possible to estimate the most appropriate number of samples needed in order to carry out the planned workflow. The necessity to complete all characterizations without falling into bottlenecks because of samples unavailability (e.g. because a sample has not been produced yet or because it's being used in other tasks) was balanced against the requirement to avoid wasting time and money due to overproduction and overloading of the production systems. Thanks to the optimization of the coated samples manufacturing, carried out in task 3.2, a high-throughput production of specimens can be achieved during WP4 and consequently, the number of samples that can be produced is subject to a less strict limitation, but proper assessment of the number of specimens to be produced for each coating type is needed. Finally, it has been estimated that:

a. In order to streamline the samples' production and preparation processes, all coatings are deposited on steel plates of the same size of 60×25×3 mm, from which the specimens required for each test can be obtained.

b. To perform the tier-1 characterizations of WP4, 12 coated plates will be needed for each coating type (composition/deposition process combination) (*Figure 3*):

- **1** plate A: will be used for microstructural and phase analysis and micro-mechanical measurements (microhardness and scratch testing).
- **2** plates B: will be used for sliding wear tests at RT.
- **3** plates C: will be used for the abrasive wear test.
- **2** plates D: will be used for the electrochemical polarization test.
- **2** plates E: will be used for salt fog testing.
- **2** plates F: will be used for adhesion tests.

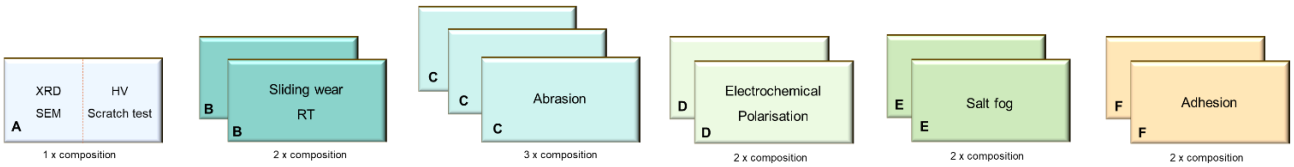


Figure 4. Coated plates to be produced for characterisation tier 1.

c. The total amount of coated plates to perform tier 2 is eight (*Figure 5)*:

- **1** plate G: for nanoindentations (high-speed and spherical) and FIB-milling (for residual stress analysis)
- **1** plate H: for interlamellar pillar splitting.
- **2** plates I: will be used for chronoamperometry tests.
- **3** plates L: will be used for sliding wear tests at HT (400 °C and 600 °C).
- **1** plate M: will be used for jet erosion test.

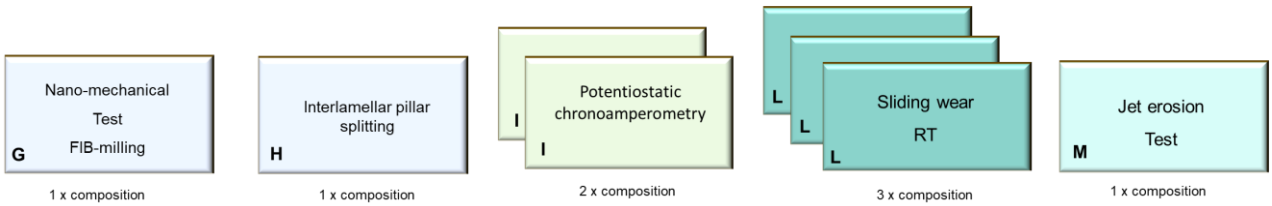


Figure 5. Coated plates to be produced for characterisation tier 2.

The total number of coated samples (plates) needed to perform the entire tier-1 + tier-2 procedure is therefore 20. Having a standard sample holder for the deposition of thermal spray coatings carrying 10 plates (as previously described in Deliverable 3.2), two identical and consecutive deposition runs will allow producing all samples needed for a coating type, which is conducive to a high-throughput workflow.

The coated samples, once produced, will need to be prepared for characterizations in order to extract the required specimens and sent to the designated partners for the specific characterizations. For this purpose, *Table* 2 specifies the preparation/finishing to be performed, the maximum specimen dimensions for each characterization technique and for each partner, the corresponding number of specimens that can be obtained out of a single coated plate, and the minimum number of replicates for each characterization test. The number of plates listed above is such that, for each characterization, a few more specimens can be extracted than are needed to attain the minimum number of replicates, so that enough are available even in case more replicates are needed (e.g. unexpected variability among the results; experimental errors compromising the results of a test; etc.).

Overall, this procedure provides clear and accurate instructions the partners in charge of the sample preparation (the samples, once created, must be given to MBN, UB, and UMR, the partners in charge of executing the sample preparation). The table also makes it clear which partner is in charge of each characterisation method, so it will be obvious who will receive the samples when they need to be sent.

Table 2. Specimen preparation guidelines.

Partner	Technique	Preparation	Dimensions	N° of specimens from a coated plate	Min. n° of replicates
UMR	SEM-EDX	Resin-mounted and polished cross-section	25 mm-wide stripe mounted in 30 mm-diameter resin	Up to 10	Porosity (all samples), oxide inclusions (metals): 8 micrographs, 3000× Chemical composition: EDX on 3 fields-of-view, 400×
	TEM	FIB milling	Standard TEM lamella	Arbitrary	1
	XRD	Cutting + surface polishing	Plate with polished surface, arbitrary size (e.g. 25×25 mm^2)	2	1
	Raman spectroscopy	Cutting + surface polishing	Samples for sliding wear testing	2	5 spectra from a wear track
	Nanoindentation (spherical)	Resin-mounted and polished cross-section	25 mm-wide stripe mounted in 30 mm-diameter resin	Up to 10	30 indents on 1 specimen
	Microhardness	Resin-mounted and polished cross-section	25 mm-wide stripe mounted in 30 mm-diameter resin	Up to 10	20 indents on 1 specimen
	Sliding wear (RT/HT)	Cutting + surface polishing	Plate, 25×25 mm^2; Ra/Sa < 0.1 µm	2	2
	Scratch	Cutting + surface polishing	Plate, 25×25 mm^2; Ra/Sa < 0.1 µm	2 (at least 6 tracks possible on each specimen)	6
	Abrasion	None	As-deposited plate	1	3
	Jet Erosion	Cutting + surface polishing	Plate, 18×18 mm^2; Ra/Sa < 0.1 µm	2	3
	Electrochemical polarisation testing	Cutting + surface polishing	Plate, 25×25 mm^2; Ra/Sa < 0.1 µm	2	2
	Potentiostatic chronoamperometrty	Cutting + surface polishing	Plate, 25×25 mm^2; Ra/Sa < 0.1 µm	2	2
UR3	Nanoindentation (High-Speed-Mapping)	Resin-mounted and polished cross-section	H=15 mm Diameter=30 mm	Up to 10	5000 indents for a map on 1 specimen
	FIB-Residual stress / Pillar Splitting	Resin-mounted and polished cross-section	H=15 mm Diameter=30 mm	Arbitrary	5
UB	Adhesion	None	As-deposited plate	1 (2 to 3 tests on one plate)	4
	Salt fog	None	As-deposited plate	1	2

The Experimental Facilities table provided on SharePoint, which includes a list of project-relevant tools available to each partner as previously described in deliverable 1.3, was used for the development of Table 2. This was created during the project's early stages to determine which partners were in charge of the

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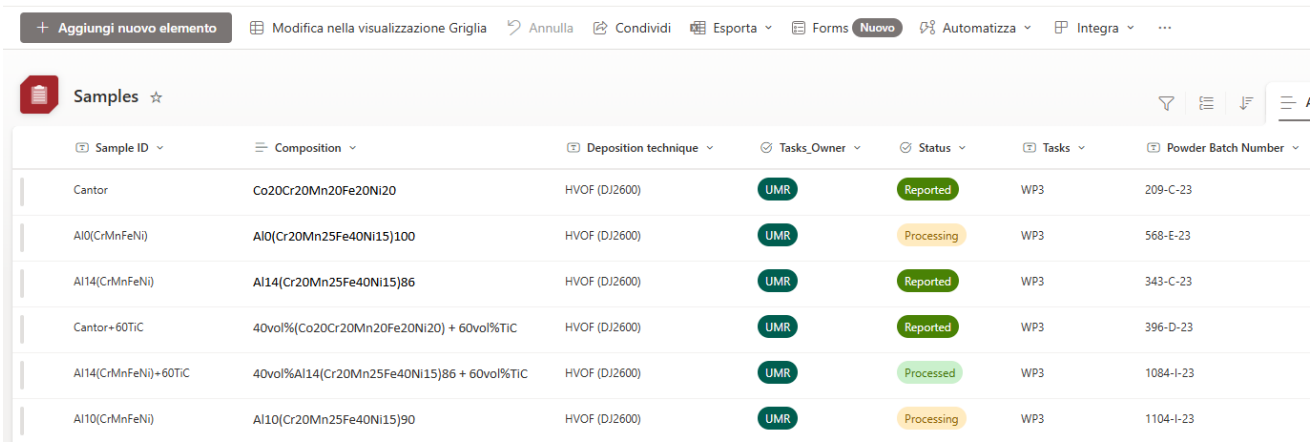
individual characterizations and, where possible, to smoothly overcome the temporary unavailability of instruments by resorting to those of another partner. In this way, greater flexibility and continuity in the characterization activities were ensured, minimizing possible delays or interruptions.

In order to facilitate the handling of samples and to have quick access to the progress of characterisation analyses, each partner is requested to fill in the SharePoint table "Samples", *Figure 6*. The producing partners must add the samples into the table after they are manufactured, with the unique name used for the project and the basic information (composition, deposition technique, batch origin of the powders, …). In the SharePoint table the Sample ID (a simplified identifier of matrix composition + vol.% of hard phase + composition of hard phase) is the unique alphanumeric identifier to be used as sample designation code for laboratory testing. This means that all laboratory samples obtained from a series of two consecutive deposition runs as specified in Section 2.1 will be given the same sample designation code to allow for rigorous but yet manageable labelling and naming schemes to avoid proliferation of codes that could become impractical for lab operators to employ in practice.



Sample ID	Composition	Deposition technique	Tasks_Owner	Status	Tasks	Powder Batch Number
Cantor	Co20Cr20Mn20Fe20Ni20	HVOF (DJ2600)	UMR	Reported	WP3	209-C-23
Al0(CrMnFeNi)	Al0(Cr20Mn25Fe40Ni15)100	HVOF (DJ2600)	UMR	Processing	WP3	568-E-23
Al14(CrMnFeNi)	Al14(Cr20Mn25Fe40Ni15)86	HVOF (DJ2600)	UMR	Reported	WP3	343-C-23
Cantor+60TiC	40vol%(Co20Cr20Mn20Fe20Ni20) + 60vol%TiC	HVOF (DJ2600)	UMR	Reported	WP3	396-D-23
Al14(CrMnFeNi)+60TiC	40vol%Al14(Cr20Mn25Fe40Ni15)86 + 60vol%TiC	HVOF (DJ2600)	UMR	Processed	WP3	1084-I-23
Al10(CrMnFeNi)	Al10(Cr20Mn25Fe40Ni15)90	HVOF (DJ2600)	UMR	Processing	WP3	1104-I-23

Figure 6. Samples table on SharePoint

The status column (Figure 7) shows the characterisation process's current state of progress, and each partner will then be responsible for updating this column whenever they change the status of a sample. If the sample is in the characterization phase, it may go from the production state to the processing state until it reaches the reported state, which details the completion of the analyses and the acquisition of the relevant data.

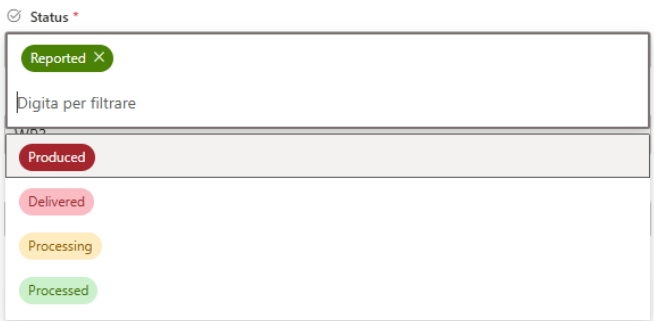


Figure 7. Sample status in SharePoint table samples

This table has been used to create a new table (available on SharePoint) that will be used during WP4. In this case, we wanted to exploit the automatically generated unique progressive numerical identifier associated with a row when it is created: the official nomenclature to be used within the project will be this identifier ID or, with a more meaningful option, the NickName, still consisting of the ID, but with the addition of a speaking abbreviation (structured like the Sample ID in the old table), *Figure 8.* In addition, the new table contains more information about the produced samples (the composition is specified both in volume, atomic and weight; the parameters used for deposition are reported) and more space is dedicated to the progress of the characterisation process: following sample receipt, each partner is required to declare the ownership of the sample by identifying themselves among the Sample Custody and to update the designated columns if they perform characterizations on the sample; in general, the update date must be changed each time modifications are made to the table.

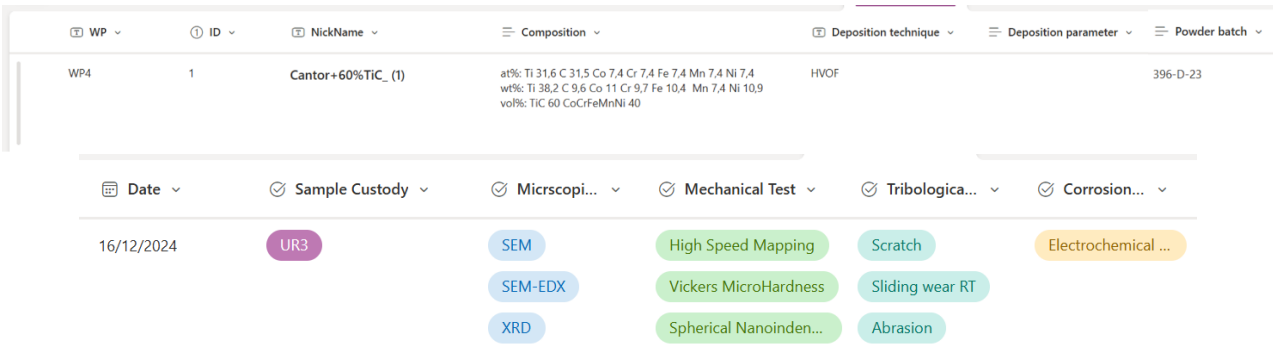


Figure 8. New samples table

3 Conclusions

This deliverable describes the workflow that will be implemented in the experimental campaign to be performed within the WP4 of the CoBRAIN project on wear-resistant and anti-corrosion coatings, obtained through three different thermal spraying techniques. The first part of this deliverable therefore describes the characterisation techniques selected, since they are considered capable of providing, on their own or integrated with each other and with modelling, the most comprehensive and detailed description of the materials. The description of the individual techniques is carried out by applying the CWA 17851, a standard that ensures the accessibility, interoperability and repeatability of characterisation procedures and the data they produce. Specifically, this document contains CHADA updates to the latest version of the CWA for the techniques considered most representative. In addition, an integrated concept map of the experimental and modelling outputs is provided: a clear idea of the data flow between the scientific domains and of any potential properties relationships helps in the creation of a relational database, on which the artificial intelligence algorithm will work.

Finally, the real characterization workflow that will be followed within the project is described. Throughout the WP3, the workflow was applied to materials identified in the literature and was revised until reaching the optimized configuration described in this deliverable. This configuration will be the one directly applied during WP4, where the last refinements will be added. The description of the workflow is, moreover, complemented by indications on the management of samples, developed based on the experience in circulating the samples among partners and exchanging data and information during WP3, and designed to facilitate the experimental procedures.

4 Attainment of Objectives

Table 3. Contribution of D3.3 toward the attainment of the general objectives of CoBRAIN

Ref.[3]	General Objective	Status
O1.3	Consistent and extended open experimental-dataset. Dataset of material and coating-specific measured properties, based on >15000 single characterization data points, obtained from a homogeneous and unbiased experimental approach. These data points are obtained on feedstock, specimens and components manufactured at industrially relevant pilot scale, and considering a minimum of 30 HHMs	The characterisation procedures described in this deliverable, as well as the guidelines for performing them as effectively as possible, are those needed to produce the homogeneous and complete experimental dataset.
O2.1	a Knowledge base repository for the ontological representation of Thermal Spray. Domain-specific TBox and ABox are developed exploiting the existing top-level and mid-level ontology released by H2020 projects. The RDF triplestore will host the semantic relations, Triples, between the results emerging from the Modelling, Characterisation and Processes Workflow. More than 1000 Triples are expected to be part of the repository.	The characterisation procedures described in this deliverable are those that need to be applied to produce the experimental dataset. In addition, the development of a characterisation concept map integrating the experimental part with the modelling part, described within this deliverable, helps in structuring the data repository.
O2.2	CHADA workflow for clear understanding of the composition-process-microstructure-properties performance correlation chain. The Documentation of the specific characterisation methods will be defined in accordance with CWA17815, as well as workflows that correlate nano- and micro-scale characterization of the coating properties with microscopy and spectroscopy methods and with standardized wear and corrosion tests that have wide industrial acceptance. More than 20 methods will be included in the CHADA.	In this deliverable, the concept map that integrates the various characterization techniques applied within the project is illustrated, highlighting the correlations between the properties. The CHADA updated to the latest version of the CWA for the most representative techniques of the project are also included.

Table 4. Contribution of D3.3 toward the attainment of the specific objectives of WP3

Ref.[4]	Work Package Objective	Status
WP3.1	Setup the equipment for the main experimental campaign in WP4	The identified characterization equipment (listed in the SharePoint table Experimental facilities) was applied to define and execute the experimental process, which is described in this deliverable. The most representative characterization techniques are detailed described within their respective CHADAs.
WP3.2	Validate the characterization workflow, the specimen amount and the consistency of the data	The deliverable includes a description of the optimised configuration (characterization workflow and specimen amount) identified by the application and review process performed in WP3; this configuration ensures the creation of the required experimental dataset.
WP3.3	Evaluate material consumption	Within the deliverable, the proper amount of specimens to be produced in order to complete the characterisation process is stated.
WP3.4	Identify criticalities before running the experimental part on the new materials from modelling	Any important issues that were discovered throughout the procedure review process have been resolved in the

		optimized configuration of the characterisation workflow, which is detailed in this deliverable.
WP3.5	Provide reference data for the modelling optimization	The integrated concept map included in this deliverable describes the data flow between the experimental and modelling part.
WP3.6	Include promising material identified in the literature in the final dataset	The analyses of the compositions identified in the literature were used in WP3 to develop and revise the characterization process: the most promising compositions, identified through this process, are included in the final experimental dataset. WP4 will instead focus on new materials identified by the ML model.

5. Annex I. Table forms from the updated CWA 17815

1. Table 1

User story	
Name	
Description	
Client/requester	

2. Table 2

Material	
Name	
Description	
Reference to standard *(Add reference to standard, if any)*	
Further documentation *(Link to a relevant document/web resource)*	
Material identifiers	
Physical structure *(if known prior to the characterisation)*	
Material composition / Chemical structure *(if known prior to the characterisation)*	
Other material properties *(if known prior to the characterisation)*	

3. Table 3

Operational context	
Description	
Further documentation *(Link to a relevant document/web resource)*	

4. Table 4

Rationale	
Description	
Further documentation *(Link to a relevant document/web resource)*	

5. Table 5

Diagram	
Put here a diagram depicting the characterisation workflow.	
Link to diagram *(In case there is a publicly available version of the diagram)*	

6. Table 6

Characterisation Workflow	
Name	
Description	
Reference to standard *(Add reference to standard, if any)*	
Laboratory	

Operator	
Further documentation *(Link to a relevant document/web resource)*	

7. Table 7

Sample	
Name (unique)	
Description	
Reference to standard *(Add reference to standard, if any)*	
Further documentation *(Link to a relevant document/web resource)*	
Identifier	
Dimensions	
Physical structure	
Material composition / Chemical structure	
Other sample properties	
Hazard	

8. Table 8

Sample Extraction	
Name (unique)	
Description	
Laboratory	
Operator	

Level of expertise of the operator and/or laboratory capabilities *(with reference to relevant standards)*		
Level of automation (*Describe what can be automated in the process*)		
Reference to standard *(Add reference to standard, if any)*		
Further documentation *(Link to a relevant document/web resource)*		
Input: Batch	Material	
	Provider	
	Identifier	
	Description	
Output: Sample *(Unique name or ID of sample, see also Sample section for describing the sample in detail*)		
Instrument	Name	
	Manufacturer	
	Model	
	Unique ID	

9. Table 9

Sample preparation	
Name (unique)	
Description	
Laboratory	

Operator		
Level of expertise of the operator and/or laboratory capabilities *(with reference to relevant standards)*		
Level of automation		
Reference to standard *(Add reference to standard, if any)*		
Further documentation *(Link to a relevant document/web resource)*		
Sample *(Unique name or ID of sample, also Sample section for a detailed description of the sample*)		
Holder *(in case the preparation involves mounting on a holder)*		
Output *(Sample preparation report)*		
Instrument	Name	
	Manufacturer	
	Model	
	Unique ID	
Parameters *(settings for the preparation)*		
Input *(from other steps of this characterisation procedure, other procedures or external data)*		
Environment	Description	

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	Properties	

10.Table 10

Sample inspection		
Name (unique)		
Description		
Laboratory		
Operator		
Level of expertise of the operator and/or laboratory capabilities *(with reference to relevant standards)*		
Level of automation		
Reference to standard *(Add reference to standard, if any)*		
Further documentation *(Link to a relevant document/web resource)*		
Sample *(unique name of the sample)*		
Output *(sample validation report)*		
Instrument	Name	
	Manufacturer	
	Model	
	Unique ID	
Parameters *(Settings for the inspection)*		

Input *(from other steps of this characterisation procedure, other procedures or external data)*	

11.Table 11

Calibration		
Name		
Description		
Laboratory		
Operator		
Level of expertise of the operator and/or laboratory capabilities *(with reference to relevant standards)*		
Level of automation		
Reference to standard *(Add reference to standard, if any)*		
Further documentation *(Link to a relevant document/web resource)*		
Reference sample *(Specify in case of a Reference Sample, Reference Material or a Certified Reference Material)*		
Output (Calibration data)		
Instrument	Name	
	Manufacturer	
	Model	
	Unique ID	
	Date of last calibration	

Parameters (settings)	
Input (from other steps of this characterisation procedure, other procedures or external data)	

12.Table 12

Measurement parameters adjustment	
Name	
Description	
Laboratory	
Operator	
Level of expertise of the operator and/or laboratory capabilities *(with reference to relevant standards)*	
Level of automation	
Reference to standard *(Add reference to standard, if any)*	
Further documentation *(Link to a relevant document/web resource)*	
Sample *(unique name of the sample)*	
Output *(Adjusted settings for the instruments)*	
Input *(from other steps of this characterisation procedure,*	

other procedures or external data)	

13.Table 13

Measurement Process		
Name		
Description		
Laboratory		
Operator		
Level of expertise of the operator and/or laboratory capabilities *(with reference to relevant standards)*		
Level of automation		
Reference to standard *(Add reference to standard, if any)*		
Further documentation *(Link to a relevant document/web resource)*		
Sample *(unique name of the sample)*		
Output *(Adjusted settings for the instruments)*		
Input *(from other steps of this characterisation procedure, other procedures or external data)*		
Instrument	Name	
	Manufacturer	
	Model	

	Unique ID	
Parameters		
Probe		
Interaction volume		
Detector		
Environment	Description	
	Properties	
Characterisation Signal		

14.Table 14

Data processing	
Name	
Type of data processing *(e.g. data normalisation, data filtering, post-processing, measurement uncertainty calculation)*	
Description	
Laboratory	
Operator	
Level of expertise of the operator and/or laboratory capabilities *(with reference to relevant standards)*	
Level of automation	
Reference to standard *(Add reference to standard, if any)*	
Further documentation	

(Link to a relevant document/web resource)	
Input *(from other steps of this characterisation procedure, other procedures or external data)*	
Output *(Secondary data)*	
Data Processing Model	
Data Processing Software	
Follows (*Unique name of the preceding step*)	

15.Table 15

Characterisation results		
Characterisation results	Name	
	Description	
	Further documentation *(Link to a relevant document/web resource)*	

6. Responses to requests for revisions

Comment	Section 4.1: material table, section of properties prior to characterization, does it mean these properties are known because of modelling?
Response	The CWA-17815 CHADA tables are meant to have general applicability to any type of characterization done for any purpose. Therefore, the "Material properties known prior to the characterisation" entry refers to any existing prior knowledge of a sample that is pertinent to the characterization activity being performed, from any source. For a well-known material, for example, properties may be an established knowledge available in the literature and/or in textbooks; alternatively, the properties may be known from prior testing or from modelling activities. The text in Section 1.2.1 introducing the general features of the updated CWA-17815 was expanded to clarify the general nature of the CHADA tables and the kind of information on known material properties that can be entered into the table.
Comment	Comment on surface roughness before spraying.
Response	While surface roughness before spraying is clearly an important parameter, it is not directly pertinent to the coatings' characterization workflow, and it is instead discussed in the deliverables that describe the coated samples' production workflows: D3.2 (sections 2.1.2, 3.1.2 and 4.1.4) and the upcoming deliverables D4.2, 4.3 and 4.4.
Comment	Comment on the adequation of the abrasion wear standard as dry abrasion (despite discussed in the meeting, I recommend including it here since ASTM g65 and b611 could also apply).
Response	The rationale for using a steel-wheel abrasion test instead of the ASTM G65 rubber-wheel test is now more explicitly laid out in the "Rationale" part of the updated CHADA of abrasion testing, page 35.
Comment	Page 9 mentions the use of ball-on-disc for adhesion wear, but D8.4 reports the use of Al2O3 ball. Please, clarify.
Response	The indication was indeed imprecise and was corrected as "sliding wear". In fact, various wear mechanisms are possible in a sliding wear test, including adhesive wear, delaminative wear, surface fatigue, and tribochemical wear (e.g. tribo-oxidation), depending on the specifics of the tribocouple and the test conditions. Adhesive wear is anyway possible even using an Al_2O_3 ball and has indeed been observed in this project e.g. with some of the metallic coatings. As explained e.g. in [G.W. Stachowiak, A.W. Batchelor, Engineering Tribology – 4th Ed., Butterworth-Heinemann, pp. 723-726], indeed, a clean metal surface can adhere even strongly to a ceramic, undergoing adhesive wear and, in some cases, interacting tribochemically. This may occur more easily when high contact pressures and correspondingly high stresses at the asperity level break the surface layers existing both on the metal (e.g., native and/or tribologically induced oxide scales, adsorbates, etc.) and on the ceramic (e.g. hydroxylated surface layers), promoting adhesive interactions e.g. between the electrophilic metal ions and the negatively charged anions of the ceramic (either an oxide ceramic, or an oxide layer on a non-oxide ceramic).

Comment	Page 50: ISO 14577 relates to hardness and not corrosion. What is the surface finish used in corrosion testing?
Response	Concerning the standard: many thanks for pointing out; it was indeed a typo. The indication was corrected to "ASTM G3-14", which is indeed the applicable standard describing the type of output parameters obtained from an electrochemical polarization test and the applicable conventions (e.g. sign conventions for potentials and currents). Concerning the surface finish: all samples used in wear, scratch, and corrosion testing are identically finished down to a 3 µm diamond paste. This results in a mirror-like finish, although the exact roughness value cannot be specified or predicted precisely a-priori, because, especially depending on the porosity of the coating, the average and maximum roughness amplitudes may change. Hence, no specific roughness value is indicated in the corresponding CHADA table. Anyway, even for somewhat porous coatings, this type of finish, by substantially eliminating most of the protruding asperities, should always guarantee that Ra/Sa are below 0.1 µm (with a very dense coating, the value may even be as low as 0.02 µm). Thus, an indication was added in Table 2, page 71, where the specifics of the specimens employed in each test are described.
Comment	Page 68: some testing include just 2 replicas, what if not enough?
Response	The original text in Section 2.2 may in fact have been misleading because it did not differentiate with sufficient clarity (even terminologically) between the number of coated plates that are produced and allocated to each characterization procedure, the number of specimens that can be obtained from a single coated plate (e.g. by cutting, etc.), and the number of replicates needed for each test. Therefore: • The terminology has been revised so that it is now unambiguously specified that the samples' counts on pages 69-70 refer to the number of full 60×25×3 mm coated plates to be produced, and how they are allocated to each characterization activity. • Table 2 (page 71) was revised by adding two columns: one with the number of specimens that can be obtained from a single coated plate, in the light of the specimen size listed in the fourth column of the same table; and one with the minimum number of needed replicates for each test. The number of plates allocated to each test is such that it is always possible to obtain an excess of specimens for each characterization, in case additional replicates are needed (e.g. higher variability than expected among the results; experimental errors during a replication; etc.). This is clearly more pertinent to some characterization procedures than others, e.g. SEM+EDX or indentation tests require small cross-sectional specimens that can always be obtained in far larger numbers than are actually needed. On the other hand, wear and corrosion tests, which use larger specimens, require a more accurate counting to make sure enough specimens can be available at all times. • The text in Section 2.2, pages 69-70, was further revised to include the above considerations.