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Integrated Computational-Experimental Material Engineering of Thermal Spray Coatings

CoBRAIN

Deliverable D3.2

Guidelines for specimen realization by HVOF, CGS, HVAF

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0.1	22/04/2024	UMR	First draft with general workflow and HVOF workflow
0.2	17/06/2024	UMR	Edits to the previous draft. Added summary, conclusions, and attainment of objectives.
0.3	21/06/2024	UB + UMR	Added CGS workflow. Further minor edits.
0.4	21/06/2024	MBN	Added HVAF workflow.
1.0	29/06/2024	UMR	Final check, finalized version.
2.0	23/09/2024	UMR	Added/updated roughness values of grit-blasted substrates according to the procedures for HVOF (Section 2.1.2), HVAF (Section 4.1.4) and Cold- Spray (Section 3.1.2). Corrected the caption and the reference to Figure 11 in the text of Section 3.1.4. Added or corrected the references to other figures throughout Sections 3 and 4.

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Publishable Summary[2]

This deliverable describes the workflows and procedures that were developed to ensure a repeatable, high-throughput production of thermally sprayed coatings by High Velocity Oxy-Fuel (HVOF), High Velocity Air-Fuel (HVAF), and Cold Gas Spray (CGS). Consistent with the general definition of thermal spraying and its objects and classes given in the ontology (Deliverable D1.1), this deliverable specializes the description of the implementation for each of the three processes. Substrate type, pre-treatment operations, deposition procedures including sample holder characteristics, practical processing steps, torch/substrate kinematics, and parameters' choice are described, and a graphical representation of the workflow is given in all cases.

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

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Abbreviations

Abbreviation	Definition
AISI	American Iron and Steel Institute
CGS	Cold Gas Spraying
HVAF	High Velocity Air-Fuel
HVOF	High Velocity Oxygen-Fuel
IR	Infrared
WP	Work Package

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1 General workflow

The workflow of a thermal spray process follows the general layout described in the A-Box that represents the mereocausal relations among the sub-processes and objects defined within the Thermal Spray Ontology, in accordance with Section 4.1.3 of Deliverable 1.1 (Figure 1). The definitions of the individual objects and processes are provided in the Ontology model described in Deliverable 1.1.

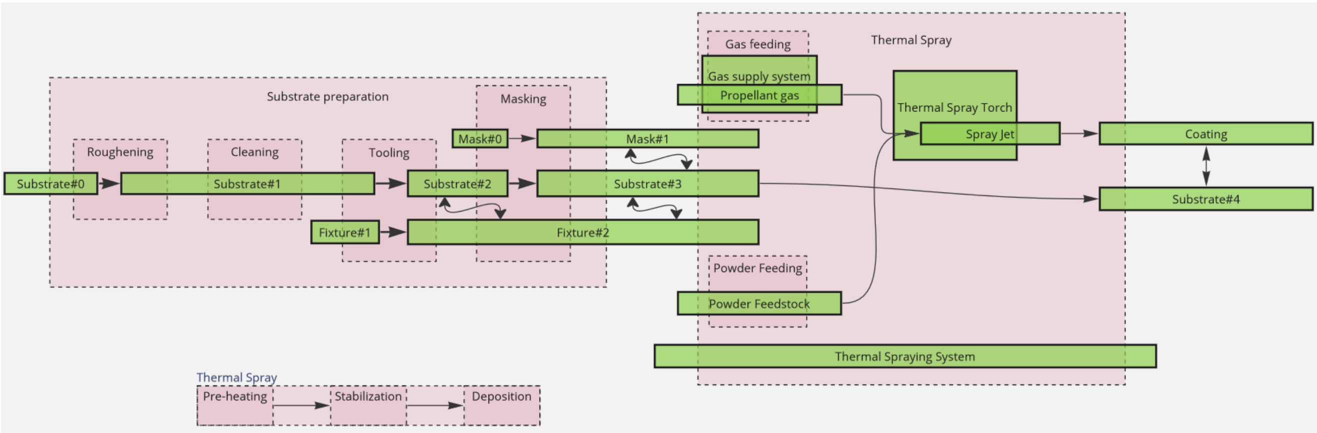

Figure 1. Ontology A-Box describing the instancing of the generic thermal spray workflow implemented in the CoBRAIN project

Briefly, a thermal spray process consists of two main sub-processes:

- Substrate preparation: it includes the sequence of operations needed to enable the deposition on the substrates.
  Substrates need to be roughened to promote mechanical interlocking of the deformed particles to the substrate asperities, which provides an important contribution to the overall adhesion force, and to increase the extension of the specific coating/substrate contact interface, which further contributes to produce a larger adhesion force as the resultant of individual adhesive interactions (mechanical interlocking, Van der Waals forces, metallurgical/diffusion bonding, etc.). If needed, substrates might be cleaned before roughening if grease, oil and/or dirt are present, lest they interfere with the roughening procedure and/or become embedded in the substrate itself. Cleaning is mandatory after roughening to remove residuals of the roughening media (e.g. loose grit residuals from grit-blasting operations).
  Once prepared, the roughened and cleaned substrates can be stored before deposition. It is important to prevent further uptake of dirt and/or oxidation of the activated surface, which would compromise adhesion and require repeated roughening and cleaning. The substrates are then mounted onto a tooling that allows them to be set into the thermal spray system. At this stage, masks can be optionally applied in case parts of the substrates need to be left uncoated. Masks can consist e.g. of heat-resistant silicone tapes, metallic tapes, or they can be integral part of the tooling, covering part of the substrates when they are mounted.
- Coating deposition: it includes the sequence of operations to perform the thermal spraying itself. The tooling with the substrates is set in the thermal spray booth (often onto vertical or horizontal turntables and/or laths); the process parameters are set into the control unit(s), including the substrate and torch motion/kinematics controls, the gas flow rates and powder feed rate, and any electrical parameters; the torch may be fitted with changeable accessories (e.g. nozzles, combustion chambers) if applicable. The feedstock powder is loaded into the powder feeder; the torch is started, gas flow and powder feed are stabilized; torch and substrate motion is started and the deposition takes place. Sensor diagnostics

can be activated at this stage if available (e.g. substrate temperature monitoring; particle in-flight diagnostic systems; etc.).

At the end of the deposition process, the tooling is removed from the thermal spray booth, masks (if any) are removed, and the substrates are taken off the tooling. Optional operations like cleaning to remove overspray dust, marking of the samples with univocal codes, and packaging of the samples can be performed at this stage.

2 High Velocity Oxy-Fuel (HVOF) workflow

2.1 Substrates and substrate preparation procedure

2.1.1 Substrate material and geometry

It was convened that HVOF depositions are carried out on AISI304 stainless steel substrates. The first series of depositions in WP3 are performed exclusively on plates of 60×25×3 mm size. These plates are easy to mount onto the tooling, their limited mass allows the use of precision scales to weigh the samples before and after the spraying process to measure the deposition efficiency, and their size is also easily handled with lab-scale metallographic preparation equipment like precision metallographic cutting machines. Thus, cutting for sample preparation (which will be part of the procedure described in the forthcoming deliverable 3.3) can be carried out at moderate stress levels, ensuring that no artefacts like cracks and burns will be introduced at that stage.

In WP4, tensile adhesion tests are also foreseen and these will be performed using the 1 in-diameter cylinders described in the ASTM C633-13(2021) standard.

The substrates are commercially procured from industrial suppliers with slightly chamfered edges and no oil/grease covering.

2.1.2 Grit-blasting

Because the substrates do not exhibit major dirt contaminations in their as-supplied conditions, they do not require pre-cleaning prior to roughening (see the outline of the General workflow).

Roughening is performed by grit blasting using a hand-held vacuum blasting equipment. Whilst the blasting conditions in a hand-held device might not be so perfectly well reproducible as in an automated device, such type of system is more suitable for lab-scale settings that must be versatile enough to handle a variety of substrate shape and sizes but are not expected to handle very large, heavy objects.

Blasting is carried out using brown corundum, a common blasting media type for general-purpose thermal spray applications that is therefore representative of possible eventual industrial applications.

The median equivalent diameter of the fresh media as measured with a laser-scattering particle size analyser (Mastersizer 2000, Malvern Panalytical, Malvern, UK) using a wet-dispersion method (Hydro-2000S wet dispersion unit) is ~655 µm (Figure 2). The media is automatically sieved during the blasting process to remove fines and recover the particles that still have suitable size.

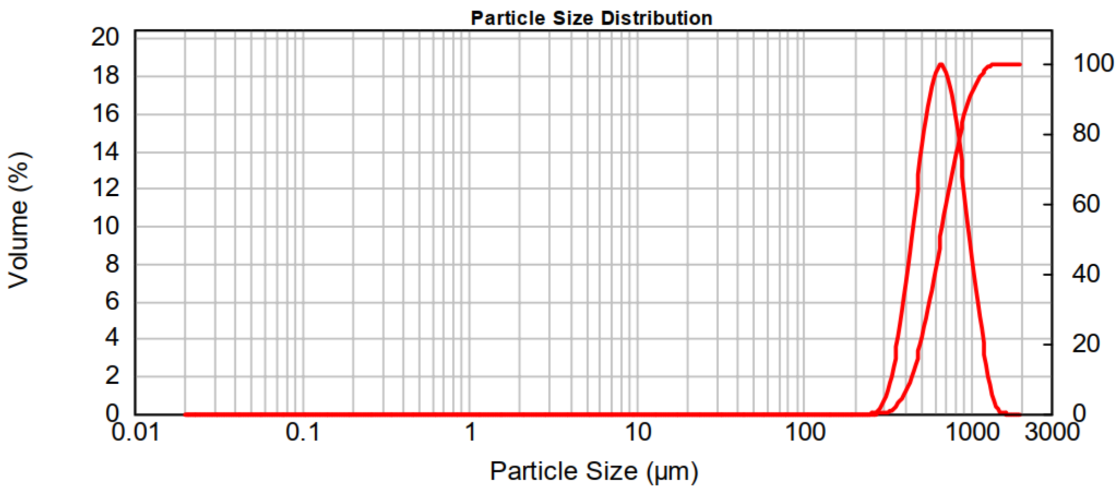

Figure 2. Particle size distribution of the brown corundum blasting media (laser scattering with wet dispersion unit)

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The blasting equipment is operated at ~5 bar pressure. The plates are kept at ~10 cm from the blasting nozzle and at an angle of ~60° between the surface plane and the blasting jet axis. Blasting is continued until the surface appears completely roughened upon visual inspection by the operator.

Surface roughness measurements performed with an optical profilometer (ConfoSurf, ConfoVis GmbH, Jena, Germany) operated in focus variation mode with a 5× objective acquiring an area of 1.7 × 1.7 mm^2 indicate that the attained roughness is Sa ~ 7 µm, Sz ~ 107 µm, Sal ~ 60 µm. Notably, the autocorrelation length Sal, which can be interpreted as the lateral "wavelength" of the profile, is comparable to or just slightly lower than the expected diameter of a lamella resulting from a powder particle with an initial diameter of 20 – 30 µm and a flattening ratio of 3 – 4 upon impact, as would be typical of HVOF spraying. Thus, the achieved surface profile appears suitable to ensure proper mechanical interlocking between the first layer of lamellae and the substrate.

2.1.3 Cleaning and storage

The grit-blasted substrates are cleaned with compressed air to remove loose residuals, then cleaned in an ultrasonic bath using acetone for at least 5 min to further remove grit residuals and any dirt left at the end of the process, and dried again with compressed air.

The samples are then kept in a stove at 60 °C to prevent the uptake of humidity until they are employed.

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2.2 Sample holder design

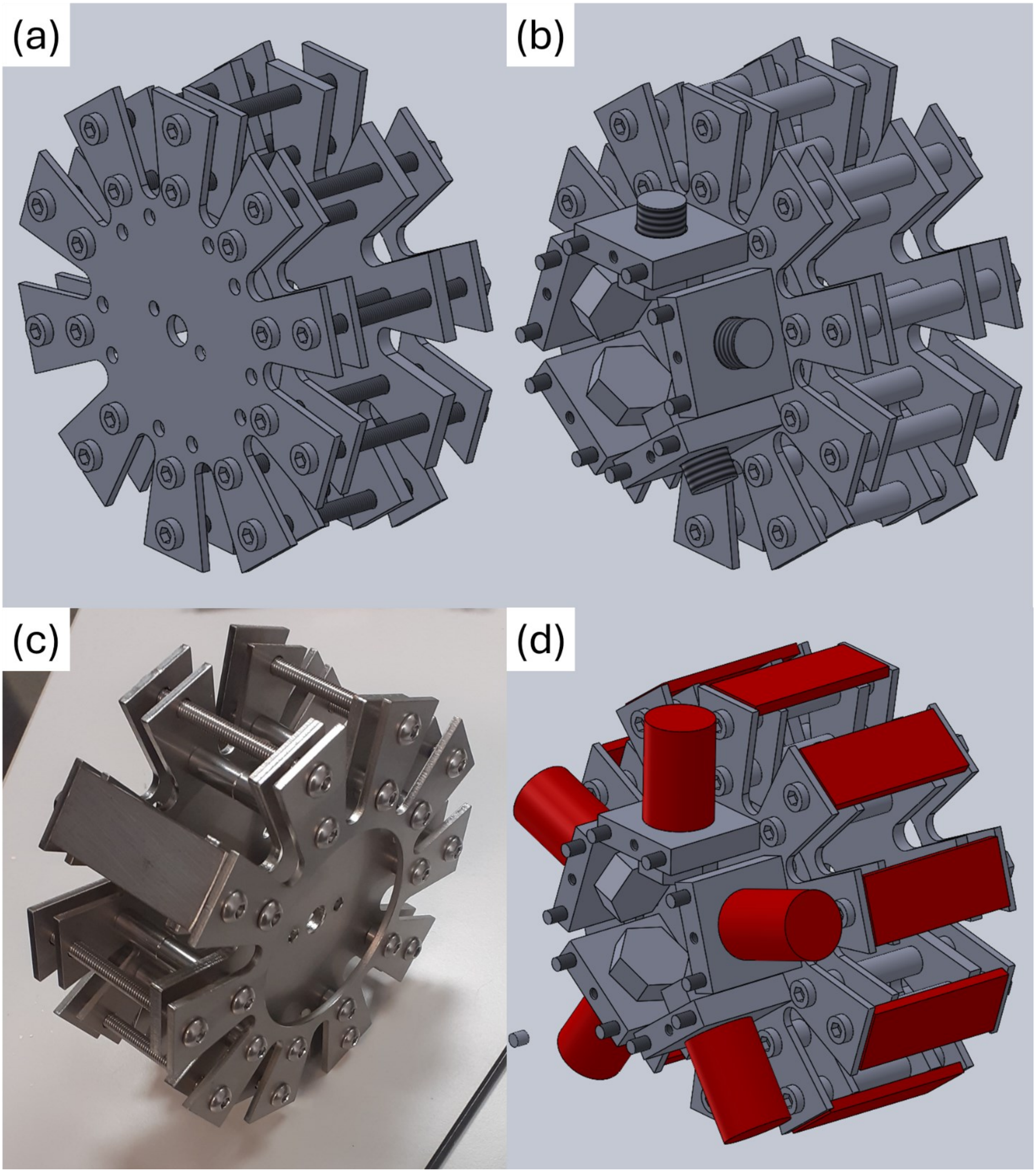

Figure 3. Sample holder design: (a) setup for plates only, (b) setup for plates and cylinders, (c) photograph of the sample holder assembled in plates-only configuration, (d) schematic of the sample holder in full configuration with projected position of plates and cylinders (in red)

A modular sample holder was designed to allow either the deposition on plates alone (WP3) or on plates and cylinders (WP4) - Figure 3. The holder is made of AISI304 stainless steel. Two inner filled plates, 5 mm-thick, provide the structural core (Figure 3a). One of the inner finned plates is designed to allow fixing with screw into a cylinder that will be mounted into the mandrel of the thermal spray booth. The other plate has a provision for the optional installation of the cylinder holders, which consist of plates with threaded nuts onto which the ASTM C633-type cylinders (having a threaded blind hole for subsequent mounting onto a tensile testing machine) can be fixed (Figure 3b, d). Importantly, the system is designed so that the surfaces of plates and cylinders are located on the same plane, so that the stand-off distance between the thermal spray torch and the substrates is always the same.

The spacing between the two plates is set using cylindrical spacers. The structural plates are then fixed with a bolt system consisting of screws of suitable length, installed co-axially along the spacers, and nuts. With this system, the holder can be adapted to host plates of virtually any length.

Two outer finned plates with 3 mm thickness are then fixed to the inner plates again using cylindrical spacers and coaxial bolts. Once the bolt connections are all securely tightened, the plates are inserted between the opposing fins of the outer plates until they lie against the tips of the fins on the inner plates. An additional bolt that connects the opposing fins of the two outer plates is then tightened (Figure 3a ,c). The deformation of the 3 mm-thick fins thus clamps the plates in place, preventing their dislodgement during the spraying process.

This system allows up to 10 plates and 5 cylinders to be coated in a single spray run using a rotating setup. The number of plates and cylinders is sufficient for the full sets of characterizations foreseen in either WP3 or WP4. The rotating configuration is advantageous because, compared to a fixed setup with the gun traversing in front of stationary substrates, allows faster traverse speeds with correspondingly lower heat accumulation in the samples, hinders the build-up of overspray dust, and allows fixed air jets to be employed to both cool the samples and blow off the overspray itself.

2.3 Coating deposition procedure

2.3.1 Filling of powder feeder

The powder feeder is a gravimetric fluidized-bed system (9MPE-DJ, Oerlikon-Metco, Westbury, NY, USA), where a pressurized hopper is vibrated with an eccentric shaft system powered by compressed air (Figure 4). The hopper is installed onto a load cell connected to a feedback-control system that maintains the mass feed rate at a preset value selected by the user thorough a PLC interface. The powder is collected from the fluidized bed by a pick-up shaft located inside the hopper, using a flow of carrier gas (argon). Two pneumatic radial pistons regulate the opening of a compressible plastic feed tube downstream of the pickup shaft. The feedback-control system therefore adjusts the hopper pressure and the pistons' pressure to control the mass feed rate.

The powder is preliminary heated to 60 °C in closed plastic containers using a stove to prevent the adsorption of humidity that would impede its flow. The container is then tumbled by the operator to counter any particle size-induced segregation that might have occurred during storage. The powder is then manually def into the hopper operating inside the cabin with adequate ventilation and PPE. The hopper should typically be loaded with between 600 g and 5 kg of powder. A powder mass of less than 600 g (also dependent on powder density) is often insufficient to guarantee a stable flow during operation and should preferably be avoided, though it can be allowed when only small amounts of powders are available for brief spray runs.

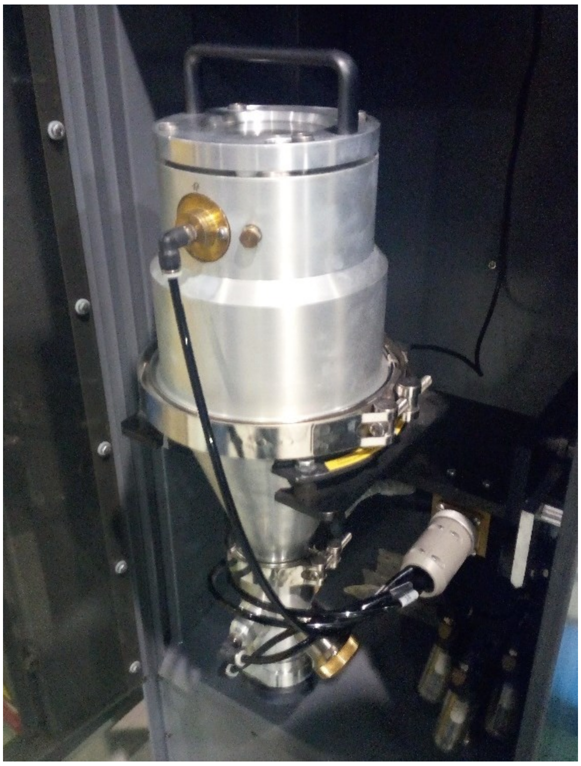

Figure 4. Fluidized-bed pressurized hopper installed onto the load cell in the gravimetric powder feed system

2.3.2 Tooling and spray system setup

The substrates are taken from the stove (see Section 2.1) and three plates for each run are weighed using a balance to at least ±0.01 g accuracy. The measured weights are marked on the rear face of the plates with a permanent marker. All the substrates are mounted onto the sample-holding tooling as described in Section 2.2. All the above operations must be performed wearing clean gloves to avoid contaminating the surface of the samples prior to spraying, which might risk impeding proper adhesion of the sprayed coating.

The tooling is then installed onto the mandrel in the thermal spray booth as shown in Figure 5.

The distance between the HVOF torch nozzle exit and the substrates is adjusted manually to the desired value (measured with a measuring tape to a precision of ±1 mm).

An infrared pyrometer (Optris CT, Luchsinger s.r.l., Curno, Italy) with laser-pointing system (Figure 5) is manually adjusted by the operator to measure the temperature in the centre of the plates at an angular position away from the intersection with the torch axis (at least 45° away from the spray position), to avoid measurement artefacts due to the irradiation from the jet itself.

Compressed air jets (Figure 5) are manually adjusted by the operator so that streams of compressed air are directed onto the substrates at an angular position approximately 90° from the spray position. Thus, the samples are cooled at each rotation and loose overspray particles are blow off without interfering with the thermal spray jet.

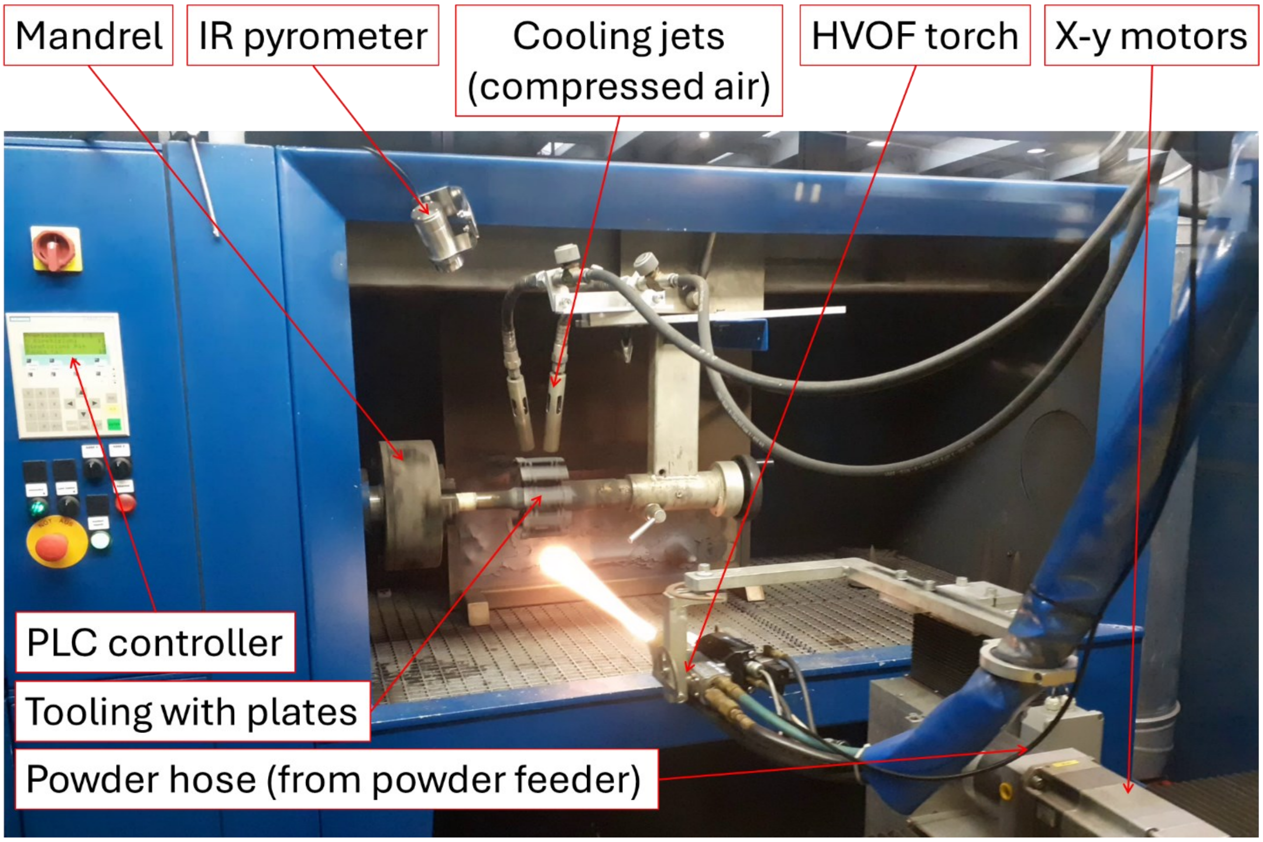

Figure 5. HVOF thermal spray system setup with the torch in the rest position between cycles

2.3.3 Spray operation

Torch/substrate kinematics settings

The traverse speed of the torch and the rotation speed of the mandrel are calculated using an Excel worksheet (Figure 6), based on the desired pass spacing and traverse speed. The operator chooses the initial and final positions of the torch so that the spray jet is entirely out of the substrates at the beginning and at the end of a cycle. The operator also selects the number of cycles between spray interruptions, the duration of the interruption, the total number of cycles, and the number of pre-heating cycles. All values are entered through the PLC interface (Figure 5).

Parametri richiesti				Parametri Robot-Traslatore	
Velocità lineare m/s	Passo deposizione mm	Diametro portacampioni mm		Velocità Mandrino rpm	Velocità torcia mm/min
0,75	7	110		130	912,0
Velocità lineare mm/min				300	12000
45000				Max valore consentito	

Figure 6. Screenshot of the Excel sheet for the calculation of the mandrel rotation speed and torch traverse speed

Gas and powder feed settings, coating deposition

The torch is started in pre-heating mode with no powder feed and no cooling air flow to the substrates. After the automatic ignition and ramp-up cycle is completed, the operator manually sets the flow rates of hydrogen, oxygen, and compressed air to the desired settings using knobs on the gas flow control unit, and if needed adjusts the respective pressures using the pressured regulators along the gas feeding lines.

The operator starts the recording process on the PC interface of the IR pyrometer and starts the pre-heating cycle. When finished, the operator switches the control panel selector from pre-heating mode to spray mode, which starts the feeding process. The operator sets the feeding gas flow rate to the desired value, the compressed air to the vibrating unit of the fluidized bed hopper to its maximum value, verifies visually that the powder feeds to the torch, and reads the actual feed rate, waiting until it is no more than ±4 g/min from the desired value, with no major oscillations. At this point, the operator starts the deposition cycle and opens the cooling air flow. The operator visually supervises the process, adjusting the gas flow rates and pressures if and as needed to maintain the pre-set values.

At the end of the process, the operator switches the system back to pre-heating mode to stop the powder flow and switches off the cooling air flow, waits until most of the powder still present along the feed line has been removed, and eventually switches to system to stop in order to trigger the automated ramp-down procedure.

The operator subsequently switches off the data recording from the IR pyrometer.

2.4 Post-coating operations

Once the powder feeder is completely de-pressurised, the operator unmounts the feeding hose from the hopper and blows it with compressed air until no powder is ejected from the front of the gun to ensure that no residual powder is left. This prevents contaminations when changing from one powder to another and avoids the unwanted projection of particles onto the substrate during the subsequent pre-heating cycle.

When the surface temperature of the substrates is below 100 °C (the lower limit of the IR pyrometer employed in the current setup), the operator removes the tooling from the mandrel and removes the coated substrates from the tooling (using suitable high-temperature gloves). The three weighed plates are weighed again, and the final weights are noted alongside the initial weights to compute the deposition efficiency.

2.5 Logbook and data storage

All deposition parameters, initial and final weights are note in an Excel logbook (Figure 7), as described in the Data Management Plan, along the operations. The operator also notes any specific issues (e.g. whether the actual powder feed rate was stable or unstable).

Pyrometer measures are saved in a txt file.

Sample ID	Feedstock	Lot. N.	Substrate	Date	Run n°	length	width	H_2		O_2		Air		Pwdr. Feed	Standoff	Pitch	Speed	n° cycles	n° passes/cycle	n° preheat passes	weight in	weight fin	weight in	weight fin	weight in	weight fin	gain	sprayed mass	efficiency	Notes
						mm	mm	FMR	psi	FMI	psi	FMI	psi	g/min	mm	mm	mm/s				g	g	g	g	g	g	g	g	g	%
AlCuCrFeNi+60TiC RUN1	Al9Cr18Cu18Fe18Ni36+60vol%TiC	072-A-24	AISI 304	19/02/2024	1	60	25	56	140	30	170	35	100	20	250	5	750	13	3	3	35,16	38,27	34,32	37,47	35,25	38,33	3,113	5,2	59,9	*New sample holder. Sli*
AlCuCrFeNi+60TiC RUN2	Al9Cr18Cu18Fe18Ni36+60vol%TiC	072-A-24	AISI 304	20/02/2024	2	60	25	62	140	32	170	35	100	20	250	5	750	13	3	3	35,07	38,08	34,62	37,47	35,1	38,11	2,957	5,2	56,9	*New sample holder. Sli*
AlCuCrFeNi+60TiC RUN3	Al9Cr18Cu18Fe18Ni36+60vol%TiC	072-A-24	AISI 304	20/02/2024	3	60	25	64	140	34	170	35	100	20	250	5	750	13	3	3	34,84	37,74	34,85	37,69	34,13	36,98	2,863	5,2	55,1	*New sample holder. Sli*
AlCuCrFeNi+60TiC RUN3	Al9Cr18Cu18Fe18Ni36+60vol%TiC	072-A-24	AISI 304	20/02/2024	4	60	25	56	140	30	170	35	100	20	250	10	750	39	2	2	34,68	37,77	34,82	37,99	35,1	38,27	3,143	5,2	60,4	*New sample holder.*

Figure 7. Example of a HVOF thermal spray logbook with indication of the 4 spray runs for each powder: "low" (Run 1), "medium" (Run 2), and "high" (Run 3) parameter settings, and parameter settings for lower surface temperatures (Run 4)

2.6 Process parameters selection

Based on preliminary tests, it is convened that all HVOF depositions for WP3 are performed using three different sets of parameters, with identical torch/substrate stand-off distance and kinematics but different gas flow rates ("low", "medium", and "high" rate settings) as shown in Figure 7. These conditions are indeed conducive to dense coatings of suitable quality, thus rendering the results of their characterization meaningful as a validation to physical models and AI predictions (Figure 8).

In addition, based on the experience from the spray runs themselves, a modified set of torch/substrate kinematics, featuring twice the pass spacing (at constant pass velocity) with no pause between cycles and double overall number of cycles, has been identified to keep the surface temperature down to a lower level of approximately 300 – 350 °C when in conjunction with the "low" gas flow settings (Figure 9: compare panel (a) with the standard kinematics and panel b with the modified kinematics) while avoiding thermal shocks to the growing coating from excessive heat/cool differences.

During the WP3 workflow, all these three runs (with an optional fourth run performed under the modified kinematics settings) are carried out on 10 plates each. Approximately 300 – 400 g of powder are employed to complete one run. Therefore, a total of 2 kg of powder are needed for each batch to be able to complete a 4-run set of experiments while keeping the hopper content above the recommended lower limit of 600 g (Section 2.3.1). During WP4, the depositions will also be carried out on 5 cylinders. This is projected to increase the powder consumption to around 500 g per run, implying the need for at least 2.5 kg of powder in each batch.

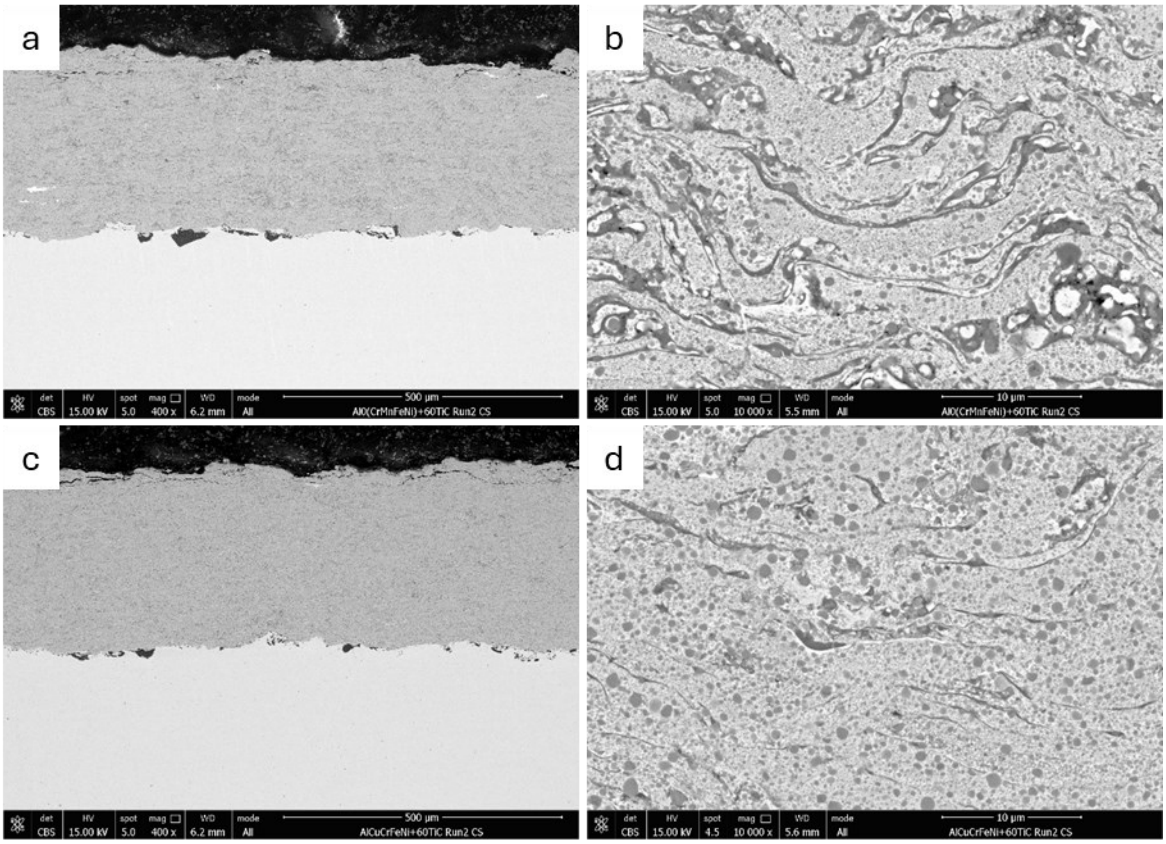

Figure 8. SEM micrographs of the cross-section of samples Al0(CrMnFeNi)+60TiC Run2 (a, b) and AlCuCrFeNi+60TiC Run2 (c, d): overviews (a, c) and magnified views (b, d).

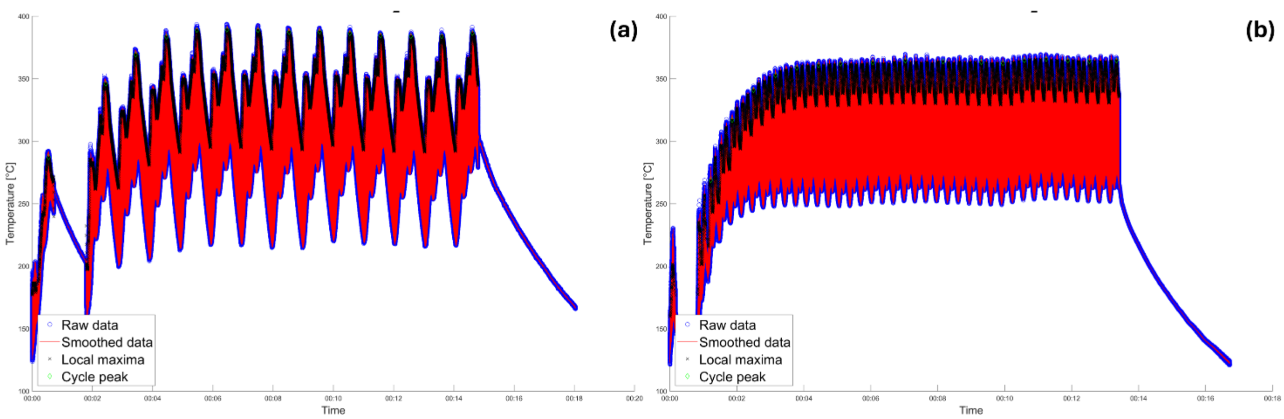

Figure 9. Examples of pyrometer outputs after the deposition of the AlCuCrFeNi+60vol.%TiC feedstock powder using “low” gas flow rates with standard torch kinematics (a) and modified torch kinematics (b).

2.7 Workflow summary

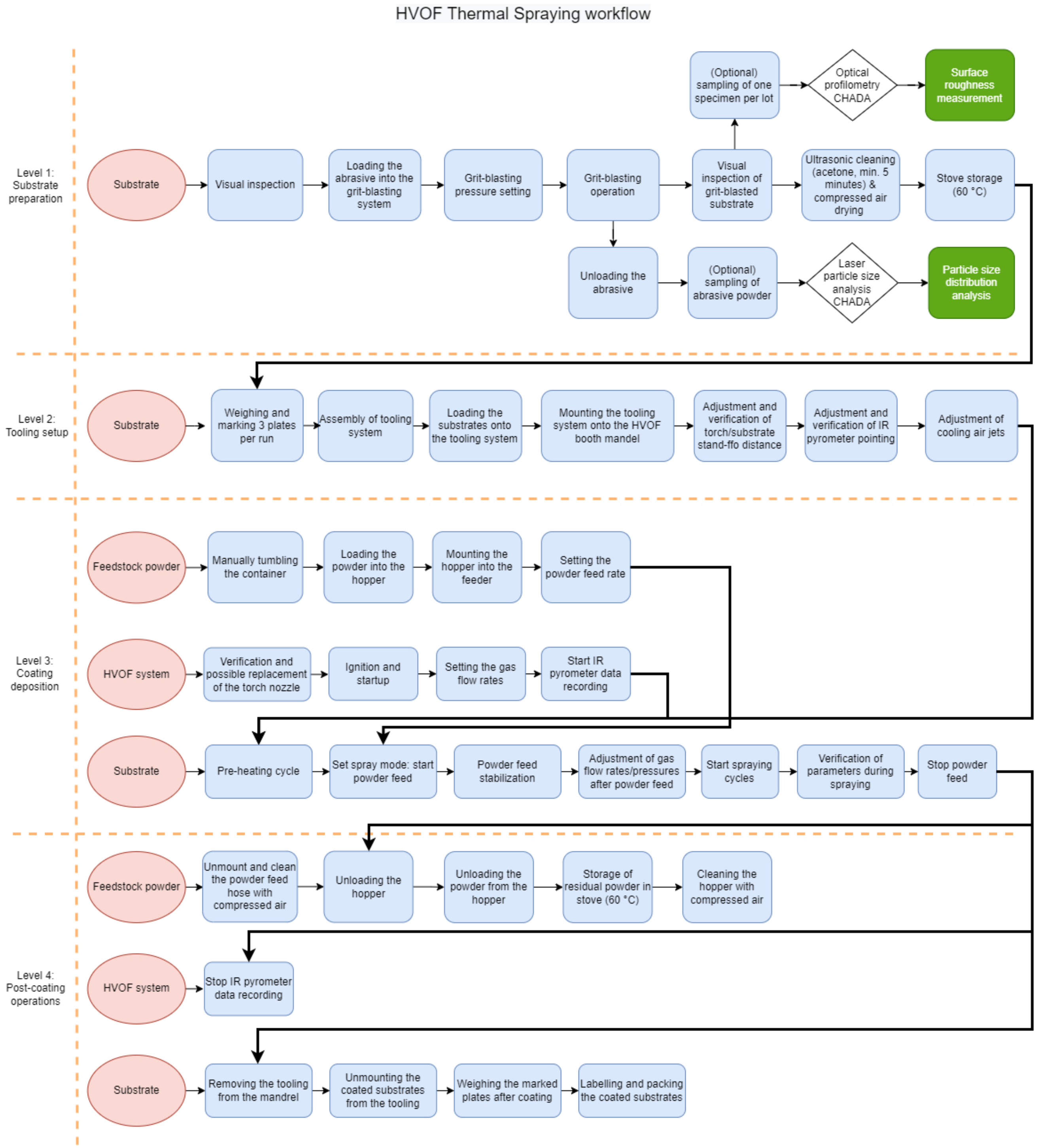

Figure 10. Summary of the HVOF spraying workflow

3 Cold Gas Spray (CGS) workflow

3.1 Substrates and substrate preparation procedure

3.1.1 Substrate material and geometry

It was convened that CGS depositions are carried out on AISI316L stainless steel substrates. The first series of depositions in WP3 are performed exclusively on plates of 50×20×5 mm size. These plates are easy to mount onto the tooling, their limited mass allows the use of precision scales to weigh the samples before and after the spraying process to measure the deposition efficiency, and their size is also easily handled with lab-scale metallographic preparation equipment like precision metallographic cutting machines. Thus, cutting for sample preparation (which will be part of the procedure described in the forthcoming deliverable 3.3) can be carried out at moderate stress levels, ensuring that no artefacts like cracks and burns will be introduced at that stage.

In WP4, tensile adhesion tests are also foreseen, and these will be performed using the 1 in-diameter cylinders described in the ASTM C633-13(2021) standard.

The substrates are commercially procured from industrial suppliers with slightly chamfered edges and no oil/grease covering.

3.1.2 Grit-blasting

Because the substrates do not exhibit major dirt contaminations in their as-supplied conditions, they do not require pre-cleaning prior to roughening (see the outline of the general workflow).

Roughening is performed by grit blasting using a hand-held vacuum blasting equipment. Whilst the blasting conditions in a hand-held device might not be so perfectly well reproducible as in an automated device, such type of system is more suitable for lab-scale settings that must be versatile enough to handle a variety of substrate shape and sizes but are not expected to handle very large, heavy objects.

Blasting is carried out using white corundum (size 24), a common blasting media type for general-purpose thermal spray applications that is therefore representative of possible eventual industrial applications.

The blasting equipment is operated at ~6 bar pressure. The plates are kept at ~10 cm from the blasting nozzle and at an angle of ~45° between the surface plane and the blasting jet axis. Blasting is continued until the surface appears completely roughened upon visual inspection by the operator. The resulting average roughness is Ra = 4.0±0.5 µm.

3.1.3 Cleaning and storage

The grit-blasted substrates are cleaned with compressed air to remove loose residuals, then kept in a stove at 60 °C to prevent the uptake of humidity until they are employed.

3.1.4 Sample holder's design

A sample holder is used to allow the deposition on plates alone (WP3) - Figure 11b. The holder is made of aluminium. The other holder allows to locate the ASTM C633-type cylinders (WP4) - Figure 11a.

A

B

Figure 11. Sample holder design mounted for (a) cylinders only, and (b) plates only.

This first holder allows up to 6 plates to be coated in a single spray planar run, and the other holder allows up to 6 cylinders to be coated in a single spray run. The number of plates and cylinders is sufficient for the full sets of characterizations foreseen in either WP3 or WP4.

3.2 Coating deposition procedure

3.2.1 Filling of powder feeder

The powder feeder is a gravimetric rotary system (Ref PCFC - 1005. PLASMA GIKEN), where a pressurized hopper is rotated and ejected by nitrogen (Figure 12). The powder feeder is placed on a scale that allows to weight the amount of powder being fed at any moment.

The powder is preliminary heated to 60 °C in closed plastic containers using a stove to prevent the adsorption of humidity that would impede its flow. The container is then tumbled by the operator to counter any particle size-induced segregation that might have occurred during storage. The powder is then manually delivered into the hopper operating inside the cabin with adequate ventilation and PPE. The hopper should typically be loaded with between 300 mL up to 5L of powder. A powder volume of less than 100mL is often insufficient to guarantee a stable flow during operation and should preferably be avoided, though it can be allowed when only small amounts of powders are available for brief spray runs.

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Figure 12. Rotary pressurized powder feed system

3.2.2 Tooling and spray system setup

The first step is to take the CGS gun at the side of the Hiwatch 2 system to measure the particle velocities at 5 different points from the nozzle exit to the focus of the camera (10 mm, 15 mm, 20 mm, 30 mm, and 45 mm). Figure 13 shows how the CGS gun is allocated and the Hiwatch system.

Figure 13. TITOMIC Hiwatch 2 system and PCS100 allocation for velocity measurements

Afterwards, 6 substrates are taken from the stove and three plates for each run are weighed using a balance to at least ± 0.01 g accuracy. The measured weights are marked on the rear face of the plates with a permanent marker. All the above operations must be performed wearing clean gloves to avoid contaminating the surface of the samples prior to spraying, which might risk impeding proper adhesion of the sprayed coating. The holder

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is then installed onto the mandrel in the thermal spray booth as shown in Figure 14. The distance between the CGS nozzle exit and the substrates is adjusted manually to the desired value (measured with a measuring tape to a precision of ±1 mm).

Figure 14. CGS thermal spray system setup with the torch in the rest position between cycles

3.2.3 Spray operation

CGS gun/substrate kinematics settings

The traverse speed of the torch, pass spacing, width, height is set directly in the ABB robot, using a program receipt setting (Figure 15). The operator chooses the initial and final positions of the torch so that the spray jet is entirely out of the substrates at the beginning and at the end of a cycle. The operator also selects the number of cycles between spray interruptions, the duration of the interruption, and the total number of cycles. All values are entered through the PLC interface.

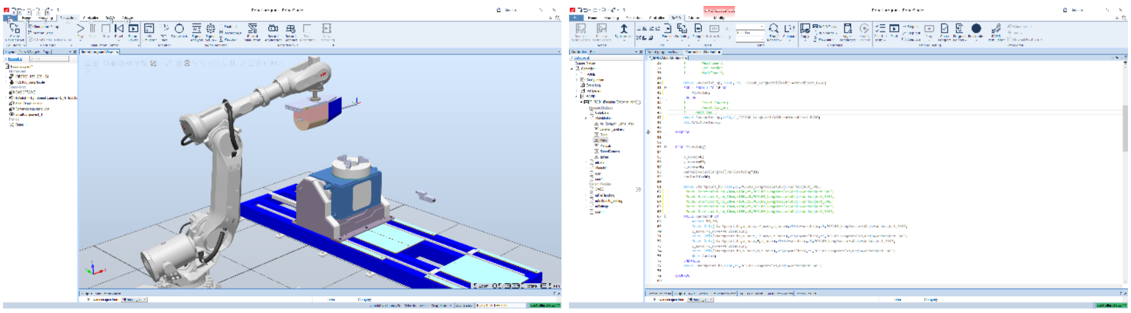

Figure 15. Screenshot of the ABB robot program and settings.

Gas and powder feed settings, coating deposition

The CGS torch is started in pre-set temperature of 800 ºC and the desired pressure for spraying, and mode with no powder feed and no cooling air flow to the substrates. After the ramp-up cycle is completed, the operator manually sets the temperature and to the desired settings using the main PLC control (Figure 16).

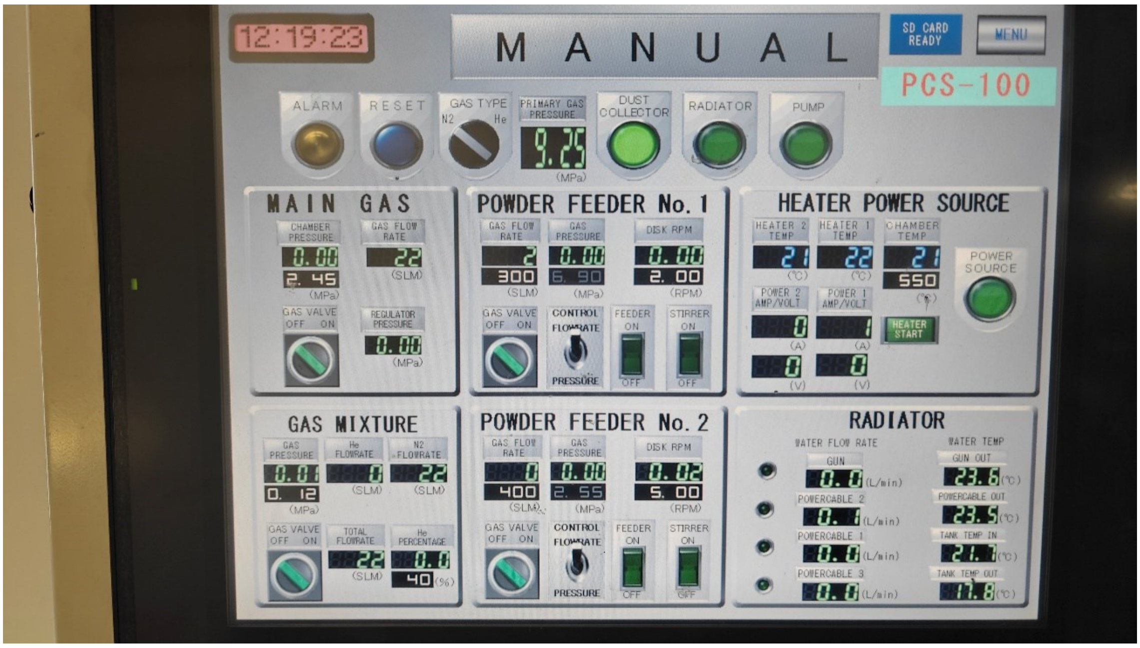

Figure 16. PLC control settings for CGS process.

When the gas flow, P and T is stable, the operator sets the feeding gas pressure to the desired value, and switches on the powder feed unit that contains the feedstock, which starts the feeding process, reads the actual disk rpm, waiting until it is no more than ± 0.1 rpm from the desired value, with no major oscillations. At this point, the operator starts the deposition cycle. The operator then visually supervises the process.

At the end of the process, the operator switches the powder feed unit off and finally switch off the power source, that stop the gas flow and switches to stop to trigger the automated ramp-down procedure.

3.3 Post-coating operations

Once the powder feeder is completely de-pressurised, the operator recovers the powder from the feeder, unmounts the feeding hose and blows it with compressed air until no powder is ejected from the front of the gun to ensure that no residual powder is left. This prevents contaminations when changing from one powder to another and avoids the unwanted spraying of particles onto the substrate during the subsequent spraying step.

The operator removes the coated substrates from the holder (using suitable high-temperature gloves). The three weighed plates are weighed again, and the final weights are noted alongside the initial weights to compute the deposition efficiency.

3.4 Logbook and data storage

All deposition parameters, particle velocities, thickness, initial and final weights are note in an Excel logbook (Figure 17), as described in the Data Management Plan, along the operations. The operator also notes any specific issues (e.g. whether the actual powder feed rate was stable or unstable).

Sample	Thickness (microns)	Powder Code	Temp.	Gas	Pressure	Powderfeeder flowrate	Feedrate gr/min	Taverse speed mm/sec	Step over	Nozzle type	Spray dist.	Gun/System	Measurement Before in mm	Mearurement After in mm	Layer thickness mm	Number of passes	Thickness per layer	Surface treatment	Particle speed	Base material
	short nozzle N2 tests																			
CGS-UB-1	132,6 ± 20,2	Cantor	1100°C	N2	7 MPa	300 SLM	3RPM	250mm/s	1mm	Glass Short	**20mm**	PCS100	4,77	4,92	**0,15**	3	0,05	Grit blasted	**735m/s**	SS316
CGS-UB-2	129,9 ± 14,4	Cantor	1000°C	N2	7 MPa	300 SLM	3RPM	250mm/s	1mm	Glass Short	**20mm**	PCS100	4,77	4,9	**0,13**	3	0,04333333	Grit blasted	**755m/s**	SS316
CGS-UB-3	121,5 ± 14,4	Cantor	1100°C	N2	6 MPa	280 SLM	3RPM	250mm/s	1mm	Glass Short	**20mm**	PCS100	4,77	4,91	**0,14**	3	0,04666667	Grit blasted	**735m/s**	SS316

Figure 17. Example of a CGS logbook with indication of 3 spray runs for CANTOR powder: at different P and T parameter settings, for a specific nozzle (glass short).

3.5 Process parameters selection

Based on preliminary tests, it is convened that CGS depositions for WP3 are performed using at least three different sets of parameters, with identical stand-off distance but different P, T for a specific nozzle, as shown in Figure 17 (short nozzle), combining also with same set of parameters for the long nozzle.

During the CGS WP3 workflow (Figure 18), all these runs are carried out on 6 plates each. Approximately 300 g of powder are employed to complete one run + Hiwatch measurements. Therefore, a total of approx. 2kg of powder are needed for each batch to be able to complete a set of experiments while keeping the hopper content above the recommended lower limit of 100mL (Section 2.3.1). During WP4, the depositions will be carried out on 12 flat substrates and 5 cylinders, implying the need for at least 2 kg more of powder in each batch.

3.6 Workflow summary

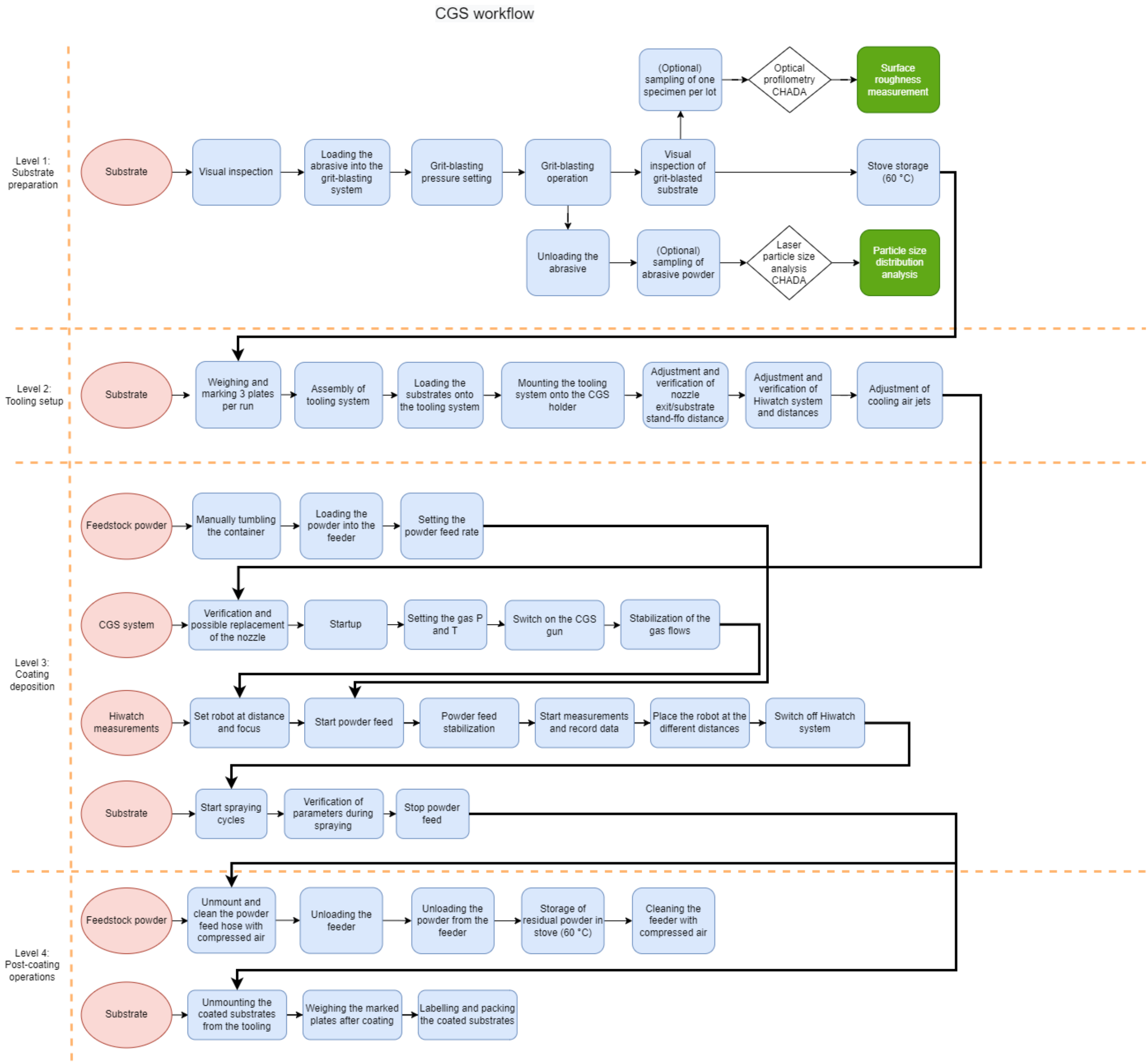

Figure 18. Summary of the CGS spraying workflow

4 High Velocity Air-Fuel (HVAF) workflow

4.1 Substrates and substrate preparation procedure

4.1.1 Substrate material and geometry

The substrate material selected for the High Velocity Air Fuel (HVAF) deposition is AISI 304 stainless steel, but due to supply issues, the first deposition trials have been conducted on S500 substrates. The deposition specimens are rectangular plates with dimensions of 60 mm x 25 mm x 3 mm. These specimens are lightweight, facilitating accurate measurement of deposition efficiency and practical handling with laboratory-scale metallographic preparation equipment, thus lowering the possible artefacts in the cutting procedures.

The substrates are commercially sourced from industrial suppliers. They feature slightly chamfered edges and are free from any oil or grease.

Each specimen is marked on the back with a unique number using a numbered punch, then cleaned with a cloth impregnated with isopropanol to remove any dirt deposited upon during transport.

The weight of each specimen is measured using a balance with an accuracy of ±0.1g. The operation is performed before the blasting procedure because the amount of material removed during sandblasting is negligible compared to the scale's sensitivity, rendering post-blasting weight measurements unnecessary.

The specimens are then mounted on the same tool used for the spraying procedure, as the blasting procedure is performed by the same HVAF system. To avoid contamination of the specimens, the operator must wear clean gloves, as any contamination may hinder the proper adhesion of the sprayed coating.

4.1.2 Sample holder design

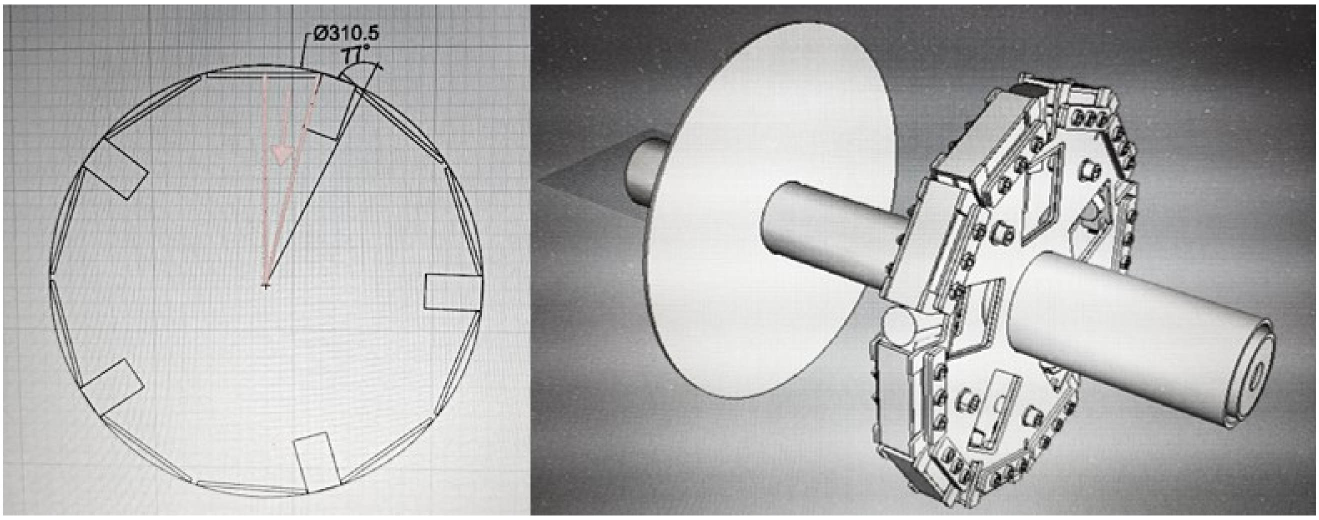

Figure 19 Sample holder design: (left) cross section detail evidencing the angle variation, (b) full configuration of the sample holder, carrying 10 plate substrates and five cylinders

The sample holder designed for the HVAF process features (Figure 19) all the specimens in the same radial section. It is designed to maximise the number of specimens and minimise the waste of powder between them.

The cons are related to the small deviation from orthogonality that the specimens encounter at its extremities.

The sample holder can accommodate also other coupon shapes and cylindrical shapes.

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4.1.3 Tooling and spray system setup

The spraying pattern is controlled prior to the spraying operations, including the blasting, which is performed by the same HVAF system (Figure 20). All the parameters needed for the program toolpath and gun configuration are controlled in the KUKA PLC interface. The spraying spot is monitored using a laser pointer installed inside the torch nozzle, which shares the same internal shape as the nozzle itself, thereby aligning the gun with the nozzle axis. Thanks to this, it is possible to have an accurate control of the path followed by the torch during the deposition procedure. A detailed explanation of the program guiding the torch in its deposition path is described in the paragraph 4.2.2.

The standoff, which is the distance between the torch nozzle extremity and the substrate, is a variable controlled in the KUKA settings. The torch position is then controlled using a measuring tape to a precision of ±1 mm.

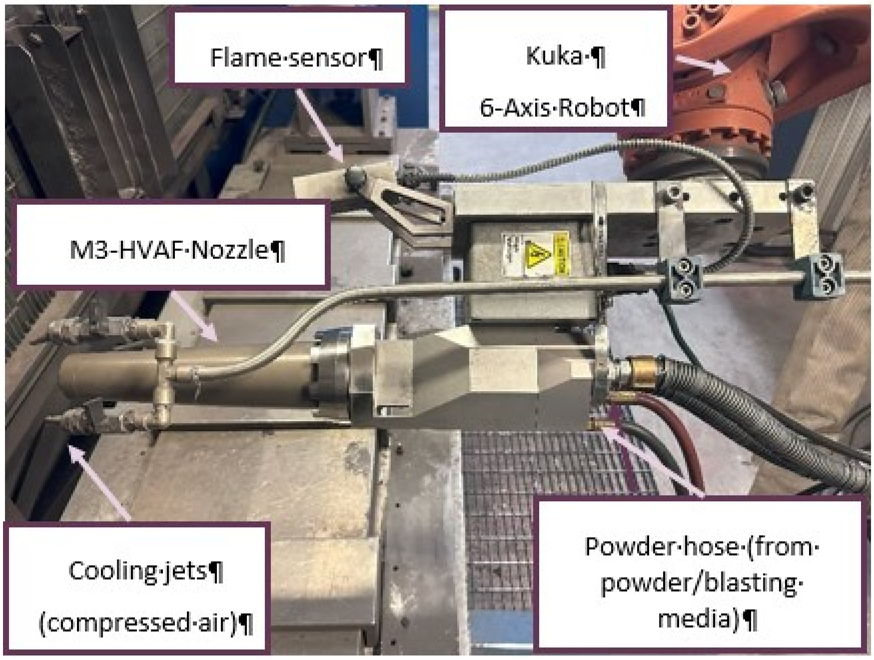

Figure 20. HVAF thermal spray system

4.1.4 Grit-blasting

The HVAF system has been developed to be able to blast in a unique solution by changing the hose connected to the V4 feeder ensuring the repeatability and consistency of the surface preparation. The spraying gun is equipped onto the KUKA 6-axis robot which is a versatile and precise industrial robot with six degrees of freedom, enabling complex and flexible movements.

The particle impact angle is maintained perpendicular to the surface. The robot keeps a constant distance of 300 mm from the substrate. The pattern is repeated 3 times: this has been verified as sufficient considering the substrate's hardness and characteristics.

The blasting media utilized in our system is industrial white corundum sand, conforming to FEPA grade F120, with a particle size range of 63-106 µm (Figure 21). The particle size distribution was determined using a laser-scattering particle size analyzer (Mastersizer 3000, Malvern Panalytical, Malvern, UK) by the dry-dispersion method, yielding a mean particle size of approximately 125 µm. The subsequent graph illustrates the particle size distribution of the blasting media.

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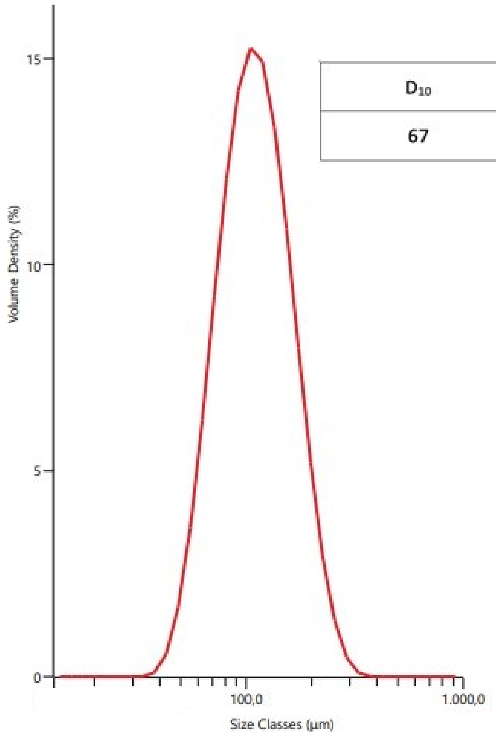

D_{10}	D_{50}	D_{90}
67	109	179

Figure 21. Particle size distribution of the blasting media

The nozzle used for blasting is consistent across all substrate materials, specifically the 4L4 nozzle, which imparts the highest kinetic energy to the in-flight particles to enhance the surface erosion of the substrate. The flame temperature is adjusted to the "high" setting (Table 1) as the substrate material does not oxidize quickly.

Table 1. “High” temperature setting

Air (Psi)	Fuel1 (Psi)	Fuel2 (Psi)	Carrier (L/min)
108	96	70	40

The powder flow is regulated by setting a percentage of the maximum angular velocity of the rotor inside the feeder, fixed at 30% for flat specimens of typical Fe-based material. The Volumetric Feeder V4 (Figure 22) minimizes friction between mechanical components delivering consistent powder flow and enabling the feeder to reliably handle agglomerating and bridging powders.

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Figure 22. V4 Pressurized hopper volumetric powder feed system

Three only blasted specimens have been sent to the University of Modena and Reggio Emilia for surface roughness evaluation following the blasting procedure. The obtained surface morphology is characterized by Sa ~ 6 µm, Sz ~ 76 µm, Sal ~ 43 µm, with a slightly lower autocorrelation length (Sal) than for the HVOF samples (see Section 2.1.2) which is consistent with the lower expected flattening ratio of HVAF sprayed particles because of their lower impact temperature, and therefore still well suited to provide good mechanical interlocking to this type of coating.

After the blasting procedure, the specimens are allowed to cool down to prevent excessive oxide growth on the surface during the blowing of compressed air to partially remove any residual blasting media that may be still attached to the surface. However, some blasting media will always remain due to the high kinetic energy imparted by the supersonic jet stream of the HVAF system.

4.2 Coating deposition procedure

4.2.1 Filling of powder feeder

The powder feeder (G4, Uniquecoat Technologies, Valpark Drive, Oilville, USA - Figure 23) is designed to deliver precise and consistent powder flow. This system utilizes a carrier gas, Argon, which passes through the bed of powder particles, fluidizing them to facilitate smooth delivery.

The regulation of the powder flow is achieved by adjusting the rotational speed of a rotor blade located inside the hopper. As the rotor blade spins, it influences the rate at which the powder particles are fed into the carrier gas stream. By varying the speed of the rotor, the system can control the amount of powder dispensed, ensuring a steady and accurate flow rate tailored to the specific requirements of the process.

Between spraying runs, the feedstock powder**s** are stored in a vacuum-sealed bag to ensure optimal preservation and quality. This storage method protects the powder from moisture, air, and contaminants, preventing clumping and degradation. Upon use, the vacuum-sealed bag is opened, and the powder is transferred into the feeder system, maintaining the material's integrity and ensuring consistent performance during the feeding process. To avoid any storage induced segregation each load of powder is evenly mixed to reach a homogeneous particle size distribution. The hopper should typically be loaded with at least 1 kg of powder to avoid flow instabilities, although some exceptions are possible, especially with high-density raw material.

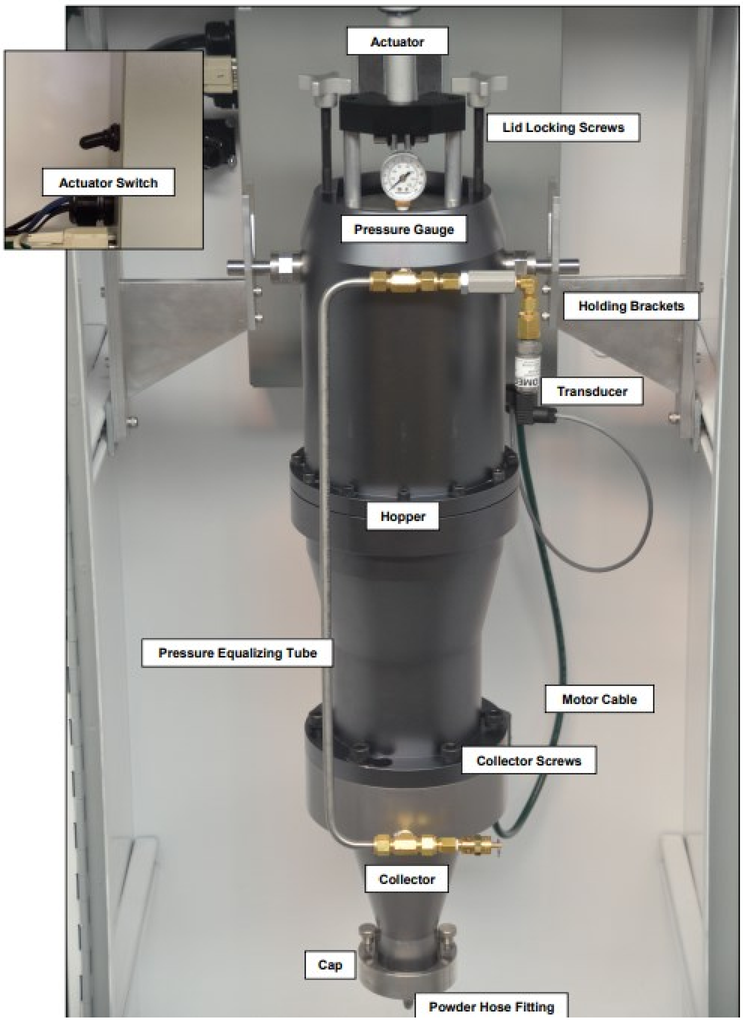

Figure 23. G4 (Uniquecoat Technologies) powder feeder

4.2.2 Spray operation

Torch/substrate kinematics settings

The robot (Figure 24) pattern during the deposition process is defined within a specific trajectory control program. For simple geometries, such as cylindrical shapes, the spraying coordinates are defined so that the robot moves in a straight line from the starting point to the endpoint of the object. The pattern outside the object is defined by a series of splines that return to the initial starting point of the deposition.

To achieve a predetermined velocity for the outer surface of the object, which corresponds to the transverse impact speed of the particles, it is necessary to set the rotating velocity of the mandrel accordingly. The speed of the torch over the object is determined by the relationships between the variables stored in the program.

The following equation defines the radius per minute of the mandrel:

$$\text{RPM} = \frac{30 \ * \ Vs}{\pi \ * \ r}$$

- RPM = revolutions per minute of the mandrel [revolutions / minute]
- Vs = desired outer surface velocity [m/s]
- r = radius of the outer surface [m]

The following equation defines the advancement speed of the gun:

$$Vt = \frac{STEP * RPM}{60}$$

- Vt = advancement speed of the gun [m/s]
- STEP = PITCH = distance between two adjacent passes of the deposition pattern onto the object [m]
- RPM = revolutions per minute of the mandrel [r/min]

The previous equations are transcribed in the KRL (KUKA Robot Language), which is a proprietary language designed specifically for programming and controlling KUKA industrial robots (Figure 25). It allows users to define the robot’s movement. KRL is similar to other programming languages but includes specific functions and commands tailored for robotic applications, such as defining paths, controlling motion, and interacting with sensors and other peripherals.

Figure 24. The robotic arm with 6 axes where the TS gun is installed

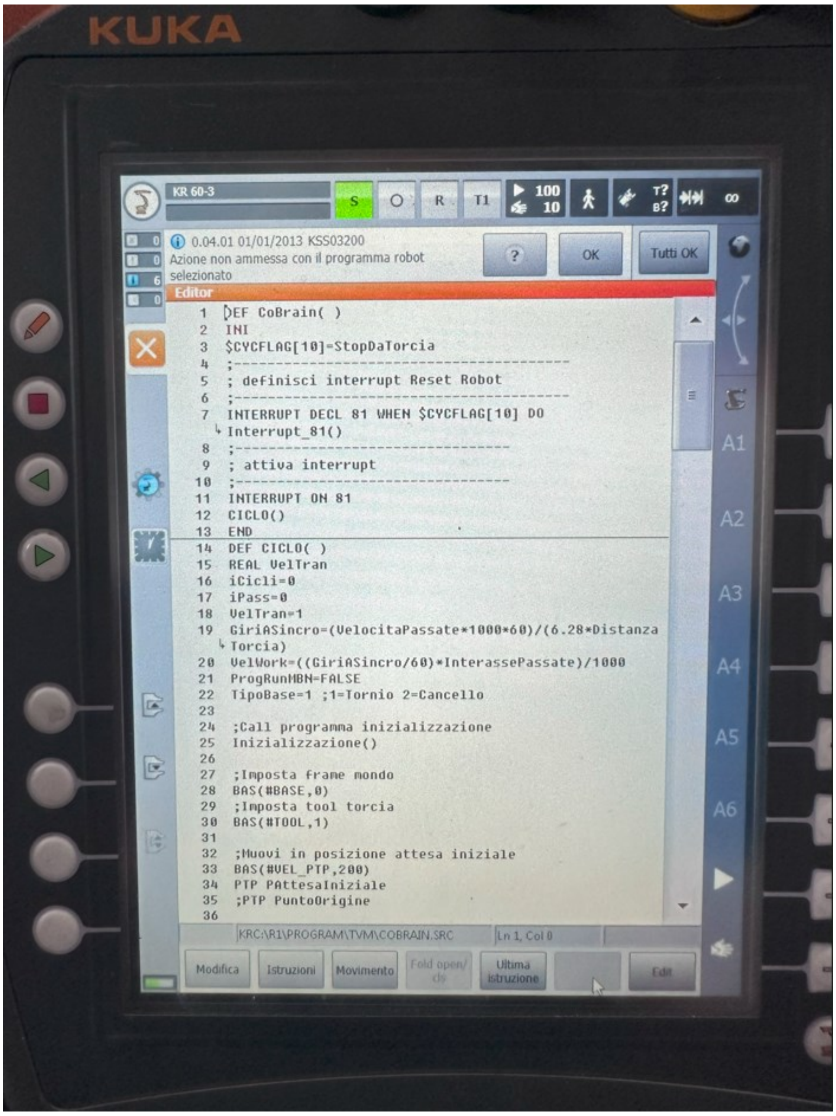

Figure 25. KUKA PLC interface – KRL program

Gas and powder feed settings, coating deposition

The initial operation required to start the HVAF (High Velocity Air Fuel) system before spraying involves enabling the aspiration within the spraying booth, which is a prerequisite for activating the vaporizer—responsible for atomizing the liquid propane fuel.

The parameters setting the thermal and kinetic energy of the spraying deposition are controlled by a totem directly linked to the Gas Distribution Cabinet, which is comprised of an electro-pneumatic pressure regulator, and solenoid valves that regulate the gas flow rates essential for system operation. The gases whose pressure is controlled include air, primary fuel, secondary fuel, and the carrier volumetric flow (in this case, Nitrogen) - Figure 26.

Figure 26. SYSTEM tab - graphical representation of the process parameters of the whole system

Thereafter, the robot is commanded to move to the initial spraying position (PL0), where a spark from a candle ignites the flame. Initially, gas flow rates are pre-set during the ramp-up cycle, to gradually reach the specified values selected in the interactive dials. The flame stabilizes after ten seconds, and after that, the values of the flame parameters can be adjusted to achieve a blue-clear jet stream. Once the powder flow stabilises, the operator initiates the deposition process by commanding the robot to start following the deposition pattern.

Upon completion of the deposition, the gun returns to its initial starting point (PL0), the flame is extinguished, and the vaporizer is powered off.

The operator monitors the deposition process by observing from the video terminal connected to the camera installed inside the spraying booth and, in case any issues arise, the operator quickly interrupts the process by pushing the emergency button.

4.3 Post-coating operations

The ventilation is kept running until most of the hazardous powder-dust aerosols are removed from the spraying booth, thereby minimizing hazardous aerosols within the chamber and reducing operator exposure.

When the ventilation is shut down, this is the condition that allows the doors to be opened from the outside and the operator can enter the spraying booth upon checking the temperature of the specimens and after letting them cool down to the right temperature to be dismantled from the tool with heat resistant gloves.

The weight of the three plates is measured and included in the HVAF logbook to calculate the deposition efficiency.

4.4 Logbook and data storage

The Excel file, referred to as the logbook (Figure 27), documents all the information about the spraying operation as outlined in the Data Management Plan.

The deposition efficiency for each individual specimen is determined by measuring the net weight of the powder deposited on the substrate. This value is then divided by the total amount of powder utilized during the actual deposition time for the specimen.

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Additional key process parameters are recorded, including the transverse speed of the gun, the stand-off distance, the spacing between consecutive passes on the specimen, and the number of deposition layers. Any issues encountered during the deposition process are documented by the operator in the logbook.

The main data included into the HVAF's and shared with the partners are: Date, Process ID, Substrate, Pre-treatment, Feedstock ID, Feedstock Description, N° of deposition cycle, N° layer per cycle, N° cooling cycles, N° of specimens, Pitch, Velocity, Stand-Off, Primary Nozzle Identifyer, Secondary Nozzle Identifyer, Air pressure, Fuel1 Pressure, Fuel2 Pressure.

Data Ctrl+d Totale: 1585 Reset View	Operatore	Progetto	Committente	Scopo	CAMPIONE Obj Nr	Substrato	POLVERE tipo	descrizione	C/S g
28-giu-2023	Rik/Marc	CoBrain	Alvise B.	Efficienza processo	**2306-020-01**	Piastrina ferro 25x3x126	AlCrFeMnNi	343-C-23 AlCrFeMnNi 15-53um	50,0
28-giu-2023	Rik/Marc	CoBrain	Alvise B.	Efficienza processo	**2306-020-02**	Piastrina ferro 25x3x126	AlCrFeMnNi	343-C-23 AlCrFeMnNi 15-53um	37,0
28-giu-2023	Rik/Marc	CoBrain	Alvise B.	Efficienza processo	**2306-020-03**	Piastrina ferro 25x3x126	AlCrFeMnNi	343-C-23 AlCrFeMnNi 15-53um	48,0
28-giu-2023	Rik/Marc	CoBrain	Alvise B.	Efficienza processo	**2306-020-04**	Piastrina ferro 25x3x126	AlCrFeMnNi	343-C-23 AlCrFeMnNi 15-53um	44,0
28-giu-2023	Rik/Marc	CoBrain	Alvise B.	Efficienza processo	**2306-020-05**	Piastrina ferro 25x3x126	AlCrFeMnNi	343-C-23 AlCrFeMnNi 15-53um	16,0
29-giu-2023	Rik/Marc	CoBrain	Alvise B.	Efficienza processo	**2306-021-01**	Piastrina ferro 25x3x126	CANTOR TiC	396-D-23 CANTOR TiC (2023) 10-45um	47,0
29-giu-2023	Rik/Marc	CoBrain	Alvise B.	Efficienza processo	**2306-021-02**	Piastrina ferro 25x3x126	CANTOR TiC	396-D-23 CANTOR TiC (2023) 10-45um	34,0
29-giu-2023	Rik/Marc	CoBrain	Alvise B.	Efficienza processo	**2306-021-03**	Piastrina ferro 25x3x126	CANTOR TiC	396-D-23 CANTOR TiC (2023) 10-45um	29,0
29-giu-2023	Rik/Marc	CoBrain	Alvise B.	Efficienza processo	**2306-021-04**	Piastrina ferro 25x3x126	CANTOR TiC	396-D-23 CANTOR TiC (2023) 10-45um	30,0
29-giu-2023	Rik/Marc	CoBrain	Alvise B.	Efficienza processo	**2306-021-05**	Piastrina ferro 25x3x126	CANTOR TiC	396-D-23 CANTOR TiC (2023) 10-45um	14,0
13-dic-2023	Rik	CoBrain	Enrico F.	Efficienza processo	**2312-010-01**	Piastrina ferro 25x3x126	WCrY	1329-N-23 WCrY 10-53um	133,0
13-dic-2023	Rik	CoBrain	Enrico F.	Efficienza processo	**2312-010-02**	Piastrina ferro 25x3x126	WCrY	1329-N-23 WCrY 10-53um	116,0
13-dic-2023	Rik	CoBrain	Enrico F.	Efficienza processo	**2312-010-03**	Piastrina ferro 25x3x126	WCrY	1329-N-23 WCrY 10-53um	75,0
26-feb-2024	Rik/Marc	COBRAIN	Enrico F.	Efficienza processo	**2402-008-01**	Piastrina ferro 25x3x126 (ferro in casa)	TiC-AlCrFeMnNi	Al14(Cr20Fe40Mn25Ni15)86 at % - TiC60v% -45+15um 1084-I-23	79,0
26-feb-2024	Rik/Marc	COBRAIN	Enrico F.	Efficienza processo	**2402-008-02**	Piastrina ferro 25x3x126 (ferro in casa)	TiC-AlCrFeMnNi	Al14(Cr20Fe40Mn25Ni15)86 at % - TiC60v% -45+15um 1084-I-23	42,0
26-feb-2024	Rik/Marc	COBRAIN	Enrico F.	Efficienza processo	**2402-008-03**	Piastrina ferro 25x3x126 (ferro in casa)	TiC-AlCrFeMnNi	Al14(Cr20Fe40Mn25Ni15)86 at % - TiC60v% -45+15um 1084-I-23	39,0
26-feb-2024	Rik/Marc	COBRAIN	Enrico F.	Efficienza processo	**2402-008-04**	Piastrina ferro 25x3x126 (ferro in casa)	TiC-AlCrFeMnNi	Al14(Cr20Fe40Mn25Ni15)86 at % - TiC60v% -45+15um 1084-I-23	36,0
27-feb-2024	Rik	COBRAIN	Enrico F.	Efficienza processo	**2402-008-05**	Piastrina ferro 25x3x126 (ferro in casa)	TiC-AlCrFeMnNi	Al14(Cr20Fe40Mn25Ni15)86 at % - TiC60v% -45+15um 1084-I-23	21,0
27-feb-2024	Rik	COBRAIN	Enrico F.	Efficienza processo	**2402-008-06**	Piastrina ferro 25x3x126 (ferro in casa)	TiC-AlCrFeMnNi	Al14(Cr20Fe40Mn25Ni15)86 at % - TiC60v% -45+15um 1084-I-23	24,0

Obj Nr	ROBOT Programma	N cicli dep Nr	N Layers per ciclo Nr	N cicli raff Nr	N pezzi Nr	Pitch mm	V m/s	Stand Off mm	GUN eder INJECTO	Nozzle Prim.	Sec.	Air PSI	Fuel 1 PSI	Fuel 2 PSI	Carrier L/min	Gun Time sec
2306-020-01	pias_test_fixpitch	4	1	1	1	3,00	1,00	300	3	**A**	**4L0**	109	97	110	80	84
2306-020-02	pias_test_fixpitch	4	1	1	1	3,00	1,00	300	3	**A**	**4L0**	109	97	110	55	53
2306-020-03	pias_test_fixpitch	4	1	1	1	3,00	1,00	300	3	**A**	**4L0**	107	104	87	80	73
2306-020-04	pias_test_fixpitch	4	1	1	1	3,00	1,00	300	3	**A**	**4L0**	107	104	87	55	40
2306-020-05	pias_test_fixpitch	4	1	1	1	3,00	1,00	300	3	**A**	**4L0**	108	96	70	50	52
2306-021-01	pias_test_fixpitch	4	1	1	1	3,00	1,00	300	3	**A**	**4L0**	109	95	110	80	185
2306-021-02	pias_test_fixpitch	4	1	1	1	3,00	1,00	300	3	**A**	**4L0**	109	95	110	55	175
2306-021-03	pias_test_fixpitch	4	1	1	1	3,00	1,00	300	3	**A**	**4L0**	107	104	87	80	103
2306-021-04	pias_test_fixpitch	4	1	1	1	3,00	1,00	300	3	**A**	**4L0**	107	104	87	55	56
2306-021-05	pias_test_fixpitch	4	1	1	1	3,00	1,00	300	3	**A**	**4L0**	108	103	70	50	64
2312-010-01	pias_test_fixpitch	4	1	0	1	3,00	1,00	300	3	**A**	**4L4**	108	97	104	65	65
2312-010-02	pias_test_fixpitch	4	1	0	1	3,00	0,70	300	3	**A**	**4L2**	108	96	100	60	59
2312-010-03	pias_test_fixpitch	4	1	0	1	3,00	1,00	300	3	**-**	**M2-3L2C**	108	97	0	65	60
2402-008-01	pias_test_fixpitch	5	1	1	1	3,00	1,00	300	10	**C**	**4L4C**	108	96	100	60	71
2402-008-02	pias_test_fixpitch	5	1	1	1	3,00	1,00	300	10	**C**	**4L2C**	108	96	100	60	65
2402-008-03	pias_test_fixpitch	5	1	1	1	3,00	1,00	300	3	**C**	**4L2C**	108	96	100	60	61
2402-008-04	pias_test_fixpitch	5	1	1	1	3,00	1,00	300	3	**C**	**4L4C**	108	96	100	60	60
2402-008-05	pias_test_fixpitch	5	1	1	1	3,00	1,00	200	3	**-**	**M2-3L2C**	85	78	0	60	60
2402-008-06	pias_test_fixpitch	6	1	1		3,00	1,00	300	3	**-**	**M2-3L2C**	110	100	0	20	66

Obj Nr	FEEDER Type	Speed %	Gas Ar/N2	Powder Time sec	Peso Polvere G4 Inizio g	Peso Polvere G4 Fine g	Note Processo
2306-020-01	G4	45	N2	45	870,0	820,0	L=110+90+110, offX=1270, offY=110, Raggiopz=0, StandOff=350
2306-020-02	G4	35	N2	37	820,0	783,0	L=110+90+110, offX=1270, offY=160, Raggiopz=0, StandOff=350
2306-020-03	G4	45	N2	35	783,0	735,0	L=110+90+110, offX=1270, offY=210, Raggiopz=0, StandOff=350
2306-020-04	G4	35	N2	38	735,0	691,0	L=110+90+110, offX=1270, offY=260, Raggiopz=0, StandOff=350
2306-020-05	G4	15	N2	33	691,0	675,0	L=110+90+110, offX=1270, offY=310, Raggiopz=0, StandOff=350
2306-021-01	G4	45	N2	39	680,0	633,0	L=110+90+110, offX=1270, offY=110, Raggiopz=0, StandOff=350
2306-021-02	G4	35	N2	40	633,0	599,0	L=110+90+110, offX=1270, offY=160, Raggiopz=0, StandOff=350
2306-021-03	G4	45	N2	35	614,0	585,0	L=110+90+110, offX=1270, offY=210, Raggiopz=0, StandOff=350
2306-021-04	G4	35	N2	36	526,0	496,0	L=110+90+110, offX=1270, offY=260, Raggiopz=0, StandOff=350
2306-021-05	G4	15	N2	33	496,0	482,0	L=110+90+110, offX=1270, offY=310, Raggiopz=0, StandOff=350
2312-010-01	G4	70	N2	35	1457,0	1324,0	L=110+90+110, offX=1270, offY=110, Raggiopz=0, StandOff=350. **Parametri come 2303-006-01**
2312-010-02	G4	60	N2	38	1324,0	1208,0	L=110+90+110, offX=1270, offY=160, Raggiopz=0, StandOff=350 **Parametri come 2312-001-02**
2312-010-03	G4	45	N2	34	1208,0	1133,0	L=110+90+110, offX=1270, offY=210, Raggiopz=0, StandOff=350. **Parametri come 1910-006-2**
2402-008-01	G4	60	N2	48	1161,0	1082,0	L=110+90+110, offX=1270, offY=110, Raggiopz=0, StandOff=350. **Parametri come 2312-001-02, ma ugello feeder N°10 (lungo). Vel. 1m/s**
2402-008-02	G4	35	N2	46	1082,0	1040,0	L=110+90+110, offX=1270, offY=160, Raggiopz=0, StandOff=350. **Parametri come 2312-001-02, ma ugello feeder N°10 (lungo). Vel. 1m/s**
2402-008-03	G4	35	N2	43	1040,0	1001,0	L=110+90+110, offX=1270, offY=210, Raggiopz=0, StandOff=350. **Parametri come 2312-001-02, ma ugello feeder N°3 (corto). Vel. 1m/s**
2402-008-04	G4	35	N2	42	1001,0	965,0	L=110+90+110, offX=1270, offY=260, Raggiopz=0, StandOff=350. **Parametri come 2312-001-02, ma ugello feeder N°3 (corto). Vel. 1m/s**
2402-008-05	G4	15	N2	45	969,0	948,0	L=110+90+110, offX=1270, offY=110, Raggiopz=0, StandOff=250. **Parametri come 2303-014-01**
2402-008-06	G4	15	N2	60	940,0	916,0	L=110+90+110, offX=1270, offY=160, Raggiopz=0, StandOff=350. **Parametri come 1711-026-1. Abbassato il carrier a 20l/min, si nota una fiamma più stabile e con uno spot meglio definito. Fatto 1 ciclo in più (6 al posto di 5)**

Obj Nr	ANALISI Codice Labo	Dose normal. s/cm²	Powder flow g/s	Dep. Area cm²	peso coating g	spessore lordo µm	spessore x layer. µm	DE peso %	DE spessore %	DE overall %
2306-020-01	830-F-23	0,13	1,11	22,5	2,20		0,0	66,0%	0,0%	4,4%
2306-020-02	831-F-23	0,13	1,00	22,5	1,70		0,0	56,7%	0,0%	4,6%
2306-020-03	832-F-23	0,13	1,37	22,5	2,10		0,0	51,0%	0,0%	4,4%
2306-020-04	833-F-23	0,13	1,16	22,5	1,60		0,0	46,1%	0,0%	3,6%
2306-020-05	834-F-23	0,13	0,48	22,5	0,70		0,0	48,1%	0,0%	4,4%
2306-021-01	836-G-23	0,13	1,21	22,5	2,00		0,0	55,3%	0,0%	4,3%
2306-021-02	837-G-23	0,13	0,85	22,5	1,60		0,0	62,7%	0,0%	4,7%
2306-021-03	838-G-23	0,13	0,83	22,5	1,70		0,0	68,4%	0,0%	5,9%
2306-021-04	839-G-23	0,13	0,83	22,5	1,80		0,0	72,0%	0,0%	6,0%
2306-021-05	840-G-23	0,13	0,42	22,5	0,70		0,0	55,0%	0,0%	5,0%
2312-010-01		0,13	3,80	22,5	0,00		0,0	0,0%	0,0%	0,0%
2312-010-02		0,19	3,05	22,5	0,20		0,0	1,5%	0,0%	0,2%
2312-010-03		0,13	2,21	22,5	0,10		0,0	1,5%	0,0%	0,1%
2402-008-01		0,17	1,65	22,5	3,00		0,0	48,6%	0,0%	3,8%
2402-008-02		0,17	0,91	22,5	1,45		0,0	42,3%	0,0%	3,5%
2402-008-03		0,17	0,91	22,5	1,48		0,0	43,5%	0,0%	3,8%
2402-008-04		0,17	0,86	22,5	1,22		0,0	38,0%	0,0%	3,4%
2402-008-05		0,17	0,47	22,5	0,50		0,0	28,6%	0,0%	2,4%
2402-008-06		0,20	0,40	22,5	0,10		0,0	5,6%	0,0%	0,4%

Figure 27. Extract of the HVAF's logbook

4.5 Process parameters selection

Initial Deposition Trials – Optimal Torch nozzle

Following a series of trials to identify the optimal secondary nozzle configuration (the 4L4C), which determines the ultimate heat and velocity to in-flight particles, additional tests were conducted to evaluate other parameter settings. The parameters values of the first combustion stage are not varied, because they

We determined that the most sensitive parameters for the deposition of Cermet coatings using the HVAF process are the Fuel2 pressure and the powder feed rate. The combination of these two parameters determines the matrix of specimens used to select the conditions for producing samples for the experimental campaign.

All other key process parameters were kept constant for all deposition runs.

The **standoff** the substrate was set to achieve the best deposition efficiency, given by the focusing of the spraying spot;

The torch traverse **speed** was maintained at the standard value, as the specimen dimensions are too short to significantly influence the overall spraying deposition time and consequently, the heat transfer to the substrate.

The **pitch** was kept constant at 3 mm, the combination of relatively big spot size and small area to be covered made this parameter not critical in determining the coating quality

The pressures of **Air and Fuel**1 determine the heat and power generated within the combustion chamber, and to ensure a complete combustion the Air/fuel1 ratio is maintained at 1.2. If the first stage establishes the initial combustion and overall power level, it is the secondary stage, controlled by Fuel2 settings, that fine-tunes the combustion process determining the optimal performance. This latter has been therefore selected as parameters to tune in the experimental matrix, choosing between 80 and 100 PSI (fuel-reach and fuel-poor regimes).

HVAF **torch system** is designed to be highly versatile, allowing for the combination of different primary and secondary nozzles to tailor the spray characteristics to specific coating requirements. Multiple combinations of the primary and secondary nozzle have been tested, considering different materials (steel, ceramic), different dimensions (long, short) and different sizes. The final torch set-up has been selected by considering, among those tested, the most stable spray jets and those that less damaged the torch itself. Combining this with the information on coating density and deposition efficiency allowed us to reliably select the one designated "4L4C" nozzle, which is a 250-mm-long de Laval nozzle with an exit diameter of 22.5 mm coupled with a ceramic primary nozzle.

The chosen parameters conditions are here summarized:

Pitch	3 mm
Velocity	1 m/s
Stand-Off	300 mm
Primary Nozzle Identifyer	Ceramic
Secondary Nozzle Identifyer	4L4C
Air pressure	108 PSI
Fuel1 Pressure	96 PSI
Fuel2 Pressure	[80] or [100] PSI
Powder Feed rate	[40] or [60] rotor speed %

4.6 Workflow summary

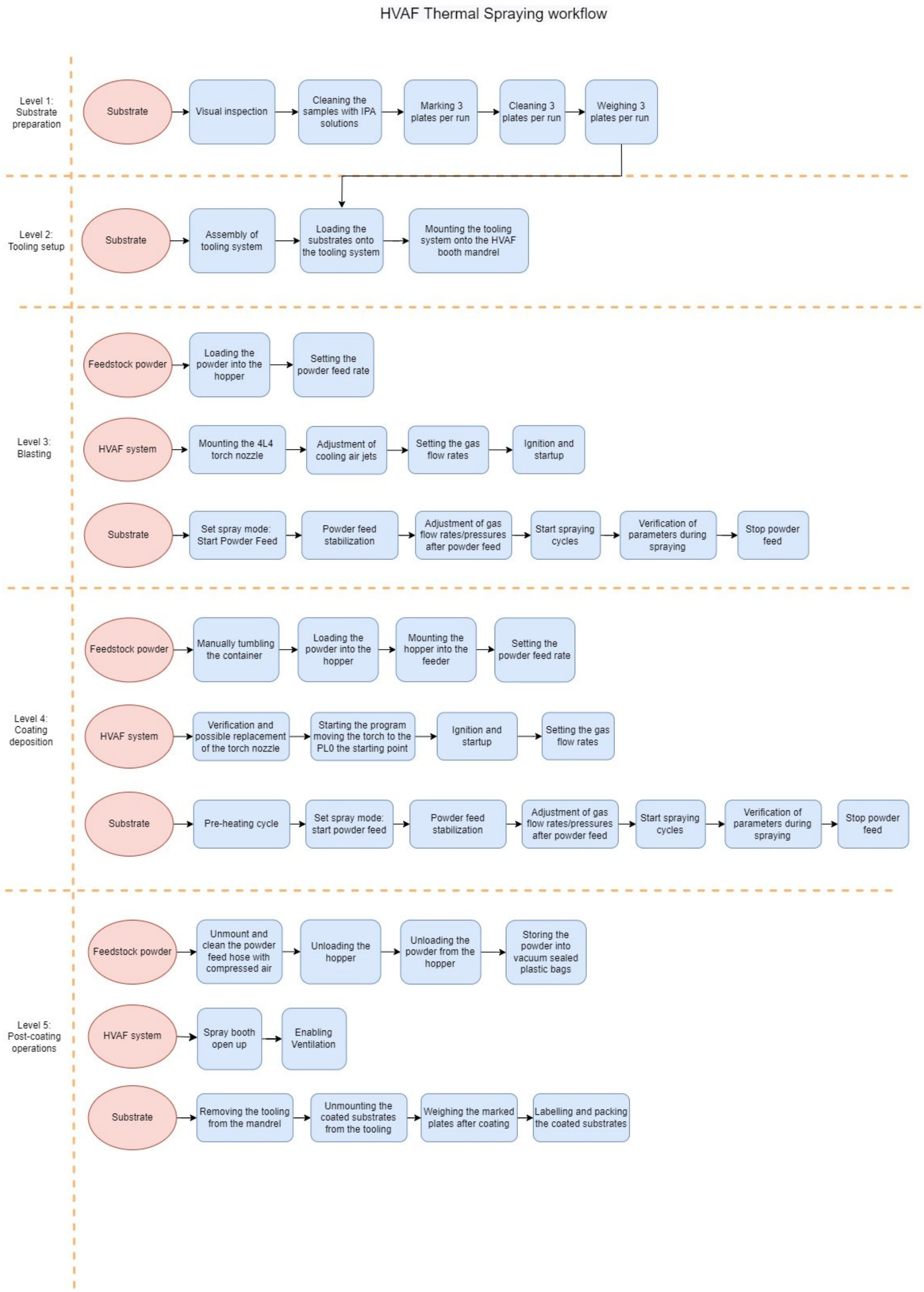

Figure 28. Summary of the HVAF spraying workflow

5 Conclusions

Based on the experience developed during the first 18 months of the CoBRAIN project, this deliverable described the workflows that will be implemented during WP4 for the high-throughput production of coatings that will be employed for the validation and refinement of the Artificial Intelligence models.

This deliverable provides an overview of the workflow as well as detailed descriptions and practical indications that need to be followed for the coatings' production, with a two-fold purpose:

- Ensuring an efficient and smooth production process that results in high productivity and cost-effectiveness in the production of samples that are as representative of an industrial process as is possible at an early TRL level.
- Ensuring that the process is fully repeatable by providing detailed, step-by-step instructions that cover all practical operations. These could also enable future third-party verifications of reproducibility by giving the necessary information to replicate the experiments using the same equipment and materials.

This deliverable should be seen in combination with D3.1, which provides an equally detailed workflow for the production of the feedstock materials.

The importance of having detailed workflows is underlined by some of the experiments described in this deliverable, which confirm that, as is known in the field of thermal spraying, there are many "hidden parameters" to be accounted for. For example, it was shown that the choice of torch/substrate kinematics (in terms of number of cycles, pass velocity, pass spacing, etc.) and/or even the sheer choice of sample holder impacts the deposition temperature more significantly than do changes to "explicit" parameters like gas flow and powder feed rates.

6 Attainment of Objectives

Table 2. Contribution of D3.2 toward the attainment of the general objectives of CoBRAIN

Ref.[3]	General Objective	Status
O1.1	Qualified powders for Thermal Spraying. HHM powders suitable for thermal spraying by CGS, HVOF and HVAF thermal spray, having a deposition efficiency above 50%, good adhesion and no coating porosity	The procedures outlined here allow to measure the deposition efficiency and provide coatings suitable for verifying adhesion strength and porosity.
O1.2	High Performing coatings for Thermal Spray. HHM coatings with high toughness and capable to displaying hardness values of 800-900 HV and sliding wear rates $<10^{-6}$ $mm^3/(N{\cdot}m)$, combined with corrosion resistance in acidic and alkaline environments	The procedures outlined in D3.2 allow repeatable production of coatings with high quality. Thus, following these procedures, (1) the resulting coatings have high likelihood to offer the best performances that the employed materials can yield, (2) the results are reliable and repeatable. This will ensure confidence in the characterization results that will validate the attainment of this objective.
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 procedures outlined here are the basis to generate the experimental dataset. The coatings produced according to these procedures have already been used to generate a wealth of data that is forming the initial part of the dataset.

[3] Reference to the general project objectives stated from page 4 of Part B of the Annex 1 – Description of Action

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Table 3. Contribution of D3.2 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	This deliverable describes the detailed setup to be employed for the experimental campaign in WP4.
WP3.2	Validate the characterization workflow, the specimen amount and the consistency of the data	The detailed (hence, repeatable) procedures laid out in this deliverable are the basis upon which reliable characterization results can be obtained to validate the corresponding characterization workflows to be described in D3.3.
WP3.3	Evaluate material consumption	Figures concerning material consumption for the experiments are provided in this deliverable.
WP3.4	Identify criticalities before running the experimental part on the new materials from modelling	Criticalities such as the need to anneal the CGS feedstock, the importance of strict control over torch/substrate kinematics and the use of a defined sample holder have been identified and addressed to ensure appropriate and repeatable samples' production.
WP3.5	Provide reference data for the modelling optimization	Repeatable coating deposition procedures are the pre-condition to obtain reliable reference data from the subsequent characterization.
WP3.6	Include promising material identified in the literature in the final dataset	The experiments to optimize the workflow have been carried out using literature materials. The results of their characterization will be part of the final dataset.

[4] Reference to the specific objectives of the workpackage as listed in Part A of Annex 2 – Description of Action

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