CRITT

Centre Régional d'Innovation et de Transfert de Technologie

INNOVATION FOR YOUR DEVELOPMENT

Guide services

MATERIAL

COATING

AND SURUFACE

TREATMENT

www.critt-mdts.com

FOREWORD

CRITT-MDTS, Regional Center of Innovation and Transfer of Technology specializing in Materials, Coating and Surface Treatment, is a research Center and a laboratory of testing and analysis on raw materials and on finished products. We work for all sectors using or transforming materials, especially metal. Our main customers work in the biomedical, aeronautics, automotive, rail and luxury sectors.

This booklet is a guide for you, so that you can have a list of the general tests and controls our company is able to realise. Another specific booklet is dedicated for the tests on biomedical parts.

The tests can be carried out in compliance with current standards (NF, ISO, ASTM,…), your own specifications, your procedure or following an established custom procedure defined together according to your needs.

CRITT-MDTS has a quality, security and environment management system, allowing to perfectly answer the customers requirements.

AFAQ certification—ISO 9001 and OHSAS 18001
COFRAC accreditation according to ISO/CEI 17025
NADCAP accreditation
Endorsed by **major customers** in the aeronautics and automotive industries

CRITT-MDTS is a member of the « Institut Carnot MICA » (label of excellence for centers of research), and certified CRT (Technological Resources Centre) by the Ministry of Research.

An up-to-date list of all our approvals, accreditations and certifications is available on our website: www.critt-mdts.com

Accredited Nadcap™ Materials Testing Laboratory

cofrac ESSAIS
Accréditations N°1-0097 & N°1-1648
Portées disponibles sur www.cofrac.fr

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centre de ressources technologiques CRT

INSTITUT CARNOT MICA Materials Institute Carnot Alsace

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PART I : RECEPTION CONTROL ON METALLIC MATERIALS

bjective: Validate the conformity of the raw material in terms of microstructure, mechanical properties, and chemical composition, ...

METALLOGRAPHIC CONTROLS

Metallographic controls can be performed on all types of metals or alloys based in titanium, aluminium, stainless, nickel, copper, etc., according to standards or the customers specifications.

Controls

Microstructure control

Exam on a metallographic cut by optical microscope in order to :

- determine the type of structure
- determine the grain size (ISO 643 / ASTM E 112)
- Identify the phases (as delta ferrite (ASTM E 407), α-case, carbides ...)
- determine the inclusion rate (ISO 4967 / ASTM E 45),

Hardness measuremenrts

Brinell HB (ISO 6506-1), Vickers HV (ISO 6507-1), Rockwell HRB/HRC (ISO 6508-1), micro-hardness, determining the depth of decarburization (ISO 3887), hardness affiliations ...

Resistance to intergranular corrosion

Determination of the resistance to intergranular corrosion of stainless steels in an environment containing:

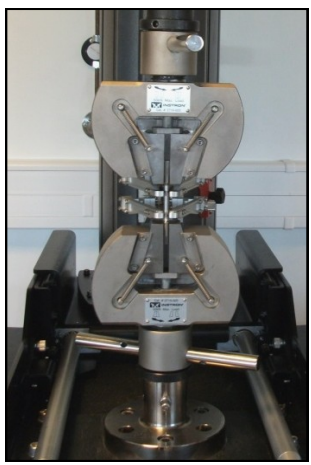
- Nitric acid: NF EN ISO 3651-1 / ASTM A 262 Practice C
- Sulfuric acid: NF EN ISO 3651-2 / ASTM A 262 Practice E

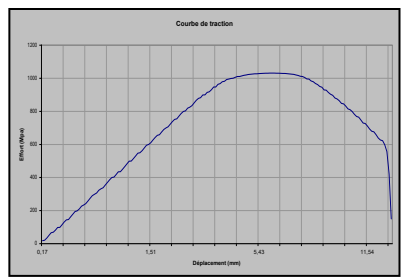
MECHANICAL PROPERTIES CONTROLS

Testing	Description
Tensile test	To determine the mechanical characteristics (maximum strength, yield strength, elongation, ...) or : - At ambient temperature (ASTM E 8 / ISO 6892-1) - At hot temperature until 800°C max (ASTM E 21 / ISO 6892-2)
Flexion par choc ISO 148-1	To determine the energy required to break in one time a metal specimen previously notched, soit : - between ambiant temperature and -60°C - at -196°C

Zoom on tensile test:

Example of a tensile test carried out on a cylindrical and standardized metallic specimen using an automatic extensometer.





ANALYSIS OF CHEMICAL COMPOSITION

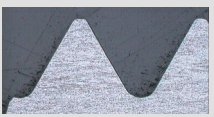
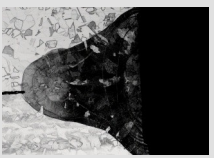
Analysis	Analysis equipments
Identification of present elements and verification of their contents	- Optical emission spectrometry (ICP-OES) - Sparks emission spectrometry - X-Ray fluorescence spectrometry
Measurement of gas content	Elemental analysers : - Hydrogen - Carbon/Sulfur - Oxygen / Nitrogen

PART II : CHARACTERIZATION OF METALLIC MATERIALS AND FINISHED PRODUCTS

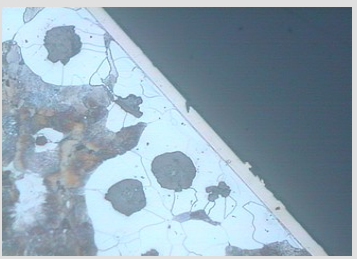
Objective : CRITT-MDTS has a full range of analytical means enabling to choose the technique the most appropriate depending on the aim researched.

METALLOGRAPHIC EXAMINATION

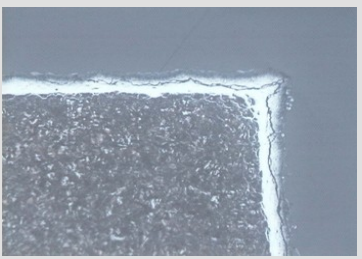
In addition of metallographic controls described on page 5, here are other possible Metallographic controls :

Controls on optical microscope	Micrographs
Thread quality Verification of screw morphology , presence of burrs, micro cracks in the thread root.	
Welding quality Verification of welds characteristics (shape, radius of connection, penetration depth,...) and of defects (porosity, cracking, blowholes...)	
Thickness measurement after treatment Nitriding, carbuurizing, galvanized, weight,...	See example below

Zoom on examples of thickness measurement after treatment :



Nickel deposit on a cast
G x 100
Thickness of deposit : 16 µm



Nitriding on 42CD4
G x 200
Nitriding of the layer : 12 µm

ANALYSIS OF CHEMICAL COMPOSITION

Analysis equipments

Optical emission spectrometer (ICP-OES)

Measurement of elements present in a solution obtained after completely dissolution by chemical attack of a sample which can be initially solid or powder form.

X–Ray diffraction (DRX)
(2 devices)

- Analysis on solid, powder, coating sample
- Identification of present crystalline phases and semi-quantification
- Estimation of crystallinity,...

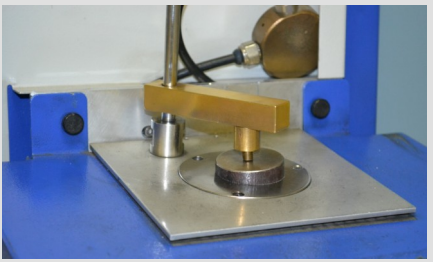
ANALYSIS OF CHEMICAL COMPOSITION

Method of analysis	
X-Ray fluorescence spectrometer	
Identification of elements present on a solid sample.	

Elementals Analysers - **Hydrogen** - **Carbon / Sulfur** - **Oxygen / Nitrogen**	*Analyser Carbone / Sulfur* 
Measurement of the amount of elements (H, C, N, O and S) directly on solid or powder samples.	

ANALYSIS OF CHEMICAL COMPOSITION

Method of analysis

Spark emission spectrometer	

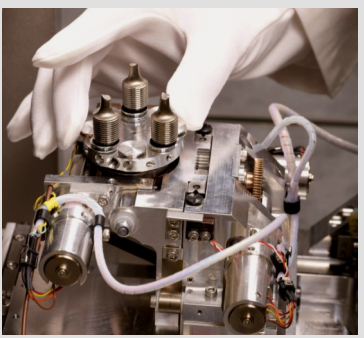
Identification of elements present on a massive sample

SURFACE ANALYSIS

Objective : CRITT-MDTS has also micro-analysis means allowing to characterize the surface of materials in order to :

- Determine the elements present on the surface
- Research the causes of any damage
- Provide solutions to specific problems

Analysis equipments

Scanning electronic microscope (SEM) coupled to an EDX probe	

- Identification and location of the present elements
- Topographic distribution of elements
- Research and localization of impurities or pollutants (deposits, solvents, residues,…)
- Observation of the surface condition, of the porosity, of the fracture surface,…

SURFACE ANALYSIS

Method of analysis

NEW

Glow Discharge - Optical Emission Spectrometry (GDOES)

Chemical composition profile as a function of depth:
- Identification of the extreme surface elements (residues, surface pollution)
- Thickness measurement of thick coatings (paint, thermal spray), thin coating (PVD)
- Possible quantification if calibration available

Comparison of two techniques of surface analysis :

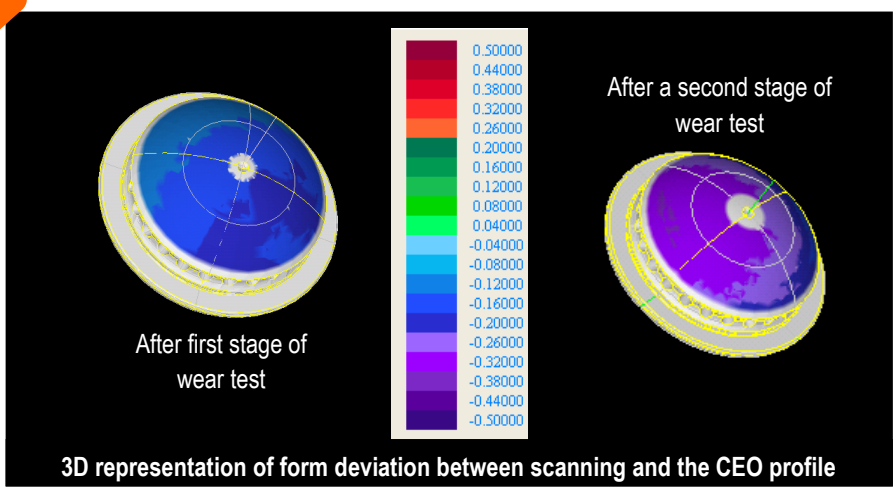
	SEM coupled to an EDS	GDEOS
Surface analyzed	From 1 µm² to 1 mm²	Circular spot of 4 mm diameter
Depth of analysis	From 1 to 1.5 µm	From 10 nm to 50 µm
Type of analysis	Semi-quantitative + cartography and photography	Semi-quantitative
Possible analysis of the elements	From Boron (Z≥5)	H, Li, B, C, N, O, F, Na, Mg, Al, Si, P, S, Cl, K, Ca, Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, Mo, Ag, Sn, Pb
Constraint	Constraints related to vacuum and SEM (dry sample, deposition of a layer of gold or carbon for non-conductive materials) Flat geometry	Flat geometry
Sample dimensions	Max. sample size (LxWxH) : 5 x 5 x 3 cm.	Minimum surface area: square 1x1cm² Maximum surface area: 30x30 cm²
Materials analysed	All types of materials	Metals, oxides, ceramics, polymers

To perform an analysis over a depth of 5 to 10 nm and obtain the chemical bonds, the X-ray Photoelectron Spectrometer (XPS) is also available.

DIMENSIONAL CONTROL AND CALIBRATION

Measurements	Description
Three-dimensional control (cf. example below)	Measurement by 3D probing combined to a comparison with the CAO file (IGES)
Control of form deviations	Measurement of Sphericality, circularity, concentricity, straightness, perpendicularity,….
Roughness measurements	Characterization of planar or curvilinear surfaces according to the ISO 4288
Calibration of measuring equipment	- Screwdrivers and torque wrenches until 1000 N.m (in our laboratory) - Marbles *(on site)* - Columns gauges *(on site)* - Profile projectors *(on site)*

Zoom on a 3D probing :



3D representation of form deviation between scanning and the CEO profile

SURFACE TOPOGRAPHY

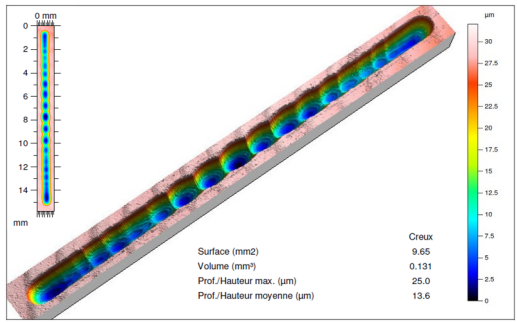
Altisurf 500

Surface measurements without contact

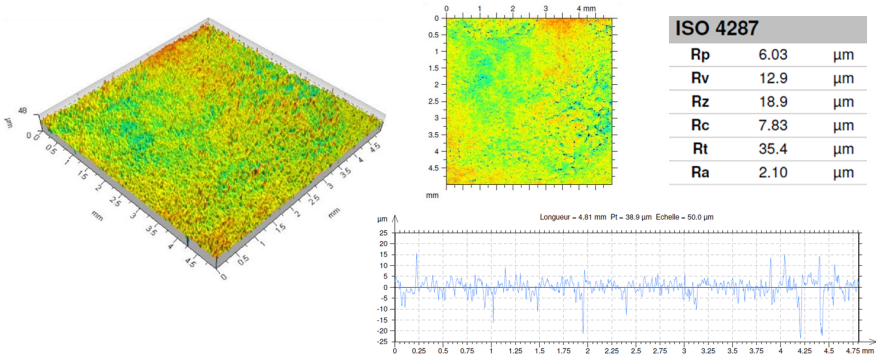
- 2D and 3D surface topography
- Linear and areal roughness measurement (ISO 4287 & ISO 25178)
- Depth and width measurement of a furrow
- Volume of groove
- Surface evolution

Zoom on examples of surface measurements without contact :

Example 1 : Wear control of a furrow after a tribometer test



Example 2 : Control of surface condition of a piece made by MIM



ISO 4287		
Rp	6.03	µm
Rv	12.9	µm
Rz	18.9	µm
Rc	7.83	µm
Rt	35.4	µm
Ra	2.10	µm

MECHANICAL TESTING

The following mechanical tests can be performed in accordance with standards or measurements according to your own specification or protocols.

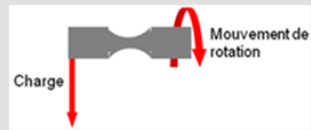
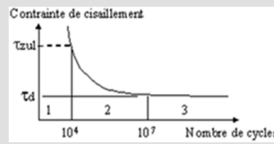
Testing	Description
Static and/or Dynamic	- Tensile - Compression - Torsion - shearing - Bending (on 1 point, 3 points or 4 points) - Impact bending - Creep or relaxation (at ambient temperature)

Our range of mechanical testing machines allows us to cover a wide working range.

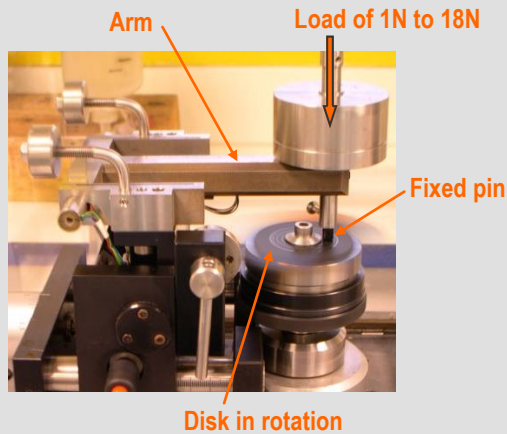
	Range capacity
Load	1,25 N to 240 kN max
Torque	0,1 N.m to 100 N.m max With the possibility to accomplished until ± 16 revolutions
Frequency	100 Hz max according to the displacement and force to applied

Do not hesitate to consult us for any specific testing.

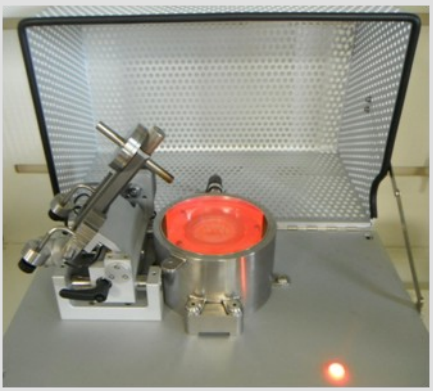
MECHANICAL TESTING

Testing	Description
Rotating bending **ISO 1143** on metallic materials Coated or not	*Rotating bending machine*  Max rotation speed = 4000 tr/min Max load = 30 N *Schematic diagram*  <u>Possible applications</u> - Application of a load and a rotational movement on a sample to determine its "fatigue limit" by drawing a Wohler curve such as :  - Biasing tensile and compression fiber - Connection fillets to target cracking zone

WEAR TESTING

Testing	Description
Pin-on-Disk testing On all the materials Coated or no	Principle : A sample (disk, plate) endures a rotary or alternating movement while being brought into contact with a stationary pin (or ball) on which a compressive axial load (1 to 18 N) is applied. The testing takes place in ambient air or lubricated environment. *Pin-on-disk apparatus* *(Set up following the standard ASTM G 99)*  - Measurement of friction of a pair of materials - Estimate the wear on simple configuration (surface condition, weight loss, the groove profiling…) - Application of a load between 1 N and 18 N - In ambient air or lubricated environment.

WEAR TESTING

Testing	Description
NEW **High temperature tribometer**	- Temperature up to 800°C - Load from 1 to 20N - Maximum speed 500 rpm - Continuous measurement of the friction coefficient - Rotary / alternative rotary mode - Possibility of fretting test  Allows to highlight specific processes of friction and wear at high temperatures, such as the impact of oxide coatings forming on the surface and their lubricating properties.

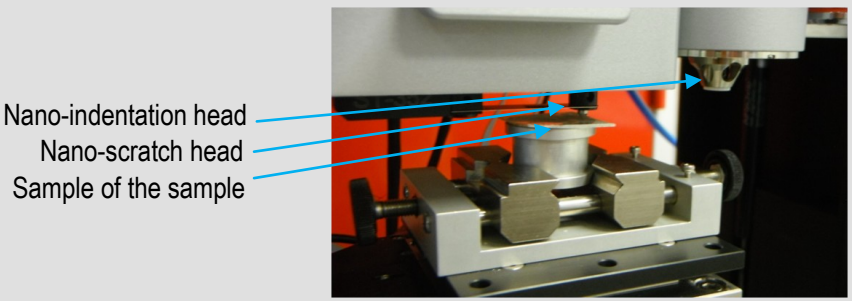
WEAR TEST

Testing	Description
Pin-on-Disk high loads testing On all the material (sample form)	*Tribometer pin-on-disc - 3 multidirectional stations* *High load multidirectionnelles*  - 3 simultaneous tests with measurement of each friction coefficient - In lubricated environment - Independent loads up to 500 N. The application of high loads in pin-on-disc test (described on the previous page) considers realistic contact pressures and the influence of the topography of sample surface. - Multidirectional kinematic (ex: triangular) due to linear movements along two directions (x, y), to solicit more realistic materials by adding shear stresses.

NANO-SCRATCH-INDENTATION PLATFORM

Description

For the characterization of hard and/or thin layers (typically less than 1 µm), and surface treatments, we have a platform of nano indentation, nano scratch test (according to the ISO 14577 and ISO 1518, respectively)
The force range for nano-indentation is from 0 to 500 mN with a Berkovich type diamond indenter.
The force range of the nano scratch is 0.08 - 5mN in addition to the micro-scratch (with 3 mN to 30 N). We have two types of scratch heads:
- Diamond spherical indenter of 2 and 5 µm radius
- Knife indenter 2 mm wide and a radius of 2µm allowing the analysis of convex surfaces



See opposite 2 examples, Nanoscratch test and Nanoindentation.

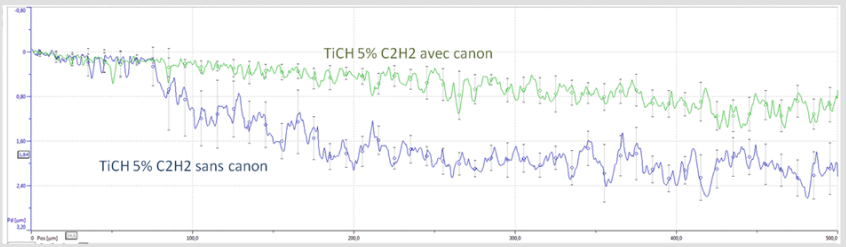
PLATFORM NANO-SCRATCH-INDENTATION

Examples of applications :

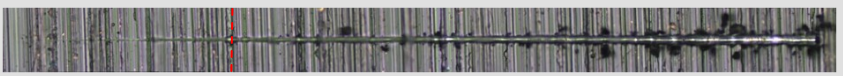
1 - Nanoscratch test :

Comparison to the nanoscratch test of the performance of a TiCH deposit with and without surface preparation prior to electron gun preparation

Evolution of the penetration depth of the indenter under load.
Mean of 3 measurements with standard deviation



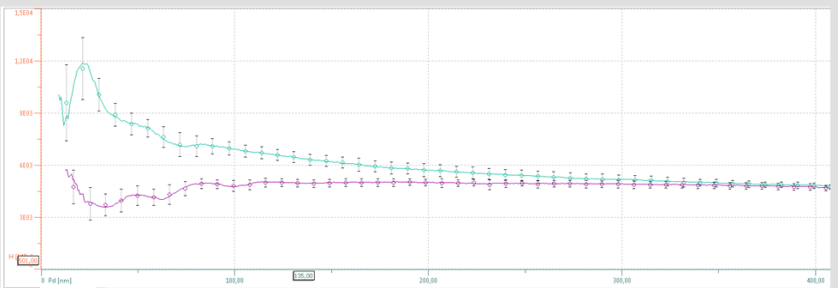
With barrel: first decohesion at 26mN



Without barrel: decohesion threshold at 16mN with massive flaking

2 - Nanoindentation :

Comparison by nanoindentation of the evolution of hardness as a function of indent depth under repeated stress (in sinus mode) of a sample with and without ion implantation :



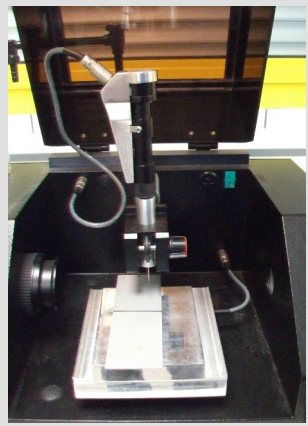
WETTABILITY TEST

Description

The study of the interactions between a solid and a known liquid is one method used to characterize the surface of solid and to predict its ability to establish links with the others materials.
The hanging drop method allows to characterize a liquid.

Measurement on all the materials with a plane surface :

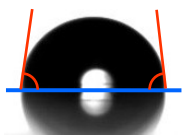
- The interfacial tension between 2 liquids
- Angle control
- Energy surface
- Superficial tension



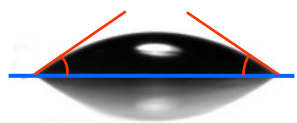
Wettability device

Zoom on the hanging drop method :

Example of measure of the angle of a drop formed in contact with a surface before and after treatment.



Before surface treatment of angle > 80°



After surface treatment angle < 40°

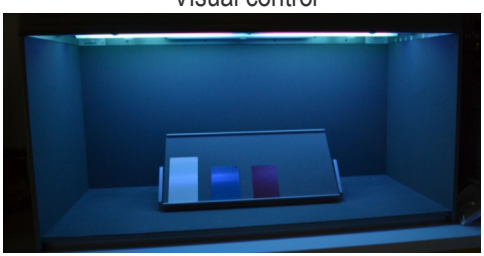
PART III : CHARACTERIZATION OF INDUSTRIAL COATINGS AND PAINTINGS

Objective : Validate the finished product before putting on the market or to compare the performances of different products.

ASPECT CONTROLS

Testing and standard	Description
Visual control **ISO 3668**	Visual comparison of colour of the paint compared to a standard and under different lights: appearance, tone, brightness. Different lights possible depending on the type of use black lights, fluorescent tube, incandescent, artificial daylight.
Measurement of colour **Former ISO 7724-2, not replaced.**	Determining the colour using a colorimeter by measuring the wavelengths emitted by the paint under a specific lighting.
Gloss measurement **ISO 2813**	Performed on a coating with a gloss which projects a light beam at a standard angle on the surface to measure the loss of intensity after reflection. Determination of specular reflection films of non-metallic paint at 20 °, 60 ° and 85 °.
Degradation **ISO 4628-1 to -6, -8 and –10**	Evaluation of degradation of the coating compared to the intensity of changes in appearance, quantity and size of defects, ...

Visual control



CHEMICAL AGENTS RESISTANCE TESTS

Standard reference	Description
Water immersion method **ISO 2812-2**	Evaluation of the action of water on the coating by partial or full immersion and assessment of the deterioration.
Method using an absorbent medium **ISO 2812-3**	Determination of the resistance of a coating with liquids or paste-like products using an absorbent environment.
Spotting methods **ISO 2812-4**	Determining of the resistance of a coating in contact with liquids or paste-like products in order to gauge the eventual damages.
Organic liquids **D27 1740 (RENAULT method)**	Method allowing to determinate the action of organic liquids *(Ex : brake fluid, cooling liquid, oil, fuels ,…)* on a paint coating. *Method realized partially (contact us)*
Customized testing	Immersion in liquids at a given temperature following the request, the ability of ventilated ovens, the type of coating, ...

GRAVEL TEST / BLASTING

Standard reference	Description
ISO 20567-1 **D24 1702 (RENAULT Method)** **D24 1312 (PSA Method)**	Resistance determination of a coating subject to the impact of one or more projectiles (stones, grit, gravel, ...) in order to quantify its alteration.

RESISTANCE TESTING AT THE HOLDING

Standard reference	Description
Bend test **ISO 1519**	Control of adhesion of a coating following the deformation of its substrate on a cylindrical mandrel with defined radius, in order to determine the resistance to cracking and peeling of the coating.
Stamping test **ISO 1520**	Slow and progressive deformation caused by the rise of a ball in contact with the paint sample in order to evaluate the resistance of a coating by measuring cracking or peeling.
Cross-cut test (control of adhesion) **ISO 2409**	This test first involves the development of a grid by incising the surface coating using a comb, then the realization of pullout testing with adhesive tape to estimate the force needed to lift the coating surface.
Choc resistance **ISO 6272-1 & -2**	Subject a coating to a rapid deformation caused by a falling weight with a spherical indenter of large area (ISO 6272-1) or small area (ISO 6272-2) in order to gauge the effects of a such deformation on the coating by the presence of cracking, delaminating, peeling, ...

HARDNESS MEASUREMENTS OF DEPOSITS AND COATINGS

Standard reference	Description
Buchholz Indentation test **ISO 2815**	Realisation of a stripe on a coating by a weighted wheel and the length of the indentation obtained allows to estimate the residual deformation of the coating.
Pencil hardness test **ISO 15184**	The pencil hardness test is to determine the film hardness of a paint by pushing pencils of known hardness on its surface.
Pendulum damping test **ISO 1522**	The pendulum damping test on a coating offer the possibility to determinate a comparative hardness value (for example) from number of oscillation measured during a given time.

ABRASION RESISTANCE TESTING (TABER)

Standard reference	Description
ISO 7784-2 **ASTM D 4060** **ASTM F 1978**	Realised by a double abrasimeter TABER® in order to measure the wear resistance of a coating using an abrasive rubber wheels. *Depending of the type of material and application, the load and the number of cycles applied can be variable characteristics during a test.*

THICKNESS CONTROL

Standard reference	Description
ISO 2808	Thickness measurement of the liquid or powder paint film after curing on steel or aluminium support by a control on a metallographic section
/	Thickness measurement of a coating on all metallic surfaces by direct reading with a coating thickness gauges of type PosiTector.

VARIOUS TESTING

Standard reference	Description
Chemical composition and groups present	Determination of groups present and the chemical composition of a coating by comparison with reference spectra thanks to a Fourier transform infrared spectrometer (FTIR)

PAINT BOOTH

Services and Equipments

CRITT MDTS has a platform with paint booth intended to parts of automotive, aeronautic, rail industries, ...

This platform is composed of:

- A box preparation equipped with a ventilation system in order to safely prepare all kinds of mixtures that emit VOCs.

- A paint booth with manual gun or robotic arm.
- A unitary oven allowing to carry out the firing of metallic or organic pieces at a temperature between 40° and 400°C.

Applications :

⇒ Development of paint deposit according to the type of material on which will be deposited the paint and its properties.
Then possibility to validate the deposits done by means of analysis equipments available in CRITT-MDTS.

⇒ Research and optimization of the bests settings (speed, scanning range, time ,…) to depositing a paint layer in the context of pilot series before launching the production .

⇒ Research to improve the resistance of successive paint layers to achieve the possible greatest cohesion between the layers

Paint booth

PART IV : ACCELERATED AGING / CORROSION

Objective : Subjecting to a sample an accelerated aging by prolonged exposure in a chamber under various conditions (temperature, salt spray, humidity, UV, ...) and according to standards or your own specifications.

ACCELERATED AGING TESTING

Testing and standards reference	Description
Tests under the influence of heat **ISO 3248**	Estimate the coating behaviour subjected to a moderately high temperature in order to identify the influence that it can have in in regard with the brightness change, colour, change blistering, cracking, peeling, ...
Neutral salt spray testing **ISO 9227** **ASTM B 117** **Self agreement Renault-Nissan RNES-G-0001 (previously D17 1058)**	Estimation of the resistance of a coating exposed to a neutral salt spray.

NEW

ACCELERATED AGING TESTING

Testing and standards reference	Description
Tests under climatic conditions **(According to specifications or custom)**	Determination of the resistance of a coating at specific or custom temperatures and humidity levels *(to study case by case => contact us)*
Humidity condition tests (Under condensation water environment) **ISO 6270-2**	Determination of the wet resistance of a coating exposed to condensation water atmospheres: - in a constant or alternate manner *(according to ISO 6270-2 standard achieved in part => contact us)*
Alternated or combined sunshine testing with climatic conditions	Exposure to UV radiation combined with climatic conditions (temperature and humidity can be adjusted) standardized or customized.

ACCELERATE AGING TESTING

Testing and standard reference	Description
Testing in cyclic conditions	
NEW **Self agreement Renault-Nissan RNES-G-005 (ECC1, previously D17 2028)**	Determination of the resistance of a coating exposed to cycles of various conditions: "Salt spray / humidity / drying." *One of our chambers meets the specifications for RENAULT ECC1 testing.* 
ISO 11997-1	Determination of the resistance of a coating subjected to cycles: "Salt spray / dryness / humidity" Standard partially met (contact us)

For any specific testing, please contact us
In order to study its feasibility.

PASSIVATION AND CORROSION TESTING

Testing and standard reference	Description
Resistance of the layer passivation on stainless steels **ASTM A 967** **ASTM 1089**	Verification by one of the following tests : - Water immersion test - High Temperature Test - Salt spray test - Copper sulfate test

Do not hesitate to contact us for the implementation of customized tests according to your specification or protocols.

PART V : EXPERTISE / FAILURE ANALYSIS

Objective : Realise a complete examination of a piece in order to determinate the failure origins (breaking, cracking, corrosion,…)

CRITT-MDTS offers you all its means of testing and analysis as well as expertise in the field of materials to :

- Identify the nature and origin of the fault.
- Reflect on the causes of its appearance.
- Help you in your search for solutions.

Examination on metallic pieces

⇒ SEM imaging and micrographic examination for :
- Define the type of damages
- Observe the fracture surface and fracture initiation area

⇒ Examination on metallographic sections to :
- Control the material microstructure (grain size, inclusions, hardness,…)
- Research of potential defects in the part of the critical areas

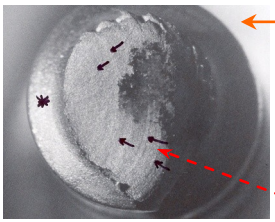
⇒ Verification of the chemical composition

⇒ Possibility to determinate the nature of pollutions ou incrusted particles

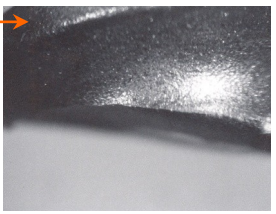
⇒ Possibility to carry out three-dimensional measurements to compare the potential form faults with the file IGES

Possible expertise on other types of matérials.
Please to consult us

Zoom on the expertise of a metallic screw after a break :



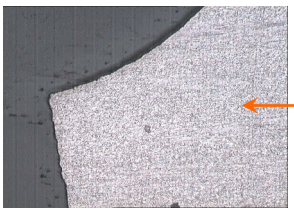
Macrographic examination with binocular magnifying glass



Tracking of the rupture initiation area (black asterisk)

The propagation lines (black arrow) Show that there is a fatigue failure

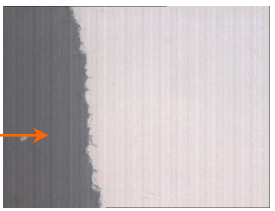
On the surface of the broken piece:
=> no traces of stroke,...



Examination on micrographic section

M x 100

M x 500



Microstructure consistent :
=> no anomaly on surface near the rupture initiation area

Fatigue rupture *(overload)* **No anomaly on the pièce**

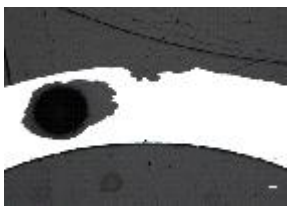
Presence of small surface cracks:
=> the rupture propagated slowly

Zoom on the expertise of corroded stainless steel piece:

Macrographic examination on binocular magnifying glass :

Observation of an area with corrosion pitting in the surface.

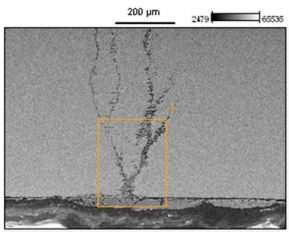
Microscopic examination with optical microscope :



Observation of an area with corrosion pitting.

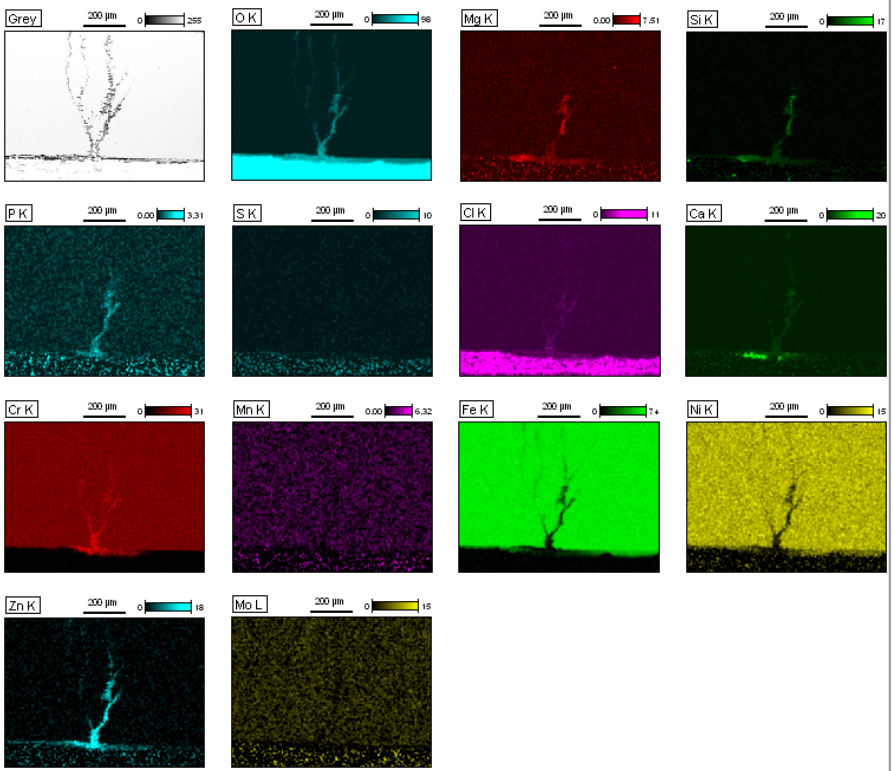
Other area observed after etching: presence of a crack

SEM analysis coupled to an EDS probe :



Analysis of fissure on the SEM coupled to the probe EDS to identify the elements present in zone 1 framed in yellow.
(cf. next page)

The cartographies below show the distribution of the various elements for the range photographed.



These cartographies provide the following information:
- Presence of Fe, Cr, Ni, Mn, Mo and Si from stainless steel.
- Presence of O, Cr, Ca, Zn, Si, Cl, Mg and P in the crack and the oxide surface.
- The sulfur is not present in the crack.

PART VI : DEVELOPMENT OF INDUSTRIAL TREATMENTS

Objective : Our industrial equipment allows to switch from a idea to its industrialization. As part of a technology transfer, studies that we carry out with this equipment, will integrate the prototyping phase until pre-production.

HEAT TREATMENTS

Service :

Development and feasibility study of specific heat treatments on different types of pieces (prototype, pre-production industrial, ...) followed or not a surface treatment.

Applications :

⇒ Improvement of the surface condition

⇒ Improvement of mechanicals characteristics *(best fatigue resistance , greater resilience,…)*

⇒ Structural homogenization

⇒ Stress-relieving

⇒ Degasification

Heat treatment and degas-sing equipments

Equipment :

- Industrial furnace gas boosted up to 1.5 bar (maximum temperature 1350 ° C - maximum load 300 kg) with a diffusion pump for a maximum vacuum level of 5.10^{-5} to 1.10^{-6} mbar.

PIM PROCESS (POWDER INJECTION MOLDING)

Presentation:

The PIM process, including the MIM and CIM (Metal or Ceramic Injection Moulding), is a combination of injection moulding and powder metallurgy for the realization of metal or ceramic pieces.

Principle and description of PIM technology:

This technology involves to inject in a press a feedstock (= mixing of metallic or ceramic powder with a binder (= polymer)) in order to obtain shaped piece (= green piece). This part is then stripped of binder and then sintered.

Equipments :

- Injection press
- Semi-industrial process of catalytic debinding, in water and thermal (2nd debinding)
- Laboratory process for the debinding with solvent (acetone or ethanol)
- Sintered furnace for metallics pieces, under partial pressure with a neutral atmosphere (N_2/Ar) ou reductrive (H_2)
- Sintered funace for ceramics pieces , with an atmospheric pressure under air.

PIM PROCESS (POWDER INJECTION MOLDING)

PIM technology positioning :

⇒ Manufacturing process adapted to :
- Mass production
- Pieces with complex or/and innovative form
- Small and medium size pieces (generally varying between 1 g and 100 g)

⇒ Wide range of materials :
- Based of metallic powder (TA6V, CoCrMo, 316L, innovents alloys ,…)
- Based of ceramic powder (zirconia, alumina,...)
- Composite (based on mixing of several powders)
- Bi-materials (by co-sintering)

⇒ This technology allows to :
- Make pieces with an excellent surface condition without required (or very few) at a remachining.
- Obtain dense or macroporous pieces (≈ 98% of theoretical value)
- Weld MIM pieces among themselves
- Co-sinter MIM pieces in order to create more complex pieces with various functions (Ex : magnetic - nonmagnetic, porous - dense and/or with gradient porosity …)

Various and diverse application areas such as :

Aeronautic, Automotive,
Micromechanical,
Electronic,

Military - Firearms,
Locksmith, Horology,
Luxury industry, etc...

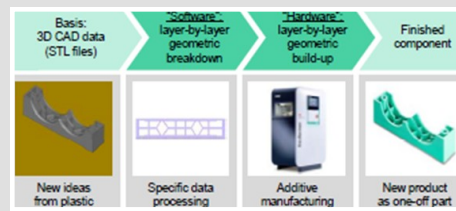
FREEFORMER — 3D PRINTING PROCESS

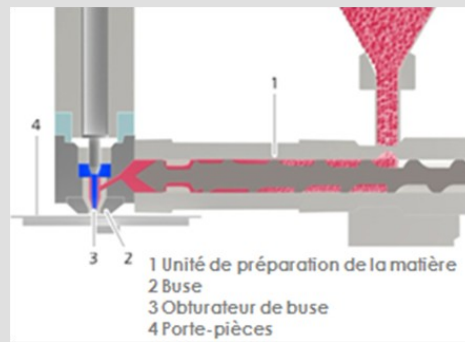
Presentation :

The Arburg FREEFORMER 3D printer allows prototypes or small series of polymer parts to be manufactured without moulds from standard plastic injection granules. Currently, this technology is capable of processing ABS, polycarbonate, polyamide, elastomers (TPE), PLLA or SAN. If the transformation parameters are within the working ranges of the FREEFORMER, we can develop the implementation of other types of polymers or elastomers.

Principle :

The part is built layer by layer from a 3D file in STL format. Two different materials can be used for the same part. The FREEFORMER machine has two nozzles, each with a 15 mm plasticizing screw. These allow the thermoplastic granules to melt. Using a piezoelectric actuator, tiny molten droplets are applied drop by drop according to the coordinates transmitted by the 3D file.





FREEFORMER — 3D PRINTING PROCESS

Applications :

- The FREEFORMER is the only additive manufacturing machine using molten polymer under pressure. This makes it possible to obtain parts with mechanical characteristics that reach 80% of those obtained by plastic injection, far above other 3D printing techniques.
- The two nozzles allow different materials to be combined for the same part with drop size accuracy. The interface between the two materials is no longer a surface, but an interpenetration of drops between them, which increases the mechanical properties at the level of the resealing area.
- By using, as a second material, a polyvinylpyrrolidone which has the property of dissolving in water, it is possible to create complex hollows or voids in the part and thus to produce shapes that are otherwise impossible to obtain.
- CRITT-MDTS carries out research to obtain, using feedstock for MIPs, metallic or ceramic parts by this 3D printing process.

Equipments :

- The chamber of work allows to build pieces of 230 x 130 x 250 mm with a precision of 0,15 mm.
- 2 available nozzles of 0,2 and 0,25 mm making respectively minimum layers of 0,25 and 0,31 mm
- Temperature of the building chamber : 50°C - 120°C
- Temperature of transformation screws : 350°C
- Pressure of the material : 500 - 1300 bar

COLD AND ATMOSPHERIC PLASMA

Presentation :

CRITT MDTS has a polyvalent platform using cold plasma technology for different sectors of Surface Treatments and Coatings composed of : 1) different atmospheric pressure plasma systems, 2) a vacuum chamber with Radio Frequency and Microwave generators, assisted by Magnetic Field and 3) independent and versatile deposition systems with flow and temperature control during the CVD deposition phase. Plasma monitoring is carried out with: mass spectrometry, optical emission spectroscopy and electronic probe.

Applications :

=> Activation of surfaces of all types of materials (metallic, polymers, mineral and ceramic glasses). Example :

- Surface preparation and cleaning (example: before gluing or painting)
- Decontamination and Sterilization of Medical Devices

=> PECVD dry deposition with different solutions (aqueous, organometallic precursors based on Si, Al, Ti or other, solvent, hydrocarbons...).

Associated characterizations :

- Scanning Electron Microscope (SEM) coupled to an EDX probe (see example on pages 36 and 37)
- X-ray photoelectron spectroscopy (XPS) see page 13
- X-ray diffraction (DRX) see page 10.
- GD-OES Glow Discharge Optical Spectroscopy (see page 13)
- Nano indentation and nano scratch platform (see pages 21 and 22)

Equipments :

- Cold plasma reactor with various generators: Microwave (MW), Radio frequency (RF) coupling with a magnetic field
- Atmospheric Plasma torch (AcXys) on 6 axes robot arms.

COLD AND ATMOSPHERIC PLASMA

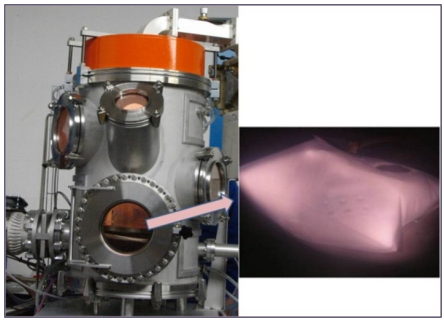


Figure 1 - Cold Plasma Platform
(on the right plasma generated only in a container)

Figure 2 - Acxys® Atmospheric Pressure Plasma Torch on Six Axis Robot

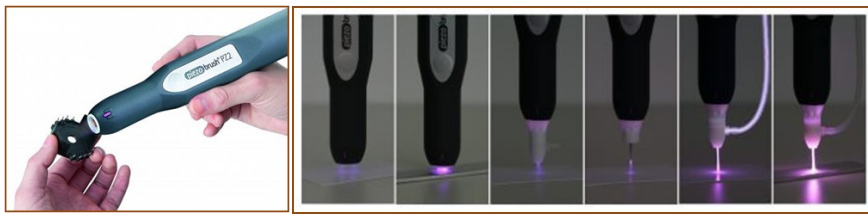


Figure 3 Manual Torch (Piezobrush® PZ2 from Relyon Plasma Gmbh) with different nozzles

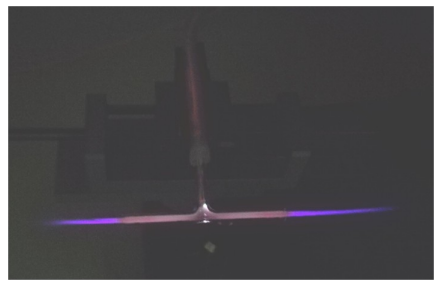
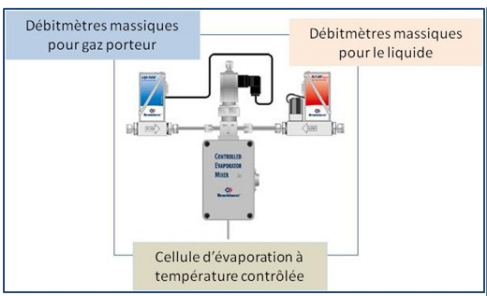


Figure 4 Plasma-Gun: Generation of a "volumic" plasma at atmospheric pressure in a T-tube



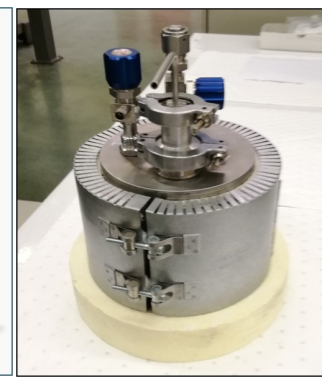


Figure 5 Precursor vaporization systems for CVD deposition

SUPPORT IN THE TECHNOLOGY TRANSFER

The main part of CRITT-MDTS's activity is to provide services to industrial customers but it is also involved in several research programs in association with other actors of the research, academic or private.

This enables to CRITT-MDTS engineers and technicians to constantly keep up-to-date and to have a dual culture that put them in the best position to understand the industrial issues and give them effective and innovative solutions.

Therefore, we can support you in your projects as follow :

- Help to define and express your needs

- Technical advice and support with an extensive bibliographic study if necessary (*cf. Monitoring tools in next page*).

- Implementation of an action plan defined together, from feasibility study to a full transfer of technology and knowledge.

PART VII : RESEARCH AND DEVELOPMENT

Objective: From our tools and the technicians and engineers experience, CRITT-MDTS will realise for you and on themes which are yours, art states of professional level, targeted, updated and exhaustive.

TECHNOLOGY SURVEILLANCE

Subscriptions to specialized scientific journals

Subscription to several scientific journals specialized in our expertise domain. These journals are read and analyzed by CRITT-MDTS technicians and engineers who keep their skills up-to-date and identify new developments that may be of interest to our customers.

Documentary database and technical data specialized in the field of materials

Access to « technical of the engineer » and complete collection of « ASM Handbooks ». These encyclopaedias are rich of technical data on processes and materials.

Access to servers of databases

Access to servers of databases, which allow to interrogate serveral hundreds of specialized databases (bibliographical, technical data,..)

Research and analyze tool on patents

Professionnal tools to consult directly patent databases from the European Patent Office (EPO) and the United States Patent and Trademark office (USPTO), extract revelant information with regard to a given subject, analyze it and the organize its surveillance.

Do not restrict your surveillance only to internet search engines

SUPPORT IN SPECIFIC REQUESTS

At your request and joining the skills of our engineers and technicians team, CRITT-MDTS can offer you specific support.
Indeed, we can support you in your development projects by means of customized service and with the possibility to mix our various activities so as to :

- Identify yours needs and/or problem
- research of solutions thanks to various data bases and monitoring tools (*see previous page*)
- carry out all types of customized analysis and testing with our equipments.

ASSISTANCE IN THE SEARCH FOR FUNDING

Help setting up a financing file

As part of ours services, CRITT-MDTS can support you in the setting up of your financing files.

Research Tax Credit and Innovation Tax Credit

As Technological Resource Centre, CRITT-MDTS is eligible to Research Tax Credit and Innovation Tax Credit.

Innovation Tax Credit is a tax reduction help calculated according to Research & Development expenditures of your company and intended to encourage the research and technological innovation.

Research Tax Credit is a help provided by tax credit on the amount committed in favour of « design, developing of prototypes or pilot installations of new products » and only the SMEs in the European sense can benefit from this tax deduction.

Therefore, this certification offers to our customers developing R&D activities the possibility to deduce the cost of some services provided to CRITT-MDTS, subject to the approval of your eligibility.

NOTES

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CRITT

Centre Régional d'Innovation et de Transfert de Technologie

CRITT Material - Coating and surface treatment

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Edition – 2019