Metamorphose raw MATERIALS

LSPM

Laboratory of Sciences of Processes and Materials — Presentation booklet

When entering the LSPM for the first time, you're a little confused at first. This research laboratory is home to unusual machines, innovative materials... You see surprising phenomena, you hear unknown words, then you try to understand and start asking yourself questions : how do LSPM researchers fabricate these new materials? How do they manage to metamorphose raw materials ?

Without providing all the answers, this booklet provides a simple approach to the materials transformation process, and offers an overview of the laboratory. It is at once a guide, a reminder and a dictionary. We hope you will find it useful and that it will make you want to know more!

Laboratory of Sciences of Processes and Materials

THE LABORATORY

The LSPM is a CNRS laboratory situated on the Villetaneuse campus of the University Sorbonne Paris Nord. What is happening inside?

How does it work?

Nearly **140 people** work in this **experimental science** research **laboratory**. Multiple subjects linked by the same theme are studied in depth: **physico-chemical processes** and their characterization.

The aim is for **researchers** to understand how to fabricate certain **materials** of interest and then to test them in order to gain a better understanding of their properties. Processes and materials are studied on a very small scale, **microscopic** or even atomic. This field of research is at the crossroads of physics and chemistry.

Through the publication of their scientific results, researchers broaden the scientific community and global knowledge, but their discoveries are also of interest to industrials partners who put them into practice. The LSPM therefore also has an **engineering** component. Nearly half of the researchers work with a variety of companies

It is organized into 3 areas of work that are interrelated : PPANAM, MINOS and MECAMETA.

The research of the 3 axes are linked to some extent and often the knowledge produce from their projects broadens their field of research.

The LSPM is a CNRS laboratory situated on the Villetaneuse campus of the University Sorbonne Paris Nord. ©LSPM

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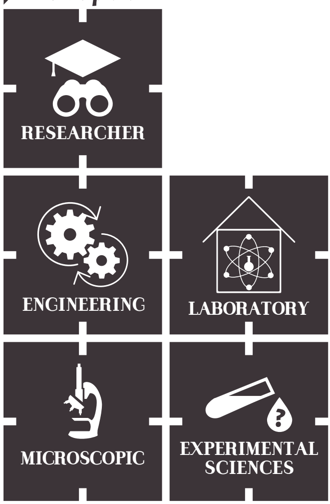

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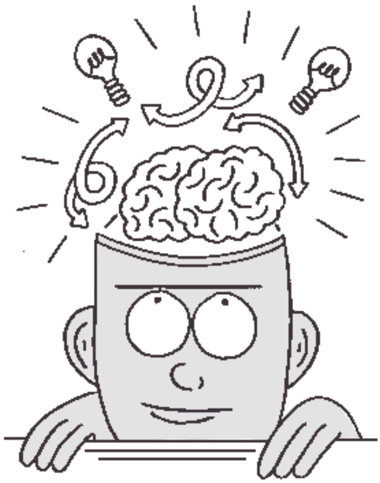


TO BETTER UNDERSTAND

The LSPM evolves continuously, in line with the discoveries and questions raised by the research activity. This laboratory is a bit like a brain where everything is interconnected : certain areas are dedicated to specific subjects and actions, but the links are made and broken according to emerging issues and questions.

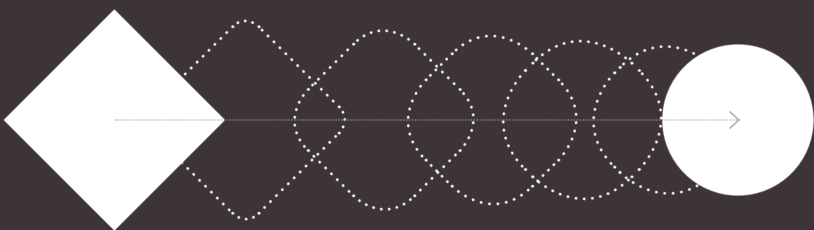
« What do you find most amazing about your job? »

« The most amazing thing is that I didn't get tired of my work. The research system allows a certain freedom of evolution. You can navigate between different forms of work: more administrative, more research, more teaching. »

« The best, or most brilliant thing is that ideas can come at any time, and not only in the laboratory : the best idea is when you don't look for it.»

Method, technique used to perform a task,
or to fabricate a material or a finished product

THE PROCESS



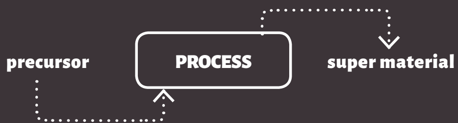
The fabrication process is the common foundation of all LSPM research projects. But what is a process?

How does it work ?

The techniques and products differ from project to project, but the principle is always the same :

First of all, one or more pure **elements** or **precursors** are needed. In order to be properly exploited, these raw materials are purchased in various forms : powder, **gas, solution...** The precursor can be exploited in different **phases**, i.e. depending on its temperature it will be solid, liquid or gaseous.

The process is in fact the **transformation of the precursor to its final form**. It may involve several physical and chemical **phenomena** as well as different techniques.



Once the process has been carried out, researchers obtain new materials from the precursors. These new materials resulting from the processes have particular properties : strength, **density**, thickness, shape, structure... which can be used in many applications.

<u>Among the processes developed at the LSPM are the following :</u>

- the growth of thin layers by atom deposition
- high-pressure and high-temperature processes to form crystallin structures
- chemical processes to create nanoparticles

Each of the LSPM's 3 research axes uses one of the 3 methods in a preferential way.

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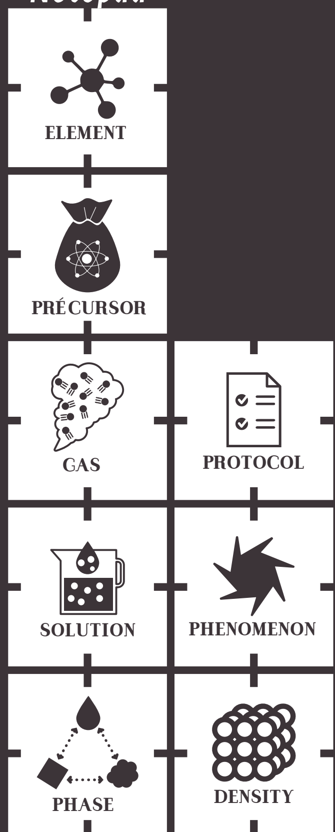
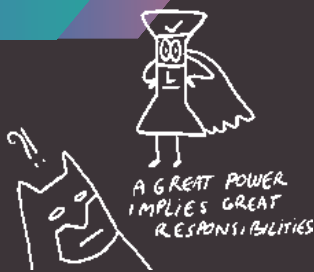


TO BETTER UNDERSTAND

The physico-chemical process can be seen as cooking. The cook elaborates a recipe by choosing the ingredients, the utensils, the way to mix them or to cook them. In the same way, LSPM researchers develop protocols from precursors that will be subjected to carefully chosen physical and chemical phenomena, temperature and pressure conditions. All this is done using appropriate instruments. The new produced material will possess new properties that meet the challenges of tomorrow.



Plasma processes, nanostructures and thin films

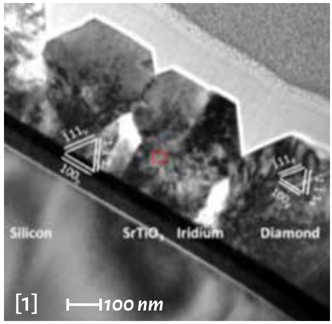
PPANAM

The PPANAM research axis uses the thin films growth by atom deposition to make new materials. What is the principle of this family of processes ?

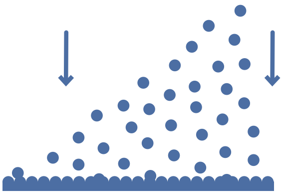
How does it work ?

On a chosen **substrate**, **atoms** are arranged to form a new material [1]. Working directly at the atomic scale allows to create very thin layers of material [2]. There are several techniques for depositing the atoms on the substrate. They can be vaporized, sputtered... All this will influence the way they organize on the substrate and modify the properties of the resulting material.

In contact with the atoms, the substrate also influences the growth of the material: this phenomenon is also studied at PPANAM.



[1] Thin layer of diamond seen in cross section with an electron microscope ©LSPM



Within the PPANAM axis, evaporation, sputtering, **Plasma** Enhanced Chemical Vapor Deposition (*PECVD*) is used. **Sputtering** consists of sputtering a **target,** which by disintegrating allows the atoms to fall towards the substrate.

Evaporation consists in heating the precursor. The vaporized element is then deposited by **condensation** on the substrate to be coated.

PECVD deposits thin films on a substrate from a gaseous state (vapor). Chemical reactions take place during the process after the formation of a plasma from the reactor gases. The plasma is usually obtained from this gas by an electrical discharge.

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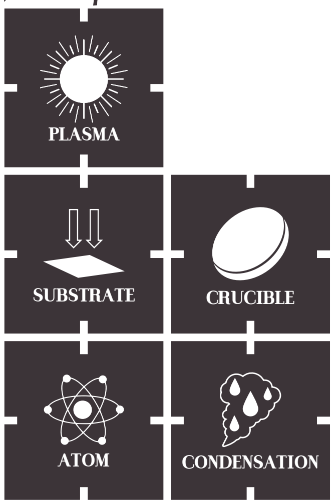
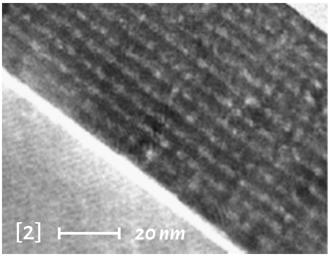


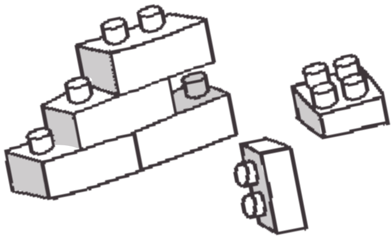


[2] Multi-layer thin film seen in cross section with the electron microscope. ©LSPM

[3] Thin-film sample for flexible electronics. In orange a flexible substrate has been used, here kapton, and in black the thin layers that have been deposited. ©LSPM

What can it be used for ?

The materials produced in PPANAM find concrete applications for their electronic properties [3], at Thales for example, their optical properties in the glass industry at Saint-Gobain or Essilor and their electromagnetic properties at Samsung for example, for the memory storage of cell phones.



TO BETTER UNDERSTAND

The different techniques used have the same goal : to master the properties of the thin layer grown, a bit like building a thin wall with Lego bricks by carefully choosing them and their positions so that the wall is solid.

Zoom on ...

Sputtering

Sputtering is a process of thin layer deposition to obtain metallic films of alloys, but also of **semiconductors** (nitrides, oxides).

This technique consists in bombarding one or several target material(s) with Argon, a **neutral gas** ❶

In the sputtering enclosure, only argon is present. A voltage is applied between the target and the substrate : this leads to the ionization of argon i.e. it loses one electron. The plasma remains neutral as Ar+ **ions** and electrons are present simultaneously. A strange light appears in the enclosure, it is due the argon becoming a plasma.❷

Under the effect of the electric field, the Ar+ ions of the plasma are attracted by the target material and starts to bombard it. The atoms are thus torn from the target and form a kind of cloud.❸

This is how the atoms of the cloud reach the substrate and are deposited there. By controlling the parameters (voltage, pressure, argon flux, etc.) it is possible to deposit very thin layers of which the thickest are only a few hundred nanometers.❹

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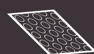
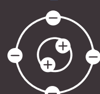
NEUTRAL GAS



SEMI-CONDUCTOR



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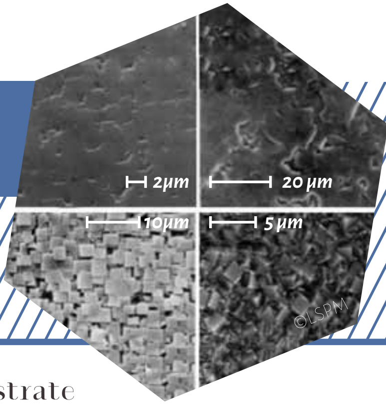
On this image we can see how the chosen substrate influences the structure of the grown material (result of diamond nucleation on different types of substrates).



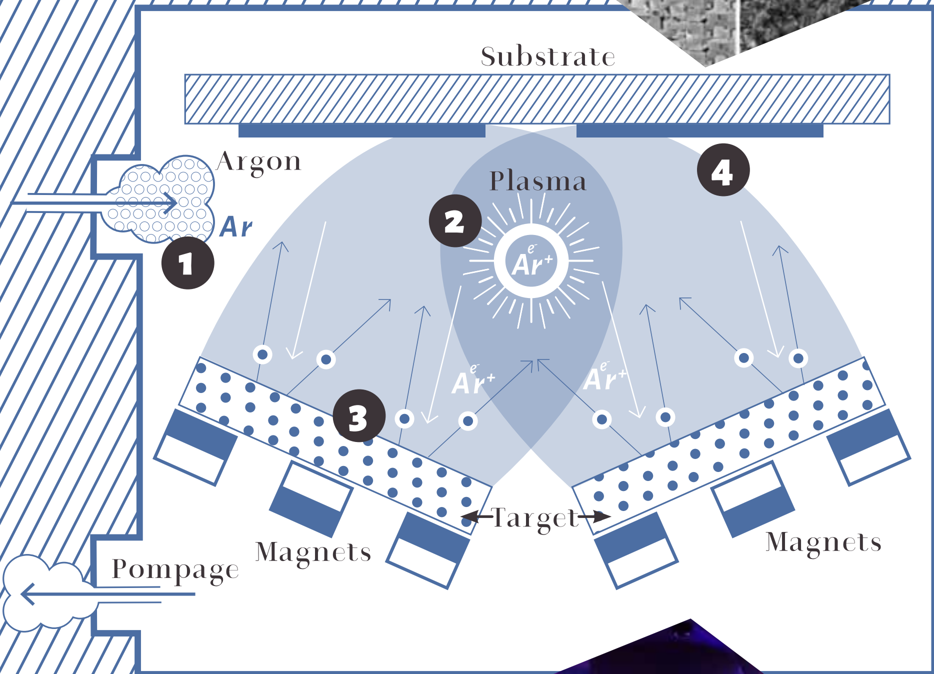


Image of the device. The light generated by the plasma is clearly visible.

photographic credits : ©LSPM

« What do you find most amazing about your job? »

« Playing alchemists. A long time ago they wanted to transform lead into gold, which they never managed to do. We transformed a simple gas into diamond! »

« We never get tired of it! » « The pleasure of constantly learning things, as if I were an eternal student. It gives the impression of staying young (and humble before Nature). »

« What's surprising is that international researchers are working on diamond synthesis for nearly 40 years. Usually if after 10-15 year a material produced in the laboratory has not been industrialized it is abandoned : diamond is an exception. »

Mechanics and metallurgy of materials

MECAMETA

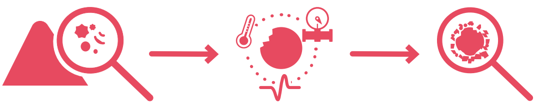
The MECAMETA AXIS
focuses on high pressure
and temperature processes.

How does it work ?

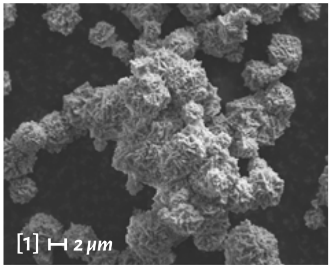
The materials are submitted to high **pressures** and **temperatures** to obtain new materials with the desired **crystal structures** and microstructures. The **properties** of these materials, such as hardness, elasticity or even density, can thus be influenced.

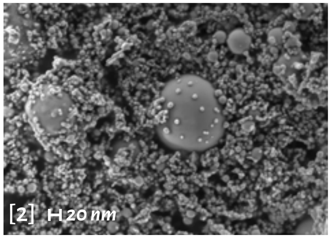
Within the MECAMETA axis, researchers work mainly on metals. The precursors can be pure but most often they are **alloys**. The selection of precursors is an important step in the research process : the right crystal structure must be selected, but also the shape of the precursor. Usually they are powder whose grain size and shape is a fundamental parameter [1,2].

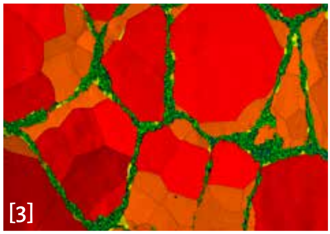
The precursor must then be subjected to various operations : for example, **impacting** or **crushing** them with a mill or **sintering** them. These phenomena make it possible to work at different scales : at the microstructural scale and at the scale of the crystalline structure.



By varying the main parameters of pressure and temperature during these steps, materials with different **mechanical properties** can be obtained [3]. To identify the characteristics of these materials, they are tested in the form of **test specimens**.







[1] Micrometric nickel powder with an appearance close to that of a Romanesco broccoli. The average particle size is 5 µm. ©LSPM

[2] Nickel nanopowder. The size of the small particles is about 100 nm with a few larger particles of about 1 to 2 µm. ©LSPM

[3] Microstructure of a titanium alloy: the alloy powder was placed in a mill and the impacted particles were then sintered. Pressure and temperature allowed the particles to bond together. The small grains (in green) were created as a result of the impacts. The larger grains inorange and red correspond to the center of the powder particles that were not affected by the impacts ©LSPM

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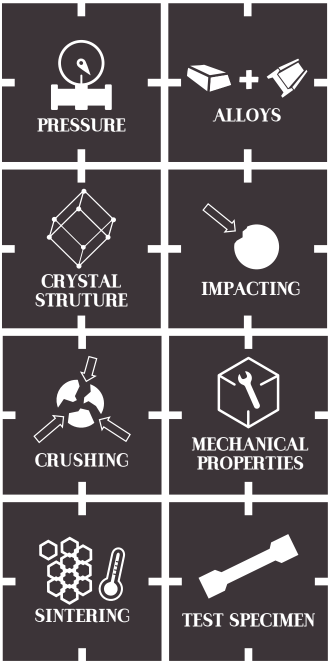
Bloc-note.



What can it be used for ?

The forged materials find concrete applications for their mechanical properties in large-scale industrial processes: research into stronger and lighter materials, impact resistance, in aeronautics or even in the medical field, where researchers are working, for example, on a new titanium alloy for hip prostheses in particular.

TO BETTER UNDERSTAND

Researchers in the MECAMETA axis are heirs to knowledge developed since the Iron Age. It was known empirically that alloying metals made it possible to create stronger materials (the hardened steel of a blade, for example), but each had its own manufacturing secrets. Today, a scientific method is being applied to understand how microscopic scale phenomena affect macroscopic properties.

Zoom on ...

The crusher

The mill is a step that allows to make first modifications on the selected powders.

In the mill, ❶ the powder precursor is placed in a container where very hard steel balls impact the surface of each particle contained in the powder.

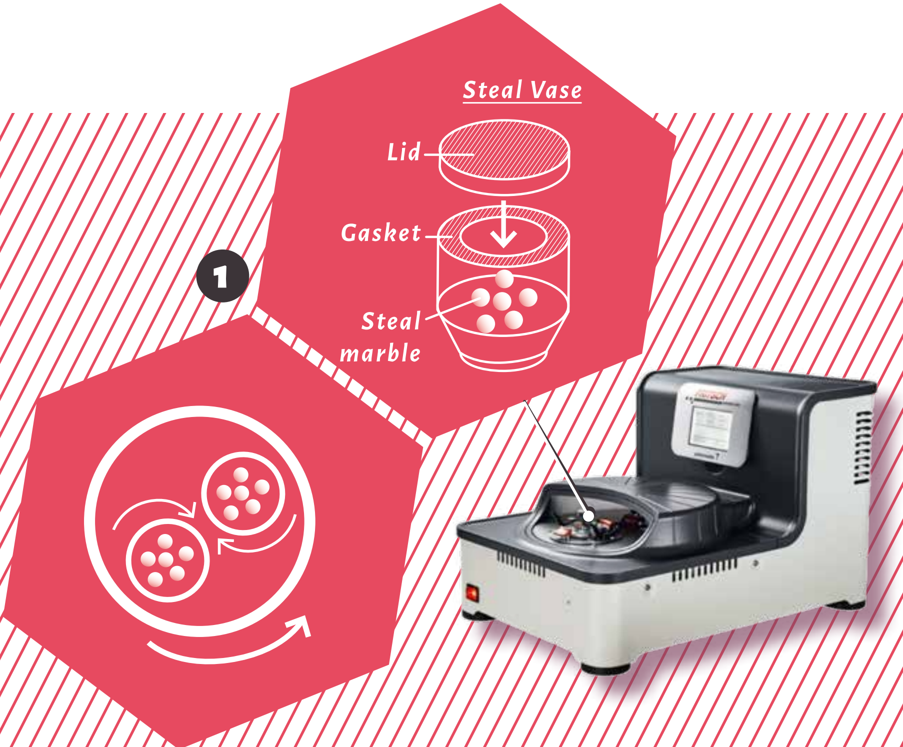
The balls can even go as far as to fragment the grains of the powder, i.e. break them up and form new, smaller grains.

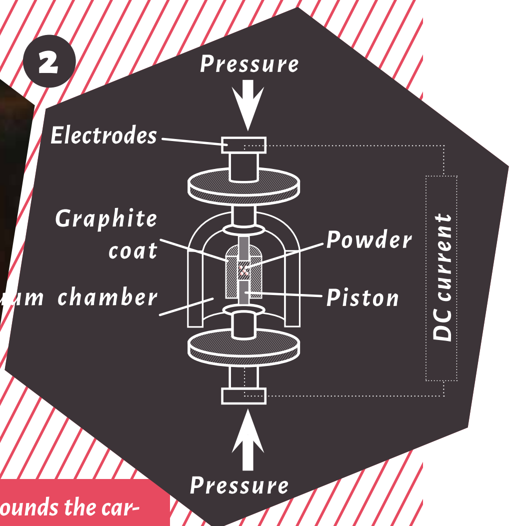
Sintering

The **SPS** or **Spark Plasma Sintering** is a furnace used to sinter powders (i.e. welding the grains together)..❷

A graphite (carbon) mould is used in which the powder is placed. Pressure is applied by means of pistons and heating is achieved by means of a flow through the mould.

To limit heat loss, the mould is surrounded by a carbon fabric called felt which is held with a carbon braided wire.





In this photograph we can clearly see the felt that surrounds the carbon mold. The mold is white because it is at high temperature.

« What do you find most amazing about your job? »

« I like to pass on my knowledge and it's a real satisfaction to help young people find their way. This is particularly true with work-study programs. »

« I love it when an idea that we sometimes had over a drink or a coffee, drawn on a corner of a table, ends up, often years later, in a real material with optimized properties. »

Inorganic Materials and Nanostructures

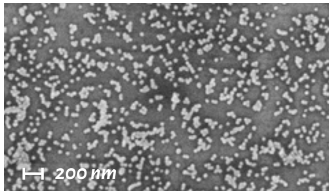
MINOS

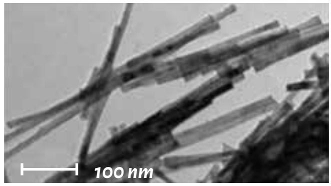
The Minos axis mainly uses chemical processes to create materials that have specific nanostructures.

How does it work ?

MINOS researchers are interested in the chemical processes used to synthesize nanoparticles.
A **nanoparticle** is a three-dimensional object whose size does not exceed one or two hundred nanometers, i.e.nanoparticles are 1,000 times smaller than the diameter of a hair ! They are not only invisible to the naked eye, but also to the optical microscope... [1]

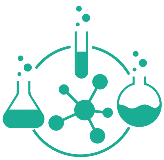
To make nanoparticles, researchers use **chemical reactions** that cause atoms to come together and create assemblies whose size is **nanometric,** halfway between the **macroscopic** and atomic scales. The advantage of being familiar with these chemical phenomena is that they can be reproduced simultaneously and in large quantities.





[1] Gold nanoparticles observed with a scanning electron microscope.©LSPM

[2]Nanoparticles in the shape of «nano-needles»©LSPM



After their synthesis, the nanoparticles are ordered to produce macroscopic material : using **magnets** for example [2],or thin film processes as in the PPANAM axis.

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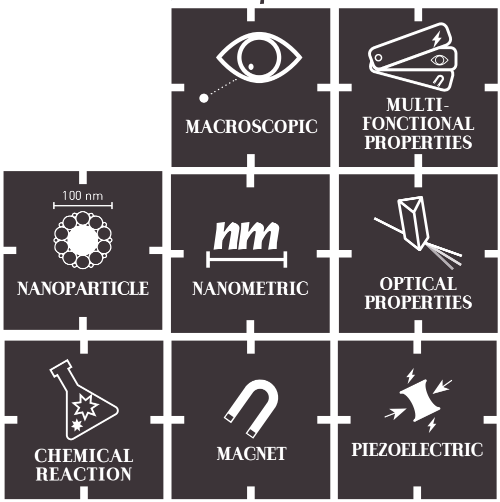

What can it be used for ?

[3] Photovoltaic panel ©Dllu - Own work /Wikipedia Creative Commons BY-SA 4.0

These processes produce materials with often hybrid and **multifunctional properties**, useful for the ecological transition : for example, materials that are both transparent (**optical properties**) and resistant to ionizing radiation, photovoltaic materials as well as materials that are both magnetic and **piezoelectric** to integrate components that consume less energy and have high data storage density for applications in microcomputing or mobile telephony. Among the materials we manufacture are also innovative electronic components that could integrate devices in the field of decarbonized vehicles or materials for hydrogen storage.

TO BETTER UNDERSTAND

One can compare the protocols followed within the MINOS axis to the work of the potter. Chemical reactions are induced to give the original material the desired shape and properties, as the potter shapes the clay, bakes it to vitrify it... We even expel air bubbles from the material as a ceramist does by hand!

Zoom on ...

Polyol and SPS synthesis processes of nanostructures under magnetic field conditions

The precursor elements are used in the form of metal salts, which are dispersed in a liquid (or solvent).

The metal atoms (cations or positive ions) of the salts are dispersed in the solvent, which is called the reaction medium. The solvent used in polyol processes is a kind of viscous oil which has the particularity of being able to heat up to more than 200°C without evaporating or degrading.[1]

After being dissolved the different precursors are heated in a **reactor**. Under the effect of heat, the atoms combine to form **nanoparticles**. [2]

The magnetic field conditions the atoms to aggregate in the form of tiny magnetic needles thinner than a hair. Like the needle of a compass, they have a north and a south pole. [3]

A larger compound (about the size of a coin) can then be formed by assembling the nanoparticles. The goal here is to ensure that this final material has a good mechanical strength but also that the magnetic needles that compose it keep their initial size and the same arrangement. For this purpose, a furnace, called Spark Plasma Sintering, is used, which allows for rapid firing (or sintering). This furnace is also equipped with a magnet which allows the magnetic needles to be aligned during the shaping step. [4]

In some cases, a second heat treatment of the resulting material is necessary to strengthen it, since air bubbles (pores) may have been trapped in the material during **sintering**. For this purpose, a **postdensification** step is carried out. The material is placed in a furnace which not only heats up but also applies a strong pressure from all sides : this is the Hot Isostatic Compaction.[5]

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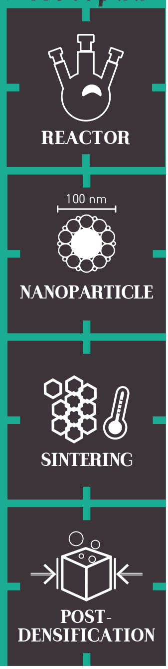
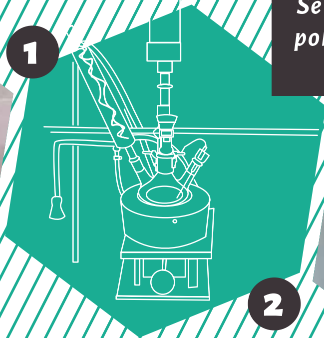
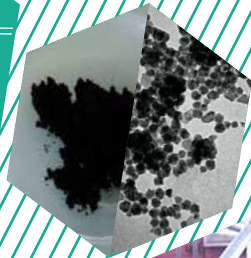


Salts at the begining



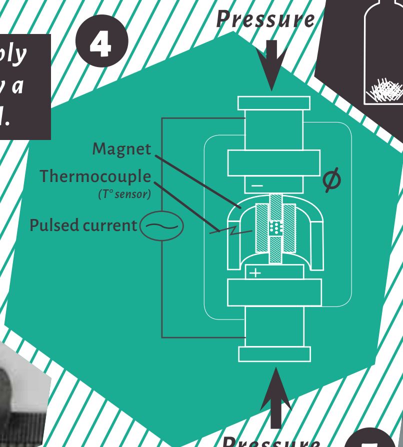
Setup of the classical polyol process (reflux heating)

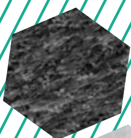


At the LSPM, the polyol process is carried out under the effect of a magnetic field: the reaction medium is placed in the center of a magnet.

Nanoparticles take the form of small needles that are oriented according to the magnetic field

The SPS assembly here assisted by a magnetic field.





the pellet of the material obtained at the end of the process.

the transparent pellet obtained after compaction

photographic credits : ©LSPM

« What do you find most amazing about your job? »

« All it takes is a small variation in one of the parameters involved in the various stages of the process to obtain a material with totally unexpected properties »

« For my part, it is the ingenuity of human kind that always amazes me. »

« The best thing is to learn new things every day, to discover new things, to develop new machines that actually work and to be able to produce materials. »

« That processes using high pressure fluids, which had begun to make their way into industry (e.g. ASEA in Sweden in the 1960s), have experienced a marked decline in Europe while they are now experiencing strong growth in Asia. »

« It's a «thankless» job in the sense that the acquisition of experimental data is cumbersome but exhilarating in the sense that it can be applied to research themes that are constantly being renewed. The high pressures make it possible to remove many technological obstacles to the development of new materials. »

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***alloy :** Metallurgical product resulting from the incorporation into a metal of one or more elements (metallic or not), carried out in order to modify some of its properties or even to give it new properties.*

***atom :** Elementary constituent of matter.*

***chemical reaction : :** Process during which the transformation of certain chemical species (molecules, atoms, ions) into others is observed. It is a process that has the particularity of preserving matter in its entirety. When brought together, the elements that make up the reagents react and are all found in the products of the reaction.*

***condensation :** Passage of a vapour into a solid or liquid state.*

***crystal structure :** The crystal structure (or structure of a crystal) gives the arrangement of atoms in a crystal. These atoms repeat periodically in space, as a pattern, and thus form the crystal structure.*

***density :** The density or relative density of a body is the ratio of its density to the density of a body taken as a reference.*

***element :** Fundamental chemical entity common to various varieties ofthe same simple body as well as to combinations of this simple body with other bodies.*

***experimental sciences :** Sciences that use the same method of testing the validity of a hypothesis by reproducing a phenomenon (often in the laboratory) and varying a parameter. The parameter that is varied is involved in the hypothesis. The result of the experiment validates or not the hypothesis. The experimental approach is applied in research in biology, physics, chemistry, psychology, or archaeology.*

engineering : *Study of an industrial project in all its aspects (technical, economic, financial, monetary and social) and which requires a synthesis work coordinating several teams of specialists.*

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gas : *Body in the gaseous state at ordinary temperature and pressure.*

---

ion : *An electrically charged particle formed by an atom or group of atoms that has gained or lost one or more electrons.*

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laboratory : *All researchers and research support staff (engineers, technicians, administrative staff) carrying out a research program in a given location.*

---

macroscopic : *Which can be seen with the naked eye.*

---

magnet : *Any substance that possesses or has acquired the property of attracting iron. In the laboratory, it is a device (permanent magnet or electromagnet) designed to produce an external magnetic field in certain processes.*

---

mechanical properties : *These relate to the deformation of a material subjected to a force, such as strength, hardness, rigidity or toughness (a material's ability to store energy).*

---

microscopic: *Can only be seen with the microscope.*

---

molecule : : *A particle made up of atoms that represents, for a pure body made up of them, the smallest amount of matter that can exist in the free state.*

***multifonctinal properties :** Properties of a material that can perform different functions, perform several tasks.*

***nanometer :** This refers to objects with dimensions in the nanometer range, where the unit of measurement for length is one billionth of a meter.*

***nanoparticle :** A particle with dimensions ranging from about 1 to 100 nm.*

***neutral gas :**A neutral gas is a gas whose composition is without detrimental consequences when used in the desired context.*

***optical properties :** Observable characteristics of a material when light passes through it.*

***phase :**State of matter. There are mainly 3 of them: solid, liquid and gaseous phases. Other unstable phases of matter are also considered in physics..*

***phenomenon :**A natural fact that is observed, susceptible to scientific study, and can become a subject of experiment: radioactivity for example.*

***piezoelectrique :** Which concerns the electrical phenomena produced by bodies that deform.*

***plasma :** Fluid composed of electrically neutral gaseous molecules, positive ions and negative electrons.*

***precursor :** A compound that precedes another in a series of chemical reactions. The word is used to describe the raw material in fabrication processes.*

pressure : *The ratio of the intensity of the force exerted uniformly on a surface and perpendicular to it, to the area of that surface.*

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protocol : *Set of rules, questions, etc., defining a complex operation: Protocol of an experiment, a test.*

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reactor :*Container in which a chemical reaction is carried out.*

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researcher : Staff who devote themselves to research on a full-time (CNRS staff) or part-time (teacher-researchers) basis.

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salt : *A pure body, of ionic structure, resulting from the action of an acid or basic oxide on a base or basic oxide, or from the action of an acid or base on a metal.*

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semiconductor : *A semiconductor is a material that has the electrical characteristics of an insulator, but for which the probability that an electron can contribute to an electric current, although small, is sufficiently high. In other words, the electrical conductivity of a semiconductor is intermediate between that of metals and insulators.*

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sintering : *: Operation carried out in powder metallurgy, to achieve by eating an agglomeration of the treated products, in order to give them sufficient cohesion and rigidity without complete fusion.*

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solution : *In chemistry, a solution is a homogeneous mixture resulting from the dissolution of one or more solute(s) (dissolved chemical species) in a solvent.*

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solvent : *A substance (usually liquid) that has the power to dissolve other substances.*

 ***specimen :*** *A sample taken from a batch, under specified conditions, and intended to be subjected to tests described in the specifications for materials supplies*

 ***substrate :*** *The infrastructure for something, that on which an action is exerted.*

 ***target :*** *Expression designating a solid precursor intended to be sputtered during a process.*

 ***viscosity :*** *Physical property of a fluid, involving a relationship between stresses and strain rates.*


APPROVED BY
SUPER ÉPROUVETTE

This project was led by Nathalie Lidgi-Guigui, associate professor att he LSPM in collaboration with Laura Bertrand and Camille Lindeperg.

We would like to thank Mr. D. Vrel and D. Faurie, directors and deputy directors of the LSPM, as well as the members of the LSPM communication unit, R. Issaoui, C. Lazzaroni and F. Cazes for their help and welcome throughout the project.

We would also like to thank all the members of the LSPM and especially those who answered our many questions F. Challali, F. Benedic, D. Tingaud, M. Traore, S. Mercone, F. Schœnstein, M. Nikravech, P. Langlois, G.Lombardi.

Finished printing in October 2020
at Paris 13 University reprography service