PRESI

PRESI LAB'NOTE

METALLOGRAPHIC PREPARATION
MEDICAL FIELD

INTRODUCTION

A multitude of materials are used in the health field, for example in medical instruments and equipment (surgical tools, mobility devices, artificial limbs, etc.) and to compensate for functional deficiencies or injuries. All these materials come under the heading "biomaterials".

Biomaterials, by definition, are materials used in a medical device intended to interact with biological systems to assess, treat, strengthen or replace a tissue, organ or body function.

Among the different biomaterials, two main categories can be distinguished:

LIVING (or natural) **BIOMATERIALS**
These are materials of biological origin. The principle is to create a natural base to accommodate the regrowth of a specific fabric allowing the replacement of the deficient function. Amongst these living biomaterials we find:
- Biological tissues (porcine valves, beef carotid, etc.).
- Grafts (xenografts, allografts and autografts).
- Coral, which is a natural porous ceramic. This porosity favours bone implantation. It is used in orthopaedic and maxillofacial surgery.
- Collagen, which is a natural protein of animal (skin) or human (placenta) origin whose applications are: cosmetology and cosmetic surgery, eye implants, tissue reconstructions, artificial skin and haemostatic dressings.
- Cellulose (dialysis membranes and cement for hip prostheses).
- Chitin extracted from crab shells (sutures, reconstructive surgery and artificial skin).
- Fucans extracted from seaweed (anti-coagulants).

INERT (or non-living) **BIOMATERIALS**
3 categories exist:
1. Metals and metal alloys
2. Ceramics (also called "bioceramics")
3. Polymers

1. Metals and metal alloys

• **Stainless steels,** which are steels (maximum 1% carbon) with at least 12% chromium content by mass. It is this chromium content that makes the steel stainless.
The types of stainless steel used in the biomedical industry are so-called "martensitic" stainless steels (magnetic and heat-treated) for surgical instruments and so-called "austenitic" stainless steels (non-magnetic and with a nickel content between 10 and 14%) for surgical implants.
Stainless steel is the most common metal used in the medical field, particularly austenitic stainless steel 316L (X2CrNiMo17-12-2).

• **Titanium and its alloys** bring very beneficial characteristics in addition to biocompatibility. Their corrosion resistance is exceptional (higher than stainless steel) and they have better fatigue properties and better elasticity than cobalt-chromium alloys and stainless steels. The density of titanium is also a big factor, since its value is low for a metal (4.5 compared to about 8 for stainless steels).
The applications of titanium in the medical field are very vast: implants, osteosynthesis, orthopaedics, prosthesis, etc... A very common grade of titanium is TA6V (TiAl6V4).

• **Cobalt-chromium alloys (stellites),** which are cobalt alloys with chromium as the main alloying element. They frequently have a high molybdenum content and a low carbon content (non-magnetic alloys).
The corrosion resistance of these alloys is excellent. Their mechanical properties are equally remarkable (high hardness, toughness and wear resistance), giving these alloys higher rigidity than stainless steels and titanium.
CoCr alloys are mainly used for hip, knee or rachis prostheses, for osteosynthesis and for dental prosthesis.

2. Ceramics

There are two types of ceramics in the medical field: "bio-inert" ceramics that do not interact with bone tissue and "bio-active" ceramics that create a bond between the material and human bone.

• **Alumina (Al_2O_3),** is a pure, dense medical-grade bio-inert ceramic. It is mainly used for its good tribological properties, ageing and mechanical resistance. It is used for the tips of hip prosthesis and in dentistry (dental implants).

• **Zirconia (ZrO_2)** has the same characteristics as alumina but with higher toughness, i.e. it has a better resistance to crack propagation.

• **Hydroxyapatite (PAH) $Ca_{10}(PO_4)_6(OH)_2$ and tricalcium phosphate β (TCP) $Ca_3(PO_4)_2$** are bioactive ceramics. These ceramics are porous, which makes them osteo-oconductive, i.e. they promote bone regrowth. They also have the advantage of being bioresorbable and are used for implants, orthopaedic surgery and dental fillings.

• **Bioglass** is a bioactive ceramic with a carbonated hydroxyapatite layer on its surface, which is chemically and structurally identical to the mineral phase of bone. A link can be made between the ceramic and bone, making osteoconduction and osteoproduction possible. Bioglasses are mainly composed of oxides, silicon (SiO_2), sodium (Na_2O), calcium (CaO) and phosphorus (P_2O_5).

3. Polymers

There are many applications for polymers in the field of biomaterials. In addition to their biocompatibility, polymers have the advantage of having easily-modulated mechanical properties according to their composition, their crystallinity rate, their shaping, etc.
The following polymers stand out:

• **Functional polymers** are used as friction surfaces (in addition to metals and ceramics), as anchoring materials for prosthesis (allowing better convalescence) and in ophthalmology, neurosurgery, cardiovascular or plastic surgery (catheters, drains, syringes, prosthesis).

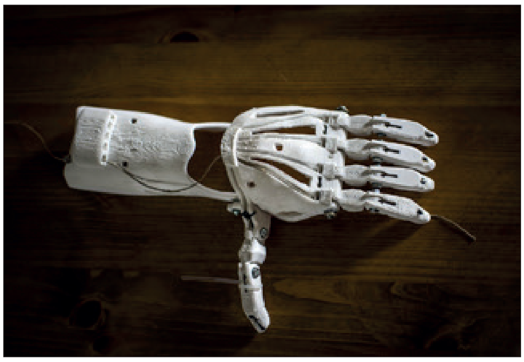
• **Resorbable polymers** make it possible to avoid further surgery. They must have sufficient mechanical properties to ensure their functions and then be resorbed afterwards. They are therefore used as surgical cements, bone fillers, diaphyseal obturators, suture threads, etc.

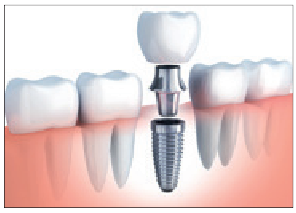
=> All of these materials make it possible to preserve the physical condition and comfort people with deficiencies. The manufacture of mobility assistance devices able to replace the functions of injured organs is now possible. Further research and medical developments are still very promising. This is a major challenge because of possible future progress and innovations.

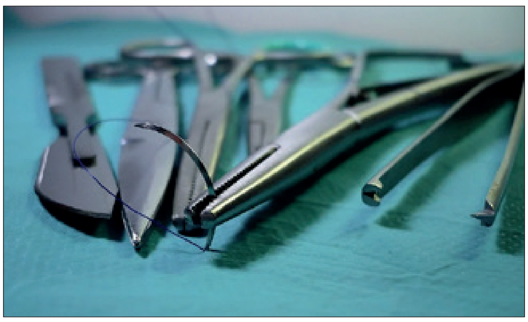
These biomaterials are developed and used in such a way that they are not rejected by the host's organism, do not contain toxic elements and offer precise mechanical characteristics that cope with the various constraints exerted by the environment.

In other words, they must comply with very demanding specifications in terms of physico-chemical properties, shaping, service life and/or deterioration, porosities, implantation or injection, etc.

It is for these same reasons that a multitude of tests must be carried out, some of which require metallographic preparation.







METALLOGRAPHIC PREPARATION

Whatever materials are being inspected, quality controls must be performed during the processes and production of workpieces. In general, processing and transformation operations and the various mechanical and thermal treatments influence the properties and microstructures of titanium and its alloys. All these influences then lead to the realization of metallographic quality controls such as: microstructure examinations, porosity and/or heterogeneity research, inclusion cleanliness, hardness tests, hardening controls, grain size controls, etc.

Obtaining an inspection surface requires a succession of operations, each as important as the next, regardless of the material. These steps are in the following order:

- The removal of the product to be examined (if necessary), called "CUTTING".
- Standardisation of the geometry of the sample taken (if necessary), called "MOUNTING".
- Improvement of the surface condition of this sample, called "POLISHING".
- Characterisation of the sample: revealing the microstructure of the sample by an etching reagent (if necessary) called "METALLOGRAPHIC ETCHING" and microscopic observation (optical or electronic).

=> Each of these steps must be carried out rigorously, otherwise the following steps will not be possible.

CUTTING

The purpose of cutting is to remove a precise section of a product, in order to obtain a suitable surface for inspection, without altering its physico-chemical properties.
In other words, it is essential to avoid heating or any deformation of the metal that could lead to degradation of the material. Cutting is a fundamental step which conditions the further preparation and inspection of parts.

PRESI's wide range of medium and large capacity cutting and micro-cutting machines can be adapted to any need with regard to cutting precision, sizing or quantity of products to be cut:

Fig 1: MECATOME T202

Fig 2: MECATOME T260

Fig 3: MECATOME T330

Each of the cutting machines in the range is equipped with the appropriate consumables and accessories. The clamping system and the choice of these consumables are always essential elements for the success of a metallographic cut.

=> Clamping, i.e. holding the workpiece, is also essential. Indeed, if the workpiece is not well held, the cut can present risks for the consumable, the workpiece and the machine.

CONSUMABLES

All cutting machines are used with a lubricating/cooling liquid composed of a mixture of water and anti-rust additive in order to obtain a clean cut without overheating. The additive also protects the sample and the machine from corrosion.

	Stainless steel	Titanium	Cobalt Chrome	Ceramics	Polymers
Micro-cutting	UTW S Ø180 AO AOF II	UTW S Ø180 MNF	UTW S Ø180 CBN	LM / LM+ LR	UTW S Ø180 MNF LM+ LR
Medium-capacity cutting	A AO AOF II	T MNF F	S CBN	LM / LM+ LR	MNF LM+ LR
High-capacity cutting	A AO	T MNF	S CBN	LM / LM+ LR	MNF LM+ LR

Table1: Choosing the right cut-off wheel type

=> The choice of the cut-off wheel type has to be adequate, in order to avoid cutting failure or excessive cut-off wheel wear or even breakage. The hardness of the workpiece determines the wheel selection.

MOUNTING

Samples can be difficult to handle due to their complex shape, fragility or small size. Mounting makes them easier to handle by standardising their geometry and dimensions.
=> Achieving good-quality mounting is essential to protect fragile materials and also to achieve good preparation results for polishing and future analysis.

Before mounting, the specimen should be deburred with coarse abrasive paper, for example, to remove any cutting burrs. Cleaning with ethanol (in an ultrasonic tank for even greater efficiency) is also possible. This allows the resin to adhere as well as possible to the sample and thus limits shrinkage (space between the resin and the sample).

If shrinkage persists, it can lead to problems during polishing. Abrasive grains may become lodged in this space and then be released at a later stage, thus creating a risk of pollution for the sample and the polishing surface. In this case, cleaning with an ultrasonic cleaner between each step is recommended.

<u>There are two mounting options</u>:

• **<u>HOT MOUNTING</u>** is to be preferred for edge inspection purposes or if the metallographic preparation is carried out in preparation for hardness testing. **This option requires a hot-mounting machine.**

Fig 4: MECAPRESS 3

<u>The machine required for hot-mounting is the Mecapress 3</u>:
- The Mecapress 3 is a fully automatic hot mounting press.
- It is easy to use, its memory, process adjustment and speed of execution make it a high-precision machine.
- The hot-mounting machine has 6 different mould diameters from 25.4-50mm.

+ POINT
One of the main advantages of this process is that it provides perfectly parallel faces.

- **<u>COLD MOUNTING</u>** Is to be preferred:
- If the parts to be examined are fragile/sensitive to pressure
- If they have a complex geometry such as a honeycomb structure.
- If a large number of parts are to be mounted in series.

The cold process can be used with:

Fig 5: Pressurized mounting device

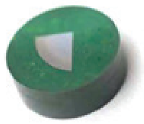
+ POINT
Substantially improves quality, in particular by reducing shrinkage, optimising transparency and facilitating resin impregnation.

Fig 6: Vacuum mounting device: POLY'VAC

+ POINT
Machine allowing vacuum impregnation of porous mounted materials using an epoxy resin.

Cold resins do not always provide a flat mounting "back" because of the meniscus of the liquid resin. Before any polishing operation, a brief step using abrasive paper will remove this meniscus. The important thing is to ensure that this operation renders the two sides of the mounting parallel.

CONSUMABLES
To meet user needs, PRESI offers a full range of cold mounting moulds with diameters from 20-50mm. These are divided into several types: optimised moulds called "KM2.0", rubber, Teflon or polyethylene moulds. Cold mounting is also more flexible, hence the existence of rectangular moulds for more specific needs.



	Stainless steel	Titanium	Cobalt Chrome	Ceramics	Polymers
Hot process	Hot epoxy Phenolic Acrylic Allylic	Hot epoxy Phenolic Allylic	Hot epoxy Phenolic Allylic	Ø	Ø
Cold process	KM-U KM-B IP / IP-FAST 2S*	KM-U KM-B IP / IP-FAST 2S*	KM-U KM-B IP / IP-FAST 2S*	KM-U KM-B IP / IP-FAST	KM-U KM-B IP / IP-FAST MA2+

Table 2: Choosing the right mounting resin type

* Suitable for very large series

Ceramics and composites are brittle materials and are sensitive to heat and/or pressure.
It is therefore not recommended to perform a hot mounting process with this type of material.

POLISHING

The last and crucial phase in the sample preparation process is polishing. The principle is simple, each step uses a finer abrasive than the previous one. The aim is to obtain a flat surface and to eliminate scratches and residual defects that would hinder the performance of metallographic control examinations such as microscopic analysis, hardness tests, microstructure or dimensional inspections.

PRESI offers a wide range of manual and automatic polishing machines, with a wide choice of accessories, to cover all needs, from pre-polishing to super-finishing and polishing of single or series samples.

Fig 7:
Le CUBE 250

Fig 8:
MINITECH 300 SP1

Fig 9:
MECATECH 250 SPI

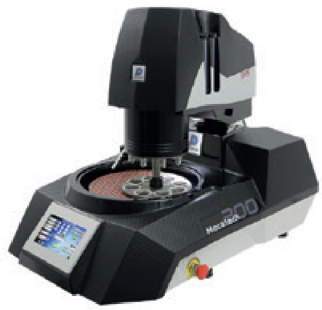


Fig 10:
MECATECH 300 SPS

The MINITECH range of manual polishers incorporates the most advanced technologies.
User-friendly, reliable and robust, they provide a simple answer to all needs.

The MECATECH range of automatic polishers allows both manual and automatic polishing. With its advanced technologies, motor power from 750-1500 W, all the PRESI experience is concentrated in this very complete range. Whatever the sample number or size, MECATECH guarantees optimal polishing.

CONSUMABLES AND POLISHING RANGE

All the polishing ranges below are given for automatic sample preparation (for manual polishing: do not take into account the parameters at the top). They are the most commonly used and are given for information and advice.

All the first steps of each range are called "levelling" and consist of removing material quickly to level the surface of the sample (and resin). Those given below are standard and can therefore be modified as required.

Applied pressures vary according to sample size, but in general the following applies: 1daN per 10mm mounting diameter for the pre-polishing steps (ex: Ø40mm = 4 daN) then reduce force by 0.5daN at each polishing step with an abrasive suspension.

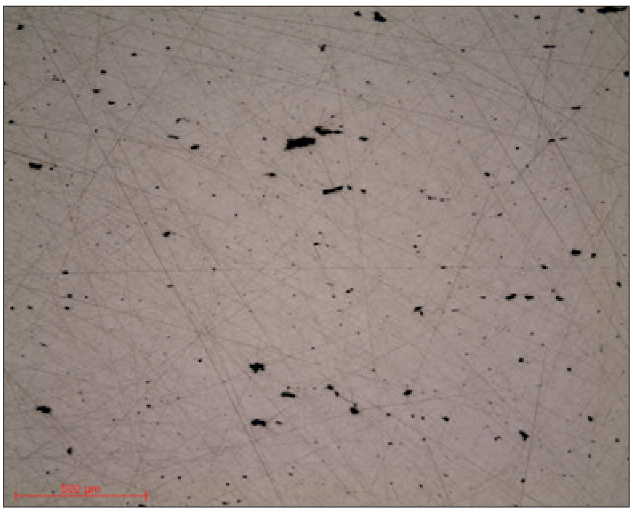
Range	N°1	N°2	N°3	N°4
Materials	Stainless steel; Cobalt-Chrome	Titanium Cobalt-Chrome	Ceramics	Polymers

Table N°3: Choice of polishing range

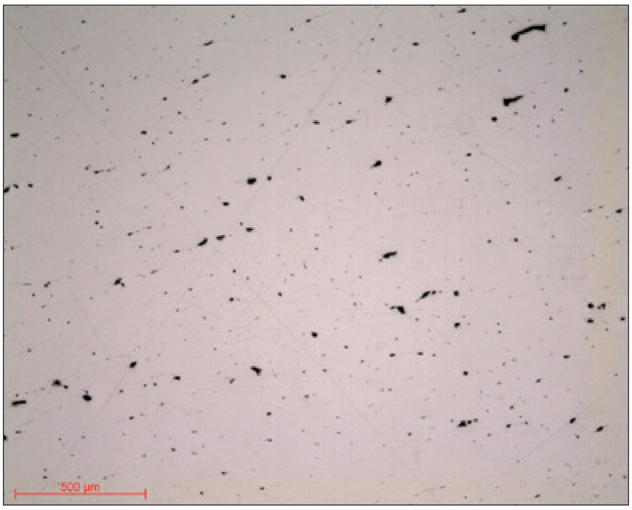
Range N°1

N°	Support	Suspension / Lubricant	Platen speed (RPM)	Head speed (RPM)	Rotation direction platen / head	Time
1	SiC P320	Ø / Water	300	150	→ →	1'
2	SiC P1200	Ø / Water	300	150	→ →	1'
3	RAM	3µm LDP / Reflex Lub	150	135	→ →	2'
4	NT	1µm LDP / Reflex Lub	150	135	→ →	1'
5	NT	Al_2O_3 n°3 / Water	150	100	→ ←	1'

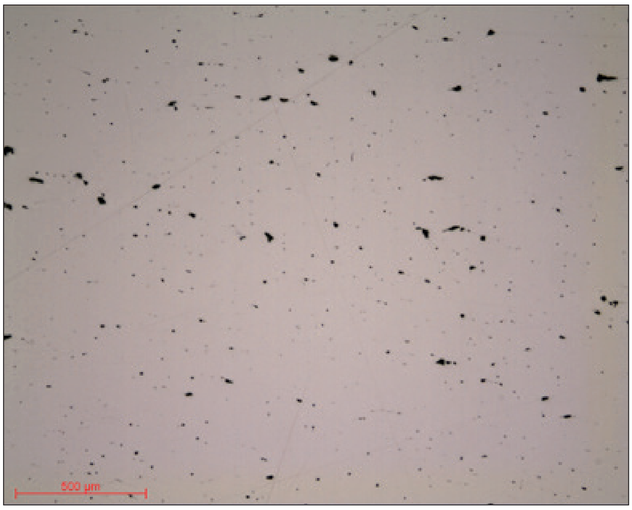
Micrograph 1: Surface condition P320 lens x5



Micrograph 2: Surface condition TOP 9 µm lens x5



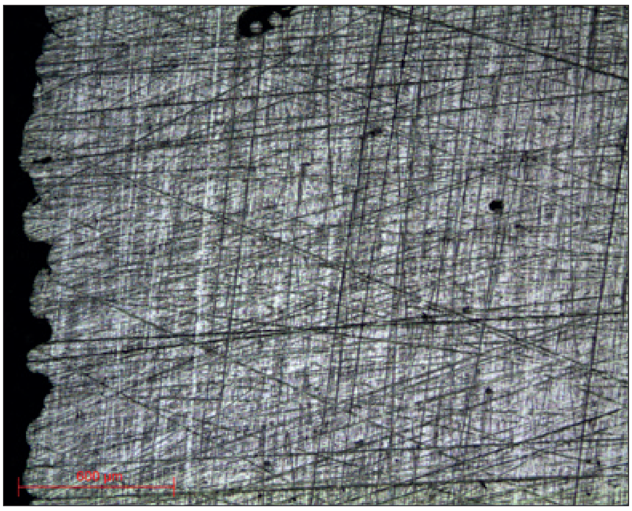
Micrograph 3: Surface condition RAM 3µm lens x5



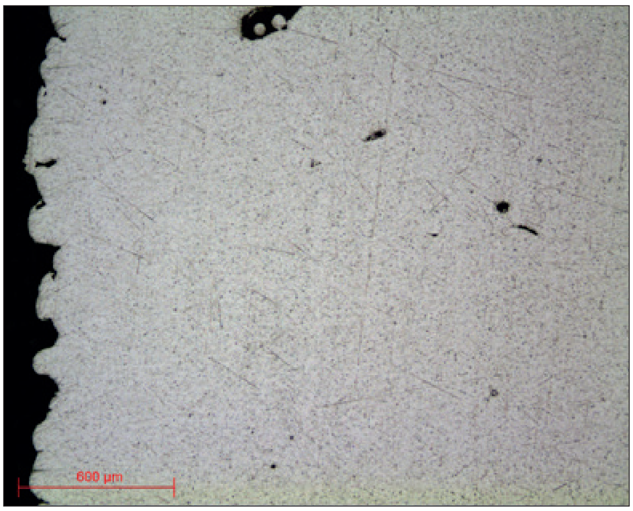
Micrograph 4: Surface condition NT 1µm lens x5

Range N°2

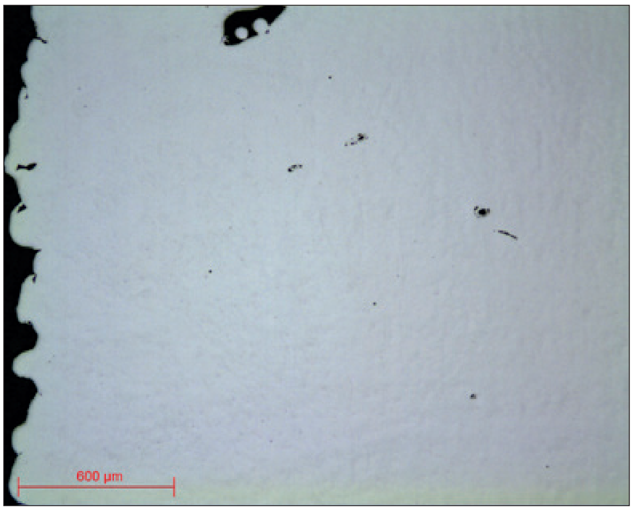
N°	Support	Suspension / Lubricant	Platen speed (RPM)	Head speed (RPM)	Rotation direction platen / head	Time
1	SiC P320	Ø / Water	300	150	→ →	1’
2	TOP	9µm LDP / Reflex Lub	150	135	→ →	5’
3	SUPRA	SPM / Water	150	100	→ ←	5’



Micrograph 5: Surface condition P320 lens x5



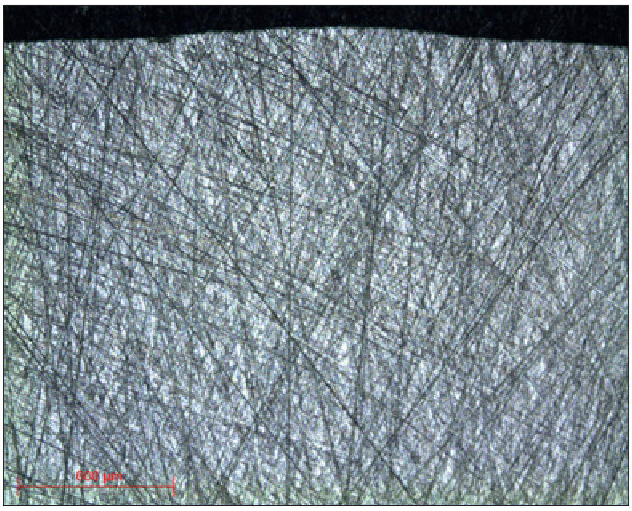
Micrograph 6: Surface condition TOP 9µm lens x5



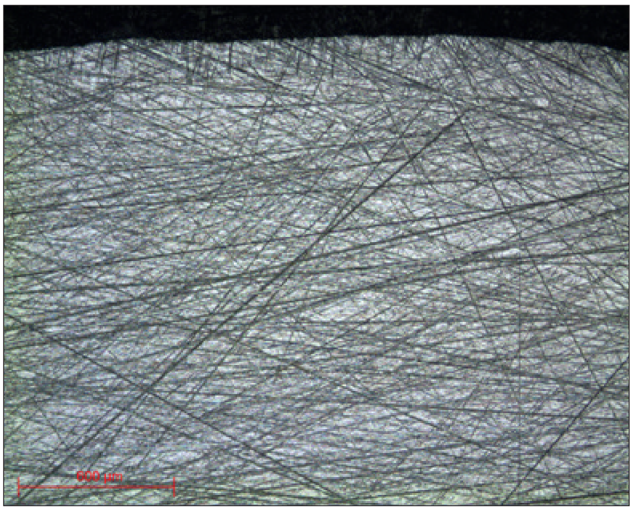
Micrograph 7: Surface condition SUPRA SPM lens x5

Range N°3

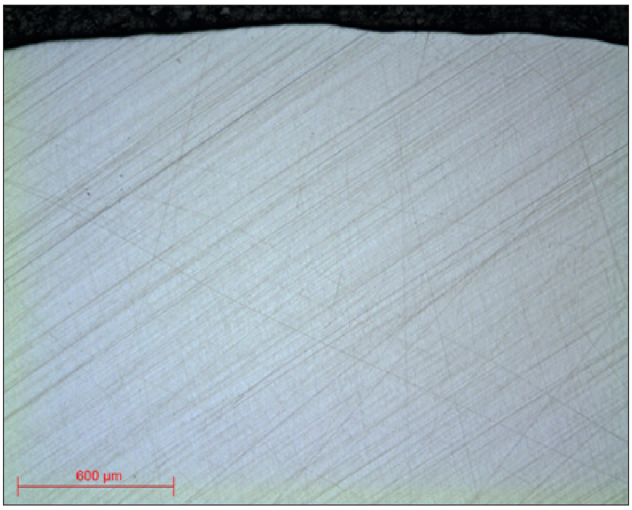
N°	Support	Suspension / Lubricant	Platen speed (RPM)	Head speed (RPM)	Rotation direction platen / head	Time
1	Tissediam 40µm	Ø / Water	300	150	→ →	2'
2	Tissediam 20µm	Ø / Water	300	150	→ →	2'
3	TOP	9µm LDP / Reflex Lub	150	135	→ →	5'
4	NWF+	3µm LDP / Reflex Lub	150	135	→ →	2'
5	SUPRA	SPM / Water	150	100	→ ←	2'



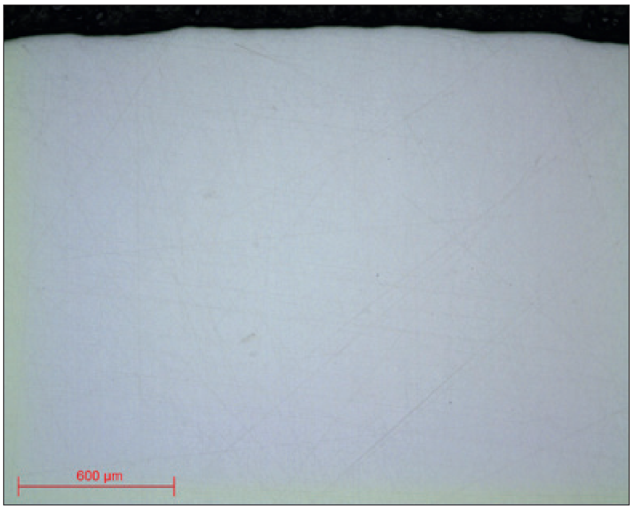
Micrograph 8: Surface condition TISSEDIAM 40µm lens x5



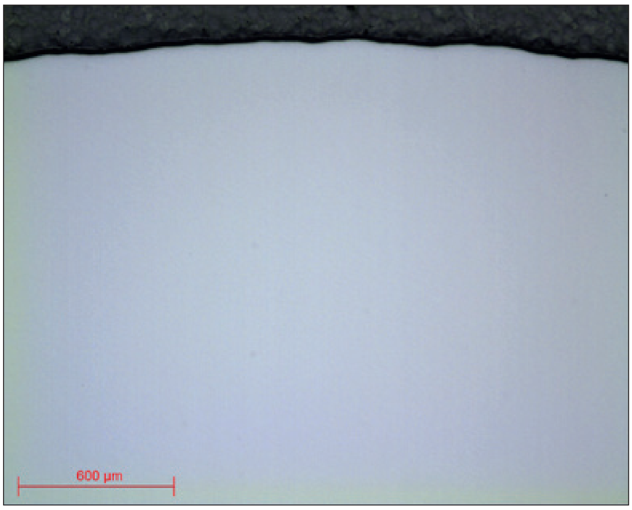
Micrograph 9: Surface condition TISSEDIAM 20µm lens x5



Micrograph 10: Surface condition TOP 9µm lens x5



Micrograph 11: Surface condition NWF+ 9µm lens x5



Micrograph 12: Surface condition SUPRA SPM lens x5

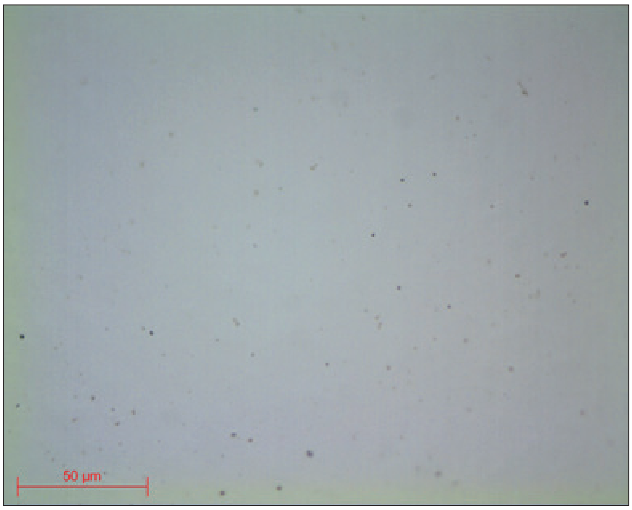
Range N°4

N°	Support	Suspension / Lubricant	Platen speed (RPM)	Head speed (RPM)	Rotation direction platen / head	Time
1	Sic P320	Ø / Water	300	150	→ →	1'
2	Sic P1200	Ø / Water	300	150	→ →	1'
3	STA	3µm LDP / Reflex Lub	150	135	→ →	5'
4	NT	Al_2O_3 n°1 / Water	150	100	→ ←	1'

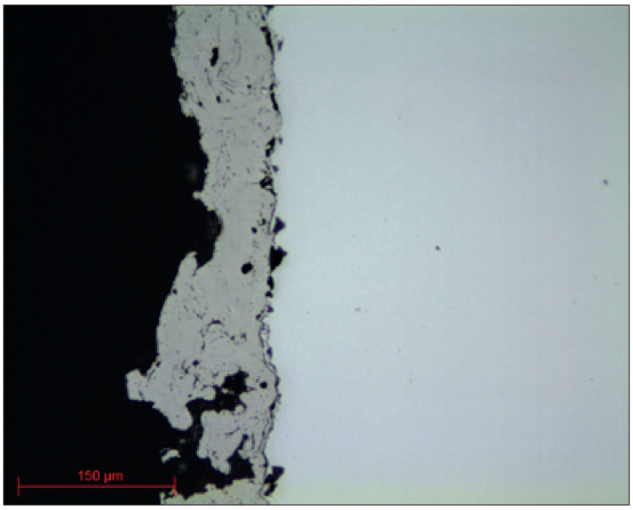
All the polishing ranges listed above are standard and versatile ranges that can be modified according to the subtleties of the samples. (Cf Lab'Notes of the material concerned for more information). Moreover, they are not necessarily to be carried out in their entirety; observations will define needs (except for titanium samples for which all the steps of the range must be performed).
At the end of this preparation phase, the polished samples can be directly observed without metallographic etching. Otherwise, metallographic etching allows differences in relief and/or colour to be made between the different components and therefore allows them to be observed. It is mainly used on metals (see Lab'Notes on the material concerned).

MICROSCOPY

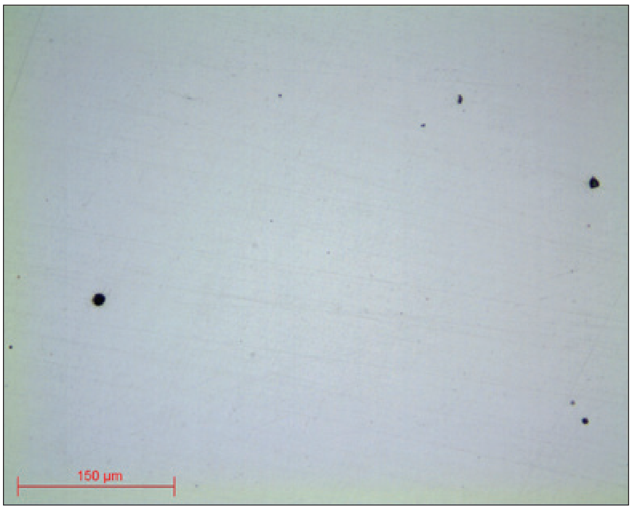
All micrographs presented were created using the **PRESI VIEW** software:



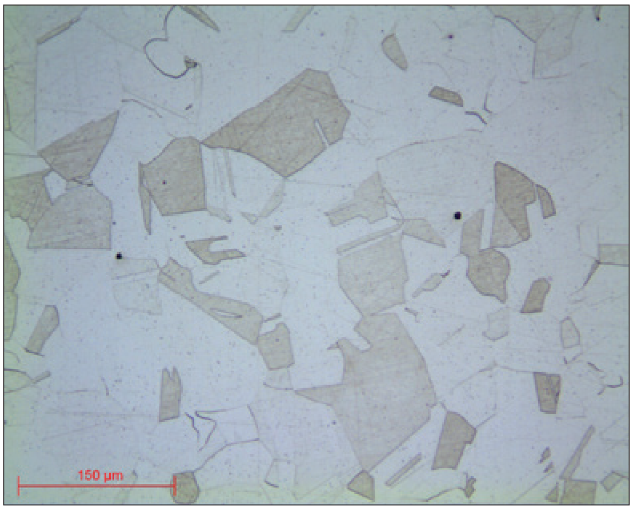
Micrograph 13: Cobalt-Chrome lens x50



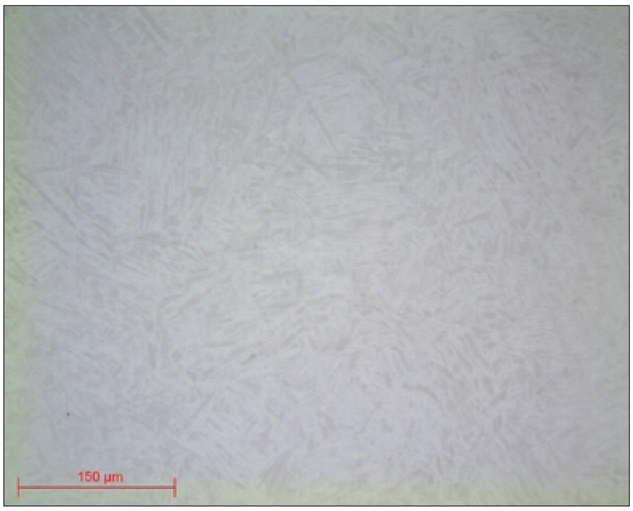
Micrograph 14: Cobalt-Chrome with titanium deposit lens x20



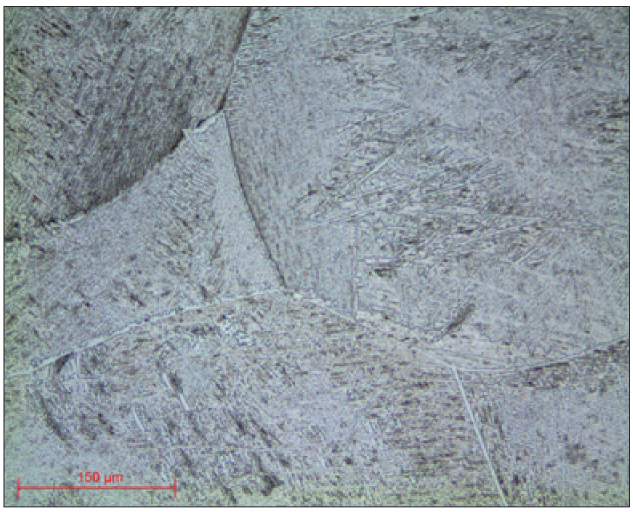
Micrograph 15: Stainless steel lens x20



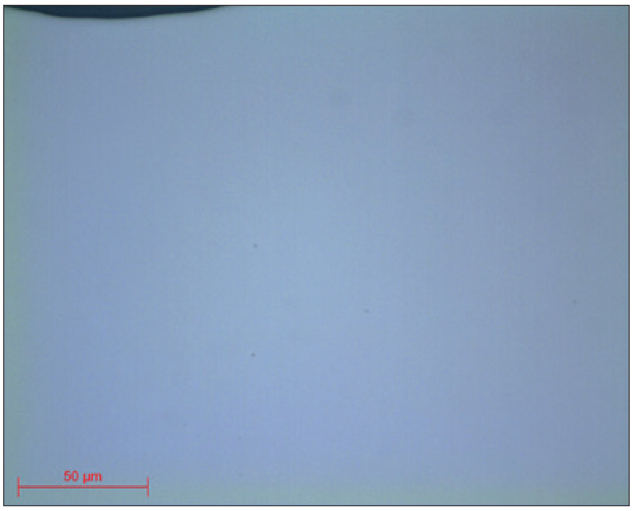
Micrograph 16: Stainless steel etched with ADLER reagent lens x20



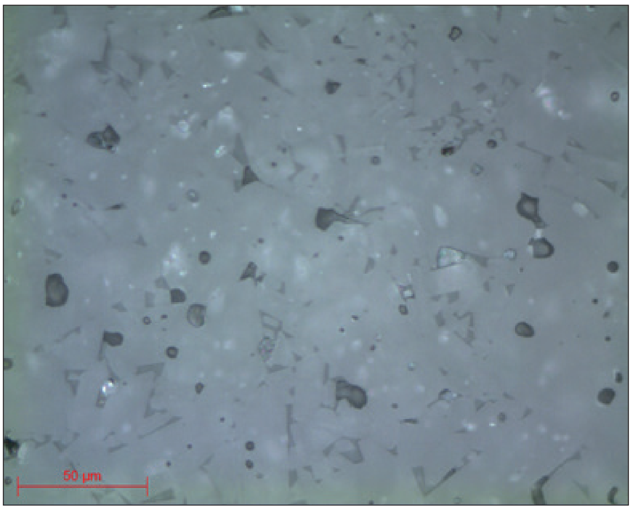
Micrograph 17: Titanium lens x20



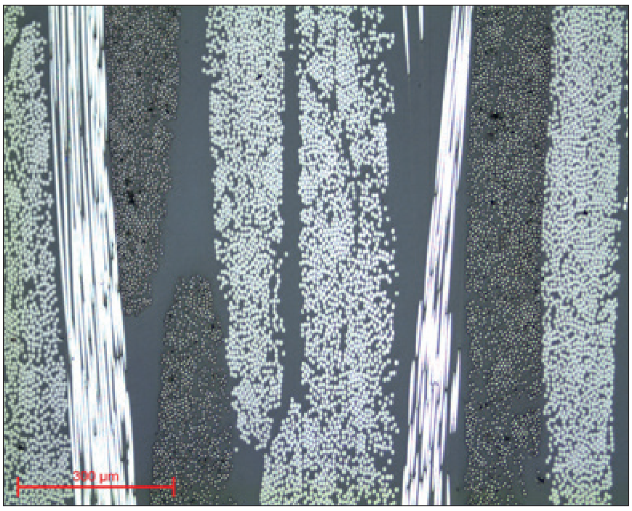
Micrograph 18: Titanium etched with KROLL reagent lens x20



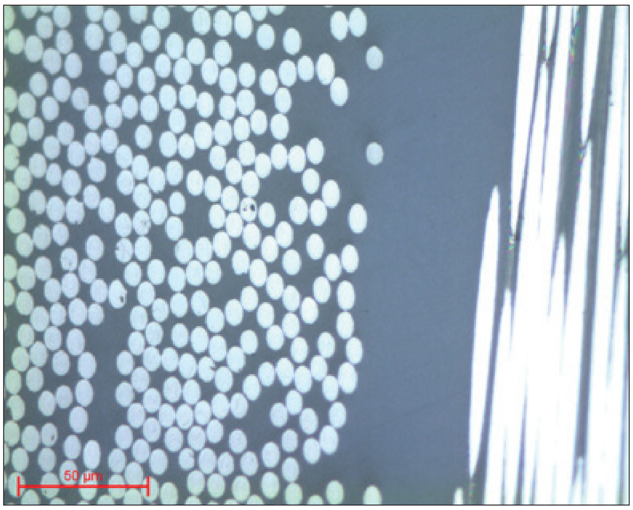
Micrographic 19: Zirconium (ZrO_2) lens x50



Micrographic 20: Alumina (Al_2O_3) lens x50



Micrographic 21: Composite polymers lens x10



Micrographic 22: Composite polymers lens x50