PRESI LAB'NOTE

METALLOGRAPHIC PREPARATION OF **PRECIOUS METALS**

INTRODUCTION

A precious metal is defined as a natural metallic chemical element that is rare and has a very high economic value. The physical and chemical properties are also important for the determination of a precious metal. They have good corrosion resistance, are ductile and have a brilliant appearance.

The three best known precious metals are gold, silver and platinum. Palladium can be added to this list depending on the country. By extension, some metals of the platinum family are considered precious metals such as rhodium, ruthenium, osmium and iridium.

These metals are rare due to their low presence in the earth's crust.
Precious metals have the particularity of being listed on the stock exchange. They exist therefore as investments, but also industrial raw materials.

To sum up, a metal is precious if it is rare, if its demand is strong and if its market value is high.

GOLD
Symbol: Au
Atomic n°: 79
Density: 19,3
Molar mass: 197 g.mol-1
Melting point: 1063°C

SILVER
Symbol: Ag
Atomic n°: 47
Density: 10,5
Molar mass: 107,9 g.mol-1
Melting point: 961°C

PLATINUM
Symbol: Pt
Atomic n°: 78
Density: 21,1
Molar mass: 195,1 g.mol-1
Melting point: 1770 °C

THE MAIN PRECIOUS METALS

GOLD

It is a metal that has been used since ancient times. It exists in pure form in its natural state or as an alloy. It is very ductile and malleable, which makes it very easy to work with. It is yellow in colour, and after polishing has a high luster.

It is chemically stable under ambient conditions. It does not oxidize in air or water. Gold is mainly used in art and jewellery. However, it is rarely used in its pure form in the making of jewellery.

Nowadays, gold is worked in the form of an alloy, which makes it rigid and allows its colour to be nuanced. Gold can be alloyed with silver, copper, platinum or palladium. These alloys will give different shades to the gold which will no longer be golden and bring other properties such as less ductility.

It is used in electronics for its good resistance to corrosion and its high electrical and thermal conductivity. It is notably used in microprocessors. Gold is also used in medicine, particularly in dentistry.

SILVER

Silver is extracted from copper, lead, zinc and gold ores. This white, shiny precious metal is, like gold, malleable and ductile. It also has great reflective power.

Silver is widely used in the field of solid metal jewellery or as a coating by electroplating. To reinforce its mechanical characteristics, silver is sometimes alloyed with copper. It can tarnish, especially in the presence of sulphide.

Silver has long been used for coinage. It is still used for this application today but mainly for collector coins or medals.
Silver is often used in the field of electronics and electricity. It a very good thermal and electrical conductor.

In the field of photography, silver (in the form of silver halide) was used in the early 1990s. Now, with the development of digital cameras, it is used much less.
Silver is also used in chemistry, optics, etc.

PLATINUM

This grey-white, shiny metal is found in its pure form, or as an ore with copper or nickel. Platinum is resistant to corrosion and oxidation at high temperatures. It is unalterable, and, like gold and silver, is ductile and malleable. This metal has both technical and decorative qualities.

Platinum can be deposited on surfaces by electroplating. This is why in the medical field it is used to cover surgical tools or to make certain prostheses or pacemakers. Platinum can also be found in dental prostheses.

Platinum is also used in the automotive industry (for catalytic converters) together with palladium and rhodium. Platinum crucibles and pots are used in chemical and pharmaceutical laboratories.

Platinum in salt form (carboplatin and cisplatin) is used in the treatment of certain cancers. Like silver and gold, it is an essential element in the making of jewellery. In jewellery, it can be 95% pure (compared to 75% for a gold alloy). As platinum is rarer than gold, it is also much more expensive.

METALLOGRAPHIC PREPARATION

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 strain hardening. 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 T205

Fig 2: MECATOME T215

For cutting precious metals, it is generally advisable to use micro-cutting machines, which are adapted to the dimensions of the precious metal samples. The Mecatome T205 or even the Mecatome T215 are both suitable for cutting jewellery pieces for example.

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. Figures 3 to 6 show examples of clamping for jewellery parts.

Fig. 3-4-5-6: Jewellery clamping

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.

	PRECIOUS METALS
Micro-cutting	S Ø180 mm UTW
Medium-capacity cutting	MNF
High-capacity cutting	MNF

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.

There are two mounting options:

• **HOT MOUNTING** 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 7: MECAPRESS 3

The machine required for hot-mounting is the Mecapress 3:
• Fully automatic hot-mounting press.
• Easy to use: memorisation, adjustment of processes 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.

• **COLD MOUNTING** 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 8 : Pressurized mounting device

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

Fig 9: 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. The cold process has different 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.

	PRECIOUS METALS
Hot process	Phenolic Allylic glass fibre
Cold process	KM-U Ma2+

Table 2: Choosing the right mounting resin type

In the case of a coating inspection, for example, it is advisable to favour the use of a resin with low shrinkage. For a general inspection, a slightly lower quality resin will be sufficient. The choice of resin should be adapted according to the final objective of the desired observations.

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 10: MINITECH 300 SP1

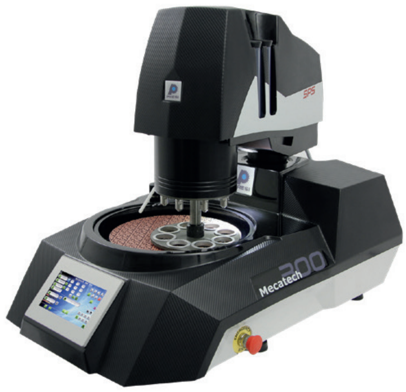

Fig 11: MECATECH 300 SPC

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.

The first range is suitable for **silver** polishing:

N°	Support	Suspension / Lubricant	Platen Speed (RPM)	Head Speed (RPM)	Rotation direction platen / head	Time
1	P600 SiC	Ø / Water	250	80	→ →	1'
2	P1200 SiC	Ø / Water	250	80	→ →	1'
3	ADR II	9µm LDP / Reflex Lub	400	80	→ →	2'
4	ADR II	3µm LDP / Reflex Lub	400	80	→ →	2'
5	RFI	Alumina n°2 / Water	200	80	→ ←	1'

Silver polishing is performed applying a rather atypical range.
The first steps are carried out using medium to fine grit abrasive papers. The speeds of rotation are also particular in order to limit the incrustation of abrasives in the material.

Steps 3 and 4 are made using ADRII cloth. This cloth has flexible fibres, which polish silver best.

Finally, the last step is an RFI cloth which is used with a suspension of Alumina Presi n°2. During this step, it is advisable to moisten the cloth for a few seconds beforehand. A rinse at the end of the step is preferable in order to clean the cloth and the sample from the Alumina suspension.
The rotation of the head is reversed in relation to the platen in order to keep the suspension on the cloth as much as possible.

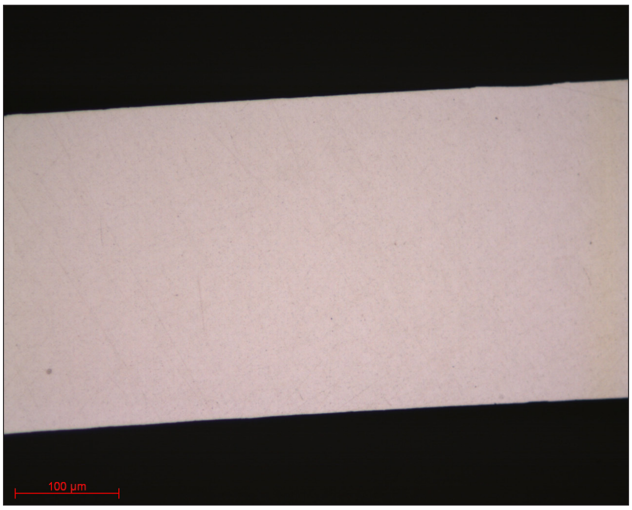

Fig. 12: Silver, alumina finish lens x20

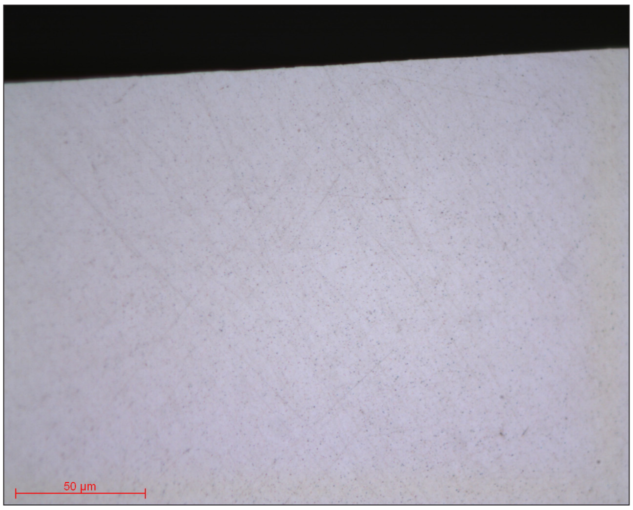

Fig. 13: Silver, alumina finish lens x50

The range below was developed for a copper sample with **silver deposit**:

N°	Support	Suspension / Lubricant	Platen Speed (RPM)	Head Speed (RPM)	Rotation direction platen / head	Time
1	P1200 SiC	Ø / Water	300	150	→ / →	1'
2	TOP	9µm LDM / Reflex Lub	150	135	→ / →	2'
3	RAM	3µm LDM / Reflex Lub	150	135	→ / →	2'
4	TFR	1µm LDM / Reflex Lub	150	135	→ / →	1'
5	SUPRA	SPM / Water	150	100	→ / ←	1'

The first step is again carried out using fine abrasive paper. Here, the range is oriented for copper polishing: we find the use of monocrystalline diamond suspension less aggressive for copper.
The last step is carried out using a colloidal silica suspension (SPM). Implementation is identical to that of Alumina.

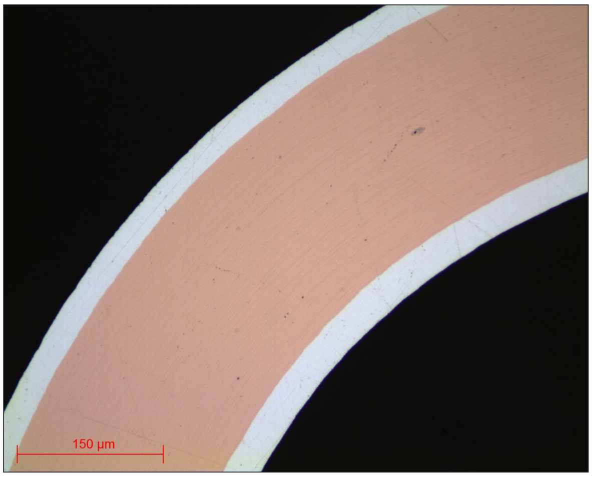

Fig. 14: Cu deposit, silver finish SPM lens x20

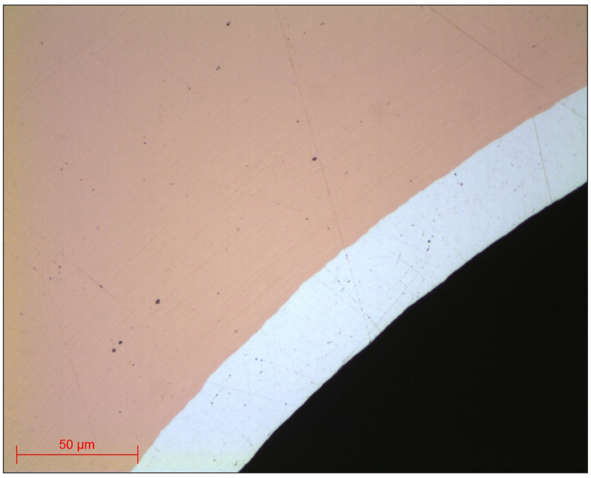

Fig. 15: Cu deposit, silver finish SPM lens x50

The range below is suitable for polishing **gold (in the form of a deposit)**:

N°	Support	Suspension / Lubricant	Platen Speed (RPM)	Head Speed (RPM)	Rotation direction platen / head	Time
1	P600 SiC	Ø / Water	300	150	→ / →	1'
2	P1200 SiC	Ø / Water	300	150	→ / →	1'
3	TOP	9µm Gel 2+ poly / Ø	150	135	→ / →	3'
4	ADR II	3µm Gel 2+ poly / Ø	150	135	→ / →	2'
5	NT	1µm Gel 2+ poly / Ø	150	135	→ / →	1'
6	SUPRA	SPM / Water	150	100	→ / ←	1'

In this case, the gold is in the form of a deposit on an electronic sample. The polishing range begins with the use of abrasive papers.

It continues with polishing using Gel 2+ polycrystalline diamond suspensions on polishing cloths (TOP, ADRII and NT). These suspensions contain the diamond abrasive and the lubricant, a 2-in-1 product. Their "gel" formula allows the product to remain on the polishing cloth longer.

The last step is also done with the SPM colloidal silica suspension.

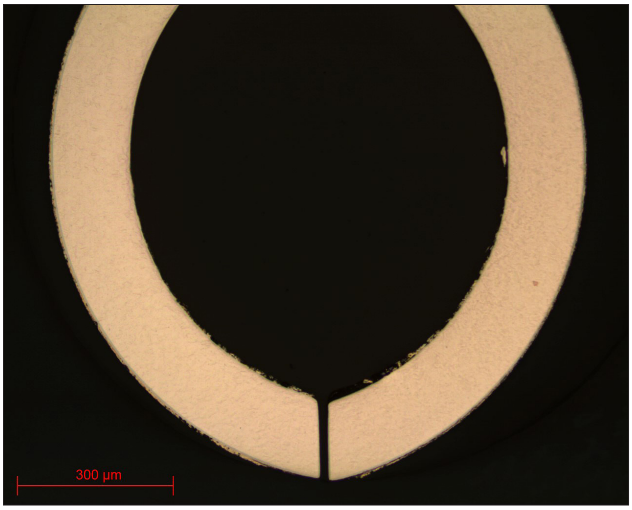

Fig. 16: Cu deposit, gold finish SPMlens x10

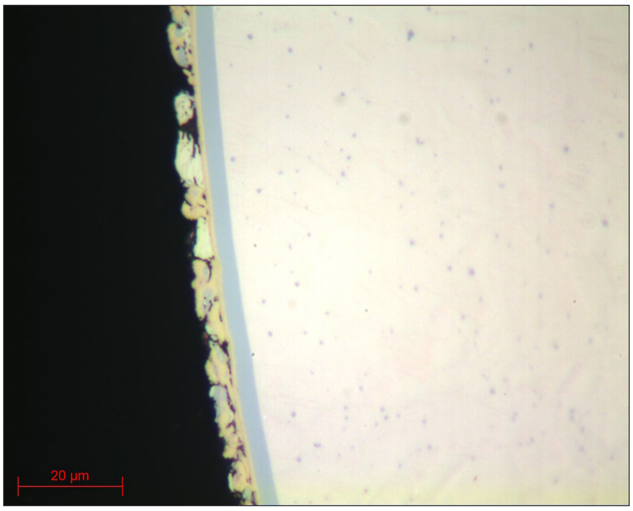

Fig. 17: Cu deposit, gold finish SPMlens x100

Gold in alloyed form can also be polished with the following range:

N°	Support	Suspension / Lubricant	Platen Speed (RPM)	Head Speed (RPM)	Rotation direction platen / head	Time
1	P1200 SiC	Ø / Water	300	150	→ →	1'
2	TOP	9µm LDM / Reflex Lub	150	135	→ →	2'
3	RAM	3µm LDM / Reflex Lub	150	135	→ →	2'
4	NT	1µm LDM / Reflex Lub	150	135	→ →	1'
5	SUPRA	SPM / Water	150	100	→ ←	2'

This polishing range is quite similar to that used for copper with silver plating.
The first step is done with a P1200 abrasive paper.
LDM 9µm, 3µm and 1µm monocrystalline diamond suspensions are used with TOP, RAM and NT polishing cloths.
The last step is carried out using colloidal silica suspension (SPM). This superfinishing step improves the surface finish for final observation.

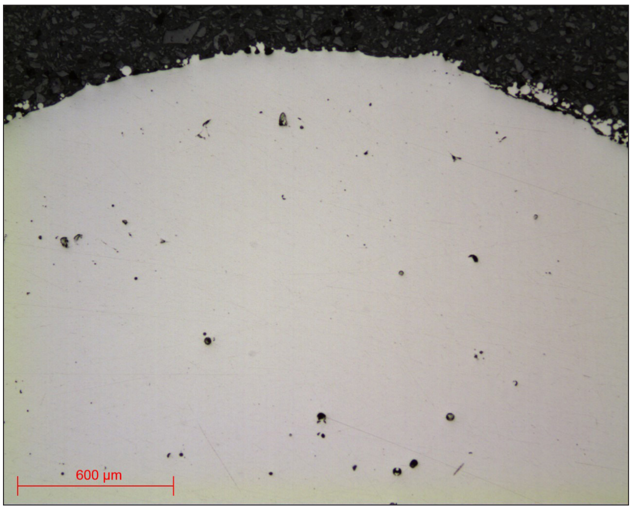

Fig. 18: Gold - SPM finish SPM lens x5

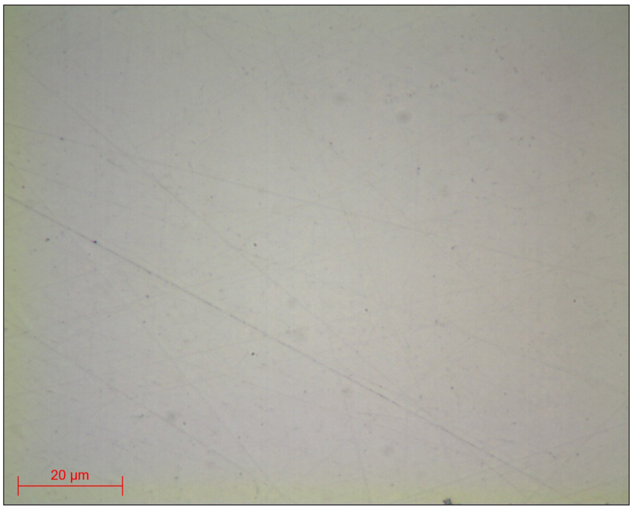

Fig. 19: Gold - SPM finish SPM lens x100