PRESI

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LAB'NOTE

METALLOGRAPHIC PREPARATION OF CUPREOUS METALS

INTRODUCTION

COPPER

Symbol: Cu
Atomic N°: 29
Density: 9.0
Molar mass: 65.3 g.mol-1
Melting point: 1085 °C

WHAT IS COPPER?

• Copper is a metal most often found in the form of rock combined with other elements (copper oxide or copper sulphide).

• A multi-stage metallurgical smelting process (Figure 1) produces pure copper.

• Low-alloyed copper also exists (4-5% alloy additions).

• Copper is a ductile material and is a good electrical and thermal conductor.

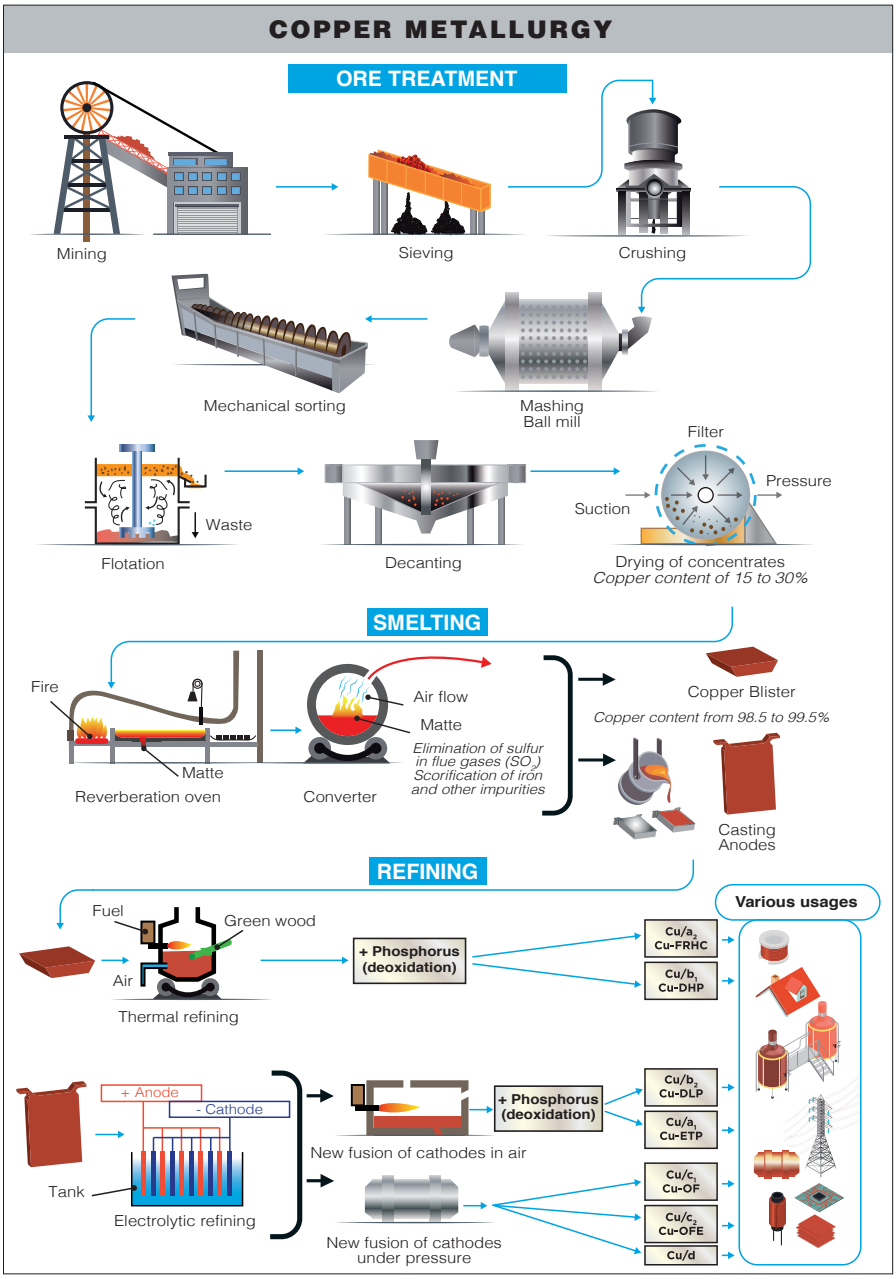
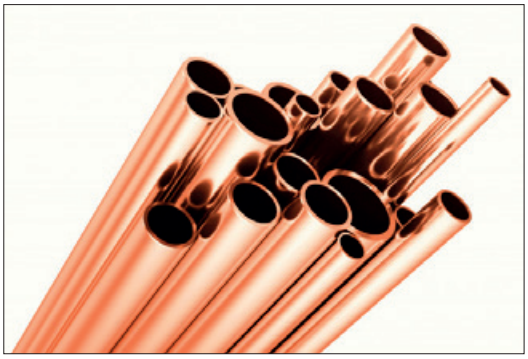
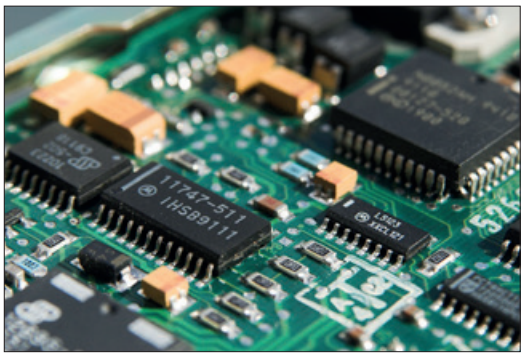


Fig. 1: Obtaining copper

The fields of application are varied: tubes, electric wires, in printed circuits, for the manufacture of kitchen utensils or heat exchangers.





MAIN COPPER ALLOYS

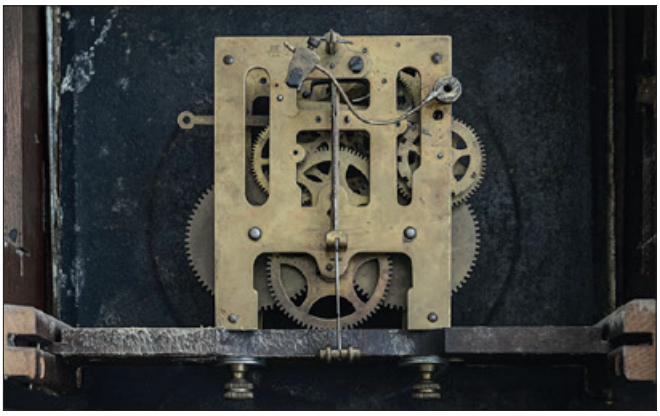
BRASS

Brass is an alloy of copper and zinc whose percentage can range from 5-40 %. This addition of zinc makes this alloy more solid, making machining easier. This alloy can be used in the screw-cutting industry, in the manufacture of parts for jewellery or watches, etc.

BRONZE

Bronze is an alloy of copper and tin. The percentage of tin can be up to 20% depending on the manufacturing process. It can also have additional elements such as lead, which allows better machinability, or phosphorus, which improves mechanical characteristics.

Bronze is more resistant than copper alone, which is why it can be used as a friction material.



METALLOGRAPHIC PREPARATION

Obtaining an inspection surface requires a series of operations, each one as important as the next and this regardless of the material. These operations come in this order:

- 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».
- Sample characterisation: to reveal 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 the physico-chemical properties of the copper.
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:

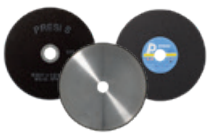
Fig 2: MECATOME T202

Fig 3: MECATOME T330

Each of the cutting machines in the range has its own customised consumables and accessories. The clamping system and choice of consumables are key factors in a successful metallographic cut.
=> Clamping, i.e. holding the workpiece, is essential. If the workpiece is not held properly, the cut can be detrimental to the cut-off wheel, 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.



	COPPER BRASS BRONZE
Micro-cutting	MNF UTW S (Ø180mm)
Medium-capacity cutting	MNF
High-capacity cutting	MNF

Table 1: 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 4: 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 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.

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.

	 **CONSUMABLES**
	COPPER BRASS BRONZE
Hot process	Phenolic Allylic
Cold process	KM-U 2S Ma2+

Table 2: Choosing the right mounting resin type

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:
MINITECH 300 SP1

Fig 8:
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 RANGES

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 following is a general polishing range for copper and its alloys:

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 LDM / Reflex Lub	150	135	→ →	4’
3	RAM	3µm LDM / Reflex Lub	150	135	→ →	3’
4	TFR	1µm LDM / Reflex Lub	150	135	→ →	1’
5	SUPRA	SPM / Water	150	100	→ ←	1’

NB: Levelling with P320 abrasive paper is enough for a sample from a metallographic cut. If additional material removal is required, a larger grit-size should be used.

For pre-polishing, the head and platen rotation direction should not be reversed, as this can detrimentally affect flatness. However, reversing rotation direction can help if a large amount of material has to be removed.

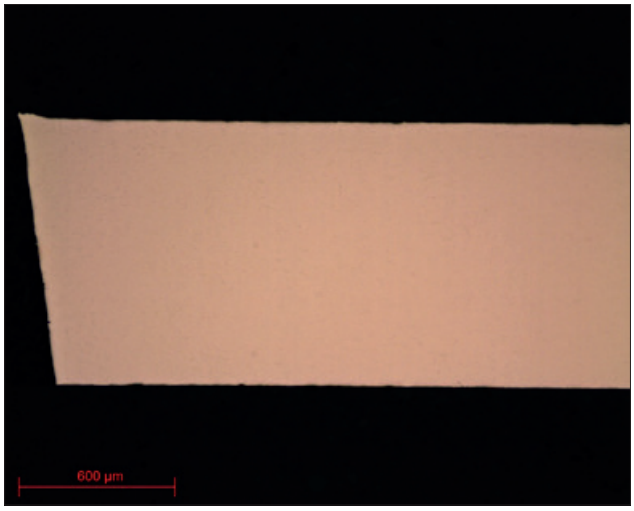


Fig. 9: Copper lens x5

Fig. 10: Copper lens x50

The important thing in copper polishing is the use of mono-crystalline diamond suspensions (LDM or Gel2+ mono) with a more rounded diamond shape (compared to polycrystalline diamond) to limit encrustations in soft materials such as copper or its alloys.

The LDM suspension is used with the Reflex LUB lubricant, which allows the fabric to be sufficiently moistened without overloading it only with diamond suspension. In any case, care must be taken not to wet the fabric too much with suspension or lubricant, so as not to «aquaplane» with the fabric, which would make it ineffective.

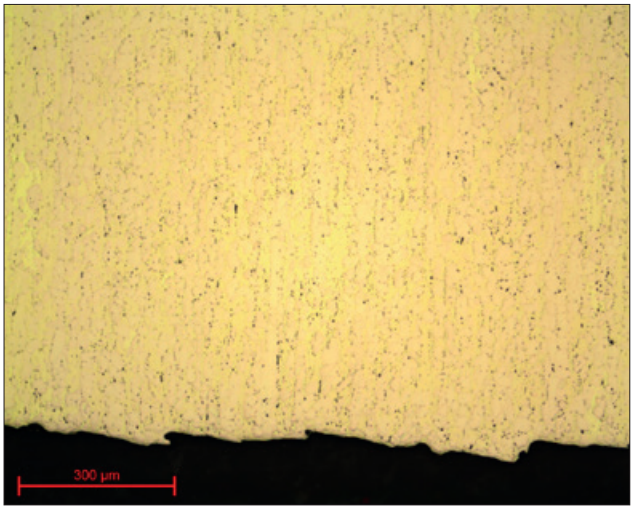


Fig. 11: Brass lens x10

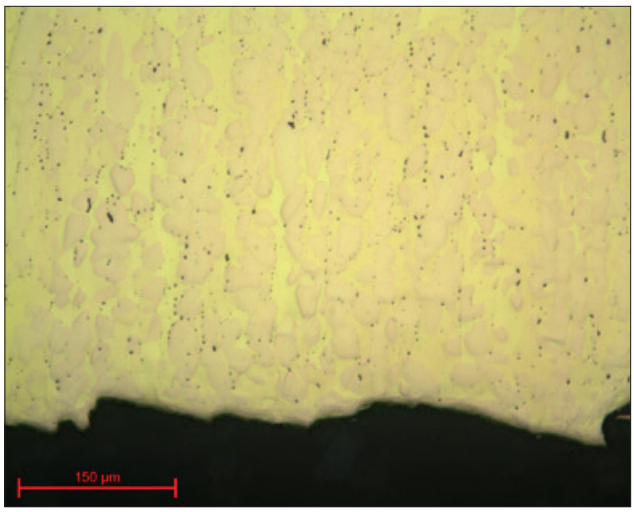


Fig. 12: Brass lens x20

The second important consideration when polishing copper is to adapt the pressure applied (manually or in semi-automatic polishing) so as not to encrust grains of abrasive paper, for example.

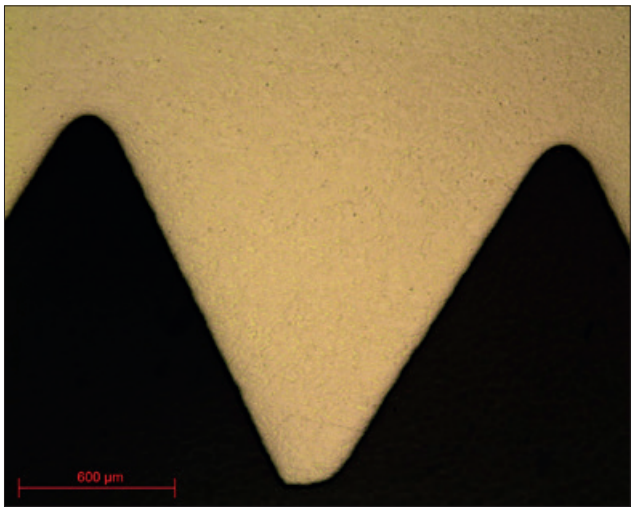


Fig. 13: Brass nut lens x5

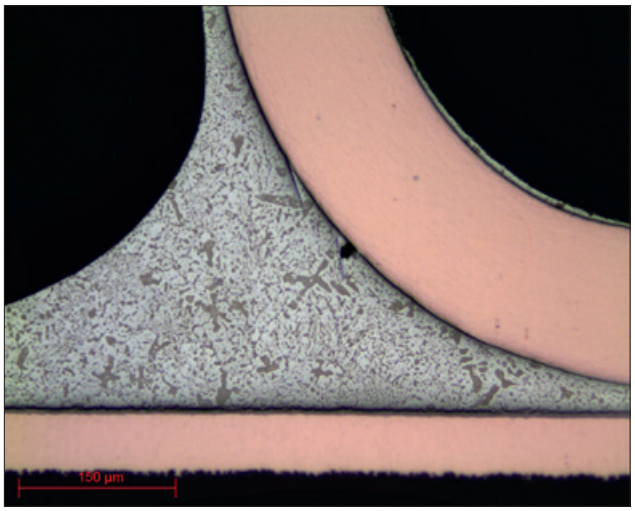


Fig. 14: Braze PCB

Finally, superfinishing is advised, using a colloidal silica suspension. This SPM suspension can be diluted up to 7 times in water.
During this stage, the rotation of the head is reversed in relation to the platen in order to keep the suspension on the polishing cloth as much as possible.

Depending on the result obtained, this stage can be replaced by the use of a PRESI n°2 alumina suspension.

Figures 9-14 show the result on copper and brass after applying the above-mentioned polishing range.

Sometimes it is necessary to adapt the polishing range according to the sensitivity of the material. Below is an example of a range for a bronze coating with lead.

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	SiC P4000	Ø / Water	300	150	→ →	1’
4	NT	Alumina n°2	150	100	→ ←	1’
5	NT	Alumina n°1	150	100	→ ←	1’

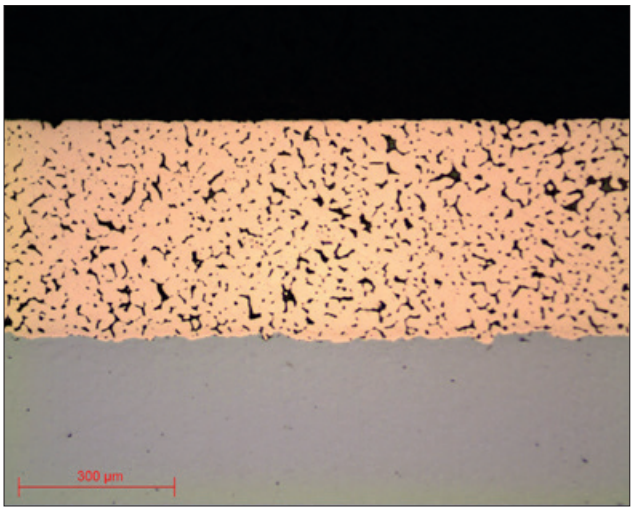


Fig. 15: Bronze with lead lens x10

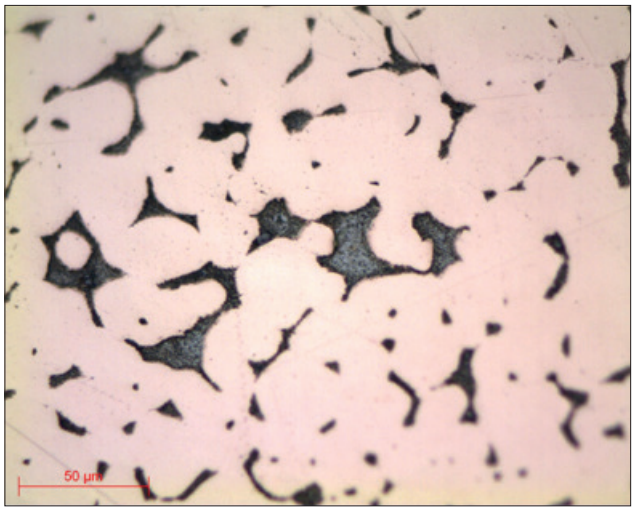


Fig. 16: Bronze with lead lens x50

MICROSTRUCTURE

The structure of the copper is easily revealed by using suitable etching reagents.
The use of etching reagents reveals grain boundaries, phases, etc...
The main reagents are:
- alcoholic solution of Fe III acid chloride (figure 17)
- potassium dichromate (Figure 18)

These reagents are available in the PRESI catalogue and other specific reagents are available on request.

All of the micrographs presented were done using the **PRESI VIEW** software:

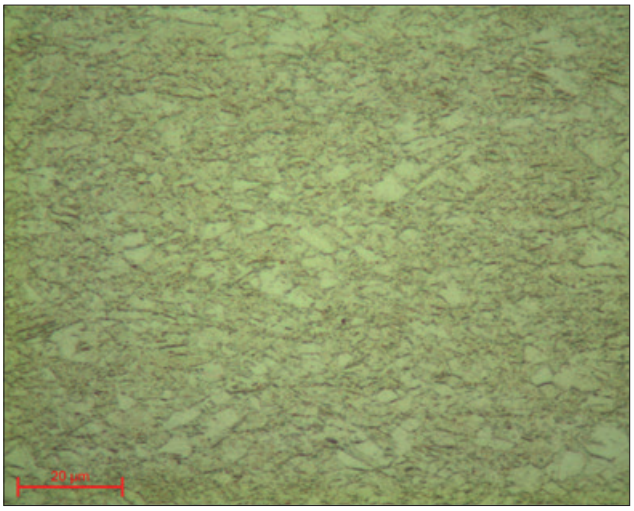


Fig 17: Brass after etching

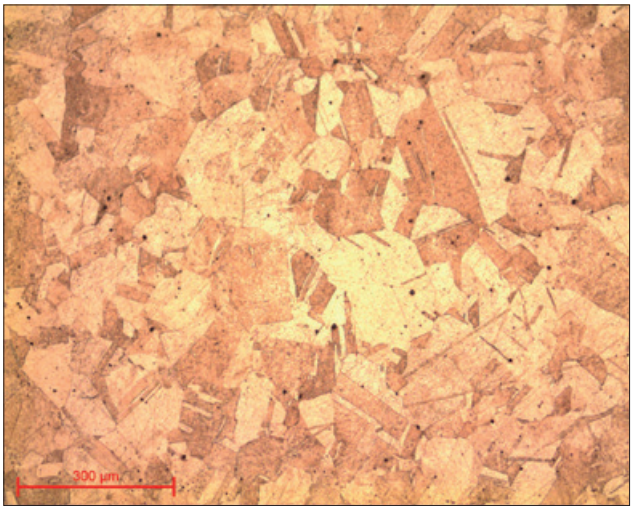


Fig. 18: Copper after etching lens x10

TO GO FURTHER

The results of the above-mentioned polishing operations allow good-quality observation under an optical microscope. For further observation for EBSD analysis under a scanning electron microscope (SEM), a **VIBROTECH** vibratory polisher can be used. This machine removes surface hardening and any residual scratches.

THE VIBROTECH is a reliable and robust vibratory polishing machine. Vibratory polishing is an ultra-smooth technique suitable for superfinishing steps for advanced inspection. Very easy to use, vibration frequency and scope can be adjusted in real time.

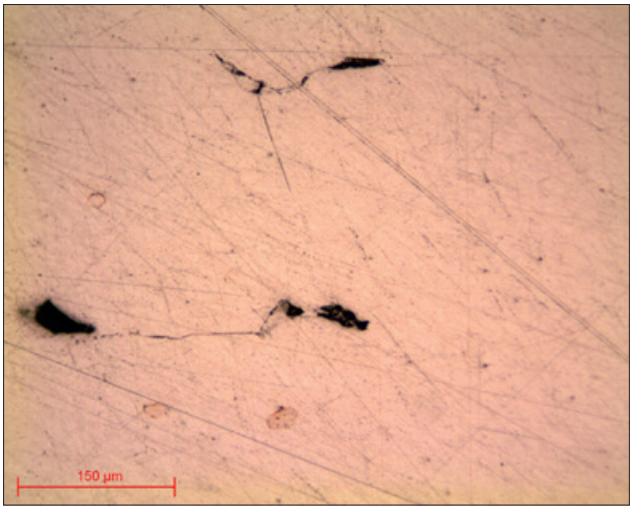


Fig. 20: Finish 1µm lens x20

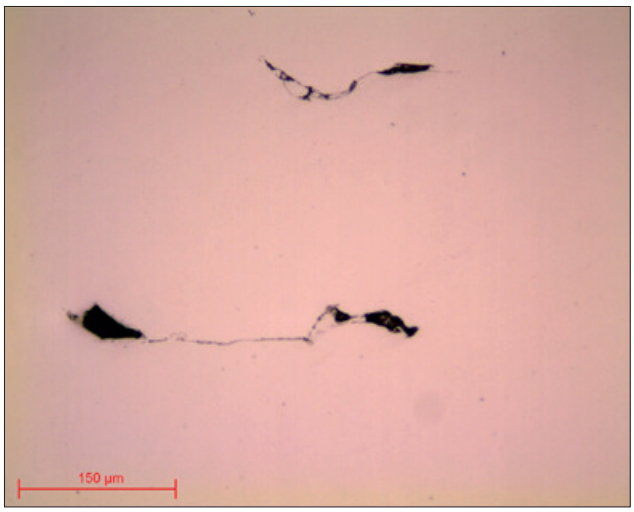


Fig. 21: SPM Finish with Vibrotech lens x20

Vibrotech removes scratches from the 1µm stage (figure 20). In this particular case, a 2 hour stage on an RFI fabric was used to achieve this result (figure 21).

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