PRESI

PRESI LAB'NOTE

METALLOGRAPHIC PREPARATION OF NICKEL

INTRODUCTION

Nickel is a very abundant metal in the universe, but despite this, it is not abundant in the earth's crust. Nickel may be present in metallic form on Earth, but only when it comes from meteorites, otherwise it is present in the form of ores. The proportion of nickel in these ores is low, i.e. the content of other elements is high.
Among them are iron, copper, cobalt, magnesium, sulphur, arsenic, etc.

Three types of ore can be distinguished: arsenic ores, sulphide ores and oxidised ores. Generally, arsenic and sulphide ores are extracted from underground, and sometimes very deep (2000m), mines, while oxidised ores are always extracted from open-pit mines since they are formed on the surface.

NICKEL
Symbol: Ni
Atomic N°: 28
Density: 8,9
Molar mass: 58,7g.mol-1
Melting point: 1440°C

PROCESSING

The multitude of elements present in the ores makes the processing of nickel complex since it requires a succession of operations. However, the objective is simple: to obtain a product with a high concentration of nickel.

The process can be summarised as follows:

• The first stage is concentration, which involves a succession of partial firings followed by reductive melting: this involves high temperatures in an oxidising atmosphere, the aim of which is to enrich the raw material with nickel by eliminating impurities as much as possible.
The material obtained is called: "speiss" if the nickel is combined with arsenic (mixture of nickel, cobalt and arsenic) and "matte" if it is combined with sulphur (mixture of nickel, copper and sulphur). A final firing transforms the materials obtained into oxides.

• The second stage is the treatment of the oxidised ores, which generally involves reducing the oxides combined with the metal or directly reducing the metallic nickel.
Due to the multitude of oxides present, several processes can be carried out involving either different heat treatments or different chemical solutions, with the aim of reducing specific oxides.

PROPERTIES AND USES

Nickel is a relatively hard, corrosion-resistant metal, with the advantage of being malleable, tough, ductile and easily rolled. It is ferromagnetic and has good electrical and thermal conductivity.

It is very rarely used pure. Nickel is mainly used as an alloying or additive element:

- **Stainless steels**, addition of nickel to improve corrosion resistance,
- **Invar/Kovar**, an iron, nickel (and chromium for Kovar) alloy with a very low thermal expansion coefficient, used in watchmaking, topography, measuring devices, etc.
- **Cunife and Fernico**, an alloy of copper, nickel and iron with the same thermal expansion coefficient as some types of glass.
- **Phynox**, an austenitic superalloy of cobalt, chromium, nickel, iron and molybdenum used for its very high corrosion resistance,
- **Nickel silver**, an alloy of copper, nickel and zinc, used for its low oxidation and silvery appearance, in jewellery, musical instruments, precision instruments, watches, etc.
- **AlNiCo**, an alloy of aluminum, nickel and cobalt used for its ferromagnetic characteristics allowing the manufacture of permanent magnets (AlNiCo magnet),
- **Gold-nickel** alloys used in jewellery for their colours and superior mechanical properties, etc.

However, its main unalloyed use is as a coating. Nickel is stainless in the ambient air and associated with its bright and aesthetic appearance, it is used for protection against corrosion, improvement of mechanical properties (resistance to wear, abrasion, friction) and/or to improve appearance.
These coating operations are called "nickel plating". There are two types: electrolytic nickel plating and chemical nickel plating.

Nickel also benefits from its own alloys (%Ni > 50%), they are practically all used for their corrosion/oxidation resistance and creep resistance, among them:

- **Copper-nickel alloys (Monel)**, which are more mechanically resistant than non-alloyed nickel and are very resistant to corrosion in chemical environments. They are heat treatable and with the addition of aluminum and titanium, the alloys retain their corrosion resistance and have enhanced mechanical characteristics. Therefore, they are used in the marine industry, oil and gas industry, in the transport of steam and water (fresh and sea water), in chains, cables, etc.
Example of grade: Ni-Cu35 (Monel or Alloy 400); Ni-Cu35AlTi (Monel or Alloy K-500).

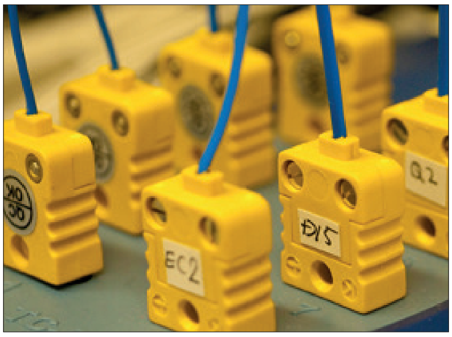
- **Nickel-chromium alloys**, which have high resistivity, very good oxidation resistance and good mechanical strength at high temperatures. Thanks to these characteristics, they are often used for electric heating resistors (electric ovens, toasters, dryers, etc.). Iron can be added to nickel-chromium alloys to increase creep resistance.
Example of grade: Ni-Cr20 (Chromel A); NiCr14Fe6 (Inconel 600).

• **Nickel-based superalloys** are high-performance alloys, which initially designate alloys developed for turbojet engine parts (aeronautics industry). Today they are also used in the power generation industry, the oil industry, the automobile industry, the chemical industry, the nuclear industry, etc.
Their main benefit lies in their excellent mechanical resistance at high temperatures (creep resistance, oxidation/corrosion, ductility, fatigue, etc.). Some alloys also have cryogenic properties.
Nickel-based superalloys have a nickel content of more than 50%. A multitude of additive elements can be added and heat treatments can be carried out with the aim of influencing mechanical properties by modifying the microstructure of the alloys.
Example of grade: Inconel (625 or 718); Hastelloy; Nimonic; Rene Alloy.

• **Other nickel alloys**, such as:
Nickel-titanium alloys (Ni-Ti) called "Nitinol" which benefit from shape memory and super-elasticity properties allowing them to withstand severe deformations but which can recover their initial shape. They are used in the aerospace sector (coupling sleeves, Frangibolt devices, solar panels), the biomedical sector (instrumentation, stents, etc.) and the commercial sector (clothing and sports equipment, etc.).

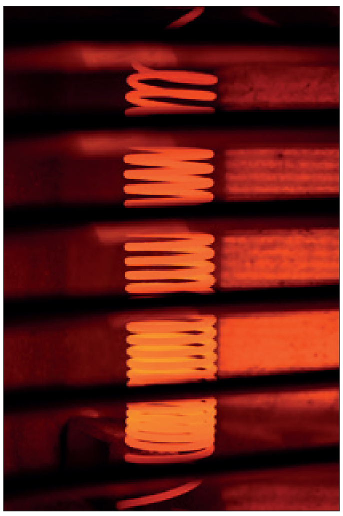
Permalloy alloys which are nickel and iron based alloys used for their magnetic properties. These include: their high magnetic permeability and magnetoresistance as well as their low coercive field and magnetostriction.

Alumel alloys, consisting of 95% nickel (with manganese, aluminum and silicon), are used to make thermocouples.







METALLOGRAPHIC PREPARATION

In general, during processing and transformation operations and various mechanical, thermal and chemical treatments, the properties and microstructures of nickel and its alloys are influenced. It is these influences that determine the performance of metallographic quality controls such as: microstructure inspections, search for porosities and/or heterogeneities, search for and inspection of inclusions and/or precipitates, hardness tests, work hardening controls, grain size, dimensioning, 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 the physico-chemical properties of the nickel.
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

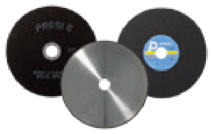
Fig 2: MECATOME ST310

Fig 3: EVO 400

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.



	NICKEL AND ALLOYS
Micro-cutting	UTW S Ø180 mm MNF AO
Medium-capacity cutting	MNF AO S
High-capacity cutting	MNF AO S

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 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.

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.

	NICKEL AND ALLOYS
Hot process	Hot epoxy Phenolic Allylic
Cold process	KM-U KM-B IP - IP FAST 2S*

Table 2: Choosing the right mounting resin type
* Suitable for very large series

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:
MINITECH 300 DP1 and DP2

Fig 9:
MECATECH 250 SPI

Fig 10:
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 750-1500W, 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

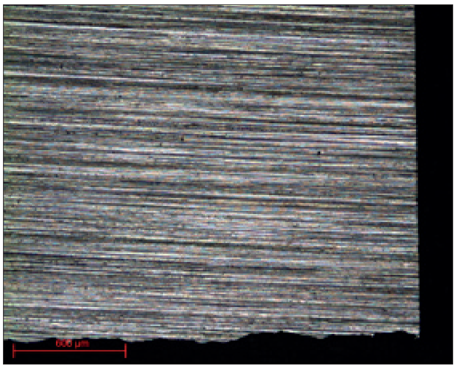
All the polishing ranges below are given for automatic sample preparation (for manual polishing: do not take into account the parameters at the top). They are the most commonly used and are given for information and advice.

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.

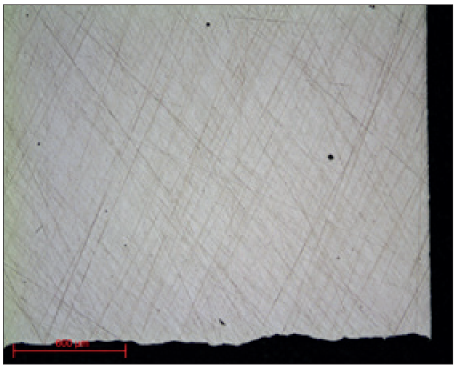
Applied pressures vary according to sample size, but in general the following applies: 1daN per 10mm mounting diameter for the pre-polishing steps (ex: Ø40mm = 4 daN) then reduce force by 0.5daN at each polishing step with an abrasive suspension.

RANGE N°1

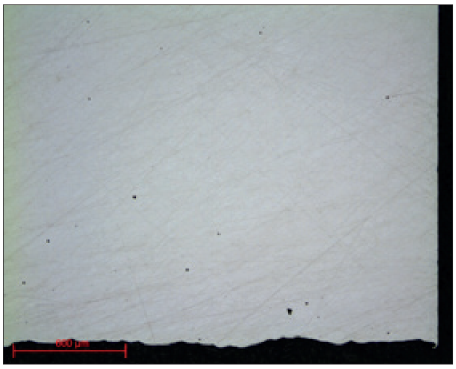
N°	Support	Suspension / Lubricant	Platen Speed (RPM)	Head Speed (RPM)	Rotation direction platen / head	Time
1	SiC P320	Ø / Water	300	150	→ →	1'
2	TOP	9µm LDP / Reflex Lub	150	135	→ →	4'
3	RAM	3µm LDP / Reflex Lub	150	135	→ →	2'
4	NT	1µm LDP / Reflex Lub	150	135	→ →	1'
5	SUPRA	SPM / Water	150	100	→ ←	1'



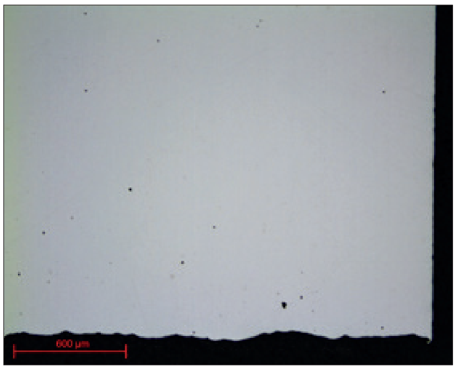
Micrograph 1:
Surface finish P320 lens x5



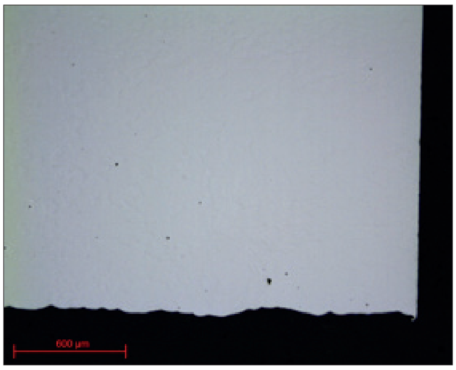
Micrograph 2:
Surface finish TOP 9µm lens x5



Micrograph 3:
Surface finish RAM 3µm lens x5



Micrograph 4:
Surface finish NT 1µm lens x5



Micrograph 5:
Surface finish SUPRA SPM lens x5

An alternative exists to the colloidal silica suspension (SPM) used for the last stage of the polishing process: N°3 alumina suspension (0.8µm).

The polishing range above is complete and is not necessarily to be carried out entirely according to the metallographic examinations to be carried out.

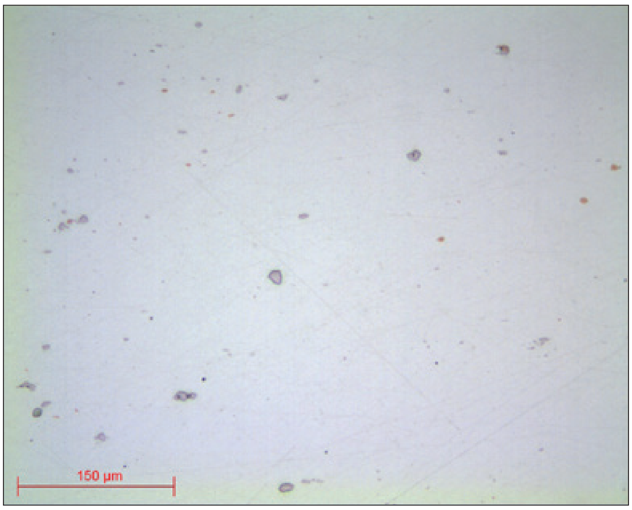
If nickel is not the main element of the material to be polished, it is necessary to adapt the polishing range according to the properties of the same material.

After this preparation, the polished specimens can be directly observed without metallographic etching.

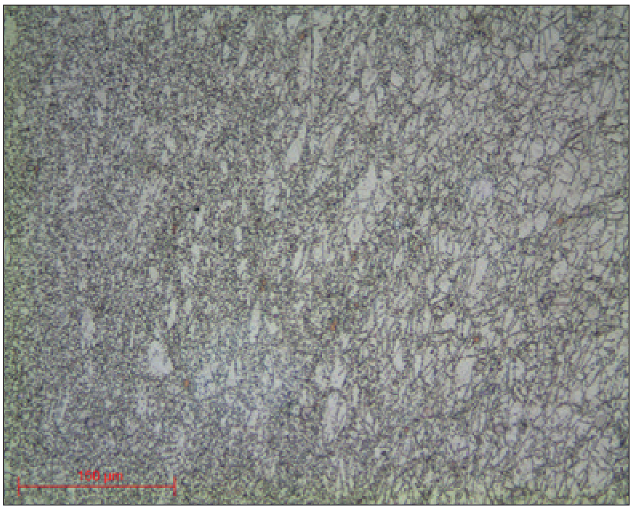
Metallographic etching of nickel and its alloys is commonly performed using ADLER reagent. The etching creates differences in relief and/or colour between the different constituents, allowing them to be observed.

MICROSCOPY

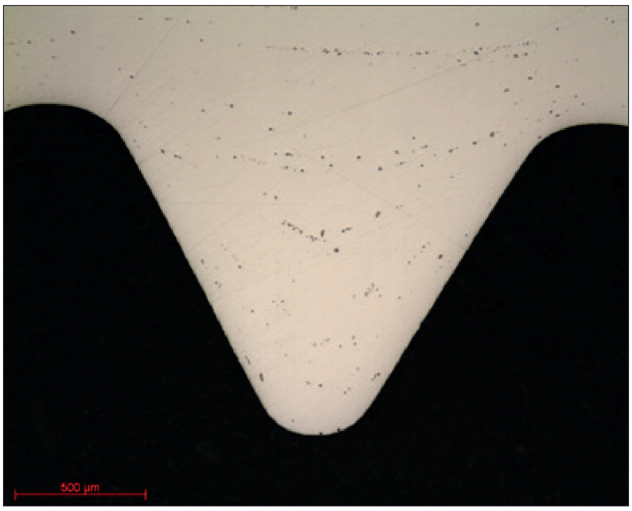
All micrographs presented were created using the **PRESI VIEW** software:

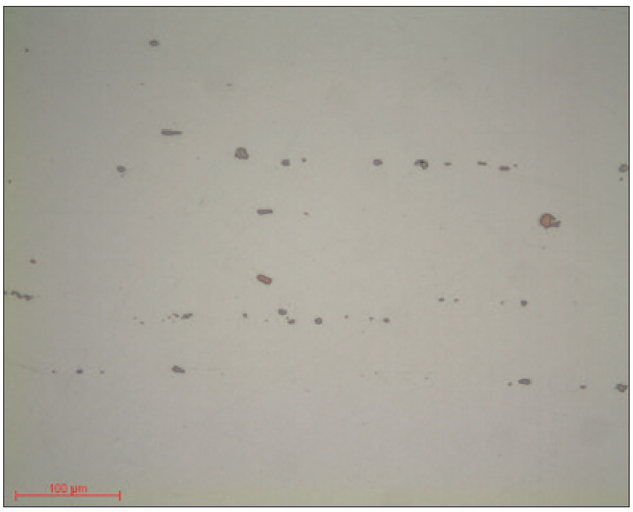


Micrograph 6: Polished nickel base up to 1µm lens x20

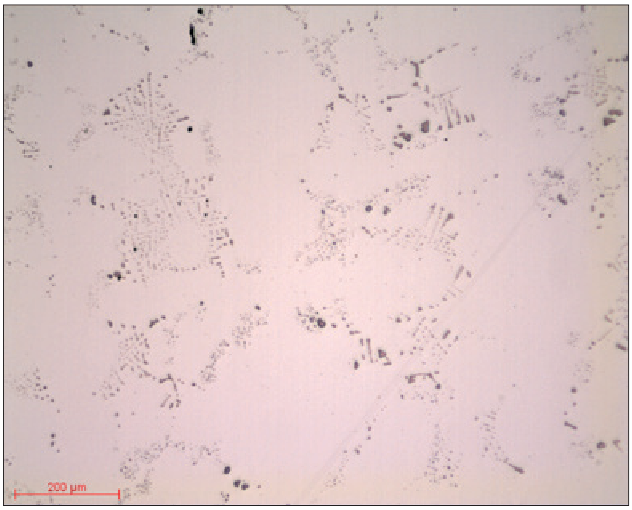


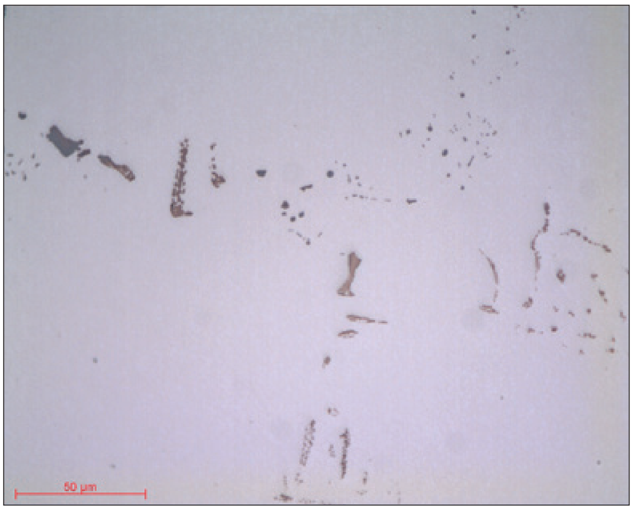
Micrograph 7: Polished nickel base up to 1µm etched with ADLER reagent lens x20



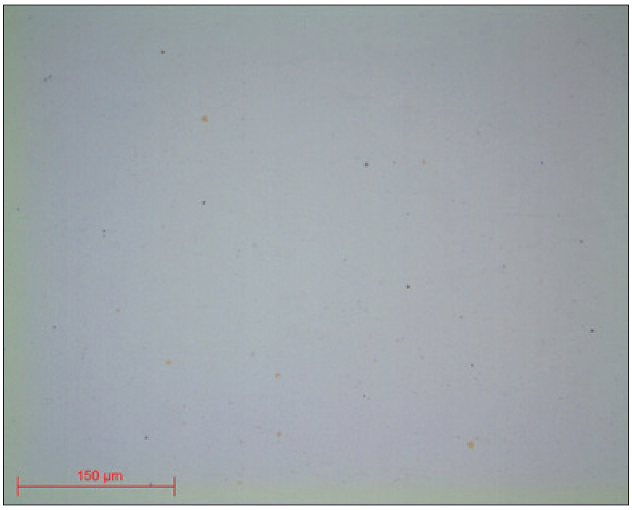


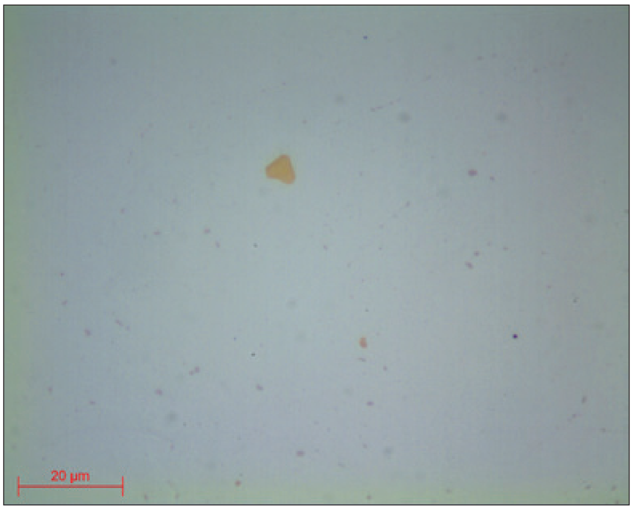
Micrographs 8 and 9: Polished nickel base screws up to 1µm lens x5 and lens x20



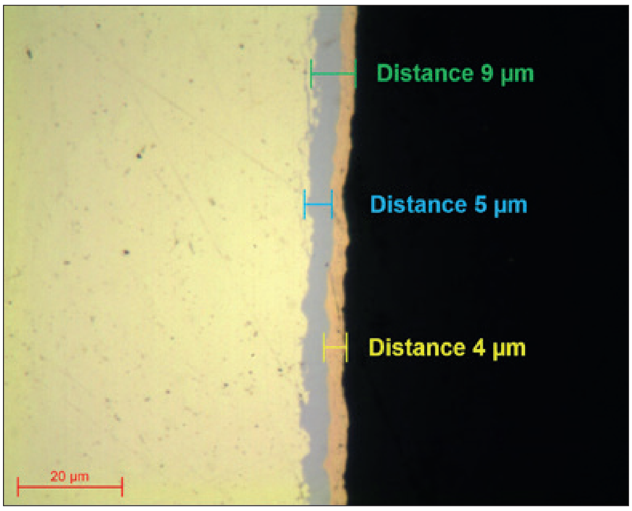


Micrographs 10 and 11: Polished superalloy up to SPM lens x20 and lens x50

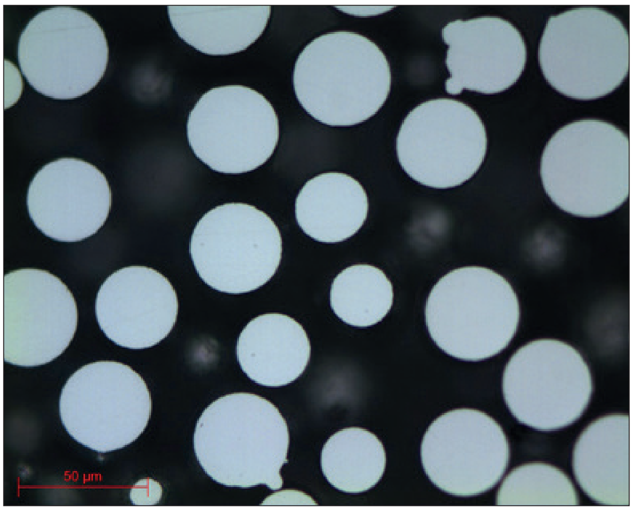




Micrographs 12 and 13: Polished superalloy up to SPM lens x20 and lens x100



Micrograph 14: Gold deposit on nickel deposit on brass substrate lens x100



Micrograph 15: Superalloy powder sample lens x50