METALLOGRAPHIC PREPARATION OF STAINLESS STEEL

INTRODUCTION

All ferrous alloys used on account of their resistance to a wide range of corrosive environments are called “stainless steel”. For a steel to be stainless (commonly called stainless steel) it must have a chromium content, which can vary, always higher than 10.5% according to the standard, 12% in practice, and a carbon content of between 0.02% and 1.2%.

IRON
Symbol: Fe
Atomic N°: 26
Density: 7.8
Molar mass: 55,8 $g.mol^{-1}$
Melting point: 1538 °C

CARBON
Symbol: C
Atomic N°: 6
Density: 2.1 – 2.3 (graphite)
Molar mass: 12 $g.mol^{-1}$

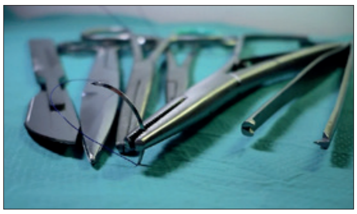
CHROMIUM
Symbol: Cr
Atomic N°: 24
Density: 7.15
Masse molaire : 52 g.mol-1
Molar mass: 1907 °C

It is this chromium content that makes steel stainless, by reacting with the oxygen in the air to form a protective layer of chromium oxide (called a passivation film):

$$4\ Cr + 3\ O_2 \longrightarrow 2\ Cr_2O_3$$

Certain other elements are added (alloy additions) producing stainless steel grade. These additions improve corrosion resistance or mechanical properties. Nickel is the most common addition, but molybdenum, copper, titanium, silicon, niobium, aluminum, tungsten, etc. are also frequently used.

Its resistance to corrosion combined with its mechanical properties makes stainless steel an essential material in many fields of application such as aeronautics, automotive, chemical and naval industries, medicine and surgery, kitchen utensils, everyday objects, tooling, etc.

STAINLESS STEEL METALLURGY

Among all stainless steel grades, five categories can be distinguished according to their composition and metallurgical make-up:

• **Ferritic stainless steels** have very low carbon content (< 0.1%) and therefore have a ferritic structure. Their resistance to corrosion increases according to their chromium content, which can range from 12% to more than 25%.
These steels are magnetic, and when stabilised (addition of titanium, niobium and zirconium) they are weldable. However, their structure limits their mechanical properties (strength and hardness in particular).
Example of grade: X6Cr17 (AISI: 430).

• **Martensitic stainless steels** have sufficient carbon content (> 0.08% and up to 1.2%). They are composed of 12-18% chromium and generally have lower corrosion resistance than other classes of stainless steel due to their martensitic structure.
This structure is obtained by heat treatment and these stainless steels behave similarly to conventional treated steels. They are therefore magnetic and are used where high mechanical characteristics are required.
Example of grade: X20Cr13 (AISI: 420).

• **Austenitic stainless steels** are the most commonly used. They have excellent corrosion resistance and high ductility. Their chromium content is 16-20% and they have fairly high nickel content, usually 8-10%. It is this nickel content that gives stainless steel its austenitic structure. Other elements can be added and/or the carbon content reduced in order to improve corrosion resistance.
This structure makes these stainless steels non-magnetic. Their mechanical properties are influenced by cold treatment (hot treatment is not possible).
Example of grade: X5CrNi18-10 (AISI: 304) or X2CrNiMo17-12-2 (AISI: 316L).

• **Precipitation-hardening stainless steels** are grades consisting of several additive elements in addition to 13-17%, chromium content, including copper, aluminum, molybdenum and niobium.
The mechanical properties of these stainless steels are improved by undergoing heat treatment to precipitate intermetallic compounds. Very often, the grades are martensitic matrix grades.
Example of a grade: W8CrNiMoAl15-7-2 (AISI: 630(17-4PH)).

• **Austenitic-ferritic stainless steels** (commonly known as duplex), have a structure with approximately equal ferritic and austenitic parts. The aim is to obtain superior mechanical properties to purely ferritic or austenitic stainless steels.
Their chromium content is high (> 20%) and they are characterised by the use of nitrogen as an additive element, which promotes structural hardening and increases toughness.
Example of grade: X2CrNiMoN22-5-3 (AISI: 2205).

METALLOGRAPHIC PREPARATION

Generally speaking, the development, transformation and mechanical, thermal and surface treatments influence the properties and microstructures inherent in stainless steels. All these influences then lead to metallographic quality controls such as: microstructure examinations, welding controls, search for porosities and/or heterogeneities, inclusion inspections, hardness tests, hardening controls, grain size controls, etc.

A succession of operations is required to inspect surfaces, each of which is as important as the next, regardless of the material. These steps are in the following 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 stainless steel.
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 T210

Fig 2: MECATOME ST310

Fig 3: EVO 400

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.

	STAINLESS STEEL
Micro-cutting	UTW S Ø180 AO
Medium cutting	A AO
High-capacity cutting	A AO

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.

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.

	STAINLESS STEEL
Hot process	Hot Epoxy Phenolic Allylic
Cold process	IP KM-U KM-B 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.

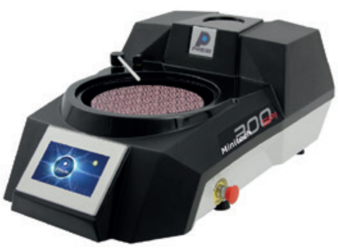


Fig 7:
MINITECH 300 SP1

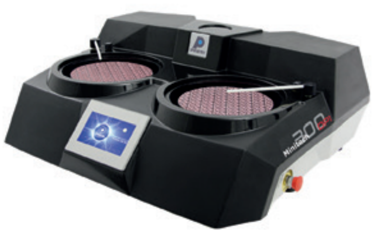


Fig 8:
MINITECH 300 DP1 and DP2

Fig 9:
MECATECH 300 SPC

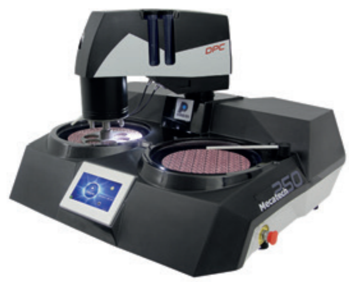


Fig 10:
MECATECH 250 DPC

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

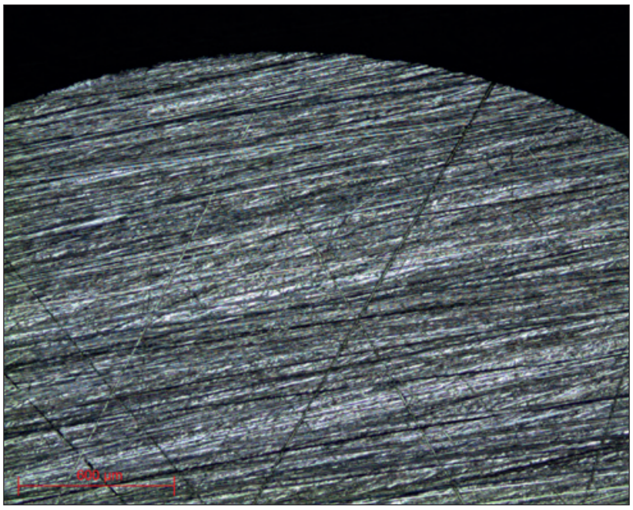
All the polishing ranges below are given for automatic sample preparation (for manual polishing: do not take into account the parameters at the top). They are the most commonly used and are given for information and advice.

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.

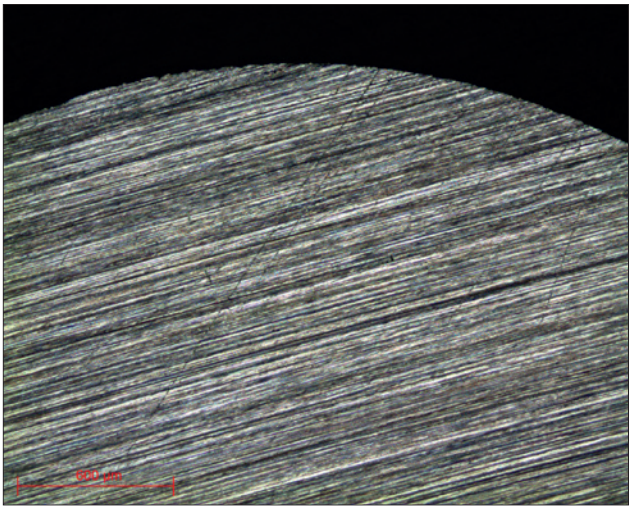
Applied pressures vary according to sample size, but in general the following applies: 1daN per 10mm mounting diameter for the pre-polishing steps (ex: Ø40mm = 4 daN) then reduce force by 0.5daN at each polishing step with an abrasive suspension.

RANGE N°1

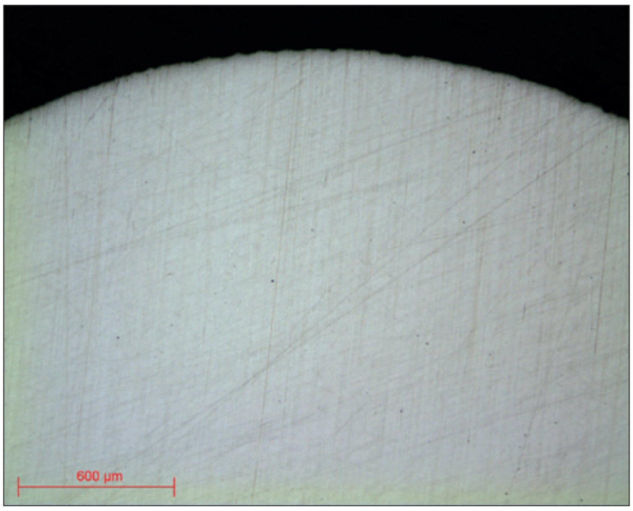
N°	Support	Suspension / Lubricant	Platen speed (RPM)	Head speed (RPM)	Rotation direction platen / head	Time
1	SiC P320	Ø / Water	300	150	→ →	1'
2	SiC P1200	Ø / Water	300	150	→ →	1'
3	RAM	3µm LDP / Reflex Lub	150	135	→ →	2'
4	NT	1µm LDP / Reflex Lub	150	135	→ →	1'
5	NT	Al_2O_3 N°3 / Water	150	100	→ ←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



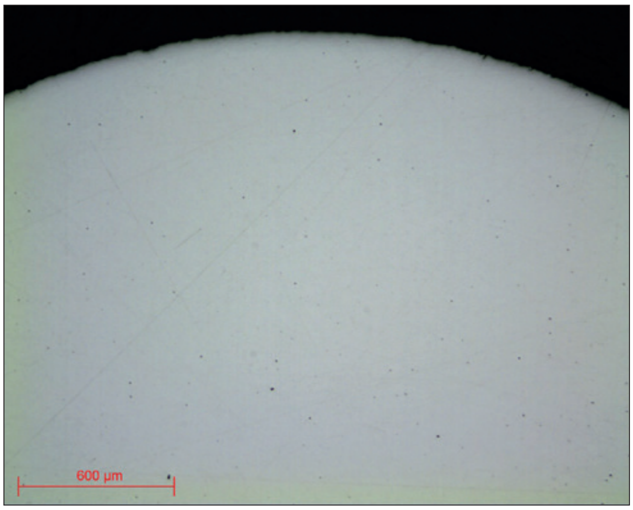
Micrograph 1:
Surface condition P320 lens x5



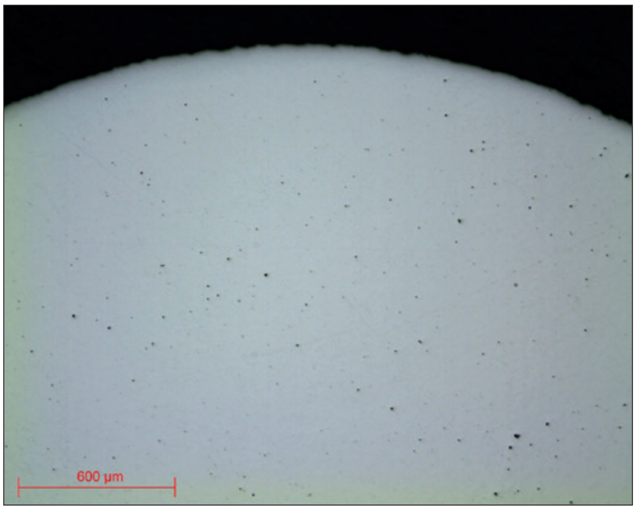
Micrograph 2:
Surface condition P1200 lens x5



Micrograph 3:
Surface condition RAM 3µm lens x5



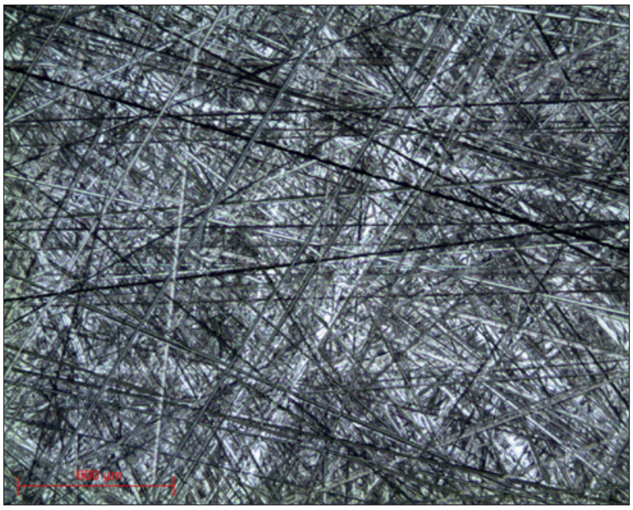
Micrograph 4:
Surface condition NT 1µm lens x5



Micrograph 5:
Surface condition Al_2O_3 N°3 lens x5

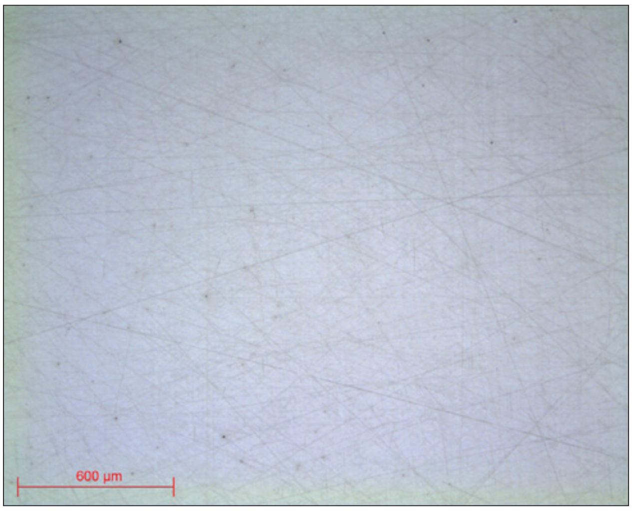
RANGE N°2

N°	Support	Suspension / Lubricant	Platen speed (RPM)	Head speed (RPM)	Rotation direction platen / head	Time
1	I-Max R 54µm	Ø / Water	300	150	→ →	3'
2	I-Max R 18µm	Ø / Water	300	150	→ →	3'
3	ADR II	3µm LDP / Reflex Lub	150	135	→ →	4'
4	NT	1µm LDP / Reflex Lub	150	135	→ →	1'
5	NT	Al_2O_3 N°3 / Water	150	100	→ ←	1'

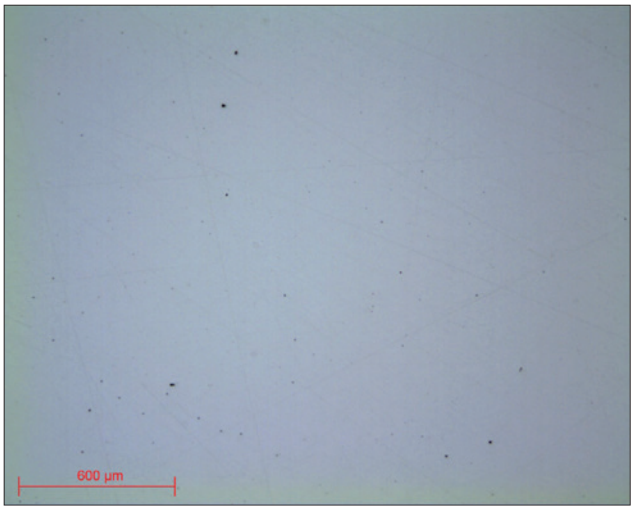


Micrograph 6:
Surface condition I-Max R 54µm lens x5

Micrograph 7:
Surface condition I-Max R 18µm lens x5



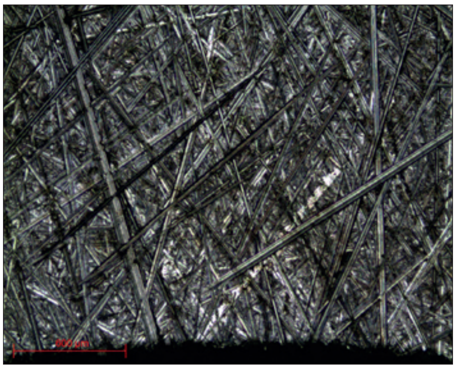
Micrograph 8:
Surface condition ADR II 3µm lens x5



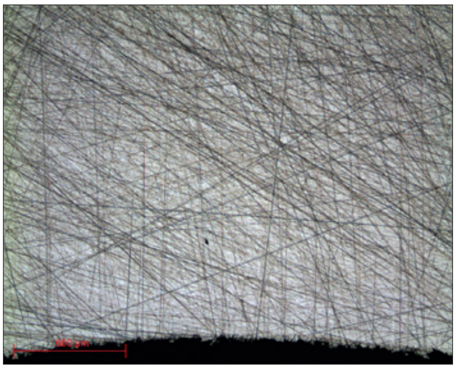
Micrograph 9:
Surface condition NT 1µm lens x5

RANGE N°3

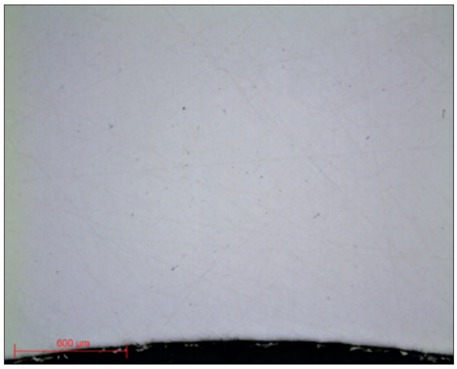
N°	Support	Suspension / Lubricant	Platen speed (tr/min)	Head speed (RPM)	Rotation direction platen / head	Time
1	SiC P80	Ø / Water	300	150	→ →	1'
2	MED R	9µm super abrasive / Ø	150	135	→ →	3'
3	ADR II	3µm LDP / Reflex Lub	150	135	→ →	3'
4	NT	1µm LDP / Reflex Lub	150	135	→ →	1'
5	NT	Al_2O_3 N°3 / Water	150	100	→ ←	1'



Micrograph 10:
Surface condition P80 lens x5



Micrograph 11:
Surface condition MED R 9µm lens x5



Micrograph 12:
Surface condition ADR II 3µm lens x5

	RANGE N°1	RANGE N°2	RANGE N°3
Stainless steels	All	Treated (hard)	All
Benefits	Flexible	• Long lasting consumables • Optimised for large series • Excellent flatness	Quick, reduced number of steps

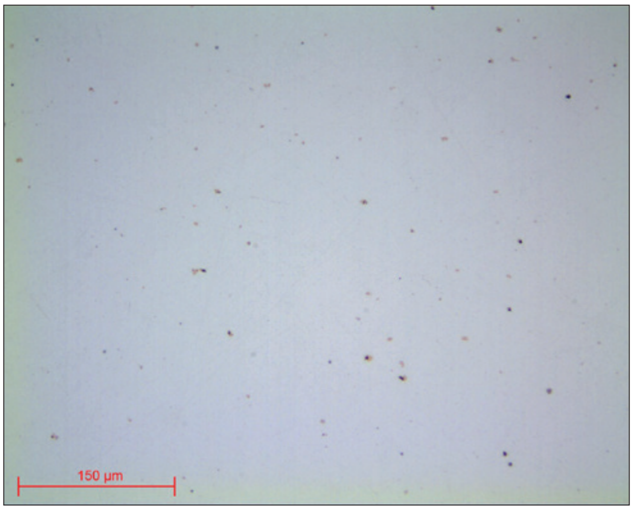
Table N°3: Choice of range

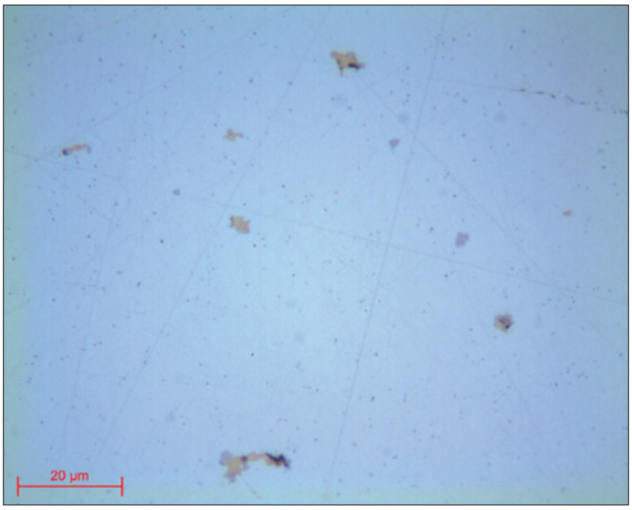
=> The polishing ranges given above are complete, but do not necessarily need to be carried out fully, depending on the metallographic examinations to be performed.

At the end of this preparation, the polished specimens can be directly inspected without metallographic etching. Otherwise, metallographic etching is commonly carried out using the ADLER reagent. It can also be done with MARBLE or KALLING reagents. Etching creates differences in relief and/or colour between the different constituents, thus allowing inspection.

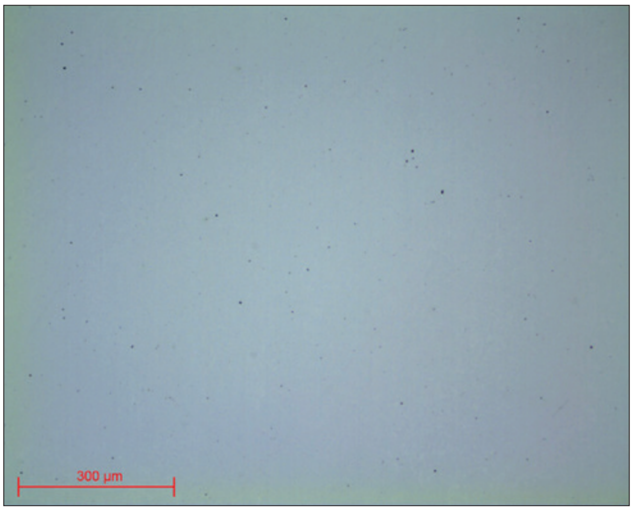
MICROSCOPY

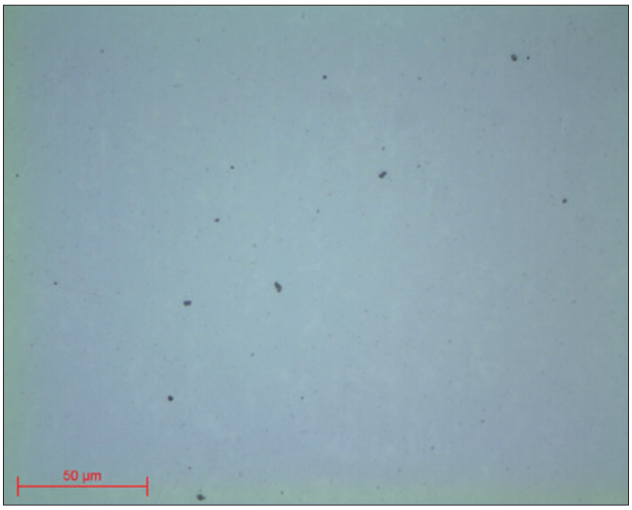
All micrographs presented were created using the **PRESI VIEW** software:



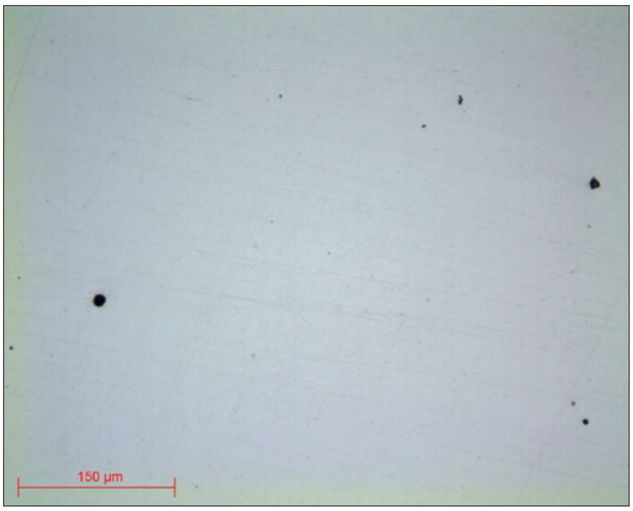


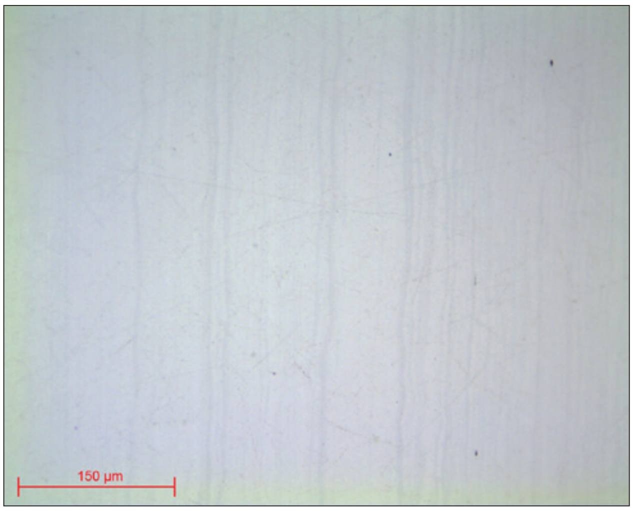
Micrographs 13 and 14:
Stainless steel polished up to 1µm lens x20 and x100



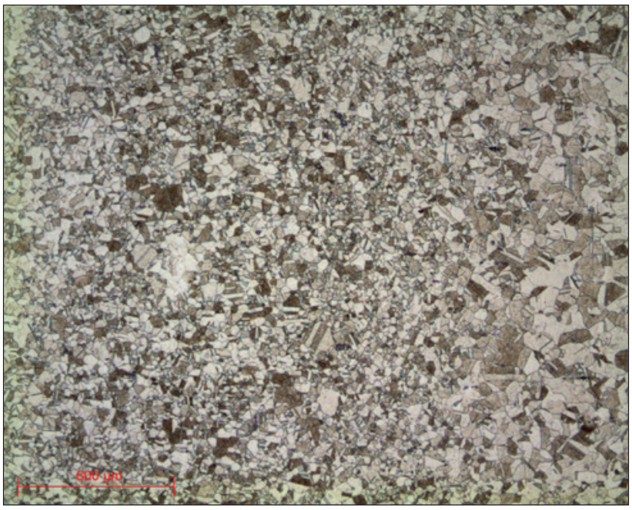


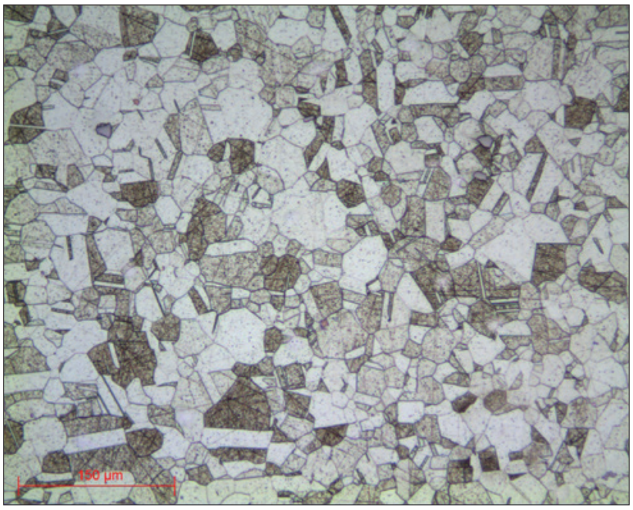
Micrographs 15 and 16:
Stainless steel polished up to Al_2O_3 N°3 lens x10 and x50



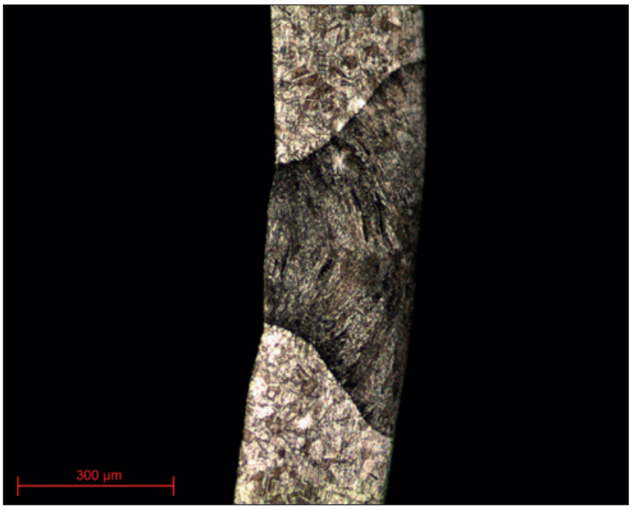


Micrographs 17 and 18:
Stainless steels polished up to Al_2O_3 N°3 lens x20

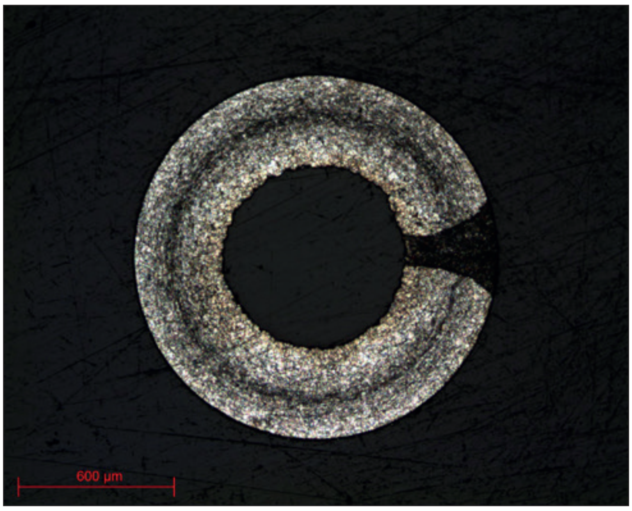




Micrographs 19 and 20:
Stainless steel etched with ADLER lens x5 and lens x20



Micrograph 21:
Stainless steel etched with ADLER lens x10



Micrograph 22:
Stainless steel etched with MARBLE lens x5

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