PRESI
LAB'NOTE

METALLOGRAPHIC PREPARATION OF **STEEL**

INTRODUCTION

What we call "steel" is a metal alloy between the elements iron and carbon. The carbon content of steel varies between 0.008% and 2.11% by mass; below that, it is simply referred to as "iron", above it, the alloy is called «cast iron».

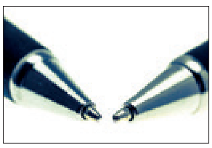
IRON
Symbol: Fe
Atomic N°: 26
Density: 7.8
Molar mass: 55.8 $g.mol^{-1}$
Melting point: 1538 °C

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

→ STEEL ←

It is mainly this carbon content that gives steel its famous properties such as resistance to plastic deformation, breaking strength, resilience and of course hardness. Elements can also be added to the steel, so-called «alloy additions», to influence its properties. These new alloys are called «grades» of steel.

However, steel has a number of disadvantages, including poor resistance to corrosion – which can be remedied by surface treatments (painting, galvanisation, zinc plating, etc.) or by using so-called «stainless» steel grades – and its difficulty in being moulded. It is therefore not recommended for large parts.





The abundance of its ore and the ease with which it can be worked have made «simple» steel inexpensive. This, combined with its properties, makes steel the preferred metallic material in most technical fields: transport, construction, the chemical industry, the nuclear industry, the military, everyday applications, the medical industry, etc.

MAKING STEEL

The iron and steel industry is the industrial sector of metallurgy that processes ferrous alloys. The production of steel involves several channels (cast iron or scrap) in which three fundamental operations are always highlighted:

• **DEVELOPMENT**
intended to produce the desired grade of steel. Steel-making is itself composed of two stages: the first is to obtain crude steel and the second is to refine this crude steel in order to achieve the desired composition and quality.

• **CASTING OF STEEL IN A LIQUID STATE**
followed by solidification of the metal.

• **FORMING** (apart from the case of casting)
which is done by rolling, either hot or cold, and which results in the production of flat products (steel sheet) or long products (bars, wires, etc.).

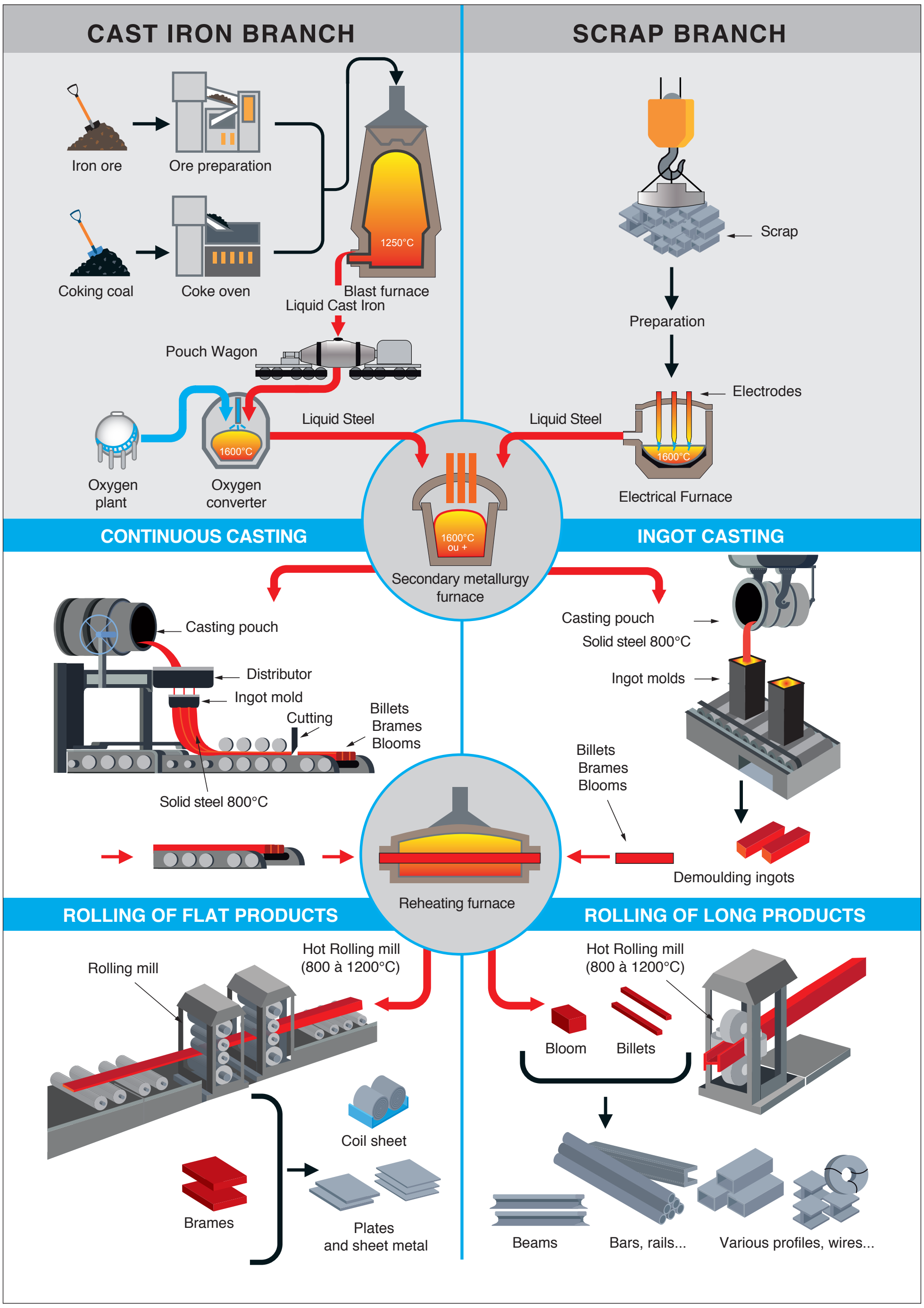


Fig 1: Steel operations

STEEL CLASSIFICATION

<u>Steels are divided into three categories</u>:

• **NON-ALLOY STEELS**
which have a very low alloy addition content, intended for general use or for heat treatment, welding, forging, etc.

• **STAINLESS STEELS**
that contain a minimum of 10.5% chromium by mass and a maximum of 1.2% carbon, which are resistant to corrosion and creep.

• **ALLOY STEELS** (not stainless)
with a higher or lower alloy addition content for hardening and tempering and for tooling.

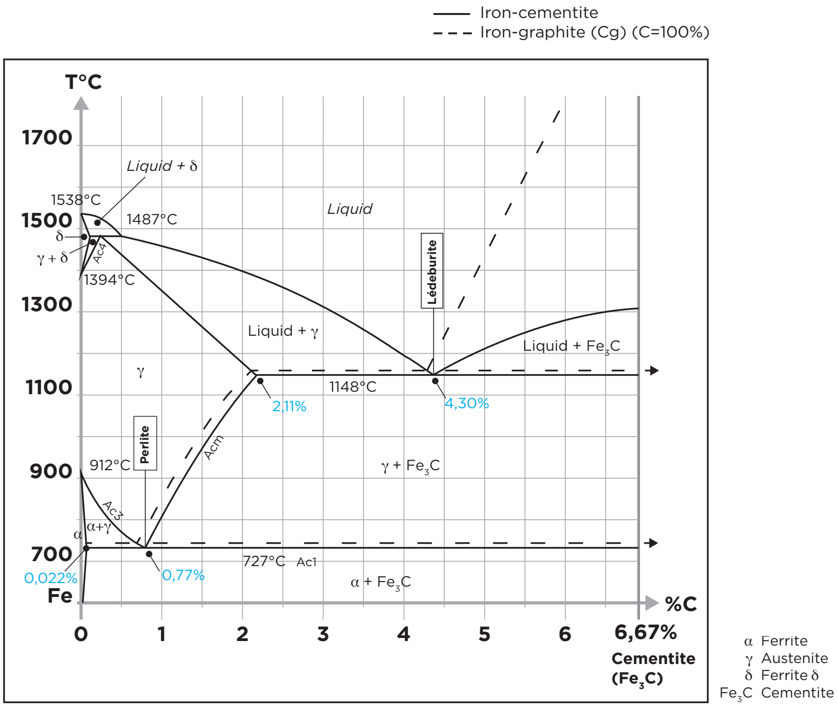


Fig 2: : Iron-cementite diagram (solid lines) and iron-graphite diagram (dotted lines)

Within these three main categories, there are several distinctions, which themselves consist of a multitude of different steel grades.
Each of these shades is also characterised by the «thermomechanical» treatment that it may have undergone, known as the metallurgical state. The purpose of these treatments is to modify the microstructure of the steel and consequently change its mechanical properties.

=> In summary, the properties of steels depend on their chemical composition and metallurgical state. **Depending on all these properties, steel's metallographic preparation can be adjusted.**

METALLOGRAPHIC PREPARATION

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

• 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.

For their metallographic preparation, steels can be divided into three categories according to their hardness:

• Steels known as «soft» or «untreated».
• The so-called «medium-hard» or «surface-treated» steels.
• The so-called «hard» or «treated» steels.

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 steel.
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 3: MECATOME T202

Fig 4: MECATOME T330

Fig 5: 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.

	STEELS «soft»	STEELS «semi-hard»	STEELS «hard»
Micro-cutting	UTW S Ø180 AOF II	UTW S Ø180 AO AOF II	UTW S Ø180 CBN
Medium-capacity cutting	A AOF II	A AOF II	S CBN
High-capacity cutting	A	AO	S CBN

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 6: 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 7: Pressurized mounting device

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

Fig 8: 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.

	STEELS «soft»	STEELS «semi-hard»	STEELS «hard»
Hot process	Phenolic Acrylic Allylic	Hot epoxy Phenolic Acrylic Allylic	Hot epoxy Phenolic Acrylic Allylic
Cold process	KM-U KM-B MA^{2+} 2S*	KM-U KM-B IP 2S*	KM-U KM-B IP 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 9: CUBE 250

Fig 10: MINITECH 300 SP1

Fig 11: MECATECH 300 SPC

Fig 12: 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

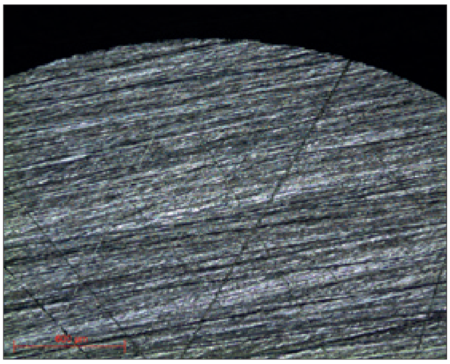
All the polishing ranges below are given for automatic sample preparation (for manual polishing: do not take into account the parameters at the top). They are the most commonly used and are given for information and advice.

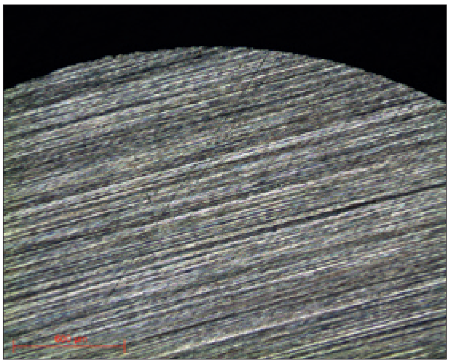
All the first steps of each range are called «levelling» and consist of removing material quickly to level the surface of the sample (and resin). Those given below are standard and can therefore be modified as required.

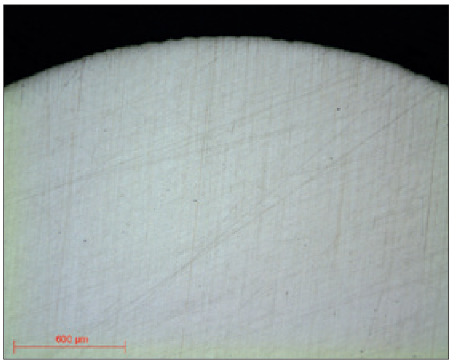
Supporting forces vary according to sample size, but in general the following applies: 1daN per 10mm mounting diameter for the pre-polishing steps (ex: Ø40mm = 4 daN) then reduce force by 0.5daN at each polishing step with an abrasive suspension.

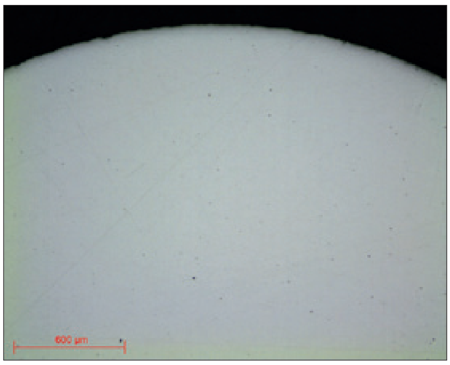
RANGE N°1

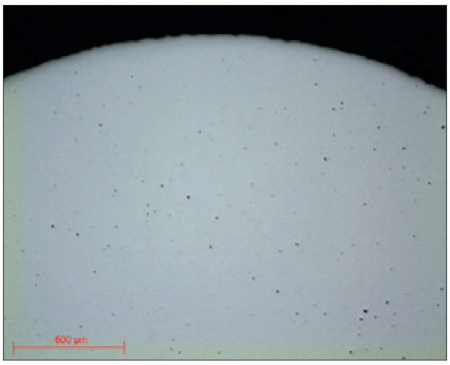
N°	Support	Suspension / Lubricant	Platen Speed (RPM)	Head speed (RPM)	Rotation direction platen / head	Time
1	SiC P320	Ø / Water	300	150	→ →	1'
2	SiC P1200	Ø / Water	300	150	→ →	1'
3	RAM	3µm LDP / Reflex Lub	150	135	→ →	3'
4	NT	1µm LDP / Reflex Lub	150	135	→ →	1'
5	NT	Al_2O_3 N°3 / Water	150	100	→ ←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


Micrograph 1:
Surface condition P320 lens x5


Micrograph 2:
Surface condition P1200 lens x5

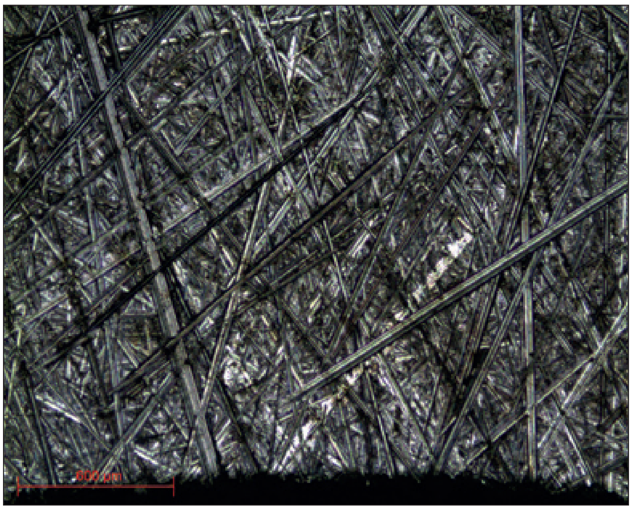

Micrograph 3:
Surface condition RAM 3µm lens x5

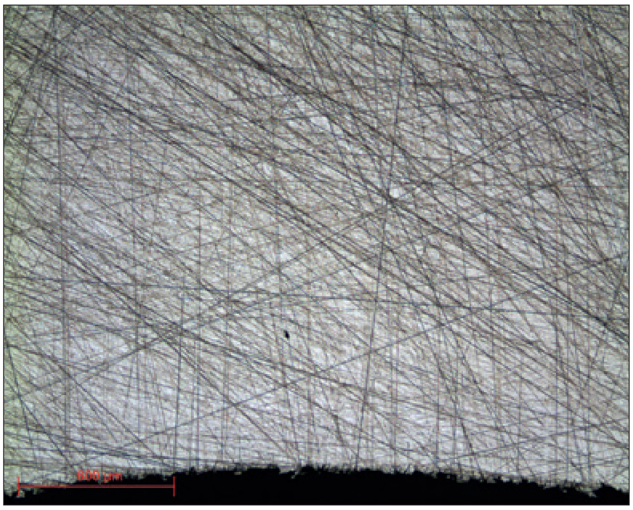

Micrograph 4:
Surface condition NT 1µm lens x5

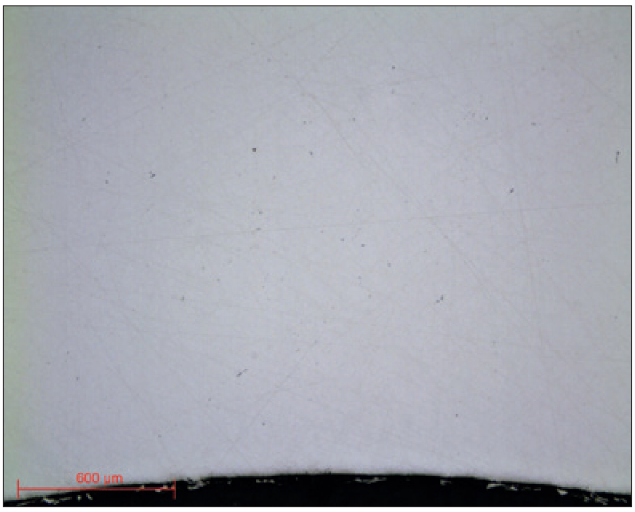

Micrograph 5:
Surface condition Al_2O_3 N°3 lens x5

RANGE N°2

N°	Support	Suspension / Lubricant	Platen Speed (RPM)	Head speed (RPM)	Rotation direction platen / head	Time
1	SiC P180	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'

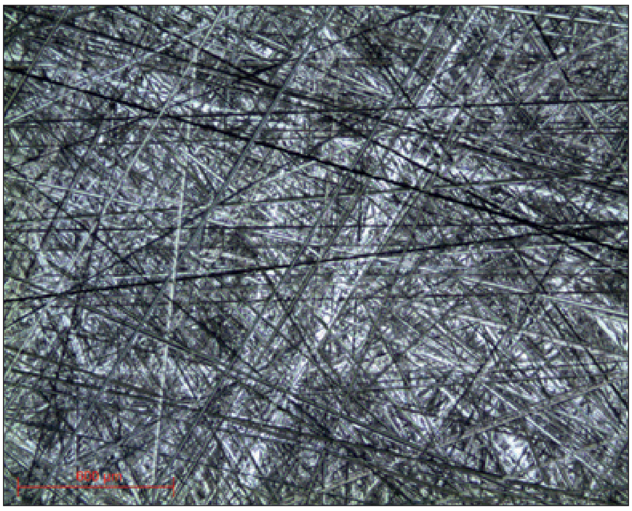

Micrograph 6:
Surface condition P80 lens x5


Micrograph 7:
Surface condition MED R 9µm lens x5


Micrograph 8:
Surface condition ADR II 3µm lens x5

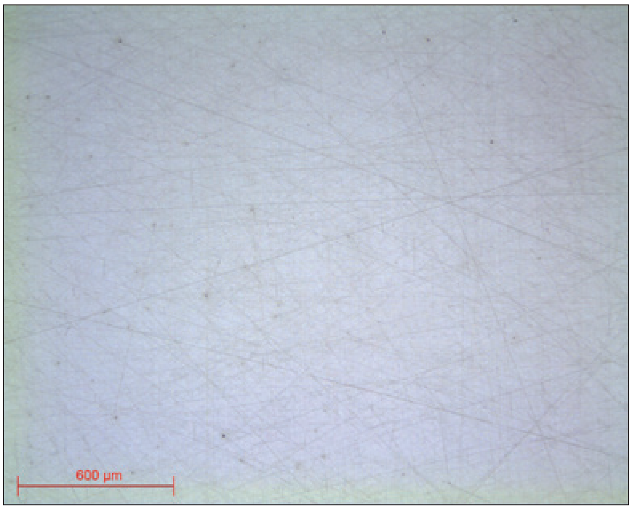
RANGE N°3

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'

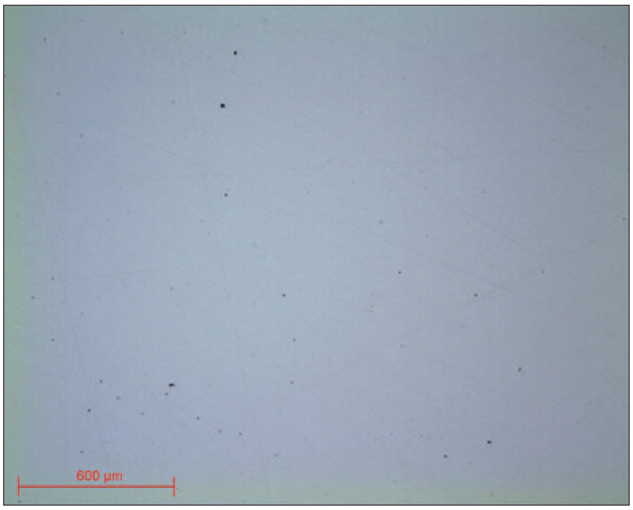


Micrograph 9:
Surface condition I-Max R 54µm lens x5

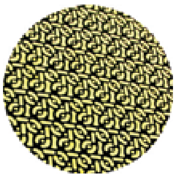
Micrograph 10:
Surface condition I-Max R 18µm lens x5



Micrograph 11:
Surface condition ADR II 3µm lens x5



Micrograph 12:
Surface condition NT 1µm lens x5



	RANGE N°1	RANGE N°2	RANGE N°3
Steels	All	All	"Semi-hard" "Hard"
Benefits	Flexible	Quick, reduced number of steps	• Long consumable service life • Optimised for large series • Excellent flatness

Table N°3: choice of range

In the specific case where the steel grade to be polished is very sensitive to corrosion, it is possible to adapt the polishing ranges. To do this, simply replace the diamond suspensions and water-based lubricant with alcohol-based suspensions and lubricant (ADS suspension and lubricant).

In some cases, and especially for the preparation of «hard steel», a phenomenon of «edge drop» can be observed, i.e. the edges of the specimen are rounded. The usage of suitable consumables such as I-Max R supports is advised in these cases. They are more rigid and offer excellent flatness.

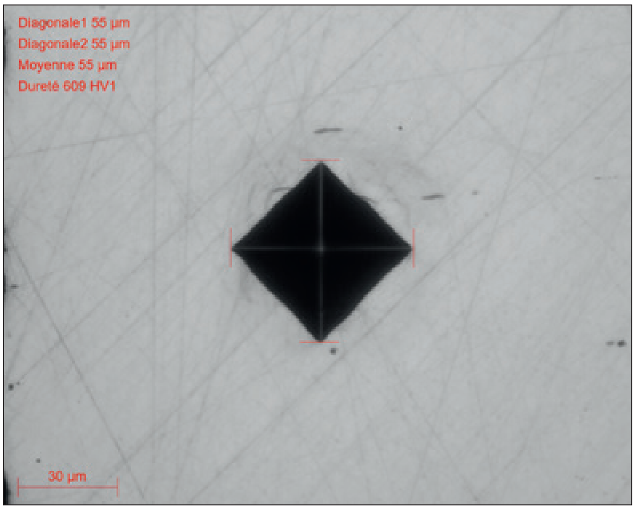
=> The polishing ranges given above are complete, but do not necessarily need to be carried out fully, depending on the metallographic examinations to be performed.

METALLOGRAPHIC ETCHING

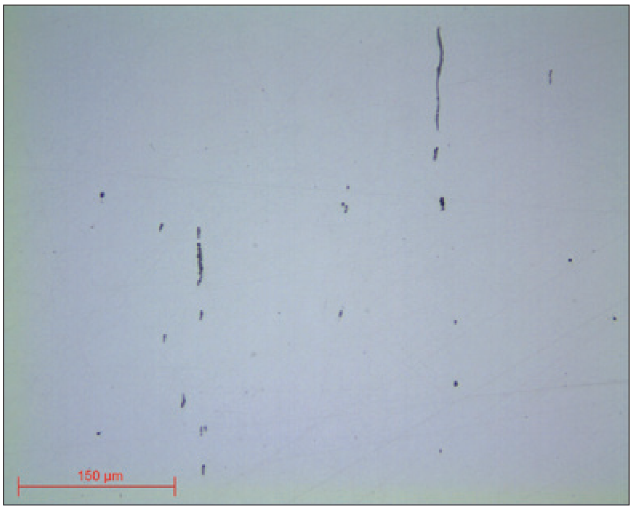
At the end of this preparation, the polished samples can be directly inspected without metallographic etching. Metallographic etching is commonly done using the reagent Nital 4: a solution of 4% nitric acid and 96% ethanol. It can also be done with a Picral reagent: 4mg of picric acid and 100mL of ethanol. Etching creates differences in relief and/or colour between the different constituents and allows their inspection.

MICROSCOPY

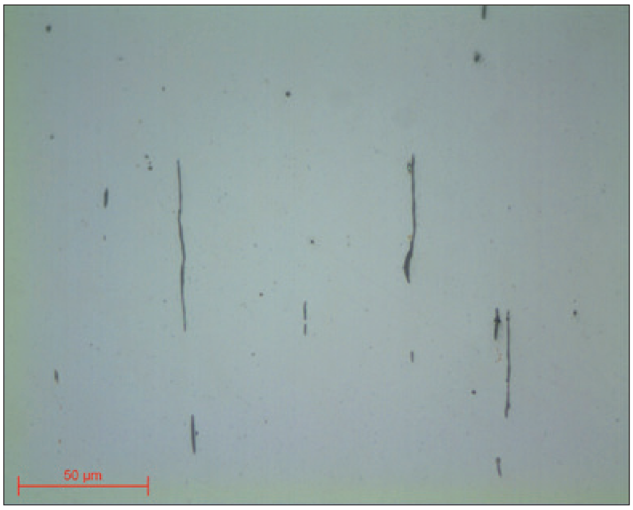
All micrographs presented were produced using **PRESI VIEW** software:



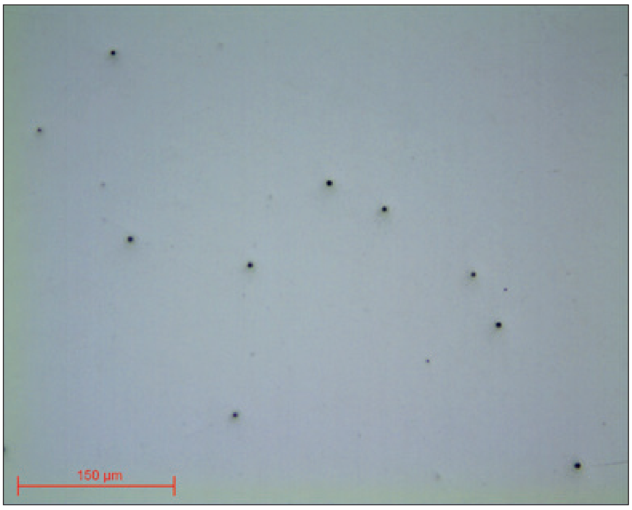
Micrograph 13: Polished treated steel up to 3µm
ideal for hardness testing lens x20

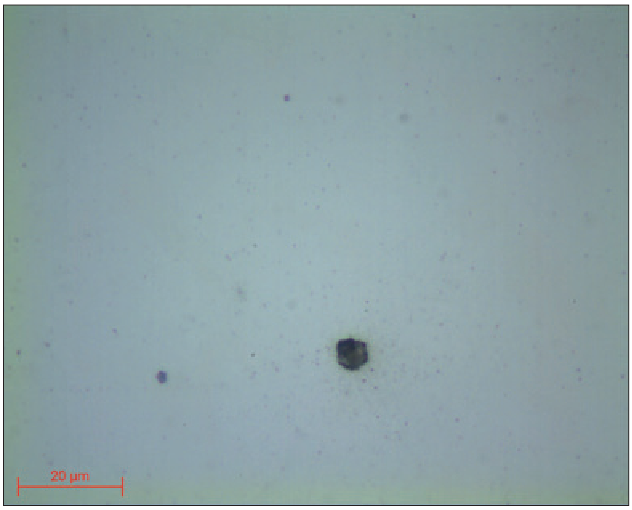


Micrograph 14: Low carbon steel polished to 1µm
Observation of sulphide and silicate lens x20 type inclusions

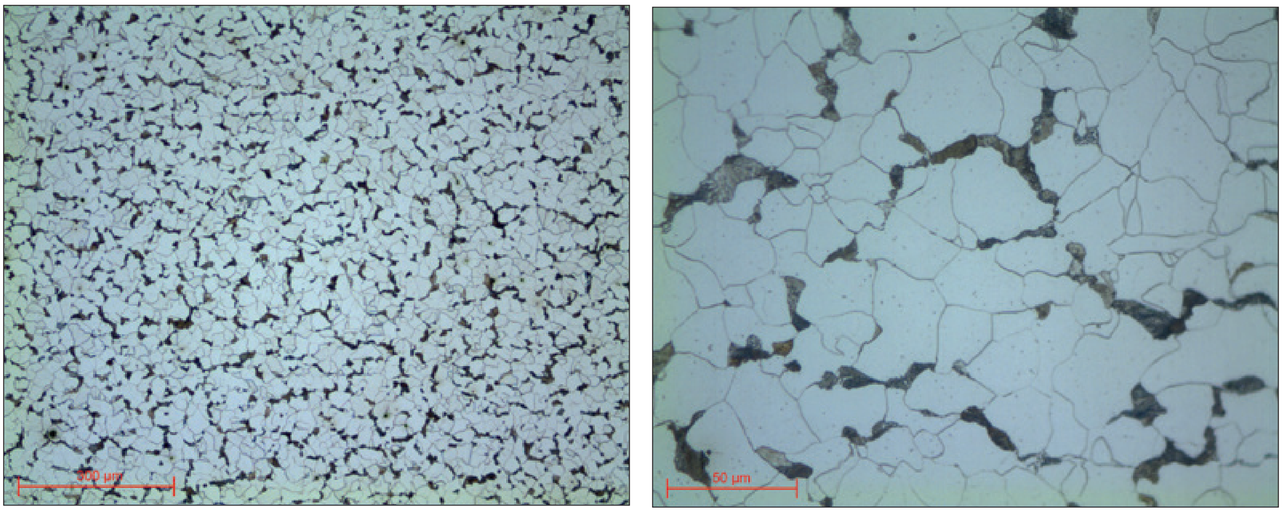


Micrograph 15: Low carbon steel polished up to Al_2O_3 N°3
Observation of sulphide and silicate lens x50 type inclusions

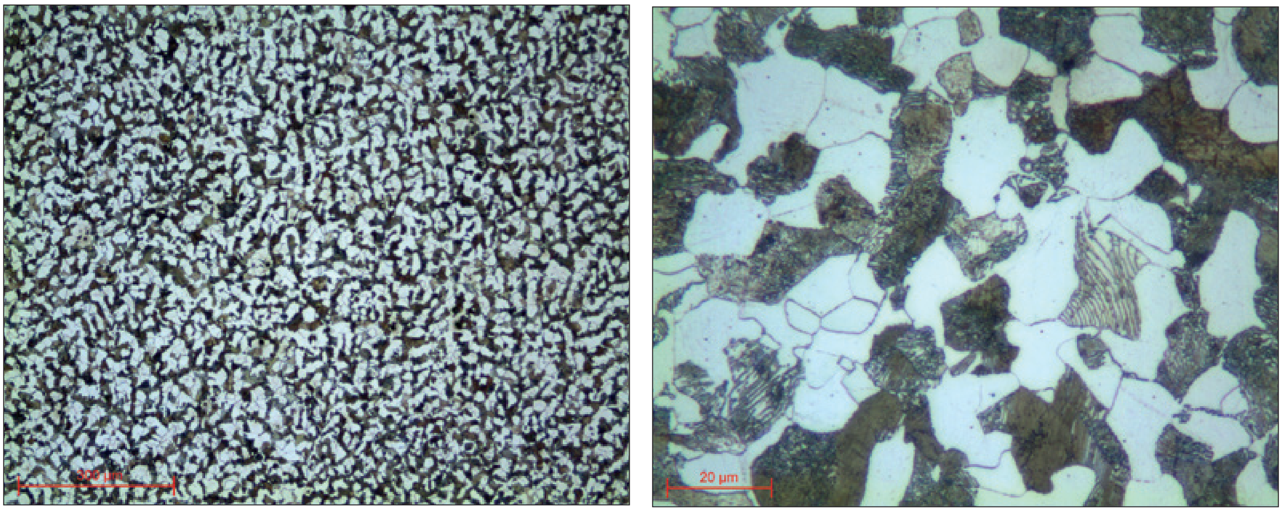




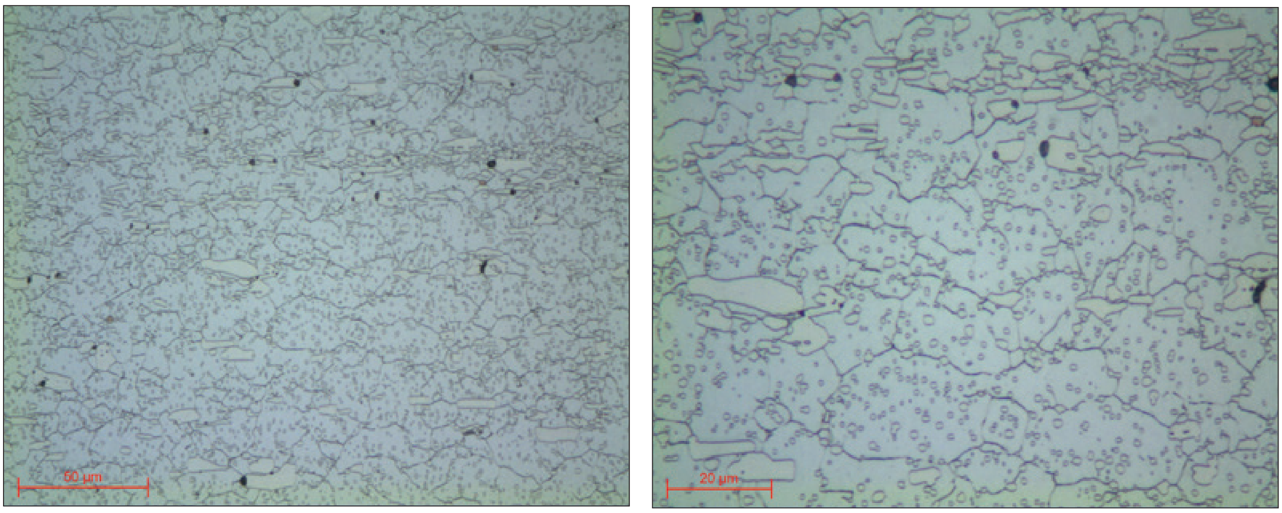
Micrographs 16 and 17:
Low carbon steel polished up to Al_2O_3 N°3
Observation of lens x20 and x100 oxide inclusions



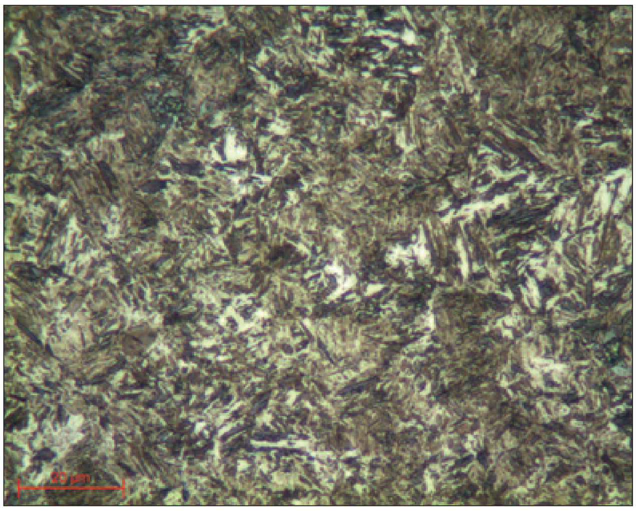
Micrographs 18 and 19:
Hypoeutectoid Steel - Ferrite and Perlite etched with NITAL 4 lens x10 and x50



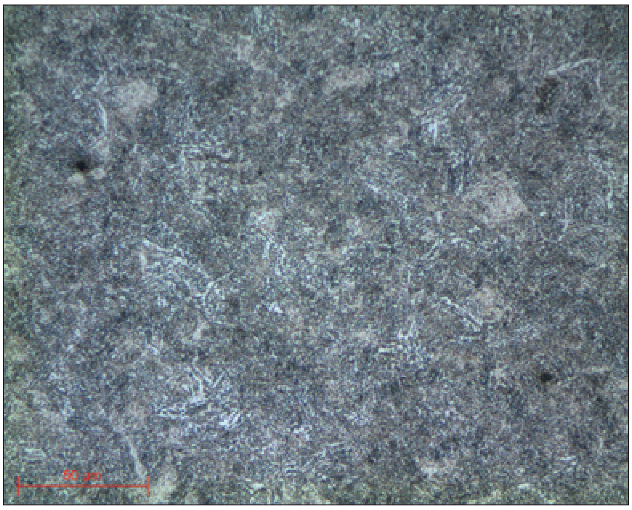
Micrographs 20 and 21:
Hypoeutectoid Steel - Ferrite and Perlite etched with NITAL 4 lens x10 and x100



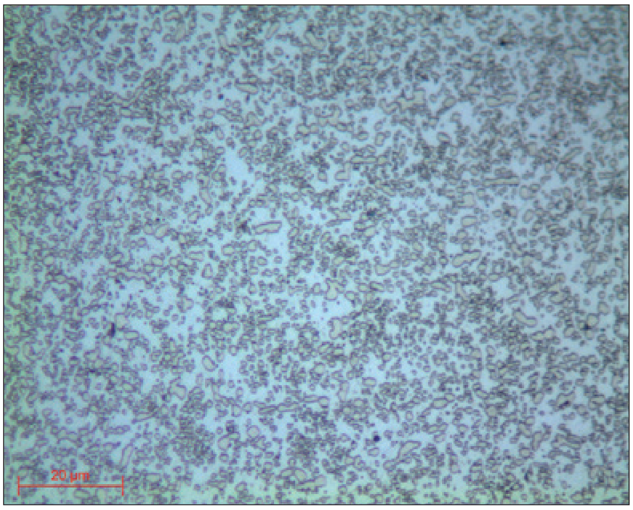
Micrographs 22 and 23:
Hypoeutectoid steel -Globular pearlite etched with NITAL 4 lens x50 and x100



Micrograph 24: Treated Steel - Martensite and Bainite
etched with NITAL 4 lens x50



Micrograph 25:
Hypereutectoid Steel - Perlite and Cementite
Etched with NITAL 4 lens x50



Micrograph 26: Tool steel - Carbides
Etched with PICRAL lens x100