PRESI

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LAB'NOTE

METALLOGRAPHIC PREPARATION OF HEAT-TREATED STEELS

INTRODUCTION

WHAT IS HEAT TREATMENT?
Heat treatment is a process by which the physical and sometimes chemical properties of steels can be modified. Heat treatment operations take place in several stages with a rise in temperature, maintaining it for a certain time and then slow or rapid cooling.

This can transform the structure of the steel in the mass or superficially.
Additive elements in steels can influence the achievement of the desired structure after heat treatment.

BENEFITS OF HEAT TREATMENT
With the help of heat treatments, it is therefore possible to increase the hardness of a steel superficially or totally (in the mass) in order to improve resistance to wear or shocks.
It is possible to restore a more homogeneous structure to steels that have undergone grain enlargement, for example.

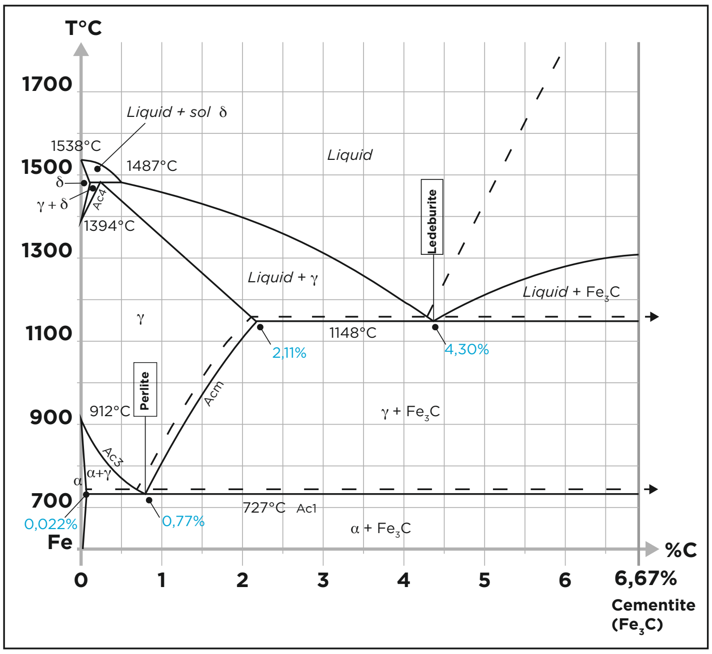
Heat treatments make it possible to increase the breaking strength, elastic limit, ductility and reduce the brittleness of a steel by removing internal stresses.
=> The aim is therefore to change its physical properties according to the final objective of the steel's use.

THE MAIN HEAT TREATMENTS

Quenching and tempering
Quenching is a heat treatment that transforms the austenite of steel into martensite. The aim is to use the Iron-Carbon diagram (figure 1) to place it in the austenitic range. This diagram is used to determine the temperature of a treatment to make phase transformations.

This heating operation is followed by rapid cooling with water, oil or gas. The CCT diagram (continuous cooling transformation), determines the cooling conditions that will transform austenite into Martensite. This type of diagram is specific to each steel grade.

Fig. 1: Fe-C diagram



Tempering is a heat treatment process generally carried out after quenching. After martensitic hardening, it is a stress-relieving temper that releases the stresses present in the material. It is performed at between 180 and 220°C. This tempering does not reduce hardness or tensile strength (or does so only slightly), however it increases ductility and yield strength.

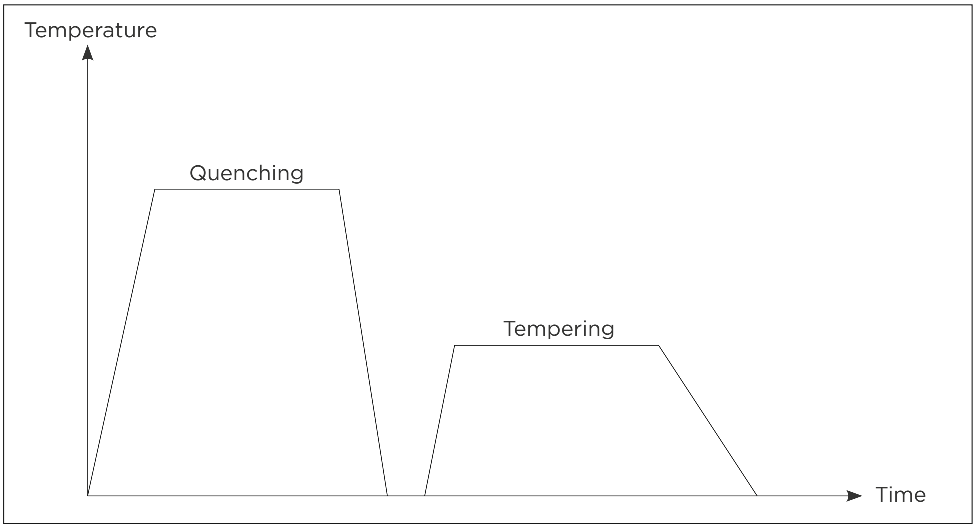


Fig. 2: Quenching and tempering curve

Other types of tempering exist and can be done at between 200 and 600°C. They are generally performed to obtain precise mechanical characteristics according to the type of steel and its future use.

Induction hardening

The principle of induction hardening consists of heating the workpiece very quickly and locally in a superficial way so that the metal is in an austenitic state, and during rapid cooling, it transforms into martensite.

Medium or high-frequency current flows through an inductor (copper tube coil) which induces an alternating magnetic field on the workpiece and thus dissipates its heat into the workpiece. This step is followed by cooling using a quenching fluid.

The cooling operation can be carried out after heating (**static quenching,** Figure 3) or after heating (**flow quenching,** Figure 4).

With this technique, treated surface areas can have a depth of 0.5-5 mm. This treatment is suitable for workpieces that are subjected to torsional stress and for impact resistance.

This treatment is performed individually, unlike carburising, which can be performed on several workpieces at the same time.

Fig. 3: Static quenching

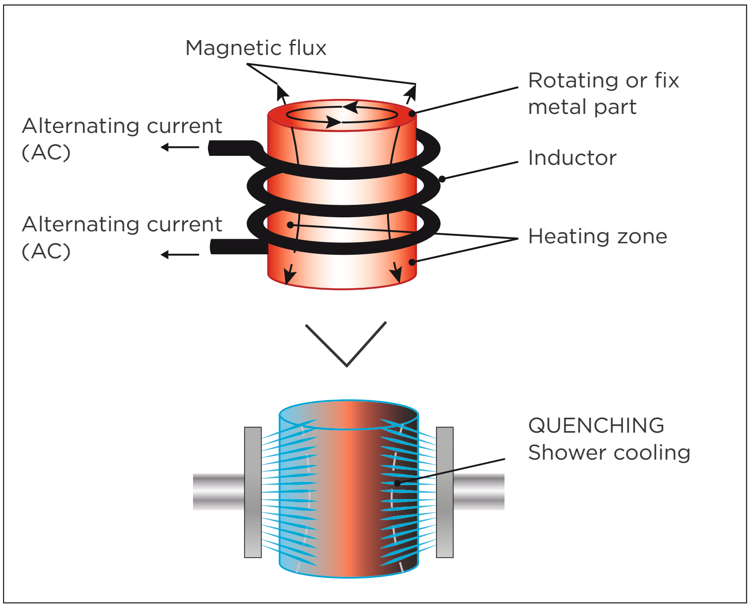
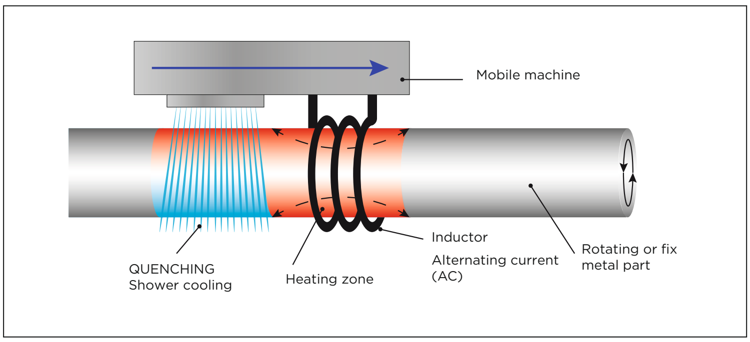
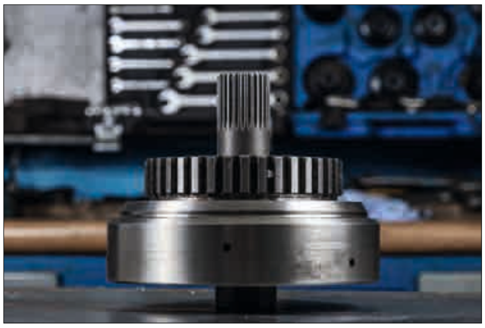


Fig. 4 Flow quenching



Applications: shaft, gears, spindle, etc.

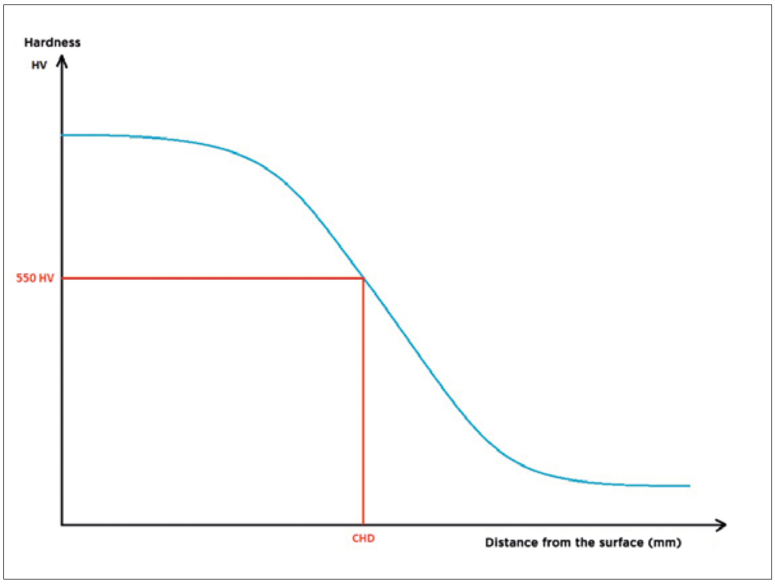



Carburizing

Carburizing is a thermochemical treatment, i.e. it takes place in a medium that modifies the composition of the base metal after exchange with the medium. In this case, carburising is the diffusion of carbon into the surface layer of the steel.

Several processes exist (case, liquid or gaseous carburising). **Gas phase carburising is the most widely used today.** The carbon-rich atmosphere (from CH4, propane or butane type gas) enriches the surface layer.

Fig. 5: Case Hardness Depth



It is used on low carbon steels and allows the surface layer to be enriched up to 0.6 to 0.9% carbon. This thermochemical treatment is followed by rapid cooling, which hardens the enriched layer.

The Case Hardness Depth (CHD) is determined by the vertical distance from the surface to the layer at a hardness limit of 550 HV. Case Hardness Depth generally varies between 0.5mm and 3mm.

Applications are mainly mechanical parts such as gears, transmission shafts, etc.


Carbonitriding

Carbonitriding is also a thermochemical treatment. It is the combination of carbon and nitrogen in the treatment environment that will result in a harder carburised layer than in conventional carburising.

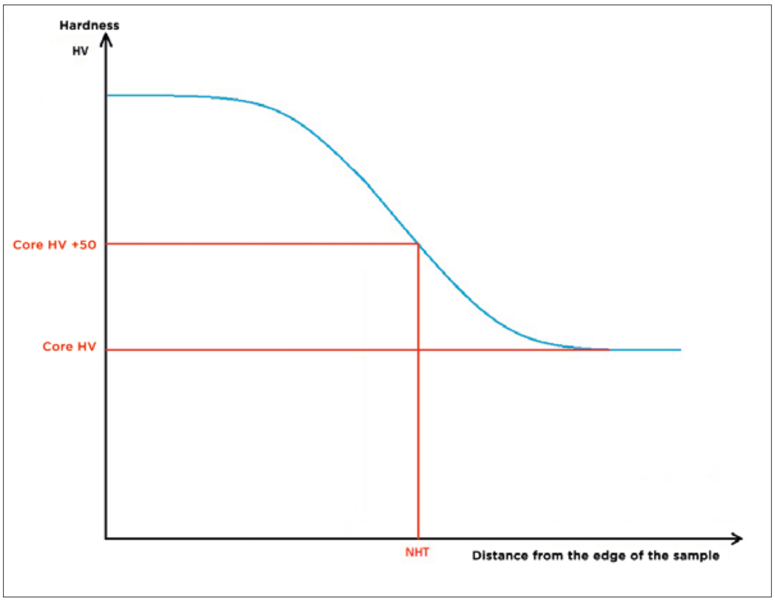
Nitriding

Nitriding is also a thermochemical treatment. It is therefore the diffusion of nitrogen on the surface of an alloy steel (which contains chromium, aluminum, tungsten, etc.). It is generally performed at between 500 and 550°C.

Nitriding consists of two layers:

• **The combination layer** ("white layer"): a nitride layer on the surface which consists of the chemical compounds present in the treatment atmosphere and the base metal. The thickness can vary from 5 to 30µm. This layer has a very high wear resistance. Its hardness generally ranges from 950 to 1100 HV.

Fig. 6: Nitriding depth

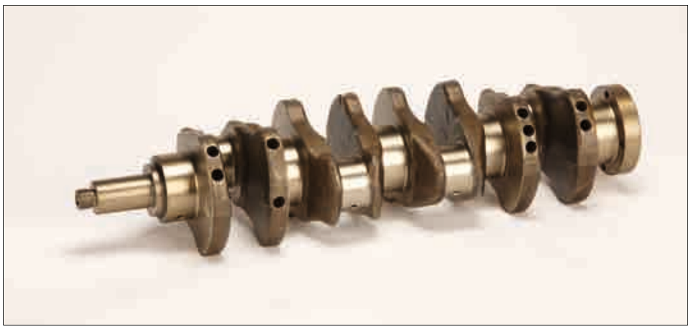


• **The diffusion layer:** Nitrides of the alloyed elements precipitate as a fine, resistant particle. This layer can vary in thickness from 0.05-0.8 mm. Its hardness can vary from 400 to 1200 HV depending on the steel used.

Several nitriding processes exist: salt bath nitriding, gas nitriding and plasma nitriding.

The nitriding depth (NHT) is determined by the hardness curve according to DIN 50190-3 or ISO 18203. After making three core hardness indentations, the nitriding depth corresponds to this value HV core + 50 HV.

Applications: Piston pins, crankshafts, valves, etc.



METALLOGRAPHIC PREPARATION

Obtaining an inspection surface requires a succession of operations, each as important as the next, regardless of the material treated. 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 steels in question.
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. 7: MECATOME ST310

Fig. 8: EVO 400

Treated steel parts are generally medium to large size parts. Two machines stand out to meet the most demanded needs in heat treatment:

• **The Mecatome ST310** is perfectly adapted to the field of heat treatment. It is a powerful and robust manual machine. The oscillating arm allows fast cuts without effort or burning.
=> The main advantage of the Mecatome ST310 is its oscillating movement. This allows the grinding wheel to never have the same point of contact with the workpiece, so there are no burns (if the chosen consumable is suitable for the material) and the cutting time is faster than with only a pendulum movement.

• **The EVO 400** is a high-capacity cutting machine. It is robust, powerful and very spacious. It is suitable for workshops and offers three cutting modes: assisted, automatic and programmable (for repeatability of cuts). Its touch screen facilitates the man-machine interface.

This machine can also offers the pulse cutting feature. It is perfectly adapted for parts with internal constraints. Pulse cutting consists of alternating feed and pause during cutting. This gives the stresses inside the material time to gradually release.

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.

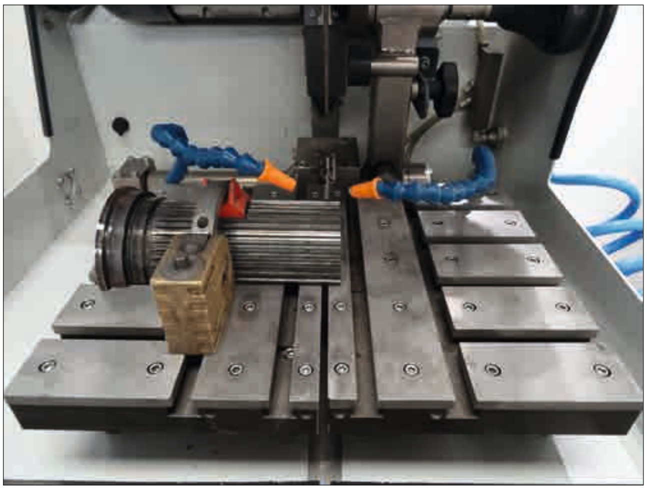


Fig. 9: Clamping on Mecatome ST310

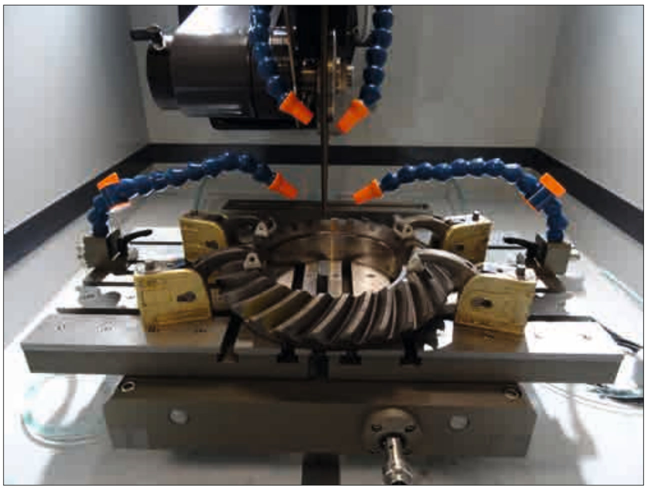


Fig. 11: Clamping on EVO 400 (crown)

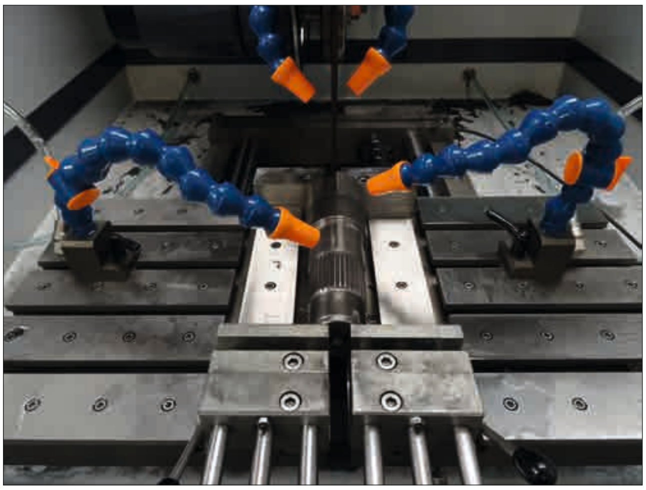


Fig. 10: Clamping on EVO 400 (pinion shaft)

Figures 9 to 11 show different clamps using Kopal clamps and quick-action vices.

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.

	Superficially-treated steels	Core-treated steels
Micro-cutting	S Ø180 mm UTW AO	S Ø180 mm CBN
Medium-capacity cutting	AO	S CBN
High-capacity cutting	AO	S

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 sample 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 12: 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 13: Pressurized mounting device

Fig 14: Vacuum mounting device: POLY'VAC

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

+ 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.

For hardness control and especially for checking the conformity of surface treatment at the edge of the sample, it is important to have a resin that offers the lowest shrinkage between the sample and the resin. Hot epoxy resin is suitable because it solves this problem with excellent characteristics and therefore, in most cases, results in limited shrinkage.

In order to reduce costs, it is possible to introduce the epoxy resin in the sole (i.e. in contact with the surface of the sample to be observed) and fill the rest with a less qualitative, and therefore less expensive, phenolic resin. If cold mounting has to be performed on treated steels, it is preferable to opt for KM-B or KM-U acrylic resin, both of which have hardnesses close to those of hot resins.

	Treated steels
Hot process	Epoxy
Cold process	KM-B (pressurised) KM-U (pressurised)

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.

For hardness testing, polishing with an automatic polishing machine from the Mecatech range seems to be the most suitable.

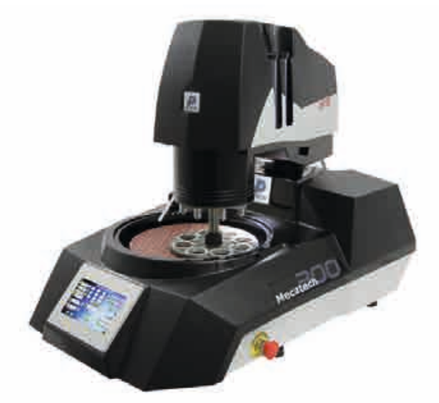


Fig. 15: MECATECH 300 SPC

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.

This range is suggested for **superficially and core-treated steels:**

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	RAM	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’

NB: Levelling with the I-Max R 54µm is sufficient for a sample issued from a metallographic cut. If more material has to be removed, an I-Max R with a larger grain size (75µm or even 125µm) should be used.

Grinding is performed using I-Max R discs. These resin-bonded diamond discs can replace several hundred abrasive papers. They provide good flatness for polishing hard ferrous materials.

When grinding, it is not necessary to reverse the rotation direction of the head and platen as this can detrimentally affect flatness. However, reversing the directions of rotation can help if a large amount of material has to be removed.

A 3-step range is sufficient for hardness testing. The scratch pattern obtained with the 3µm suspension is fine enough to allow the hardness indentations to be read. The RAM cloth can also be replaced by ADRII cloth.

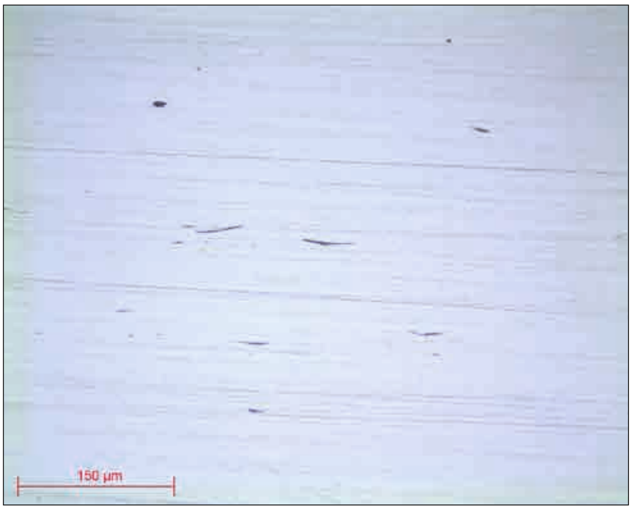


Fig. 16: Finish 3µm lens x20

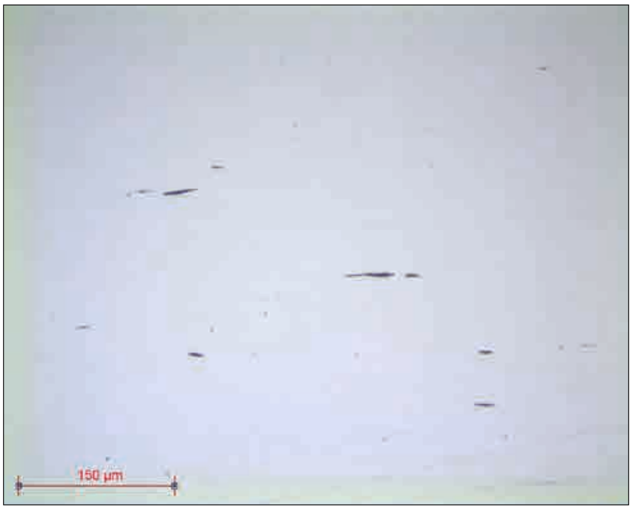


Fig. 17: Finish 1µm lens x20

If structural observation is required, a 1µm finish on an NT cloth can follow.
Finally, a superfinish with PRESI n°3 alumina can be used to make an inclusionary check for example.

Fig. 18: Finish 1µm lens x50

Fig. 19: Finish Alumina n°3 lens x50

This is a second possible range for **superficially or core-treated steels:**

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	MED-R	9µm super abrasive / Ø	150	135	⇉	4’
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’

For this second range, step 2 is replaced by an MED-R disc. This disc, composed of resin pads, makes it possible to replace several cloths and maintain good flatness. It is used with a super abrasive suspension for MED-R which is a 2-in-1 product (the abrasive suspension and lubricant are already mixed and ready to use).

For diamond polishing, steps 3 and 4 are carried out using LDP concentrated polycrystalline suspensions. Polycrystalline diamond has sharp angles which are suitable for polishing medium-hard to hard materials.

If the materials to be polished are sensitive to corrosion, LDP diamond suspensions can be replaced by alcohol-based, anhydrous polycrystalline ADS diamond suspensions.

Sometimes it is necessary to adapt the range according to the treatment, especially in the case of **nitrided steels.**

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	→ →	3’
4	NT	1µm LDP / Reflex Lub	150	135	→ →	1’
5	NT	Al_2O_3 n°3 / Water	150	100	→ ←	1′

<u>NB</u>: Levelling with P320 abrasive paper is sufficient for a sample issued from a metallographic cut. If more material needs to be removed, a larger grit size abrasive paper should be used.

For nitrided steels, polishing with I-Max R or MED-R is too aggressive and could damage the combination layer. It is therefore preferable to replace the I-Max R with a P320 abrasive paper.

The following steps are carried out conventionally using polishing cloths and LDP diamond suspensions and the associated lubricant Reflex LUB. Finally, superfinishing is optional using alumina on an NT flocked cloth.

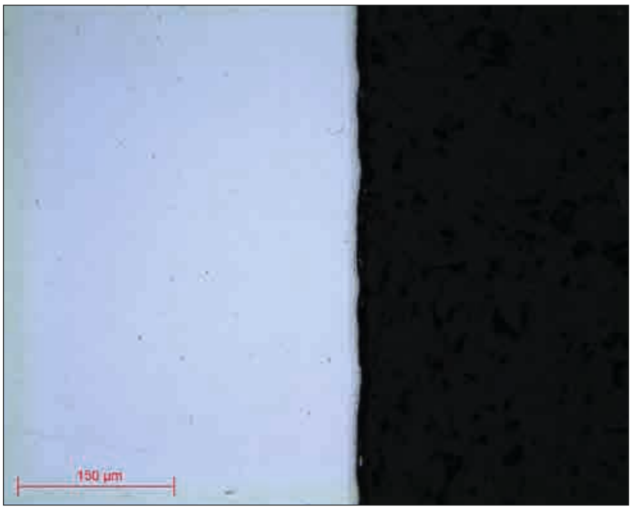


Fig. 20: ¼ µm nitrided steel finish lens x20

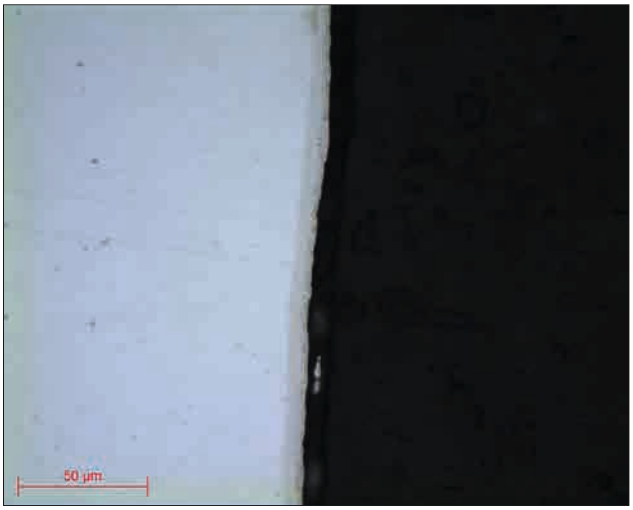


Fig. 21: ¼ µm nitrided steel finish lens x50

MICROSTRUCTURE

Treated steels are generally etched using Nital 4% etching solution (except for stainless steels which have their own etching solution). This reveals the structure and, in the case of nitrided steels, highlights the combination layer (white layer). All of the micrographs presented were produced using the **PRESI VIEW** software:

Fig. 22: Martensitic hardening lens x50

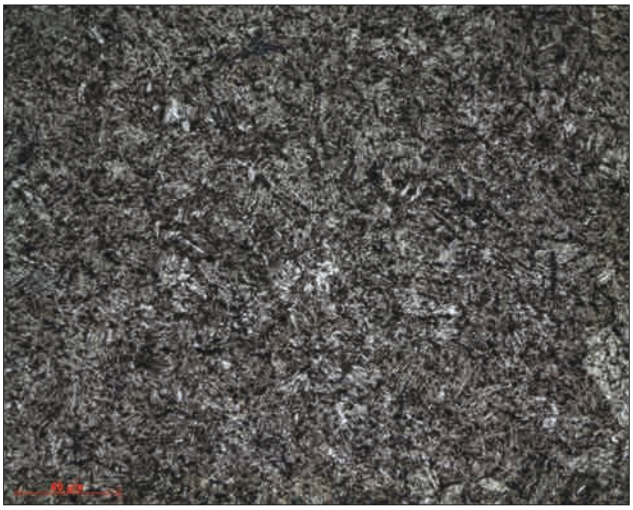


Fig. 23: Martensitic hardening-tempering lens x50

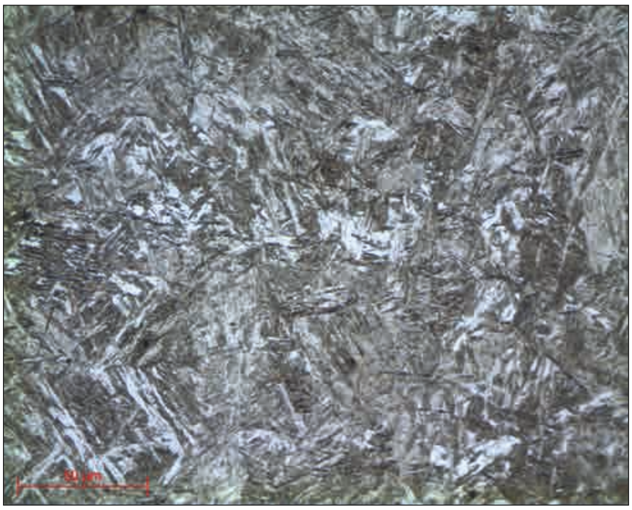


Fig. 24: Martensitic structure, edge lens x50

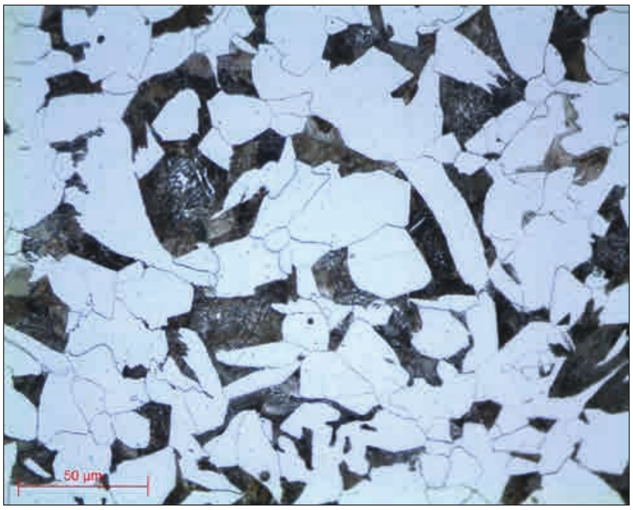


Fig. 25: Ferrito-perlitic structure, core lens x50

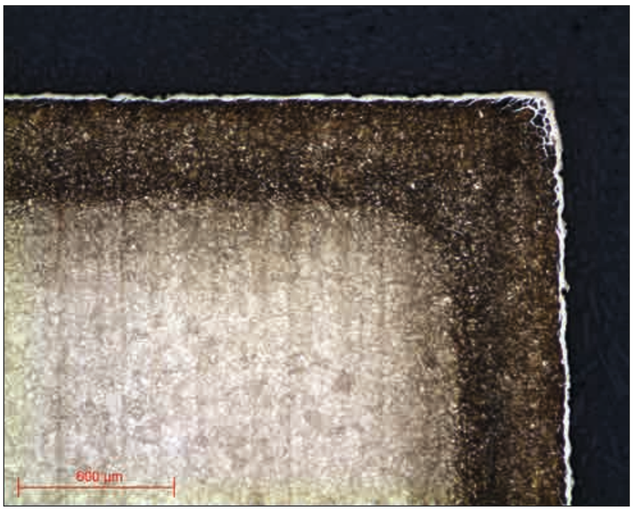


Fig. 26: Nitrided steel structure lens x5

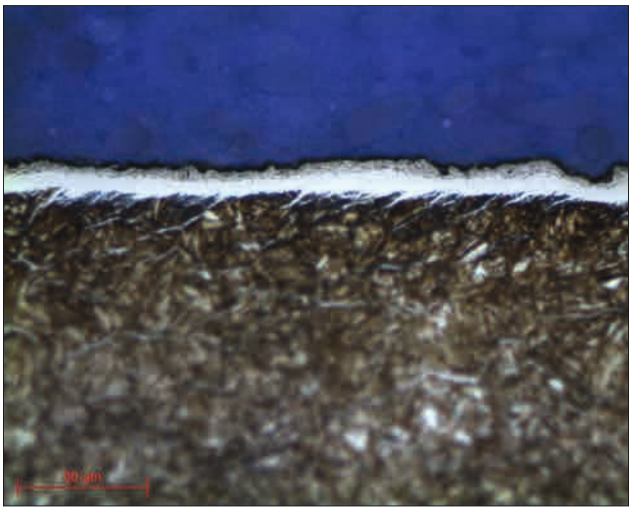


Fig. 27: White layer – nitriding lens x50

Figures 22 to 27 are examples of structures that may be encountered when inspecting steel that has been heat-treated.

HARDNESS PATTERNS

Usually, hardness tests are carried out to check the conformity or otherwise of the heat-treated part. This will therefore show variation in hardness if the treatment is superficial (harder externally and less hard throughout) and a lesser variation (with fairly similar hardness values) throughout the workpiece if it has been treated in the mass.
To carry out these hardness tests, **the HZ 10-4 hardness tester should be used, with the PRESI Touch Pattern software.**

Fig. 28: HZ 10-4 hardness tester

Fig. 29: Presi Touch Pattern screen

The HZ 10-4 hardness tester has a load range of 10 g to 10 kg. It is equipped with a single Vickers or Knoop indenter and it is possible to have up to 4 lenses, allowing magnification from x 200 to x 2000.

A macroscopic camera is available as an option and provides an overall view of the part (facilitates the positioning of patterns).

The PRESI Touch Pattern software shows automatic patterns for heat treatment, CHD, NHT, etc.

Navigation is intuitive and all functions are directly accessible. Data and results are displayed at all times.

Patterns can be regular or irregular and it is possible to make isolated points. A preview of the indents helps to position the pattern before testing.

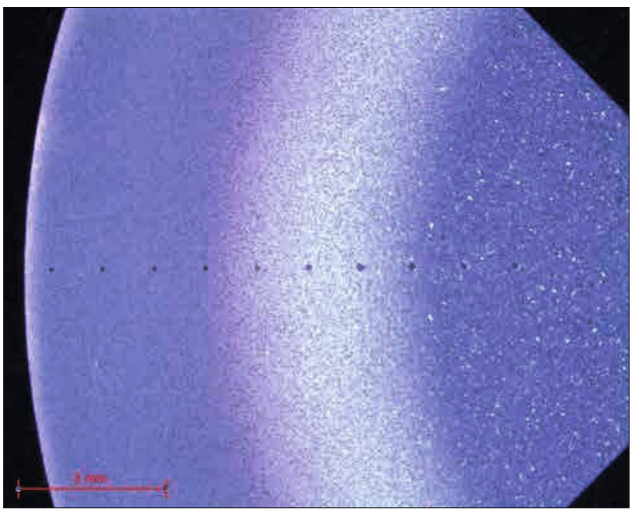


Fig. 30: Workpiece with hardness pattern

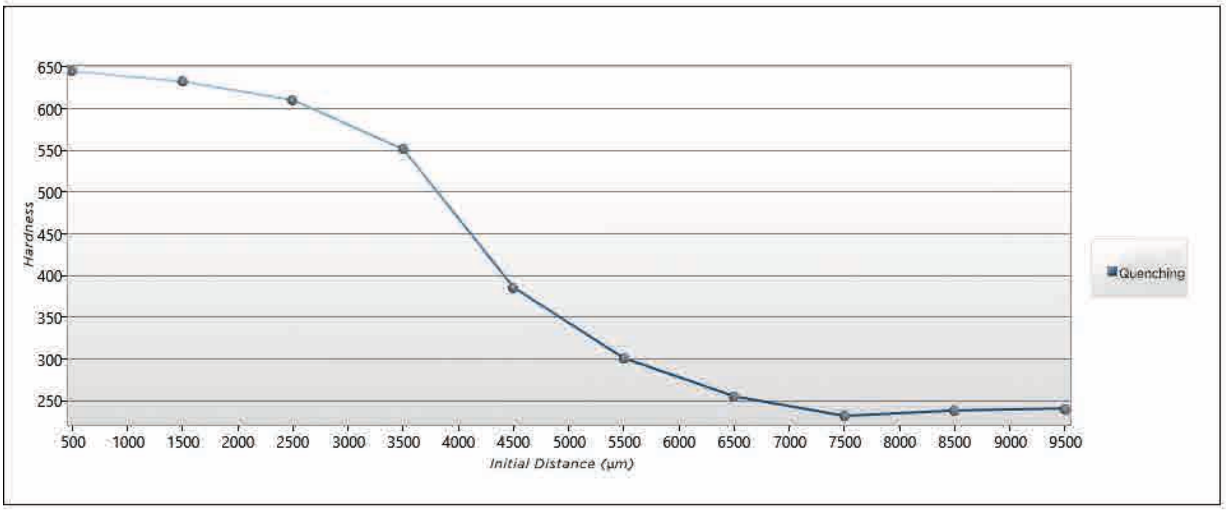


Fig. 31: Graph of induction-treated steel