

METALLOGRAPHIC PREPARATION OF TITANIUM

INTRODUCTION

Titanium is a metal that is abundant on Earth, but its extraction and purification are not easy. It was discovered at the end of the 18th century by the mineralogist William Gregor, but it was not until 1939 that William Justin Kroll developed an industrial manufacturing process. As a result, one of the major drawbacks of titanium is its still relatively high cost.

TITANIUM

Symbol: **Ti**

Atomic n°: **22**

Density: **4,5**

Molar mass: **47,9g.mol⁻¹**

Melting point: **1670°C**

PROCESSING

The manufacturing process of titanium bears the name of its developer: the Kroll process, which consists of two stages.

- The first stage consists of reacting the titanium oxides with carbon and chlorine (an operation called "carbochlorination") in order to obtain titanium tetrachloride. This reaction is written:



- The second step allows solid titanium to be obtained by reducing the TiCl_4 with magnesium:



The titanium obtained is in the form of a porous solid called "sponge". This sponge is then melted down to obtain pure titanium or the desired titanium alloy (after additions to the molten metal). The titanium is resolidified into an ingot.

These ingots can be transformed into semi-finished products (slabs, billets, blooms) by machining or forging, then into finished products (bars, coils, plates, cables, etc.) by various operations (rolling, forging, extrusion, etc.).

PROPERTIES AND USES

Titanium is a very remarkable metal. It has excellent mechanical properties (good fatigue strength, high resilience, resistance to heat and cryogenics, acceptable creep resistance, etc.) for a density of about 60% of that of steel ($D_{\text{titanium}} = 4.5 < \text{Steel} \approx 7.8$). Its specific strength (mechanical strength/density ratio) is therefore higher than that of aluminum or steel.

In addition to its very good density, titanium has exceptional corrosion resistance (higher than that of stainless steels) even in the most aggressive environments. This resistance combined with its biocompatibility and low modulus of elasticity (110,000MPa) makes it an ideal metal for the medical field.

It should be noted that heat and mechanical treatments are carried out on titanium in order to modify its physico-chemical properties.

[There are four classes of titanium alloys:](#)

UNALLOYED TITANIUM

is mainly used in the chemical field for its corrosion resistance properties and its cold deformability. It has excellent weldability. Example of grade: T40.

THE ALPHA ALLOYS α

also have excellent weldability but are difficult to cold-form and not heat-treatable. They are used for cryogenic applications, aerospace parts and in the chemical industry. Example of grade: TA5E (TiAl5Sn2.5).

THE ALPHA-BETA ALLOYS $\alpha+\beta$

offer the most possibilities due to their ability to be heat-treated, their compositions and therefore their different properties. They are used for aeronautical parts (structures, turbojets, etc.) or for marine and biomedical applications. On the other hand, they are more difficult to weld.

Example of grade: TA6V (TiAl6V4).

BETA ALLOYS β offer an excellent combination of mechanical properties: hardness - ductility - fatigue resistance. They are weldable but nevertheless not heat-treatable. These alloys are mainly used for highly stressed structural aerospace parts.

Example of grade: Ti.10.2.3 (TiV10Fe2Al3).

=> A fifth category can also be defined: **INTERMETALLIC TIAL COMPOUNDS**. They are alloys of titanium, aluminum (generally between 45 and 48%) and additive elements. They are of interest for high temperature applications (aeronautics, automotive, etc.)

Example of grade: TiAl47Nb2Cr2.

However, the majority of titanium is used in the form of titanium dioxide (TiO₂) which is an excellent pigment and/or thickener. It is subsequently used for paints, plastics, paper, cosmetics, sun creams, etc.



METALLOGRAPHIC PREPARATION

In general, processing and transformation operations and the various mechanical and thermal treatments influence the properties and microstructures of titanium and its alloys.

All these influences then lead to the realization of metallographic quality controls such as: microstructure examinations, porosity and/or heterogeneity research, inclusion cleanliness, hardness tests, hardening controls, grain size controls, 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 titanium.

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 T210



Fig 2: MECATOME T330



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.

In addition, titanium is very sensitive to burning during metallographic cutting, which makes it all the more important to determine the appropriate consumables and parameters.

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.

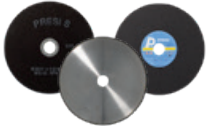
	
TITANIUM AND ALLOYS	
Micro-cutting	UTW S Ø180 MNF
Medium-capacity cutting	T MNF F
High-capacity cutting	T MNF

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 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 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.

TITANIUM AND ALLOYS	
Hot process	Hot epoxy Phenolic Allylic Conductive phenolic** Acrylic copper powder**
Cold process	KM-U KM-B 2S* KM-CO**

Table 2: Choosing the right mounting resin type

* Suitable for very large series
 ** Conductive resins for SEM observations

Titanium is very often observed by a Scanning Electron Microscope (SEM).

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 SPI

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 750-1500 W, all the PRESI experience is concentrated in this very complete range. Whatever the sample number or size, MECATECH guarantees optimal polishing.

As a material, titanium polishes in a very special way, it is relatively soft and prone to incrustations and plastic deformation. As a result, during a polishing process where the material is usually cut/removed by the abrasive, the material will in this case have a tendency to "bend" back on itself. This is known as "buttering". This phenomenon is undesirable and greatly hinders microscopic observation. Titanium therefore has its own specific polishing ranges in order to guarantee an excellent examination surface.

CONSUMABLES AND POLISHING RANGE

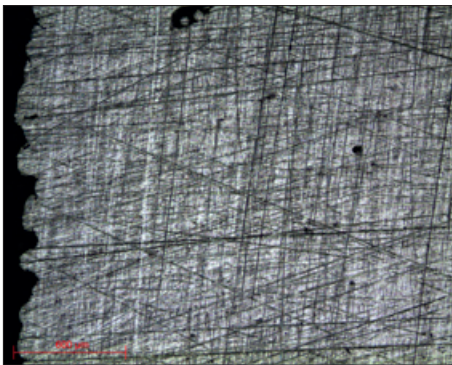
All the polishing ranges below are given for automatic sample preparation (for manual polishing: do not take into account the parameters at the top). They are the most commonly used and are given for information and advice.

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.

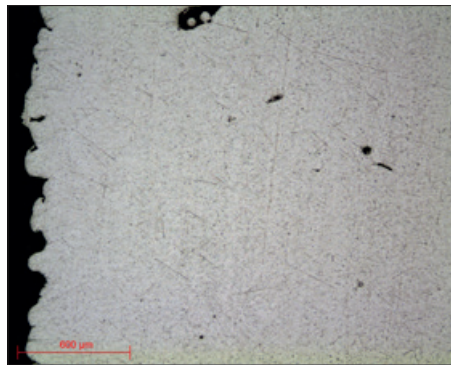
Applied pressures vary according to sample size, but in general the following applies: 1daN per 10mm mounting diameter for the pre-polishing steps (ex: Ø40mm = 4 daN) then reduce force by 0.5daN at each polishing step with an abrasive suspension.

RANGE n°1

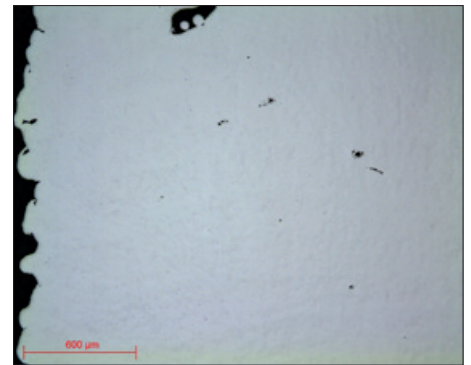
N°	Support	Suspension / Lubricant	Platen Speed (RPM)	Head Speed (RPM)	Rotation direction platen / head	Time
1	Sic P320	Ø / Water	300	150	→ →	1'
2	TOP	9µm LDP / Reflex Lub	150	135	→ →	5'
3	SUPRA	SPM / Water	150	100	→ ←	5'



Micrograph 1:
Surface condition P320 lens x5



Micrograph 2:
Surface condition TOP 9µm lens x5



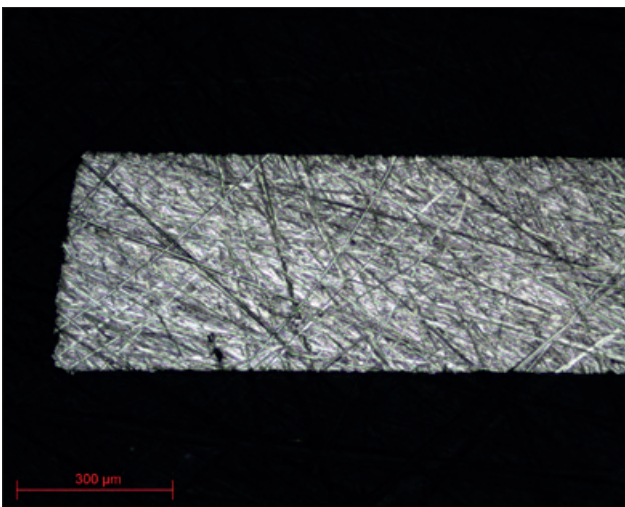
Micrograph 3:
Surface condition SUPRA SPM lens x5

RANGE n°2

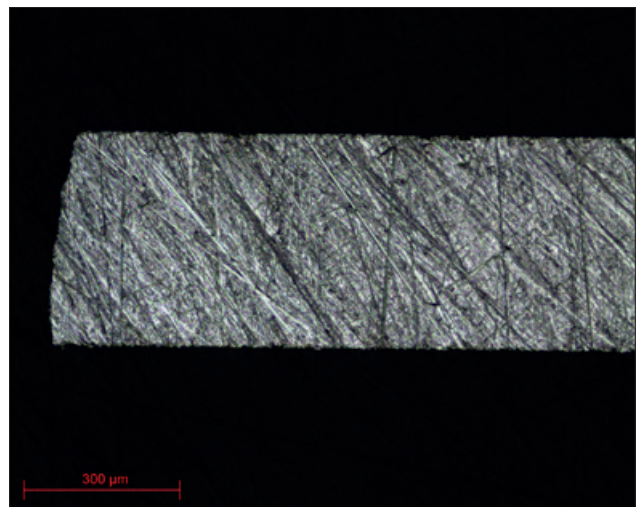
N°	Support	Suspension / Lubricant	Platen Speed (RPM)	Head Speed (RPM)	Rotation direction platen / head	Time
1	SiC P320	Ø / Water	300	150	→ →	1'
2	SiC P600	Ø / Water	300	150	→ →	1'
3	SiC P1200	Ø / Water	300	150	→ →	1'
4	SiC P2400	Ø / Water	300	150	→ →	1'
5	SiC P4000	Ø / Water	300	150	→ →	1'
6	SUPRA	SPM / Water	150	100	→ ←	5'

RANGE n°3

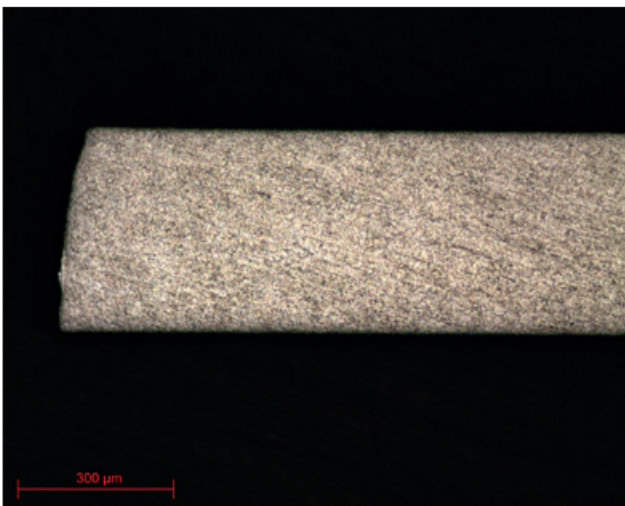
N°	Support	Suspension / Lubricant	Platen Speed (RPM)	Head Speed (RPM)	Rotation direction platen / head	Time
1	SiC P320	Ø / Water	300	150	→ →	1'
2	SiC P1200	Ø / Water	300	150	→ →	1'
3	RAM	Al ₂ O ₃ N°4 / Water	150	100	→ ←	2'
4	SUPRA	SPM / Water	150	100	→ ←	3'



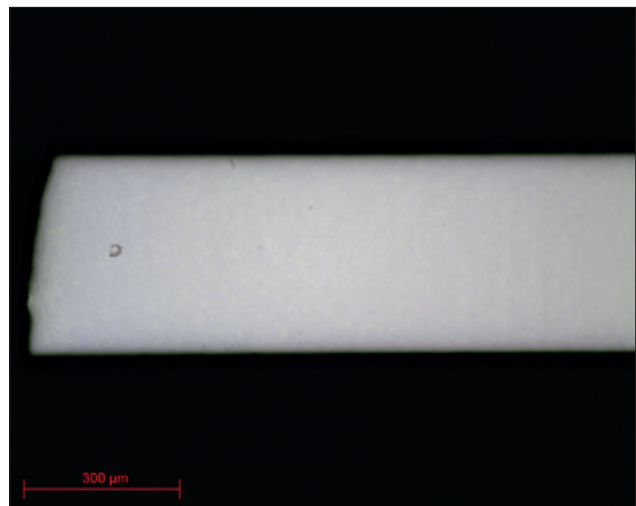
Micrograph 4: Surface condition P320 lens x5



Micrograph 5: Surface condition P1200 lens x5



Micrograph 6: Surface condition RAM Al₂O₃ N°4 lens x5



Micrograph 7: Surface condition SUPRA SPM lens x5

The polishing ranges given above are complete and are to be carried out in their entirety. The super-finishing step using colloidal silica solution is compulsory. It delivers an excellent inspection surface free of any scratch or buttering phenomenon.

However, an alternative has been developed in the particular case where colloidal silica is not suitable. The SPM can be replaced by a suspension of Al₂O₃ N°2 with the same parameters.

=> **Range N°1** is the most versatile range, is effective on most titanium grades and provides an excellent inspection surface.

=> **Range N°2** is the traditional titanium polishing range, its advantage being the absence of polishing steps using diamond suspension. Diamond polishing should be restricted to the bare minimum, because it tends to encrust and "butter" the surface of the specimen.

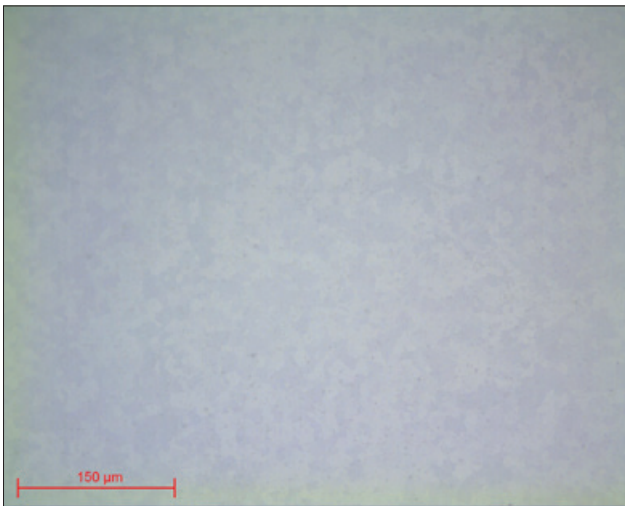
=> **Range N°3** was developed specifically for polishing a very soft titanium grade, i.e. unalloyed titanium (T40 for example). The diamond suspension must not be used in this case.

After this preparation, the polished specimens can be observed directly without metallographic etching. Moreover, titanium's crystallography allows very good inspection of the structure under an optical microscope, revealing it with the help of a polarised light filter.

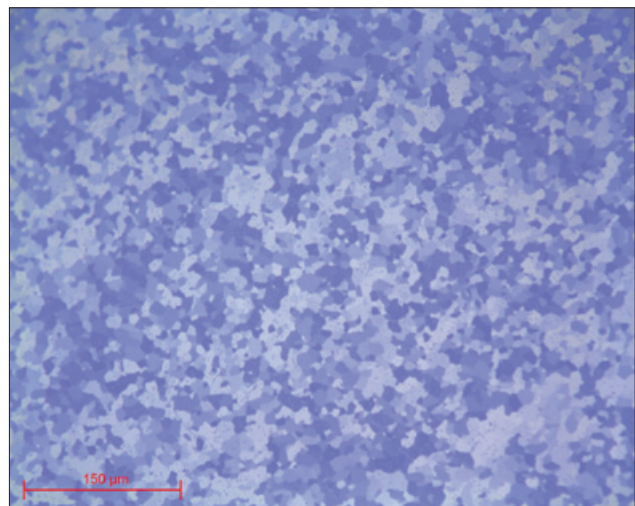
Metallographic etching of titanium is commonly done using Kroll's reagent: a solution of 3mL of hydrofluoric acid and 6mL of nitric acid for 100mL of water. The etching creates differences in background and/or colour between the different constituents, allowing them to be inspected.

MICROSCOPY

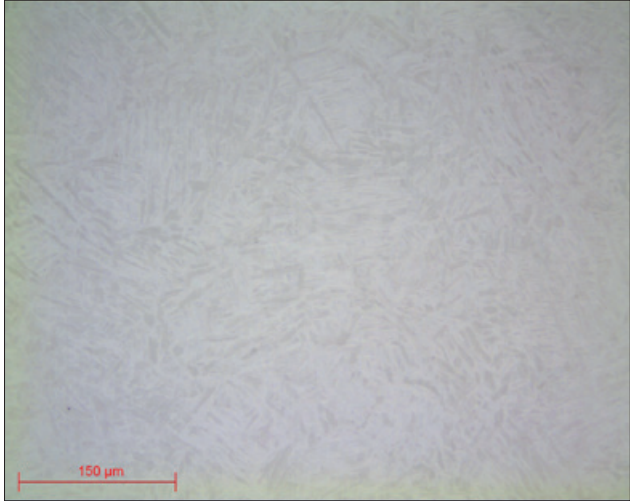
All micrographs presented were created using the **PRESI VIEW** software:



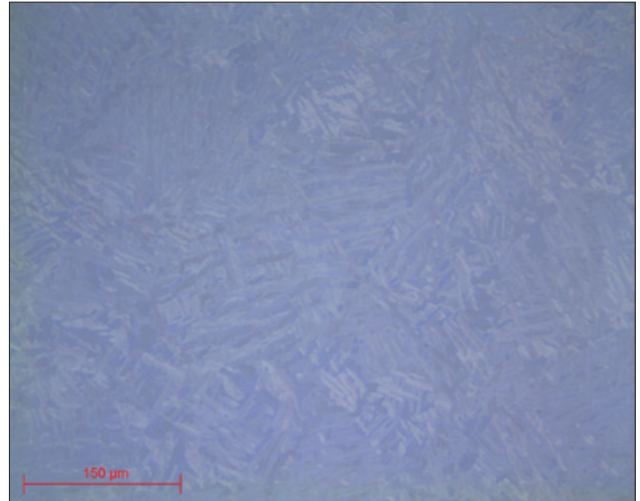
Micrograph 8: TA6V untreated polished up to SPM lens x20



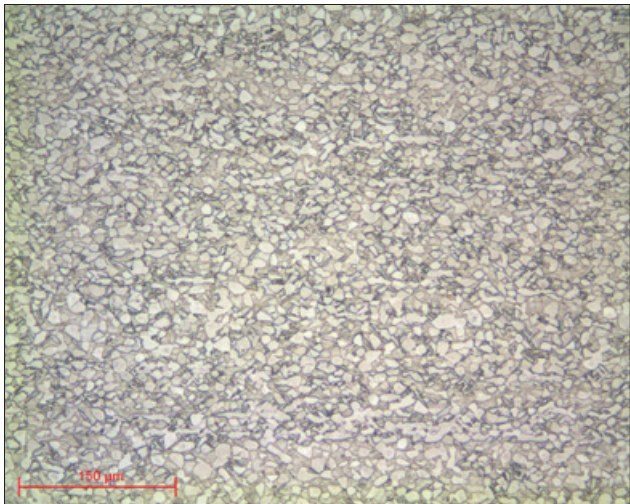
Micrograph 9: TA6V untreated polished to SPM and observation under polarized light lens x20



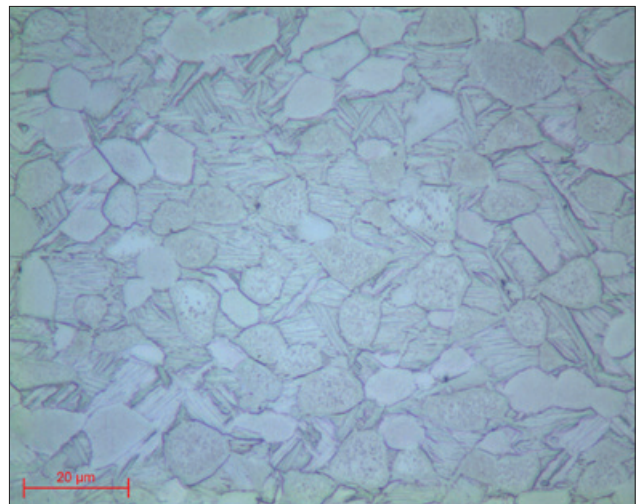
Micrograph 10: TA6V polished up to SPM lens x20



Micrograph 11: TA6V polished to SPM and observation under polarised light lens x20



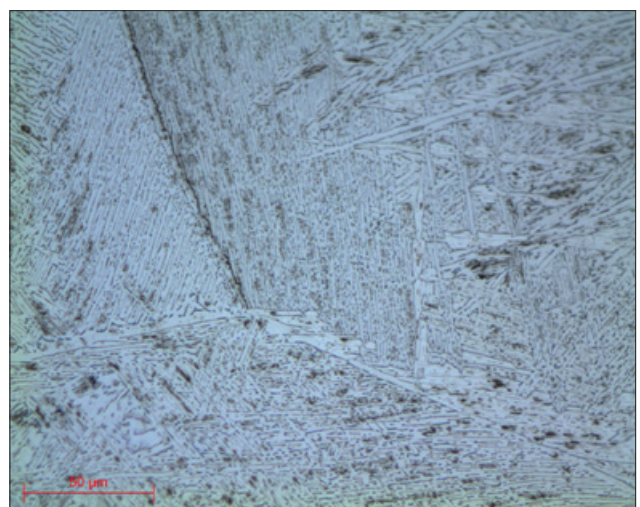
Micrograph 12: TA6V untreated Kroll lens x20 TA6V reagent etched



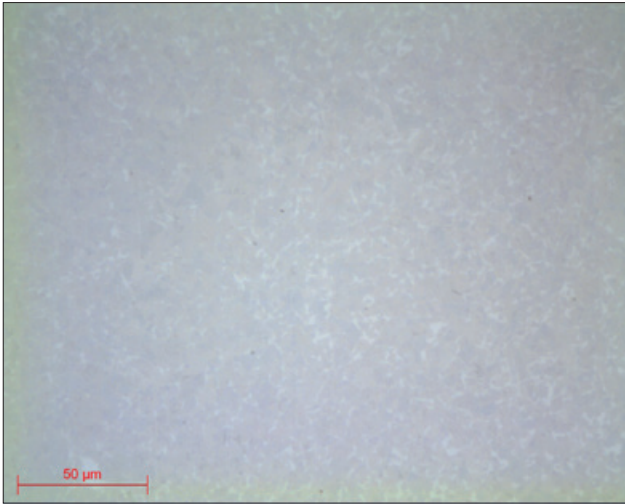
Micrograph 13: TA6V untreated Kroll lens x100 TA6V reagent etched



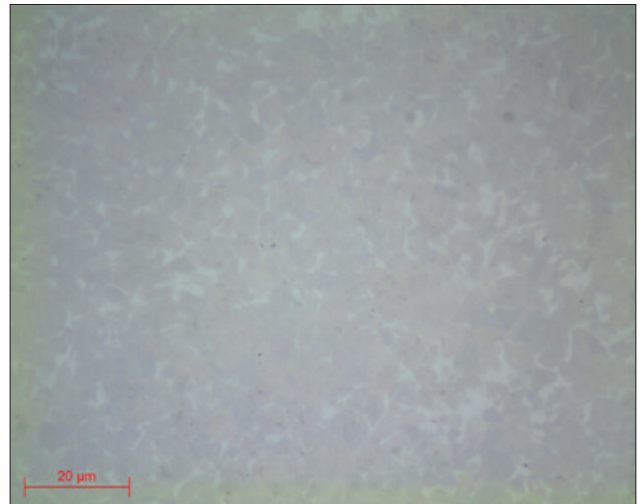
Micrograph 14: TA6V treated with Kroll lens x20 reagent etching



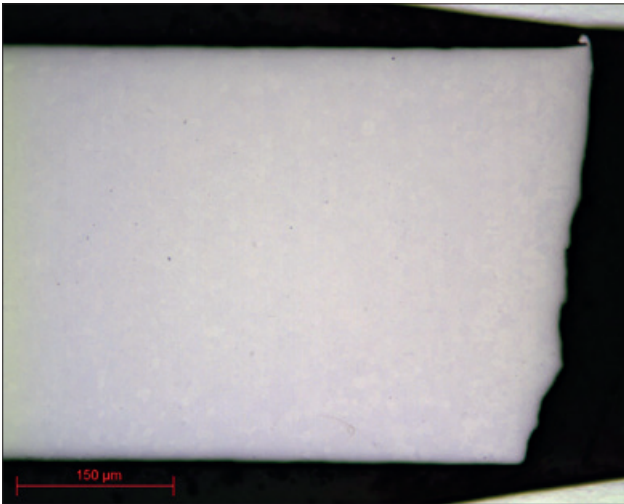
Micrograph 15: TA6V treated with Kroll lens x50 reagent etching



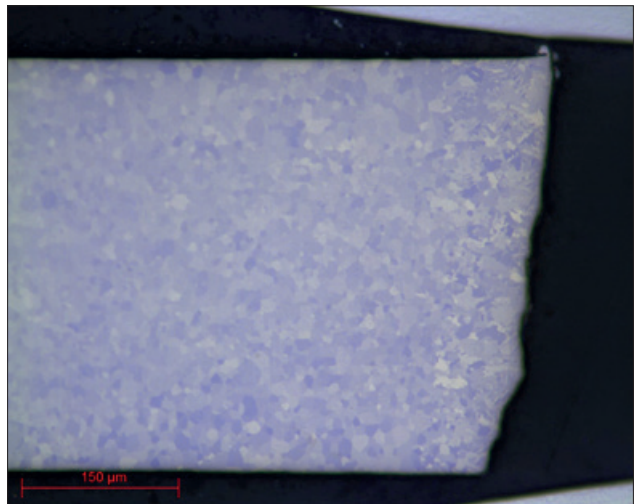
Micrograph 16: Intermetallic compound TiAl polished to Al₂O₃ N°2 lens x50



Micrograph 17: Intermetallic compound TiAl polished to Al₂O₃ N°2 lens x100



Micrographs 18: T40 polished up to SPM lens x20



Micrograph 19: T40 polished to SPM observation under polarised light lens x20

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