PRESI
LAB'NOTE

METALLOGRAPHIC PREPARATION OF CERAMIC MATERIALS

INTRODUCTION

Ceramics (called "technical ceramics" in the industrial field) are non-metallic and inorganic materials obtained by high-temperature processing. Their composition is very varied; ceramics contain a large proportion of the elements in the periodic table.

They have high melting points, very high hardness and no ductility. Common manufacturing processes (machining, casting, plastic deformation, etc.) are therefore either unsuitable or only marginally so. Manufacture involves manipulating a powder before the product consolidation operation.

PRODUCTION PROCESS

There are several methods of manipulating ceramics to obtain a granular structure:

LIQUID PROCESSES

which consist of dispersing ceramic powders in a solvent (water, alcohol, etc.) in order to obtain a suspension with the desired properties (solvents are then eliminated during subsequent thermal cycles). This suspension obtained makes it possible to carry out casting operations in moulds or tape and deposits by dipping.
Example of tape casting: the suspension is laminated and then dried by infrared radiation before being laser-cut and assembled into multiple layers.

PLASTIC PROCESSES

which involve mixing technical ceramic powders with organic bonds in order to obtain a fluid (bonds are removed in subsequent thermal cycles). It is then shaped by injection or extrusion.
Example of injection: the "ceramic fluid" is fed into the hopper, then heated, compressed and injected into the mould before being cooled and removed from the mould.

DRY GRANULAR PROCESSES

to agglomerate powder particles for mould filling, giving them sufficient plasticity for deformation during pressing.
Example of single-axis pressing: the mould is filled with the ceramic powders which are then pressed and subsequently removed from the mould.

Once the granular structure is obtained, the last step in the manufacture of technical ceramics is high-temperature consolidation called **"sintering"**. The principle is to assemble the grains constituting the ceramic by heating to very high temperatures (but below the ceramic's melting point) for a long period of time. This operation also densifies the ceramic (reduction of porosities), enlarges grains and eliminates the various bonds used during shaping.

Technical ceramics have a variety of physical properties that offer suitable solutions where metallic materials and polymers may be ineffective. Among these properties, the most important are the following:

- **Mechanical properties**: their extreme hardness offers very good resistance to wear, abrasion and compression.
- **Thermal properties:** resistant to very high temperatures (up to 2,000°C), ceramics are the reference in refractory materials.
- **Electrical properties:** some ceramics are excellent electrical insulators and others, on the contrary, are (super)conductors.
- **Chemical properties:** some possess chemical inertness, biocompatibility and vacuum tightness.
- **Optical properties:** some transparent ceramics have exceptional optical properties (visible, IR or UV ranges).

=> All these properties make technical ceramics remarkable materials with numerous industrial applications:

The electronics field accounts for 70-75% of global turnover for technical ceramics. Their various compositions and properties of use mean electronic ceramics can be used in a variety of applications: electrical insulation (Al_2O_3, SiO_2, MgO, etc.), semiconductors (SiC, Cu_2O, TiO_2, etc.), electrical conductors (ReO_2, $MoSi_2$ LaB_6, etc.) and magnetic ceramics (Fe_3O_4, $NiFe_2O_4$, etc.).

The medical field also uses ceramics, commonly known as "bioceramics". They are used in medical instruments and systems, reconstructive surgery (prostheses, implants, bone substitutes, etc.) and in the dental field (implants, bridges, etc.). Alumina (Al_2O_3) and zirconium dioxide (ZrO_2) are the most commonly-used ceramics due to their density, purity, tribological qualities and mechanical resistance.

Technical ceramics are commonly used as filters or membranes in the **energy and environment fields**. Particle filters or catalytic converters (some with honeycomb structure) are made using ceramics and allow the filtering and/or degradation of gas pollutants (SiC often used for its thermal conductivity but also Al_2O_3, CeO_2, ZrO_2 on which noble metals are deposited in the particular case of catalytic converters). Ceramics are also used as fuels in the nuclear field (UO_2, PuO_2, etc.).

The use of ceramic components in the **telecommunications** field now holds sway, in particular due to their resistance to their environment and the stresses to which they are subjected (humidity, vibrations, temperature variations, etc.).

In the **aerospace field too**, technical ceramics have a multitude of applications (turbine blades, telescope mirrors, sensors, combustion chambers, engines, etc.).

In order to compensate for their fragility, ceramics can be used as components of composite materials. Ceramics generally make up the matrix containing a set of fibres (glass, carbon, silicon carbide, etc.) called “strengthening”. These composites are called "ceramic matrix composites" (CMC).

METALLOGRAPHIC PREPARATION

In general, during the elaboration, powder forming, processing operations and/or various mechanical, thermal and chemical treatments, the properties and structures of ceramic products are influenced. All these influences lead to metallographic quality controls such as: examinations of porosities, dimensioning, tearing, structures and microstructures, searches for heterogeneities, search for and examination of inclusions and/or impurities, hardness tests, 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: 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 ceramic product.
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 T205

Fig 2: MECATOME T215

Fig 3: MECATOME T265

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.

	**Ceramics**
Micro-cutting	LM / LM+ / LR
Medium-capacity cutting	LM / LM+ / LR
High-capacity cutting	LM / LM+ / LR

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.

As ceramic materials are fragile, the hot mounting process is not possible because the pressures exerted are too high and could therefore damage the samples. In such cases, the cold mounting process is to be preferred. Some complex geometries also require cold mounting to allow the resin to impregnate the specimen as well as possible.
This type of mounting is also preferred if a large number of parts are to be mounted in series.

The cold process can be used with:

Fig 4: Pressurized mounting device

+ POINT

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

Fig 5: 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.

	**Ceramics**
Cold process	KM-U KM-B IP - IP FAST 2S*

Tableau 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 6:

MINITECH 300 SP1

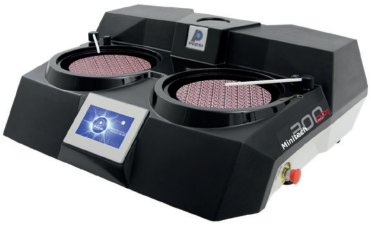


Fig 7:

MINITECH 300 DP1 et DP2

Fig 8:

MECATECH 250 SPI

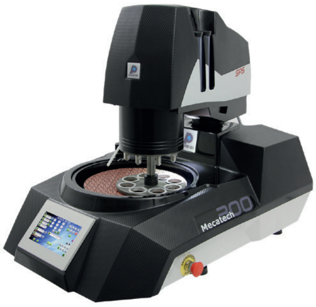


Fig 9:

MECATECH 300 SPS

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

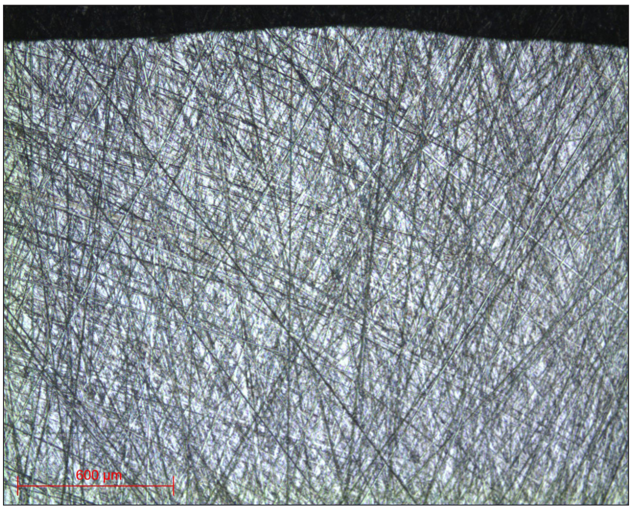
All the polishing ranges below are given for automatic sample preparation (for manual polishing: do not take into account the parameters at the top). They are the most commonly used and are given for information and advice.

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.

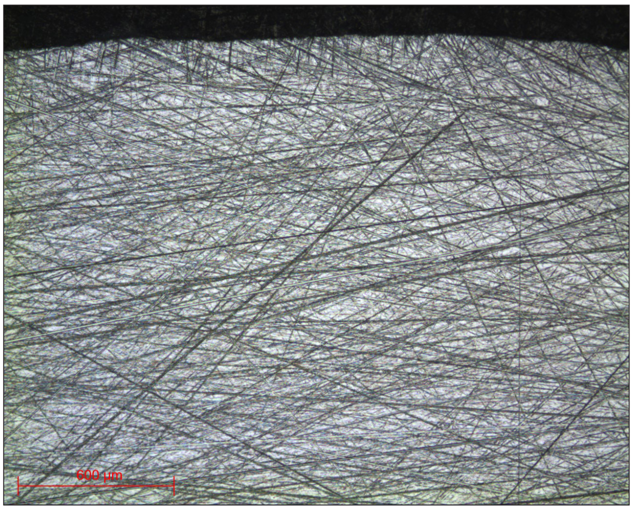
Applied pressures vary according to sample size, but in general the following applies: 1daN per 10mm mounting diameter for the pre-polishing steps (ex: Ø40mm = 4 daN) then reduce force by 0.5daN at each polishing step with an abrasive suspension.

Range N°1

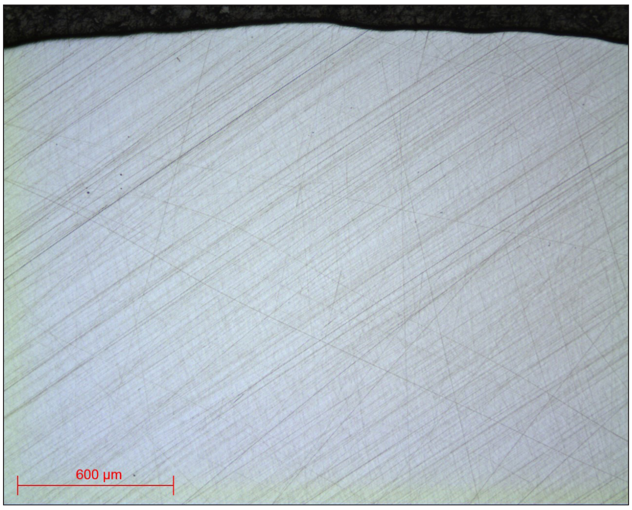
N°	Support	Suspension / lubricant	Platen Speed (RPM)	Head Speed (RPM)	Rotation direction platen / head	Time
1	Tissediam 40µm	Ø / Water	300	150	→ →	2'
2	Tissediam 20µm	Ø / Water	300	150	→ →	2'
3	TOP	9µm LDP / Reflex Lub	150	135	→ →	5'
4	NWF+	3µm LDP / Reflex Lub	150	135	→ →	2'
5	SUPRA	SPM / Water	150	100	→ ←	2'



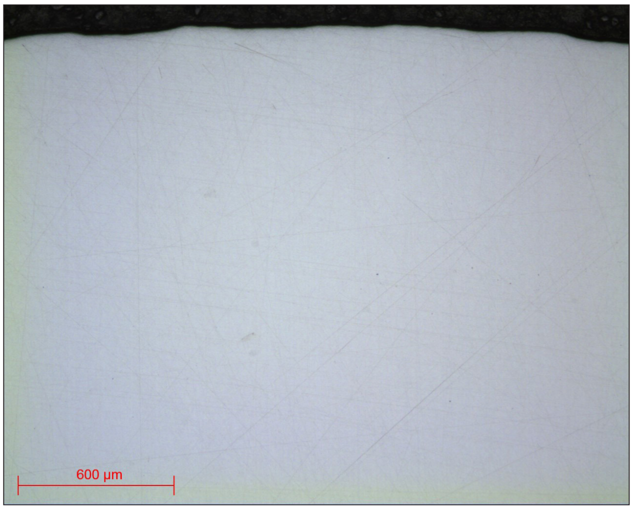
Micrograph 1: zirconium dioxide ZrO_2
Surface condition TISSEDIAM 40µm lens x5



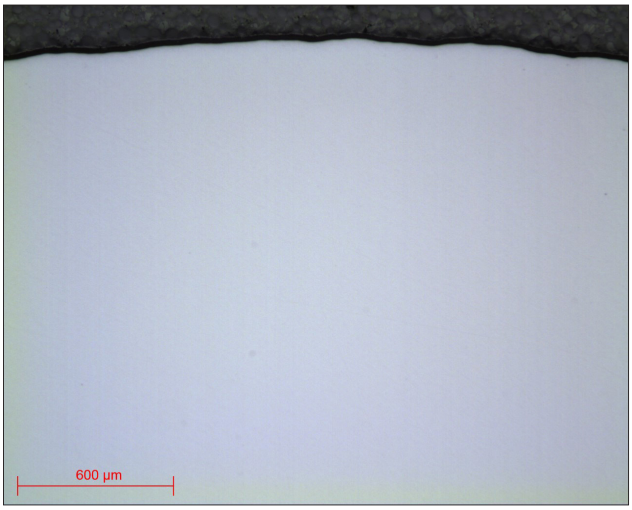
Micrograph 2: zirconium dioxide ZrO_2
Surface condition TISSEDIAM 20µm lens x5



Micrograph 3: zirconium dioxide ZrO_2
Surface condition TOP 9µm lens x5



Micrograph 4: zirconium dioxide ZrO_2
Surface condition NWF+ 3µm lens x5



Micrograph 5: zirconium dioxide ZrO_2
Surface condition SPM 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	TOP	9µm LDP / Reflex Lub	150	135	→ →	4'
4	RAM	3µm LDP / Reflex Lub	150	135	→ →	3'
5	NV	CeO2 / Water	150	100	→ ←	1'

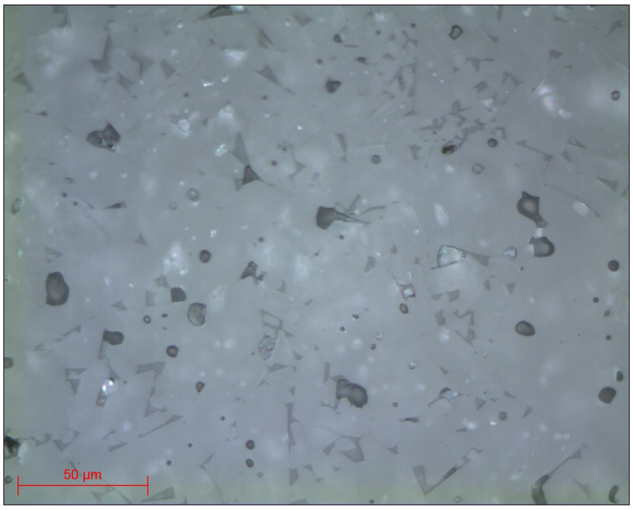
Polishing range N°2 includes supports and suspensions that offer an alternative to those used in Polishing range N°1.

All the polishing ranges listed above are standard and versatile ranges that can be modified according to the subtleties of the samples. (Cf Lab'Notes of the material concerned).
Moreover, they are not necessarily to be carried out in their entirety; observations will define needs (except for titanium samples for which all the steps of the range must be performed).

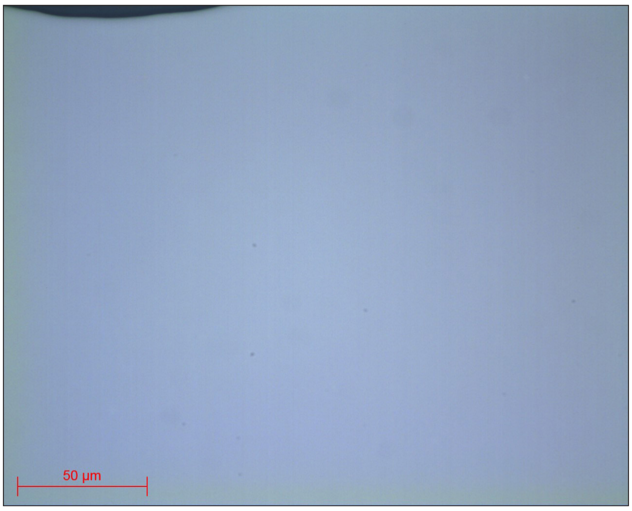
At the end of this preparation phase, the polished samples can be directly observed without metallographic etching. Otherwise, metallographic etching allows differences in relief and/or colour to be made between the different components and so allows them to be observed. It is mainly used on metals (see Lab'Notes on the material concerned).

MICROSCOPY

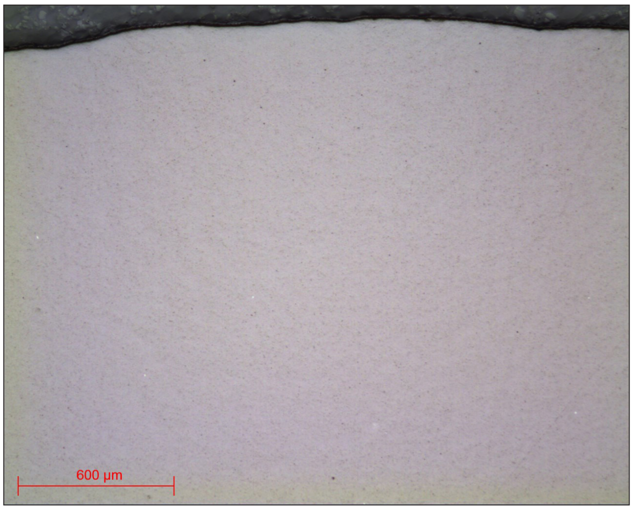
All micrographs presented were produced using **PRESI VIEW** software:



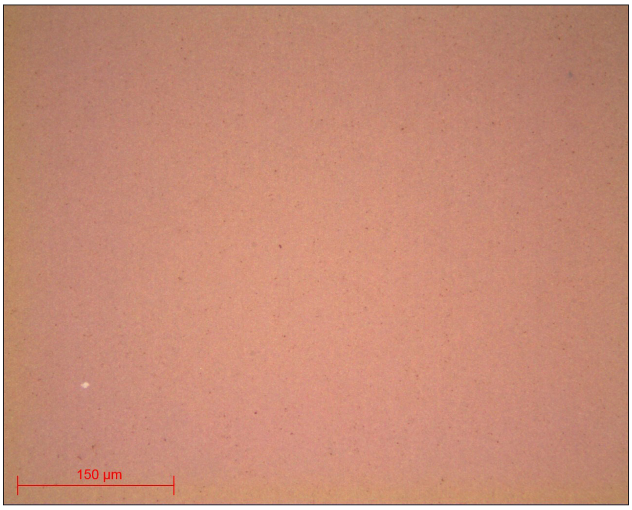
Micrograph 6: Alumina Al_2O_3
Polished to SPM lens x50



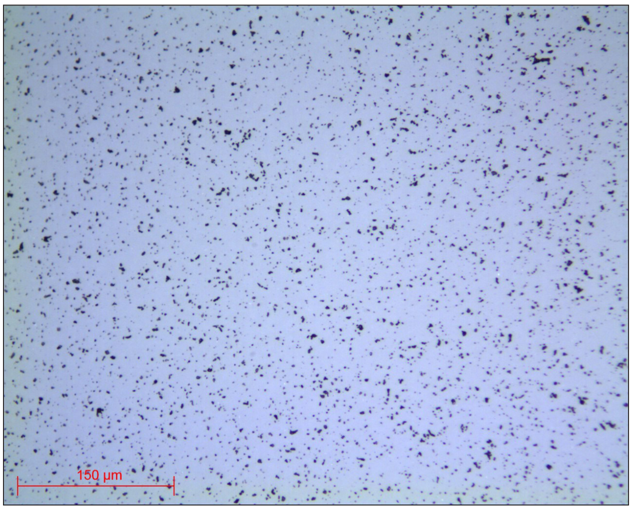
Micrograph 7: Zirconia ZrO_2
Polished to SPM lens x5



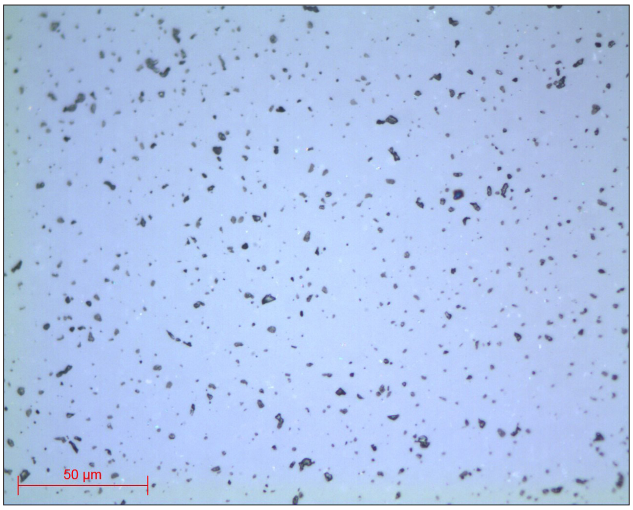
Micrograph 8: Zirconia hardened alumina ZTA
Polished to SPM lens x5



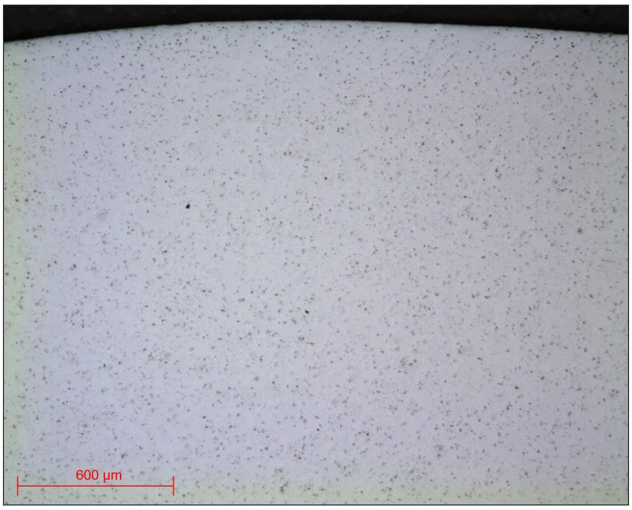
Micrograph 9: Zirconia hardened alumina ZTA
Polished to SPM lens x20



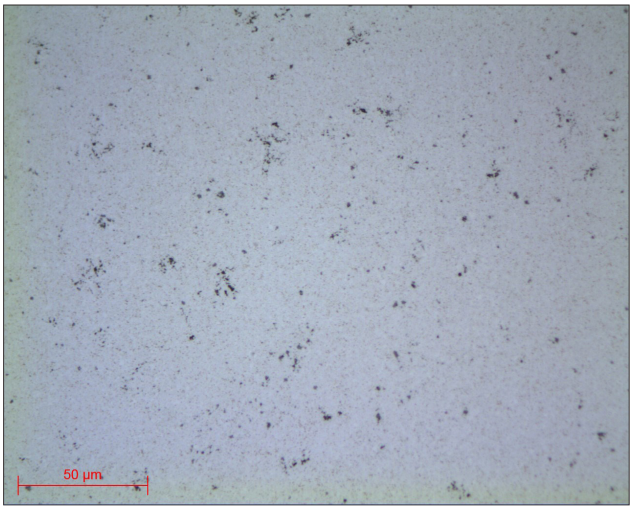
Micrograph 10: Silicon Carbide SiC
Polished to SPM lens x20



Micrograph 11: Silicon Carbide SiC
Polished to SPM lens x50



Micrograph 12: Tungsten carbide WC
Polished to SPM lens x5



Micrograph 13: Tungsten carbide WC
Polished to SPM lens x50