Metallographic preparation of powder metallurgy parts

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Application Notes

Next to casting, mechanical forming and machining, powder metallurgy (P/M) technology is an important method of manufacturing metal parts. Undesirable characteristics of ingot-based metals can be greatly reduced, and desired properties of metals which would normally not alloy easily can be achieved by combining different metal powders or mixtures of metal and non metal powders.

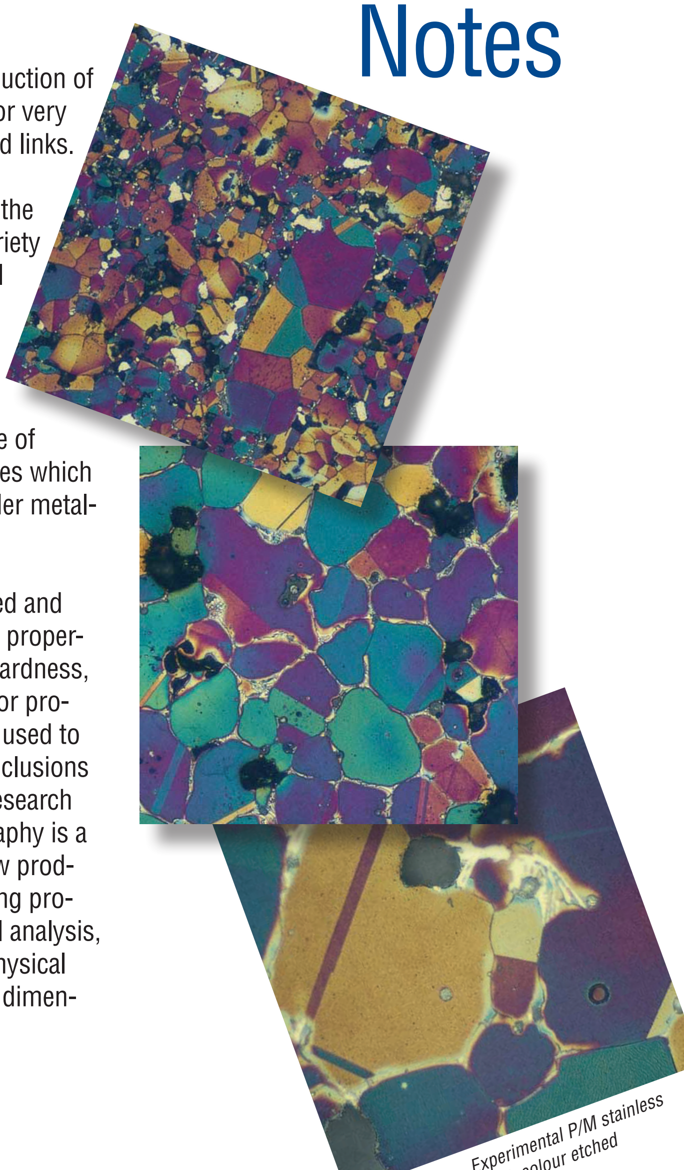
The process of making powders, compacting them into useful shapes and then sintering them is costly, but the finished parts have some specific advantages over wrought or cast parts.

The main advantages are:
- the possibility to make fine grained homogenous structures
- the ability to form complicated shapes with close dimensional tolerances
- and the ability to produce parts with a superior surface finish.

Costly machining processes are thus reduced or eliminated and consequently there is less scrap loss compared to other forming methods. It is therefore most economical to use powder metallurgy for the high volume production of small, intricately shaped, and/or very precise parts such as gears and links.

In addition, the process offers the potential to produce a wide variety of alloys with different material properties such as high temperature toughness and hardness. High speed cutting tool bits from sintered tungsten carbide powder are an example of the variety of different properties which can be achieved with the powder metallurgical process.

As the density of the compacted and sintered part influences its key properties of strength, ductility and hardness, a specific porosity is critical. For process control, metallography is used to check porosity, non-metallic inclusions and cross-contamination. In research and failure analysis, metallography is a major tool used to develop new products and improve manufacturing processes. In addition to chemical analysis, quality control also includes physical methods for checking density, dimensional changes, flow rate etc.



Experimental P/M stainless steel, colour etched

Difficulties during metallographic preparation

Grinding and polishing:
To reveal the structure with the correct and representative porosity.

Solution:

Sufficiently long polishing

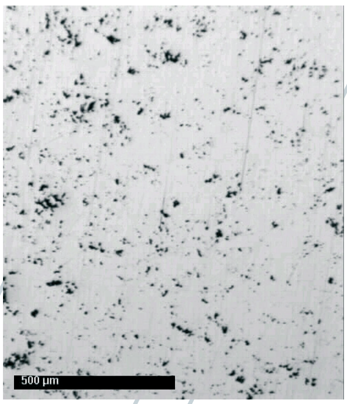


Fig.1: Porosity of a P/M steel sample after a 4 min. polish with 3 µm diamond

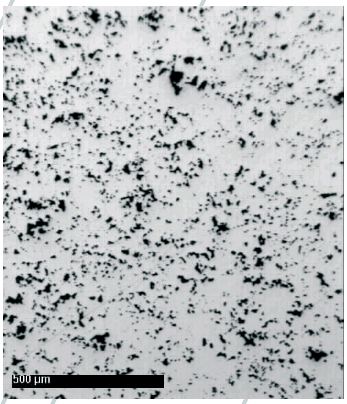
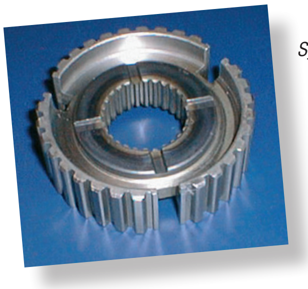


Fig. 2: Same sample as Fig.1 after 8 min. polish with 3 µm diamond

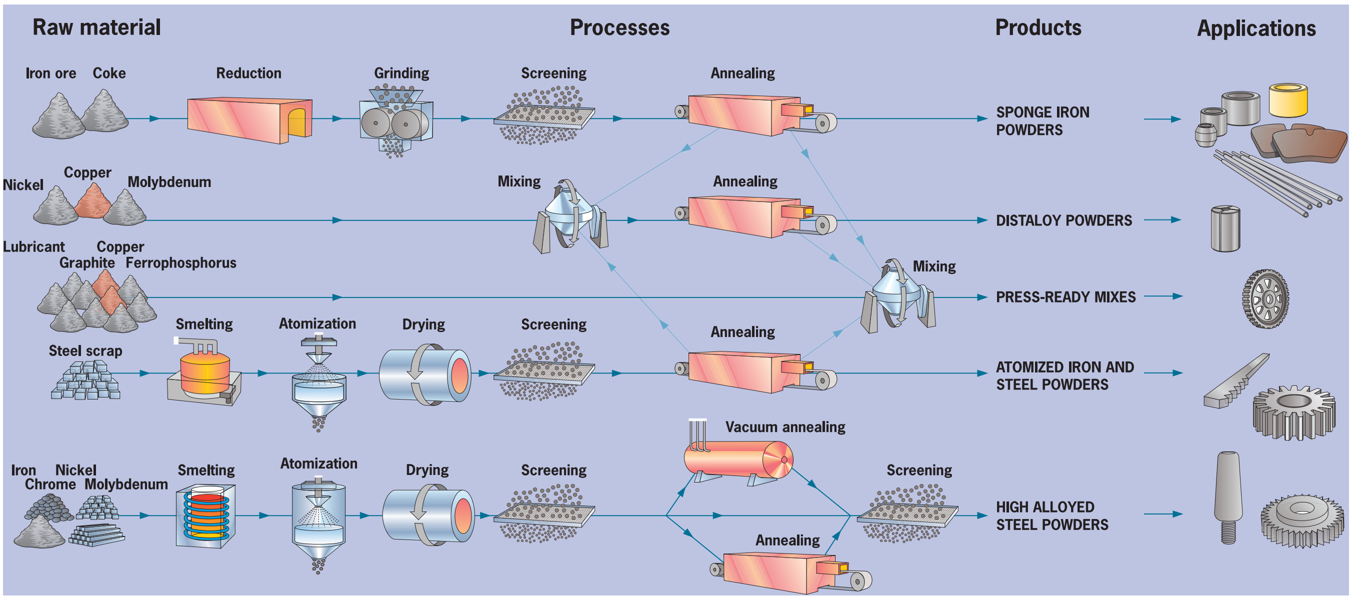
Different powder metallurgy steel components

Production of powder metallurgy parts



Synchronising hub

Manufacturing process of iron and steel powders



Production
To achieve the desired structure and near net shape of a powder metal part, stringent process control of the following production steps is required:

- Making the powder
- Mixing the powder with additions such as lubricant, carbon and/or alloying elements
- Compacting the powder in carbide dies
- Sintering at high temperature (1100-1200°C) in a protective atmosphere

Chemical and atomisation methods are the two most common methods for powder production. The chemical method converts metal from ore oxides directly to metal powder at a temperature below the melting point. For example, iron powder is made through direct reduction from iron ore into sponge iron. The sponge iron is then mechanically crushed to powder, which is further refined through annealing under reducing atmosphere to produce pure iron powder. It is used for alloying and low density applications, such as bearings.

In the atomizing process molten metal of the desired alloy flows through a nozzle and is struck by high pressure water or gas jet. Small droplets are formed which solidify into particles. Atomized powders result in higher densities than mechanically crushed powders, therefore all steel powders are produced by atomizing.

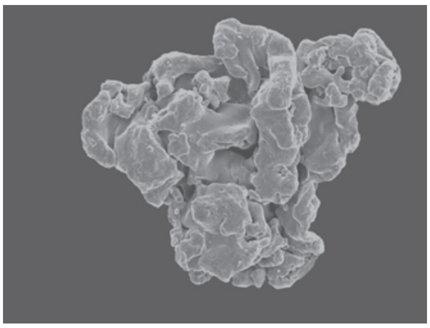
Copper powder is made by atomizing or electrolytic methods. Tungsten carbide powder is produced by adding controlled amounts of carbon to the tungsten powder and carburising it at 1400 - 2650°C

Powder production and mixing is a highly specialized and complex process which produces custom made powder mixes designed to satisfy the needs of a specific application.

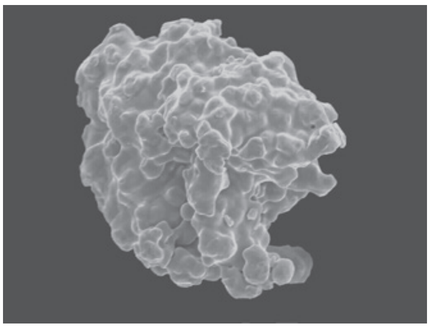
A good powder mix not only has the ability to produce the required properties of a specific alloy, but also needs to facilitate handling, compacting and sintering.
For instance, the easy flow of powder and its capability to mix evenly with other powders is important for an even powder distribution before pressing, and ensures uniform properties of the finished part.

For the production of components the mixed powders are first compacted under high pressure in a carbide die. At this stage the part has the geometrical features of the finished component, but not its strength and is called the “green” part. In order to develop the mechanical and physical properties of the material, metallurgical bonding has to take place through sintering at high temperature in a sintering furnace. The bonding occurs through diffusion between adjacent particles. To avoid oxidation, which would impair the inter-particle bonding, the sintering process is conducted in a protective atmosphere.

The bonding increases the density, and pressed and sintered powder metal parts generally contain between 5 and 25% residual porosity. Depending on the application some parts may need an additional hot isostatic pressing for better dimensional accuracy, or surface finish or impregnation with oil. Final treatments, such as surface hardening, plating or coating can be applied.



Sponge iron powder, SEM



Atomized iron powder, SEM

Difficulties in the metallographic preparation of powder metallurgy parts

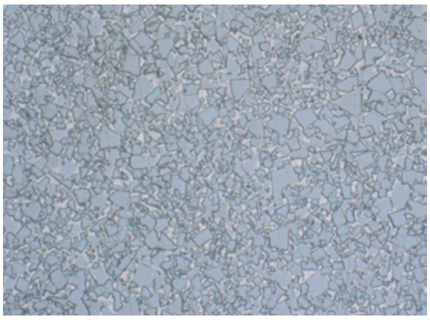


Fig. 3:
Sintered tungsten carbide (WC/Co), etched with Murakami's reagent.
1500 x

The sintering of cemented carbides is carried out in a vacuum sintering process.
The carbide powder is mixed with 3-25 w% cobalt and small amounts of titanium and tantalum carbides are added to inhibit grain growth. This mixture is pressed and sintered. At 1280 – 1350°C the liquefaction of cobalt takes place and results in the formation of a eutectic-like phase of WC/Co. Densification begins at lower temperatures and reaches a theoretical 100% shortly after the liquefaction has occurred. During liquefaction the part shrinks up to 40% in volume.

Applications
Components made by powder metallurgy are mainly used for the following applications:

- Mechanical and structural parts, mainly iron based, but also from copper, brass, bronze and aluminium. The largest user of P/M parts is the automotive industry. Component suppliers make connecting rods, synchronizing hubs, chain sprockets, cams and gears.
- Refractory metals which, due to their high melting points, are difficult to produce by melting and casting.
- Porous material in which controlled porosity serves a specific purpose, for instance self lubricating bearings.
- Composite materials that do not form alloys, for instance copper/tungsten for electrical contacts, cemented carbide cutting tools (Fig. 3), materials for brake linings and clutch facings, diamond cutting tools, or metal matrix composites.
- Special high-duty alloys, such as nickel and cobalt based super alloys for jet engine parts, and high speed tool steels, which have an even distribution of carbides and have isotropic qualities (Figs. 4 and 5).

In addition, different powders and powder mixtures for thermal spray coatings are produced and are also subject to metallographic quality control.

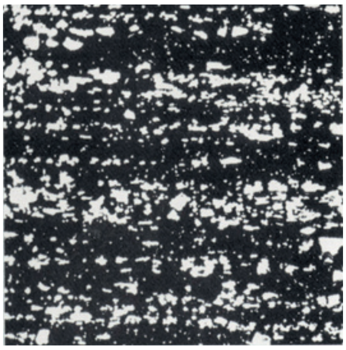


Fig. 4: Carbide distribution in conventionally produced steel

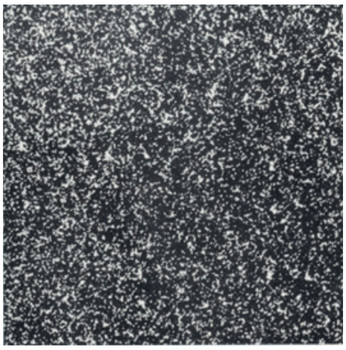


Fig. 5: Carbide distribution in powder metallurgically produced steel

The main challenge during the preparation is to show the true porosity after grinding and polishing. Depending on the hardness of the material this can be more or less successful. During the grinding of soft metals, abraded metal is pushed into the pores, which then has to be removed by polishing.
Samples from parts in which hard and soft materials are mixed are prone to show pronounced relief. The preparation of green parts needs particular care and patience as they are very fragile.

Recommendations for the preparation of powder metallurgy parts

Cutting
For sectioning a powder metallurgy component of a specific metal or alloy one can select an appropriate cut-off wheel using the recommended charts and guidelines. For mixed materials it is recommended to use a cut-off wheel suitable for cutting the material which constitutes the major part. For sintered carbides a resin bonded diamond cut-off wheel is recommended (e.g. B0DXX). Green parts need to be mounted in cold mounting resin (see section "Mounting") before cutting, so that they are not crushed by clamping.

Mounting
In order to assure good adhesion of the mounting resin to the sample material it is essential to degrease the sample thoroughly with acetone, toluene or Isopar C* before mounting (Use proper safety precautions when handling solvents!).

Sintered parts can be mounted with a resin suitables to the mechanical properties of the sample material: For hot compression mounted either phenolic resin (MultiFast) or resins containing harder filler material (DuroFast, LevoFast). For a cold mounting acryllic resins with filler (DuroCit-3 or LevoCit) can be used. For more information see www.struers.com
Green parts need to be re-impregnated after sectioning under vacuum with a cold mounting epoxy resin (CaldoFix-2, EpoFix, SpeciFix-40).
Powders can be mounted by mixing a small amount (about 1/2 teaspoon) of powder with a slow curing epoxy resin and pouring it into a mounting cup. During the approx.12 hours curing process the particles will form a layer by settling at the bottom of the cup.

**Available from Exxon*

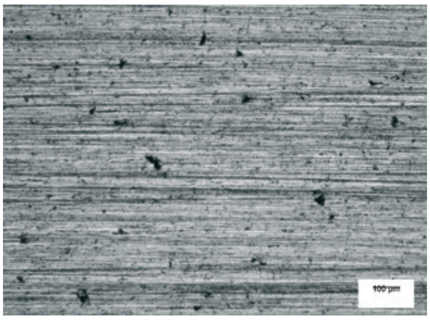


Fig. 6:
Surface of P/M steel after fine grinding on MD-Allegro

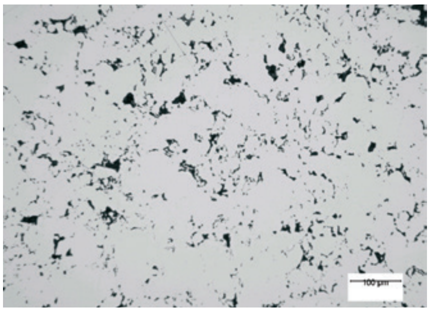


Fig. 7: Same sample as in Fig. 6 showing insufficient polish

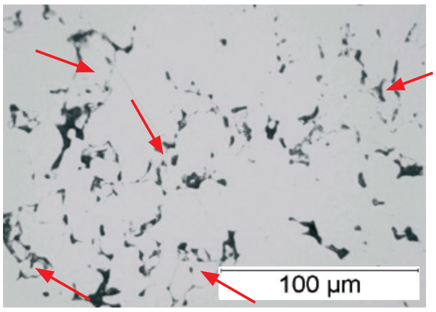


Fig. 9: Larger magnification of surface from Fig. 7, showing metal "lids" covering pores

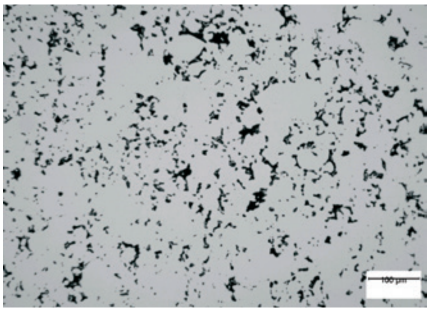


Fig. 8: Same sample as in Fig 7 after longer polish showing correct porosity

Hard metal powders can be hot compression mounted by mixing with one measuring spoon of the fine grained mounting resin DuroFast.

The mixture is then poured into the cylinder of the mounting press and is topped with phenolic resin.

Grinding and polishing
The grinding and polishing procedure for powder metals follows the same rules which are applied to prepare ingot based samples of the same material.
Plane grinding high volumes of samples of materials >150 HV can be carried out on an aluminium oxide grinding stone or a diamond grinding disc (MD-Piano). Materials <150 HV can be plane ground on silicon carbide Foil/Paper.

For fine grinding with diamond the fine grinding disc MD-Allegro is suitable for materials >150 HV and MD-Largo for materials <150 HV. This is followed by a thorough diamond polish with 3 µm and a brief final polish with 1 µm or with oxide polishing suspensions. As one of the major goals of preparing a powder metallurgical sample is to show the true porosity, it is important that the diamond polishing step is carried out long enough to achieve this goal (see Figs. 6-9). For large or soft samples it can take up to 10-15 minutes of diamond polishing to remove the metal pushed into the pores during grinding and reveal the correct porosity. For soft metals the final polishing time should not be prolonged unnecessarily, as this leads to rounding of the pore edges. Starting with 500# or 800#, green parts are ground manually on silicon carbide Foil/Paper to 4000#. The surface is re-impregnated as needed. Polishing can be done on a semi-automatic polishing machine for individual samples.

In order to establish the exact polishing time for specific alloys and parts, it is recommended to check the structure during polishing every two minutes with the microscope, and only proceed to the next polishing step when all residual metal has been removed from the pores.

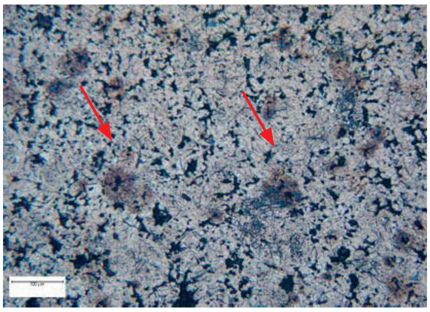


Fig.10: Water stains from cleaning can lead to misinterpretation of structure

Cleaning and drying
After polishing it is essential to clean the sample with a water/detergent mixture to remove remnants of the polishing suspension and lubricant from the pores. The sample is then rinsed with water, followed by a thorough rinse with isopropanol and dried with a warm stream of air, holding the sample at an angle. Do not blow the stream of air directly

Preparation method for P/M bronze

Grinding

FG	Step		PG	
			Foil/Paper	MD-Largo
	Abrasive	Type	SiC	Diamond
		Size	#320	9 µm
	Suspension/ Lubricant		Water	DiaPro Allegro/Largo 9
	rpm		300	150
	Force [N]/ specimen		35	25
	Time (min)		As needed	4

Polishing

	Step		DP	OP
	Surface		MD-Mol	MD-Chem
	Abrasive	Type	Diamond	Silica
		Size	3 µm	0.04 µm
	Suspension/ Lubricant		DiaPro Mol R3	OP-S NonDry
	rpm		150	150
	Force [N]/ specimen		25	25
	Time (min)		4	1

Table 1: Preparation method for 6 samples of P/M bronze, mounted, 30 mm dia., using the semi-automatic Tegramin, 300 mm dia.

As an alternative to DiaPro polycrystalline diamond suspension P, 9 µm, 3 µm and 1 µm can be used together with red and green or blue lubricant.

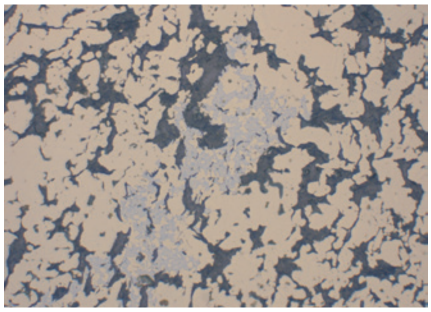


Fig.11: P/M bronze, unetched, containing graphite (grey), and α-δ eutectoid (blue) 500x

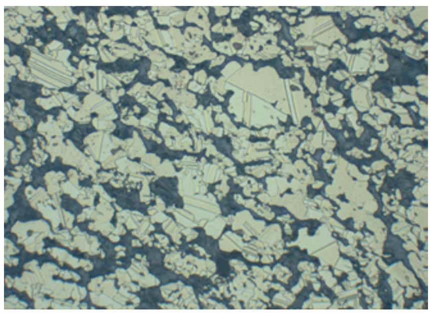


Fig.12: Same as Fig.11, etched with iron-III-chloride, showing grain structure of bronze 500x

Preparation method for P/M steel

Grinding

Step			PG	FG
	Surface		Foil/Paper	MD-Allegro
	Abrasive	Type	SiC	Diamond
		Size	#220	9 µm
	Suspension/ Lubricant		Water	DiaPro Allegro/Largo 9
	rpm		300	150
	Force [N]/ specimen		35	30
	Time (min)		As needed	5

Polishing

Step			DP 1	DP 2
	Surface		MD-Mol*	MD-Nap
	Abrasive	Type	Diamond	Diamond
		Size	3 µm	1 µm
	Suspension/ Lubricant		DiaPro Mol B 3	DiaPro Nap B 1
	rpm		150	150
	Force [N]/ specimen		30	20
	Time (min)		6-8	0.5 - 1

Table 2: Preparation method for 6 P/M steel samples, mounted, 30 mm dia., using the semi-automatic Tegramin, 300 mm dia.

As an alternative to DiaPro polycrystalline diamond suspension P, 9 µm, 3 µm and 1 µm can be used together with green or blue lubricant.

**Alternatively MD-Dac/DiaPro Dac 3 can also be used.*

from the top onto the sample surface as it forces the liquids out of the pores, which will leave stains on the surface (Fig.10). It is important to use a high quality alcohol for rinsing to keep staining to a minimum.

Etching

It is recommended to examine the unetched sample first to check the density, shape and size of the pores, oxidation and inclusions, sintering necks and free graphite (see Figs.11 and 12). It is important to know the theoretical density in order to compare it to the porosity.
After this initial inspection it is recommended to etch the sample immediately to avoid drying stains, which form when cleaning and drying liquids are gradually released by the pores. For very dense sintered carbides this is not as important as for any powder metal with a certain amount of porosity.

To reveal the structure, the common chemical etching solutions for metals and their alloys, that are recommended in the literature, can be used. The following procedure is recommended for etching: wet the surface with isopropanol, immerse sample face up into etchant and slightly agitate. When the appropriate etching time has elapsed, take the sample out of the etchant and rinse either with isopropanol or water depending on the etchant (see below) and dry with a stream of warm air. Interpretation and photographic documentation should take place immediately after drying.

Etching time depends on the alloy and it needs some experience for the correct timing. Etching too short will not give sufficient contrast of the different phases. If the sample is overetched, it is difficult to distinguish the various phases (see Figs.15-17). When working with unfamiliar material it is recommended to etch shorter rather then longer and to check the result with the microscope first. More etching can be carried out if necessary, but if the sample is overetched, it needs to be repolished. Following are some of the common etchants. *Follow standard safety precautions when mixing and working with chemical reagents!*

Copper and copper alloys:

1. 100 ml water
   20 ml hydrochloric acid
   5 g Iron-III-chloride
   10-20 sec
   Rinse with water followed by isopropanol
2. 100 ml water
   10 g ammonium persulfate. Use fresh only!
   Rinse with water followed by isopropanol

Steel:

1. 1-3% Nital for iron-carbon alloys, iron-carbon-copper alloys and pre-alloyed iron-molybdenum:
   100 ml ethanol
   1-3 ml nitric acid
   10-60 sec depending on carbon content.
   Rinse with isopropanol (Fig.14)
2. Picral to develop difference between martensite and austenite:
   100 ml ethanol
   4 g picric acid
   10-60 sec. depending on carbon content.
   Rinse with water followed by isopropanol (Fig.13)

Preparation method for sintered carbides

Grinding

Step			PG	FG
	Surface		MD-Piano	MD-Allegro
	Abrasive	Type	Diamond	Diamond
		Size	#220	9 µm
	Suspension/ Lubricant		Water	DiaPro Allegro/Largo 9
	rpm		300	150
	Force [N]/ specimen		30	30
	Time (min)		As needed	4

Polishing

Step			DP	OP
	Surface		MD-Dac	MD-Chem*
	Abrasive	Type	Diamond	Silica
		Size	3 µm	0.04 µm
	Suspension/ Lubricant		DiaPro Dac 3	OP-U NonDry
	rpm		150	150
	Force [N]/ specimen		30	20
	Time (min)		4	1

Tabel 3: Preparation method for 6 samples of sintered carbides, mounted, 30 mm dia., using the semi-automatic Tegramin, 300 mm dia.

As an alternative to DiaPro polycrystalline diamond suspension P, 9 µm, 3 µm and 1 µm can be used together with green/blue lubricant.

**Optional step*

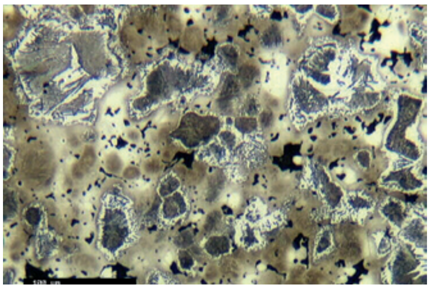


Fig.13: P/M steel with 0.5%C, diffusion alloyed with Ni, Cu and Mo. Etched in picral, showing areas of fine pearlite surrounded by ferrite, martensite, bainite and Ni-rich austenite.

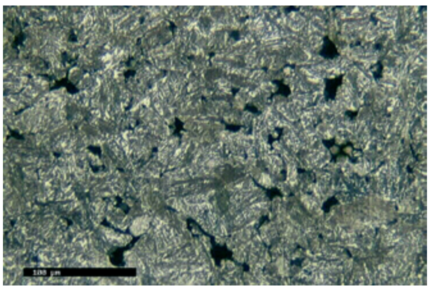


Fig.14: P/M steel with 0.8% C, pre alloyed with 1.5% Mo. Etched with nital, showing dense bainite

3. For contrast between pearlite, bainite and martensite:
   200 ml ethanol
   4 g picric acid
   1-2 ml nitric acid
   20-100 sec, depending on carbon content and alloying elements.
   Rinse with water followed by isopropanol

Stainless steels:
Vilella's reagent:
45 ml glycerol
15 ml nitric acid
30 ml hydrochloric acid
30 sec to 5 min
Rinse thoroughly with water followed by isopropanol

Sintered tungsten carbides:
Murakami's reagent
100 ml water
10 g potassium or sodium hydroxide
10 g potassium ferricyanide
Etching by immersion or swab etch
Rinse thoroughly with water followed by isopropanol

Summary

Powder metallurgy is a method of producing parts from metals which normally would not alloy easily, or from mixtures of metals and non-metals, taking advantage of their combined properties. Although the powder metallurgical process is costly, it has the advantage of economically producing large amounts of small and intricately shaped parts with homogenous structures. The density of compacted and sintered parts affects their strength, ductility and hardness. Therefore metallographic control of porosity is an integral part of quality control.
During metallographic grinding, metal is pushed into the pores and if the subsequent polishing steps are not carried out properly, residual metal "lids" covering the pores will obstruct the evaluation of the correct porosity. Thorough grinding and polishing with diamond, and microscopic checks between polishing steps, assure a true representation of the structure. The given procedures for automatic preparation and the chemical etchants have been used successfully in day to day practical laboratory application and give reproducible results.

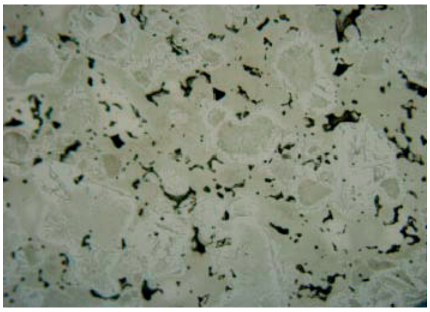


Fig.15: Etched too short

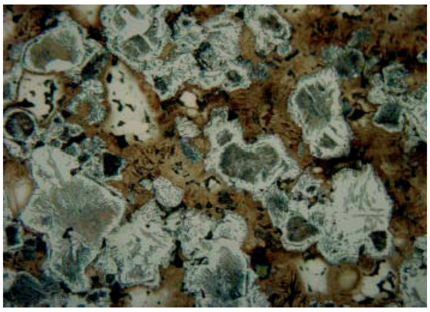


Fig.16: Etched too long, over etched

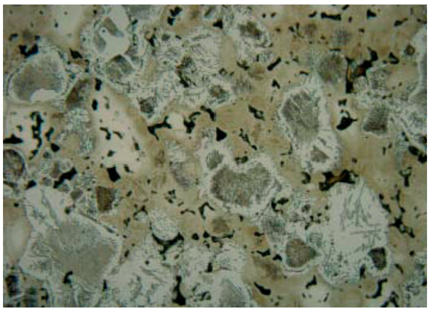


Fig.17: Correct etching

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Acknowledgements
We wish to thank Höganäs AB, SE 26383 Höganäs, Sweden, for the co-operation and supplying information and material for reproduction: all photos of P/M components, diagram and SEM photos on page 2, Figs. 1, 2, 6, 7, 8, 9, 10, and 13 -17. Special thanks go to M. Carlsson who generously shared her professional experience with us.
Figs. 4 and 5 courtesy Böhler Edelstahl GmbH, Kapfenberg, Austria

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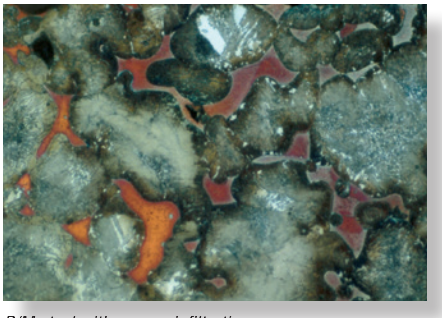

P/M steel with copper infiltration

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28.02.2019 / 62140805