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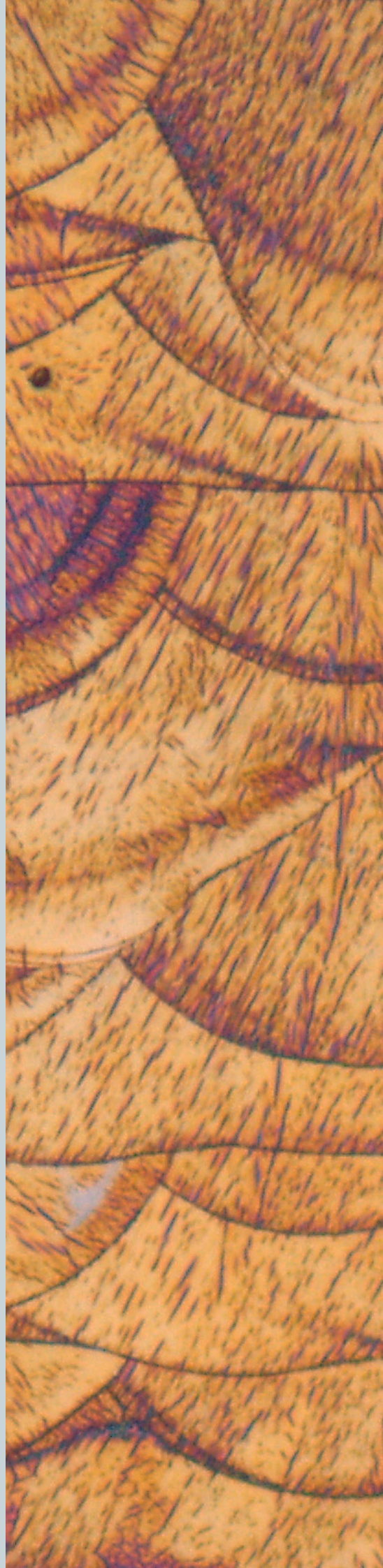
ADDITIVE MANUFACTURED MATERIALS

Proven materialographic knowledge



DANISH
TECHNOLOGICAL
INSTITUTE

APPLICATION NOTE



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METALLOGRAPHY OF ADDITIVE MANUFACTURING

What is AM?

As the name implies, AM adds material to create an object. The term “additive manufacturing”, also known as 3D printing, refers to technologies that grow 3D objects one superfine layer at a time. Each successive layer bonds to the preceding layer of melted or partially melted material to create components, parts, and even complete products.

AM can rightly be called the opposite of subtractive manufacturing. Rather than chipping pieces of a large block to reveal the “hidden” part inside, AM assembles the material “chips” into a new part.

Advantages of additive manufacturing

For prototypes and models, the advantage of AM is straightforward. You can easily make a one-off 3D representation of your design or even a functioning prototype that can be used to test the performance of the design. The cost is often a fraction of what it would cost with conventional manufacturing methods to manufacture a one-off build. Today, however, the advantages of AM for some products or applications are so strong that they are taking over the serial production of components from conventional manufacturing. The advantages that drive the transformation towards AM are:

Design improvements

AM can create parts with complex geometries as a single object. Therefore, multiple parts can be integrated into one in the design phase, which allows for improved strength, less weight, and higher durability.

For tools used in production, AM can play a role in improved work processes, increasing output, and producing better parts. The centering tool from Danfoss, shown in Fig. 1, is an example where weight reduction saved the operators from lifting additional kilos every day. The Marel case in Fig. 3 (see page 3) also illustrates a case where a support element used in industrial meat production was designed improved for better performance in terms of food contact properties.



Fig. 1: Manufacturing tools and aids.

The DanSTAR case in Fig. 4 (see page 4) is another example of AM used for design improvements, which ensures the high performance operation. The 3D rocket nozzle takes advantage of the design freedom that AM offers.

Design improvements are also used in the Hydrauvison case in Fig. 5 (see page 5), to improve performance for the optimized hydraulic block.

Cost savings

The production of near net shape parts reduces material wastage, especially interesting for high-value parts. Elimination of expensive tools for conventional manufacturing and saving labour-intensive assemblies due to parts consolidation are also driving manufacturing costs down for AM. One example of this, where labour-intensive manual work can be exchanged for AM, is the cookie extrudes used by Bühler-Meincke for their cookie machines. See Fig. 6 (see page 5).

DANFOSS: CENTERING TOOL

Danfoss has begun 3D-printing a centering tool used to assemble an electric motor in one of the company's compressors. The background for 3D printing the tool was a wish to remove all unnecessary material to reduce weight and price - but at the same time retain enough mass for the tool to be sufficiently robust.

Facts and economy when redesigning to AM:

-  With AM, the tool became much lighter:
From 1,100 g (Aluminum) to 382 g (Titanium)
-  Designed with complex geometry that enables weight reduction
-  3D printed with SLM technology in titanium powder
-  The reduced weight makes the tool easier to manage for operators who lift it 180 times a day
-  But the 3D-printed tool has a significantly longer service life, so the price per year is reduced by 63 percent

Lead time reduction

Due to the near net shape technology, the manufactured parts can be ready to use right out of the machine or with limited post-processing. In the design phase, this can facilitate fast market introduction, in the manufacturing process, it can save time and cost or produce unique parts, and in end-of-life services facilitate delivery of spare parts without storing parts for aftermarket sales.

AM PROCESSES

History of AM

The birth of 3D printing is credited to Hideo Kodama, who in 1981 invented the concept of 3D printing that was patented in 1983 and later commercialized by Charles Hull. While the other basic forms of manufacturing have been known long before the industrial revolution, accelerated manufacturing to what we know today, AM is a digital newcomer still playing catch-up.

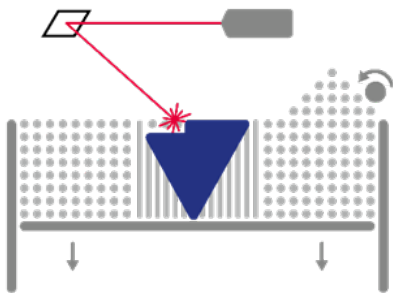


Fig. 2: Laser Powder Bed Fusion (L-PBF).

The first 3D printers used light to activate photopolymer resins, and this created polymer parts. The first commercially available metal printer, the EOSINT M250, was launched by EOS GmbH in 1994 – but only with the capability to sinter a material blend consisting of bronze and nickel. AM has developed from a tool to manufacture models and prototypes to full-scale production of parts within the span of a few decades. Today, metal AM components operate in highly critical functions, such as; torsion springs for F1 cars, brake calipers on the Bugatti Chiron hypercar, most of the Czipper C21 car, fuel nozzle and turbine blades on the LEAP and GE9X jet engines, the SpaceX Dragon rocket thrusters, medical implants, and recently on the Perseverance rover that landed on Mars. Metal AM has entered critical applications where quality control and assurance are key. While AM has entered the market for critical high-performance parts, this is not the sole area of application today. AM is also used for tools and moulds with conformal cooling, machine parts, and grippers for industrial applications, as well as con-



Marel: Support element for industrial meat cutting

For a new industrial portion cutting module, Marel needed a support element that could stop the meat on the conveyor belt, hold it while the knife cut, and subsequently move the cut pieces forward. The desire was low weight and few assemblies where bacteria could accumulate. Additive manufacturing was chosen for several reasons.

Facts and economy when redesigning to AM:



From 42 components to one component



From 45 assemblies to 6 assemblies



Previously machined in stainless steel 316L - now printed in titanium



Weight reduction from 600 g to 250 g



Same price as before

Fig. 3: Food contact approved machine part.

sumer goods like high-end gramophone pick-ups and racing bicycle pulley wheels.

The field of AM is littered with different process abbreviations – mainly because different machine manufacturers have trademarked the same processes under different brand names. While numerous brand names and abbreviations exist, ISO/ASTM 52900:2017 defines a common terminology for AM as well as a framework categorising the different processes into seven main types of AM: Vat Photopolymerization, Material Extrusion, Sheet Lamination, Powder Bed Fusion, Binder Jetting, Material Jetting, and Direct Energy Deposition.

This application note focuses on Laser Powder Bed Fusion (L-PBF) – also known as Direct Metal Laser Melting (DMLM) and Selective Laser Melting (SLM). The technology basically consists of laser welding fine metal powder particles together, one layer at a time. See Fig. 2. The L-PBF technology includes countless process parameters which can influence the properties and quality of the final component.



The laser scan

Initially, a fine metal powder of spherical particles – 15-60 μm in diameter - is spread evenly across a base platform consisting of a material compatible with the build powder. When the powder has been spread in a thin layer only 30-90 μm thick, the laser scans across the powder, heating and welding it together, as well as welding it to the layers directly below. For the first few layers, this also means welding the part to the build platform. After the laser has scanned the layer, the platform is moved a layer increment downwards, and the next layer of fine metal powder is distributed, after which the laser scans and welds the new layer. Thus, the process repeats numerous times until the often thousands of stacked layers make up the final component. Laser power, scan speed, hatch distance or spacing between laser scans, powder layer thickness, and the scan strategy. These are all process parameters that affect how the layers are built in the component. To add enough energy and create a complete melt, both the laser power and scan speed are adjusted. The spacing between the laser scans is typically set lower than the width of the melt pool to ensure overlap between adjacent laser scans. The layer thickness is usually determined by the size of the metal powder and the necessity for full penetration of the melt pool with the underlying material.

The atmosphere

The process is situated in an enclosed chamber with either a vacuum, or more commonly, a protective atmosphere of nitrogen or argon, depending on the reactivity of the print material being processed. The protective gas is blown across the build surface to also help remove slag and spatter from the welding process from in upcoming welding area. The build platform is heated to adjust the cooling rate of the build material as it is welded. This aids in controlling the metallurgy formed in the part during the printing process and reduces residual stress formation.



DanSTAR: 3D-printed rocket nozzle

Students from the DanSTAR project have 3D printed a combustion chamber in stainless steel for a rocket that the students designed and developed from scratch. 3D printing was the obvious solution for the combustion chamber, as it consists of very complex internal geometries that are nearly impossible to achieve with other production technologies.

Facts when designing for AM:



The only flight-proven student-made liquid rocket engine in Europe, and it is built to reach an altitude of 30,000 feet



The design freedom resulted in an intelligent design where the fuel is circulated through the monolithic AM engine in channels that run inside the pressure-bearing wall itself, prior to injection into the combustion chamber



Printed in 316L stainless steel with a weight of 5.7 kg – and no machining was needed after 3D printing and initial post processing



AM and the regenerative cooling principle is also used by larger space engine manufacturers – e.g. SpaceX and Orbex

Fig. 4: Rocket nozzle for a student project.

Inside the build chamber, atmosphere-related parameters like inert gas type and its purity, chamber pressure, and temperature can typically be controlled. The build process is usually carried out with a small overpressure to avoid oxidation if there were a leak and to stabilize the melt pool by exceeding a force.

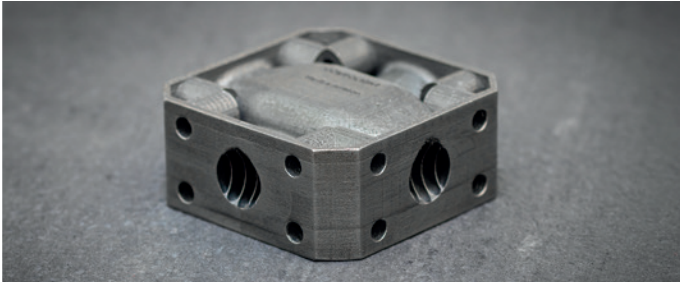
Destressing and heat treatment

After the build process has finished, the parts are often destressed in a gas-protected environment for multiple hours. If not destressed, some materials and geometries will crack and deform due to residual stresses as soon as they are cut from the build plate. Since the parts

built using L-PBF are all one large welding, they inherently come with a very fine microstructure that has cooled rapidly. In most cases, this results in a high-strength, low-ductility part. Via heat treatment, however, the mechanical properties of the L-PBF parts can be tailored to specific mechanical properties or corrosion resistance.



AM MATERIALS



Hydrauvision: Compact hydraulic block

In the EU-funded Compolight project, the DTI printed a more compact and optimized hydraulic block for the Dutch company Hydrauvision. The new 3D-printed hydraulic block improved the flow in the hydraulic systems, thereby eliminating large energy losses and at the same time significantly reducing weight and size

Facts when redesigning to AM:

-  Significant size and weight reduction - from 20.15 kg and 230x230x54 mm to 0.95 kg and 80x80x50 mm
-  Greater power with a 25% reduction in pressure loss - by reducing the pressure loss in the system, you do not need as large a hydraulic system as before to handle the task
-  The design freedom resulted in a new solution without straight angles and with a much more complex construction, enabling an uninhibited flow of oil and air throughout the system



Bühler-Meincke: Nozzles for cookie production

Bühler-Meincke started with metal AM for their cookie nozzles. This gave them great design freedom and they could make geometries that were not possible with traditional manufacturing - before, production was manual and dependent on one man, who soldered the nozzles together from several parts.

Facts and economy when redesigning to AM:

-  Ultimate design freedom - and redesign without large tool costs
-  No joints or welds where bacteria can accumulate
-  Full traceability through production and the ability to print in several materials - all approved for food contact
-  Simplified production and no variation in the result
-  Faster production at a lower price

Fig. 5: High pressure component with reduced flow resistance.

Fig. 6: Volume production of complex parts.

The primary limiting factor today, regarding the availability of alloys for L-PBF, is the required processing parameters. Different alloys must be processed using different tailored parameters to ensure fully dense materials, minimise residual stress build-up, achieve the best possible surfaces, and optimise production speed to limit machine time and thus reduce cost. In some cases, tailored heat treatments are also required to ensure the desired final microstructure and mechanical properties.

In some cases, the hardware can be a limiting factor. Some alloys require a heated build chamber, special atmospheric conditions, and/or a high laser power to be manufacturable. The available metal alloys were initially ported from industries driving the development of AM – mainly the car

industry, aerospace industry, and medical industry. This meant that the initial development within alloys and production parameters for AM alloys was focused on TiGr5 for aerospace usage, Ti Gr1 or Gr23 for use with implants. AlSi10Mg, as a well-known casting alloy within the car industry, tool steels like H13, a few variations of Inconel, and other nickel-based alloys, as well as 316L stainless steel, were also adopted for use in AM. Multiple new and redeveloped alloys keep entering the market. The Senvol Materials Database contains, in 2021, more than 1200 material entries for L-PBF metal alloys.

Materials characteristics

In general, the mechanical properties and densities of AM parts are on par with or better than traditional manufactured parts.

The large amount of process parameters related to the L-PBF process requires careful control and adjustment to avoid initiation of defects and to ensure final part quality. The most common defects and deviations related to L-PBF are described and categorized into 3 main groups:

Surface quality

With no post processing, the high surface roughness of AM parts is the most obvious difference, and changing the surface through machining, polishing, glass blasting, etc., is an integrated part of manufacturing. Surface-related defects like roughness and its morphology are highly influenced by in-process parameters and laser specifications, such as scan speed, scan strategy, hatch distance, and spot size.



METALLOGRAPHIC PREPARATION FOR MICROSTRUCTURAL INVESTIGATION OF AM MATERIAL

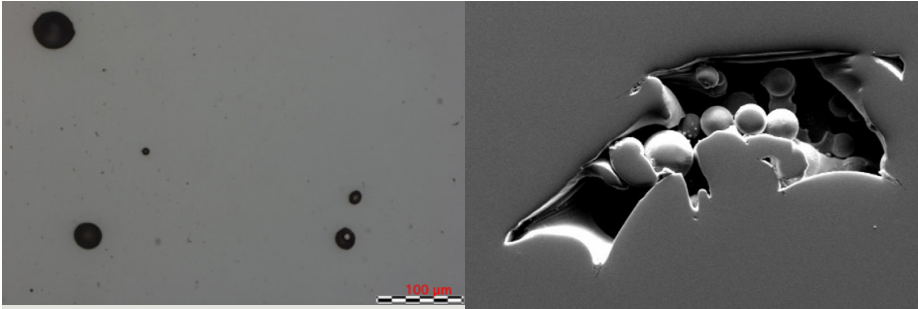


Fig. 7: Polished cross section of L-PBF part. Example of gas porosity (left). Example of lack of fusion porosity (right).

Geometry and dimension deviation

Thermal shrinkage due to cyclic heating may lead to dimensional deviations compared to the CAD model. Shrinkage can take place in both X, Y, and Z directions and can often be decreased by adjusting parameters like laser power and scan strategy.

Microstructure defects

Porosities are a frequent and well-known microstructural defect in relation to L-PBF. The amount, size, and shape of the porosities directly affect the density and the mechanical properties. The types of porosities are typically categorized as lack of fusion porosities or gas porosities. Lack of fusion porosities is often a result of imbalance between in-process parameters like laser power, scan speed, and hatch distance, resulting in too low heat input, lack of penetration, or missing overlap between laser scans.

Lack of fusion porosities are often irregular in shape and may act as stress concentrations, leading to reduced fatigue properties, see Fig. 7, right.

Gas porosities are spherical in shape and can typically be related to entrapped gases in the raw metal powder particles or trapped inert gases during the L-PBF manufacturing process, see Fig. 7, left. Heterogeneity may co-occur in the microstructure as the printed material undergoes cyclic sequences of vertical heat conduction, repeated melting, and rapid solidification. Also, the geometry of the printed part may potentially affect the solidification condition, hence leading to variations in the microstructure. Anisotropy developed by the L-PBF process introduces differences in mechanical properties at different angles relative to the build direction. This means that the part orientation should be considered before starting the print process.



Fig. 8: Variety of metallic samples made by additive manufacturing (laser melting processes).

AM Samples

The feedstock powders are in certain situations investigated, regarding their particle size distribution, shape, or structure.

The final printed workpiece is not destroyed, and therefore, the test samples for microstructural analysis are small rods, bars, or cubes printed together with the workpiece, to mirror the laser melting conditions and microstructural properties in each single layer. Figure 8.

When the aim of the metallographic investigation is to analyse a pore, a crack, an impurity, or any other microscopic attribute or flaw the sample is categorized as a target sample.

Preparation of AM in general

For all 3 types of AM samples; feedstock powder, printed workpiece or target sample, the requirements are as we know them; surface must be plane and scratch free, all structural elements must be clearly visible, and the microstructure after preparation should be ideally free of deformations, and artefacts. Generally, all steps known from conventionally manufactured materials are applicable and metallographic preparation rules are, as always, determined by the material properties themselves. These properties are mainly hardness, ductility, and chemical resistance.

The materialographic process follows the conventional sequence of; cutting, mounting, grinding, mechanical or electropolishing, and a final etch if needed. The process steps will have different importance depending on the type of samples, and the equipment used will be selected according to sample type, and aim of the metallographic investigation.



Materialographic preparation of feedstock powders

Cold mounting in epoxies is the best method when investigation of powders is needed. The powder can be slowly stirred into the resin to have a larger amount of powder to investigate. (Fig. 9). Depending

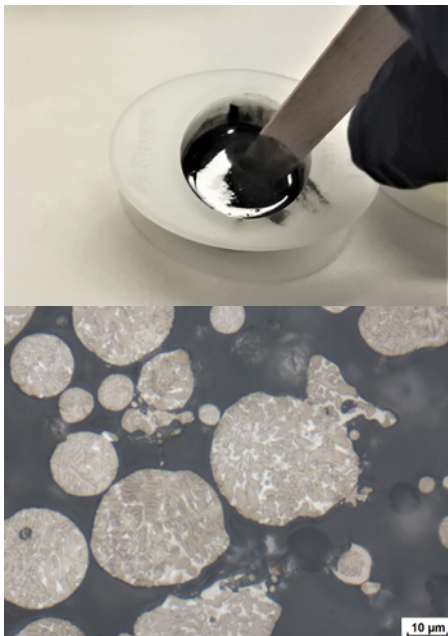


Fig. 9: Metallic steel powder stirred in FixiForm with CaldoFix-2 (top). Polished and etched with modified Klemm etchant (bottom). Bright field.

on the particle size, agglomeration state, material density, and resin viscosity, the larger powder fractions might sink down as a layer or remain in balance. When sinking of small-sized powder fractions or lightweight materials is desired, heating up the resin for some minutes enables easier sinking of small-sized powder fractions or lightweight materials.

To get a full particle size distribution examined, making a longitudinal cut through the mount and remounting it at an angle of 90° is useful. (Fig. 10).

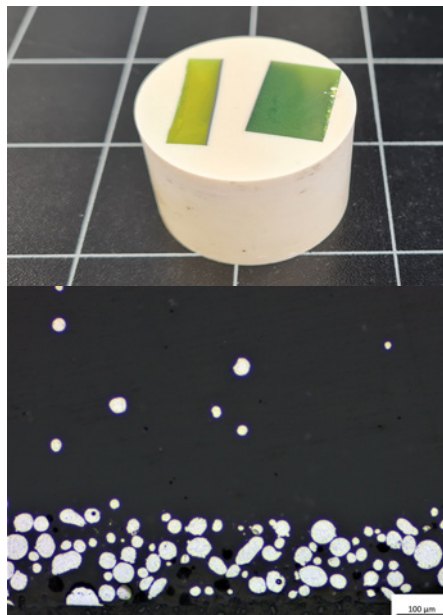


Fig. 10: Cross section through a mount with AlSiMg10 powder in EpoFix, remounted in DuroCit-3 (top). The polished sample shows microscopic distribution, size and shape of the powder material (bottom).

Hot mounting of powders is possible and requires resin without metallic fillers unless additional etching can help to distinguish powder from resin. In some cases, pressing a tablet of powder-enriched resin can be helpful. The tablet itself can be glued temporarily on a dummy sample or a metallic support block with the same diameter (Fig. 11).

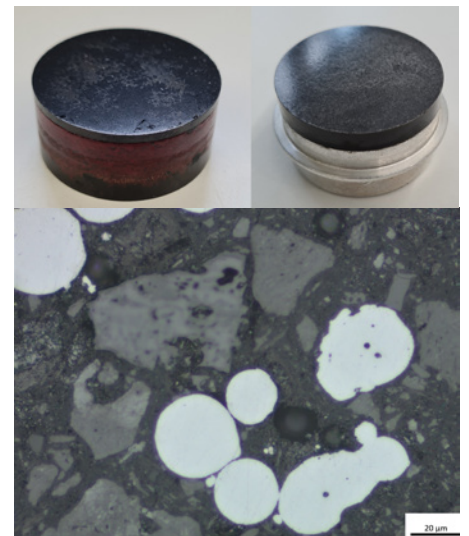


Fig. 11: Tablets of metallic powder enriched PolyFast, pressed in CitoPress, glued temporary on MultiFast dummy sample (top left). Tablet glued on metal support block (top right), for later investigation in a SEM. Microscopic image of steel powder particles (bottom) in PolyFast. Bright field.



Materialgraphic preparation of AM test samples

The area of interest in a printed workpiece must be set and defined. This is predefined by cutting the sample.

The so-called build-up direction must be taken into consideration before the cutting starts.

Mostly used orientation is parallel to the build-up direction and can be called a “longitudinal section”. Another important view into microstructure is perpendicular to the build-up direction and will be called “cross section” (Fig. 13)



Fig. 12: Clamping of AM titanium sample in Secotom-60

All other defined angles to the build-up process are possible, but this is not so common. In such a case, the cutting angle must be captured.

Since AM test samples are small parts representing the large workpiece, an accurate cut is needed, and therefore, precision cutting solutions are preferred. The solution also needs to include clamping tools for square, round, or long samples as well as adaptors for holding the sample in the precise position, for example, a specific angle or distance to the cut-off wheel.

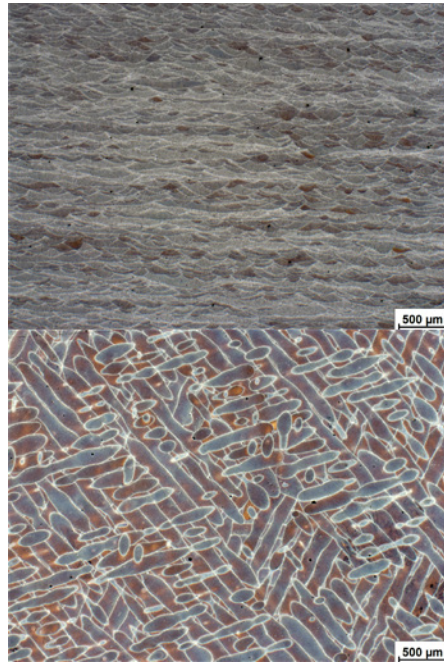


Fig. 13: Longitudinal (top) and cross section (bottom) of AlSi10Mg alloy etched with Fuss etchant. Bright field.

In Fig. 12, a Secotom-60 is shown with a clamping solution for the accurate positioning of small workpieces. Positioning is assisted by a laser line. The precision cutting equipment facilitates the accurate cut using relatively small and thin cut-off wheel, which is selected by material and the supposed material hardness. If very small samples need to be cut or the geometry is very complex, cutting of the sample after mounting in transparent epoxies (EpoFix, CaldoFix-2) can help to fix and protect.

For AM test samples the mounting process will not be as important as it is for powder or target preparation. Special features for the resin like, transparent or conducting, will be parameters that go into the selection of resin as well as the material hardness.

Depending on the sample material, size, and geometry, after initial cutting, both hot mounting and cold mounting processes are possible. Cold mounting is recommended if the samples are fragile, small or have a complicated shape. The following section will show standard surface preparation methods for 4 typical material groups used in AM.



TITANIUM

Because of its extreme ductility, titanium is prone to mechanical deformation and scratching during metallographic preparation. Diamond polishing should be avoided, especially with pure titanium, as it introduces scratches and smearing. Once introduced, this layer of deformation is difficult to remove. To avoid this issue, a chemical-mechanical polish can be used directly after plane- and fine grinding. The three-step method in Table 2 (page 10) is a proven procedure that gives excellent, reproducible results for titanium and titanium alloys.

A fast and low deformation option, especially useful for the less alloyed titanium alloys, is electropolishing.

Equipment:	LectroPol-5
Electrolyte:	A3
Mask size:	1 cm ²
Temperature:	Room temperature 18-20°C
Flow rate:	10-15
Voltage:	34-45 V
Time:	20-30 sec

Table 1: Preparation method for electrolytic polishing of Titanium.

The sample can be polished to a mirror-like surface after a few seconds, ready for investigation. A method for electrolytical polishing is shown in Table 1. Before electrolytic polishing can be performed, a pre-ground or pre-polished surface is recommended. The time required to perform electrolytic polishing is dependent on the surface roughness, i.e., finer surfaces require less time. The method in Table 1 is suitable for equipment like Struers LectroPol-5. A microstructure of an electropolished sample from additive-manufactured titanium is shown in Fig. 15.

To reveal more specific information of the titanium sample, etching is needed after either mechanical or electrolytic preparation. The chemically resistant characteristics of titanium cause a need for aggressive chemical etching with etchants containing hydrofluoric acid. The amount of HF varies, and frequently used reagents are Keller, Kroll or Fuss, see Fig. 16. Polarized light is an optical etching technique especially suitable for all hexagonal structured materials, like titanium. It can be used after oxide polishing, see Fig. 14, or after special additional etching methods like Keller, see Fig. 17.

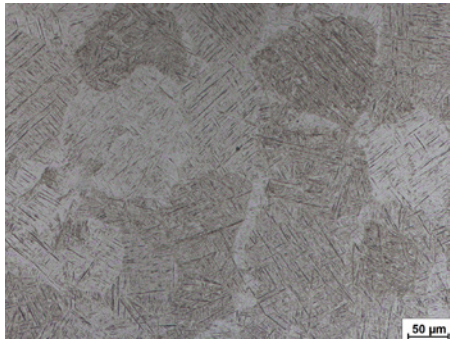


Fig. 16: Titanium alloy electrolytically polished and chemical etched with Fuss etchant. Bright field.

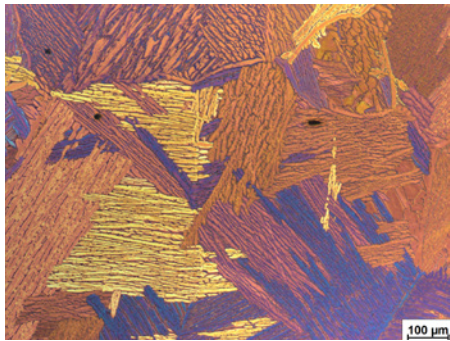


Fig. 17: Titanium alloy after etching with Keller etchant. Polarized light.

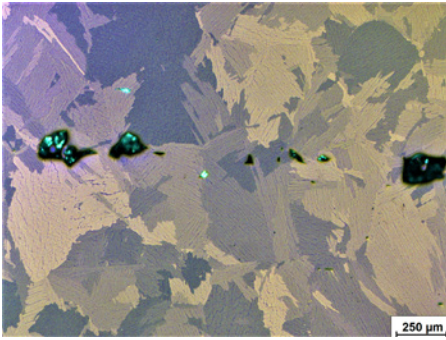


Fig. 14: Final polished titanium sample with pores. Polished on MD-Chem with OP-S NonDry and additive, microstructure visible in polarization contrast.

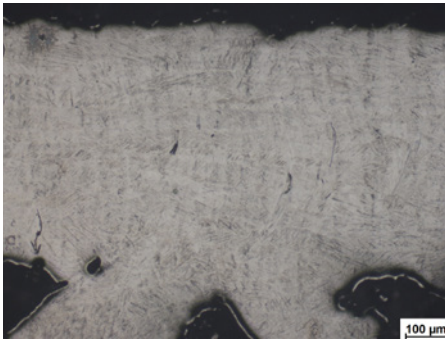


Fig. 15: Titanium alloy in bright field mode. Electrolytical preparation with A3, not mounted. Bright field.



RECOMMENDATIONS FOR THE PREPARATION OF TITANIUM AND ALUMINIUM

Table 2 - Grinding and polishing preparation method for unmounted 30 mm samples of titanium

		Grinding		Polishing
STEP		PG	FG	OP
 	Surface	MD-Mezzo	MD-Largo	MD-Chem
	Abrasive	Type	Diamond	Colloidal Silica
		Size	#220	9 µm
	Suspension/ Lubricant	Water	DiaPro Allegro/Largo 9	OP-S*
	rpm	300	150	150
	Force [N]/ specimen	40***	30	15
	Time (min)	Until Plane	5	5**

* Mix 70-90% OP-S with 10-30% H2O2 (of 30% concentration).
** The polishing time depends on the sample area. Very large samples require more polishing time than small ones.
*** Decrease to 25 N to avoid pencil shapes in single sample preparation of mounted samples.

Table 3 - Grinding and polishing method for unmounted 30 mm samples of aluminium

			Grinding		Polishing	
STEP			PG	FG	DP	OP
 	Surface		MD-Mezzo	MD-Largo	MD-Mol	MD-Chem
 	Abrasive	Type	Diamond	Diamond	Diamond	Colloidal Silica
		Size	#220	9 μm	3 μm	0.25 μm
 	Suspension/ Lubricant		Water	DiaPro Allegro/Largo 9	DiaPro Mol R3	OP-S NonDry
	rpm		300	150	150	150
	Force [N]/ specimen		25	30	25	15
	Time (min)		Until Plane	4	3	1



ALUMINIUM

Aluminium is, in general, soft but alloying elements can change mechanical properties drastically. The challenges in the preparation of aluminium are the introduction of deep deformations during grinding or embedding of hard abrasives in the material during grinding or polishing. That might occur if the selected preparation surface is too hard or aggressive. The quality of the polishing result is also a challenge for aluminium, as a well-polished surface is needed to create a sufficient contrast for microstructure analysis.

A preparation method for aluminium is shown in Table 3. A rigid plane grinding surface, MD-Molto, was specifically developed for aluminium alloys above 50 HV1. MD-Molto has good removal rates and eliminates the risk of edge rounding, which can be seen when using SiC papers or foils. For the fine grinding, MD-Largo with a 9 µm diamond suspension (e.g. DiaPro Allegro/Largo) is suitable for many types of materials, and is chosen for aluminium to address the challenge of deformation and scratches and the risk of incorporating diamonds into the soft material. The trade-off for this gentle fine grinding is lower removal rates, resulting in longer fine grinding times.

The need for a thorough polishing process of aluminium is addressed by the combination of a diamond and an oxide polish step. A soft woollen cloth is used, MD-Mol, with 3 µm diamond suspension, followed by an oxide polishing step with colloidal silica, OP-S. Final polishing suspensions should be used on a surface like MD-Chem. For better flatness and edge retention, another cloth with lower resilience, such as MD-Nap, can be used.

To reveal more, or specific details in aluminium structures both chemical, electrochemical, and optical etching methods, or a combination, can be used. In Fig. 19, a polished sample is shown using differential interference contrast. With this technique, topographic information of the specimen surface is emphasized. In Fig. 18, samples electrolytically etched with Barker's reagent are shown.

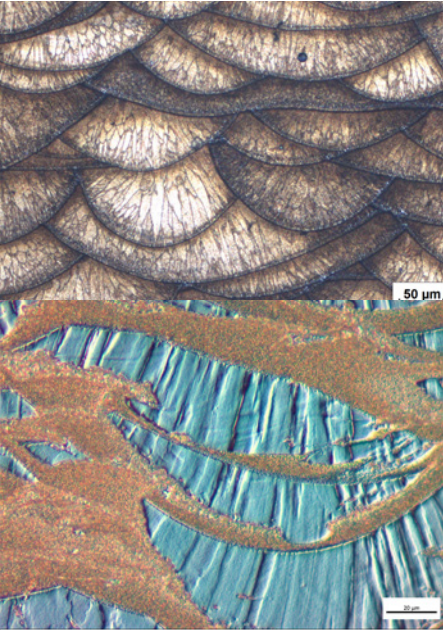


Fig. 18: Different aluminium alloys after Barker's etching in bright field (top) and in differential interference contrast, DIC, (bottom).

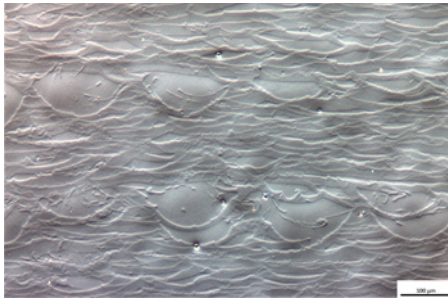


Fig. 19: Fine polished surface of aluminium alloy. Polished with MD-Chem and OP-S. Differential interference contrast, DIC, unetched.

In Fig. 22 (page 12), a sample after etching with Barker's reagent is shown in polarized light. A method for electrolytical etching of aluminium with Barker's reagent is shown in Table 4. The method is suitable for a Struers LectroPol-5 (Fig. 20).

Equipment:	LectroPol-5
Electrolyte:	200 ml water + 10 ml fluoroboric acid (35%) (Barker's)
Mask size:	2 cm ²
Voltage:	24 V
Flow rate:	8

Table 4: Preparation method for electrolytic polishing and etching of aluminium.



Fig. 20. Struers electrolytic preparation equipment, LectroPol-5.

STAINLESS STEELS AND NICKEL-BASED ALLOYS

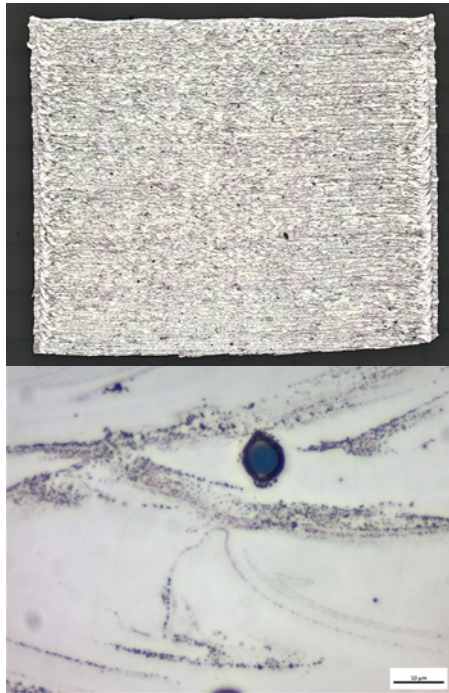


Fig. 21: Overview of an AM aluminium alloy, etched with Barker's (top). Detail of the aluminium alloy, with precipitation in high magnification (bottom). Bright field.

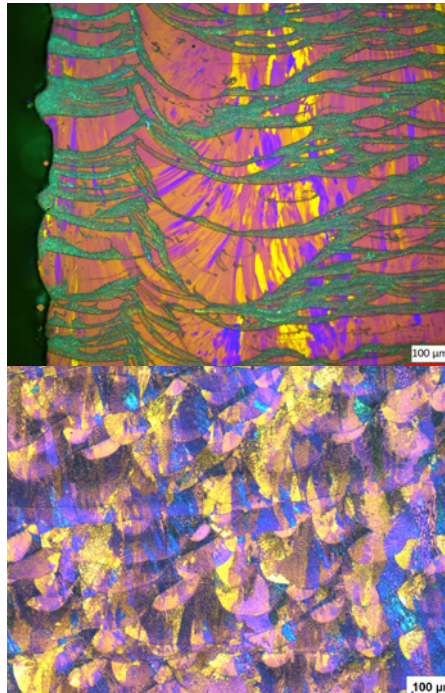


Fig. 22: Aluminium alloy after Barker's etching (top). Inconel etched with Adler (bottom). Polarized light.

Stainless steels and nickel-based alloys are often soft and ductile. Therefore, the use of very coarse grinding surfaces and high pressure should be avoided. To address this, a dedicated plane grinding disc for these materials, MD-Alto, is used (Table 6.) Fine grinding is carried out with diamond suspension on a rigid disc (MD-Largo) or, as an alternative for some types of stainless steels, on a MD-Plan cloth. Fine grinding is followed by a thorough diamond polish on a medium soft cloth, MD-Dac, and the final polish with colloidal silica (OP-S), or alumina (OP-A) removes the fine scratches. This step should be very thorough, and it can take up to several minutes. A good final polish increases the chance for a better contrast.

The method can also be used for samples when performing porosity evaluation of additive-manufactured samples. In that case, the final oxide polishing step is left out, as the 3 µm polishing on a medium/hard acetate cloth, MD-Dac, reveals the true porosity of the sample, see Fig. 23 (page 13).

An alternative to mechanical polishing is electrolytical polishing, which is done with an electrolyte based on perchloric acid (A3). A method for Struers LectroPol-5 is shown in Table 5 (page 11).

Table 6 - Grinding and polishing method for unmounted 30 mm samples of stainless steel

STEP		Grinding		Polishing	
		PG	FG	DP	OP
	Surface	MD-Alto	MD-Largo	MD-Dac	MD-Chem
	Abrasive	Type	Al ₂ O ₃	Diamond	Colloidal Silica
		Size	#180 / 220	9 µm	3 µm
	Suspension/Lubricant	Water	DiaPro Allegro/Largo 9	DiaPro Dac 3	OP-S NonDry / OP-A
	rpm	300	150	150	150
	Force [N]/specimen	25	40	20	15
	Time (min)	As needed	5	4	2-3

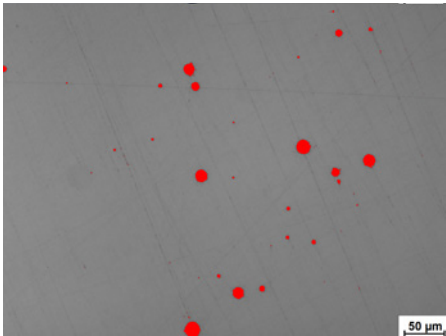


Fig. 23: Measured porosity in additive manufactured stainless steel after 3 μm polishing step on MD-Dac, unetched, bright field. The visible scratches are from the 3 μm diamond polishing suspension.

Equipment:	LectroPol-5
Electrolyte:	A3
Area:	1 cm ²
Voltage:	35 V
Flow rate:	13
Time:	25 sec

Table 5: Preparation method for electrolytic polishing of stainless steels. Initial grinding on SiC foil/paper #320, #500 and #1000.

Austenitic and corrosion-resistant steels, as well as nickel super alloys, can be etched with acids like V2A lye, Adler etchant (Fig. 25), or colour etchants like Beraha II or III or Lichtenegger and Bloech. Also, optical etching with differential interference (DIC) contrast filter can be useful on stainless steels.

See Fig. 24 after oxide polishing and Fig. 26 after electrolytical etching.

The electrolytic etching method, using 10% oxalic acid in water, is very common, see Fig. 26 and Table 7 (see next page). For duplex steels electrolytic etching with NaOH (20% in water) delivers good results. Polarized light is an optical etching method which can be used after special additional etching, such as oxalic acid for austenitic steels, or Adler for Ni-based superalloys, see Fig. 25.

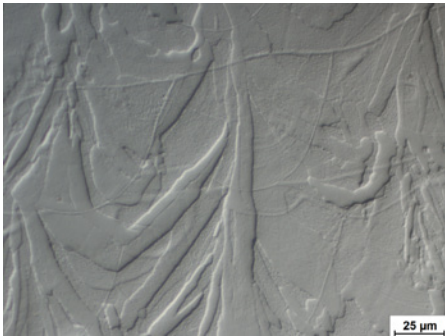


Fig. 24: Austenitic steel after polishing with OP-S on MD-Chem. Structure visible without chemical etching. Differential interference contrast, DIC.



Fig. 25: Inconel 718, etched with Adler etchant. Bright field.

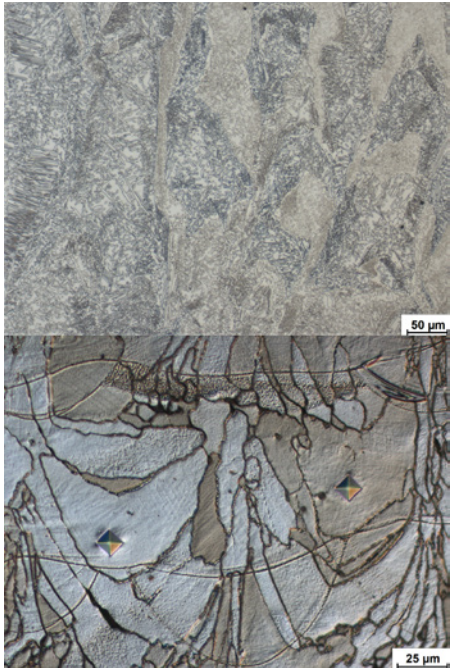


Fig. 26: Duplex steel 1.4462 after electrolytic etching with oxalic acid (10%). Bright field (top). Duplex steel 1.4410 with micro hardness indents, after electrolytical etching with 20% NaOH in water, DIC (bottom).



TOOL STEELS

Tool steel is a high alloy steel made with large amounts of alloying elements, such as chromium, nickel, vanadium, tungsten, or molybdenum. The main difficulty during grinding and polishing of high alloy tool steels is ensuring that carbides and non-metallic inclusions are retained.

Depending on the state of the microstructure, carbides can either fracture during grinding or be pulled out from the softer matrix during grinding or polishing.

For these types of ferrous metals, ductility or proneness to smearing and scratches must be taken into consideration, see Table 8. Diamond plane grinding discs like MD-Piano can be used with its high material removal. For the fine grinding step, the metal bonded composite disc, MD-Allegro, with a 9 μm diamond suspension is used before the diamond polishing steps.

For high alloyed tool steels, a modified Klemm etchant can be used, see Fig. 27.

Equipment:	LectroPol-5
Electrolyte:	10% aqueous oxalic acid
Voltage:	15 V
Time:	60 sec

Table 7:. Preparation method for electrolytic etching of Stainless Steel

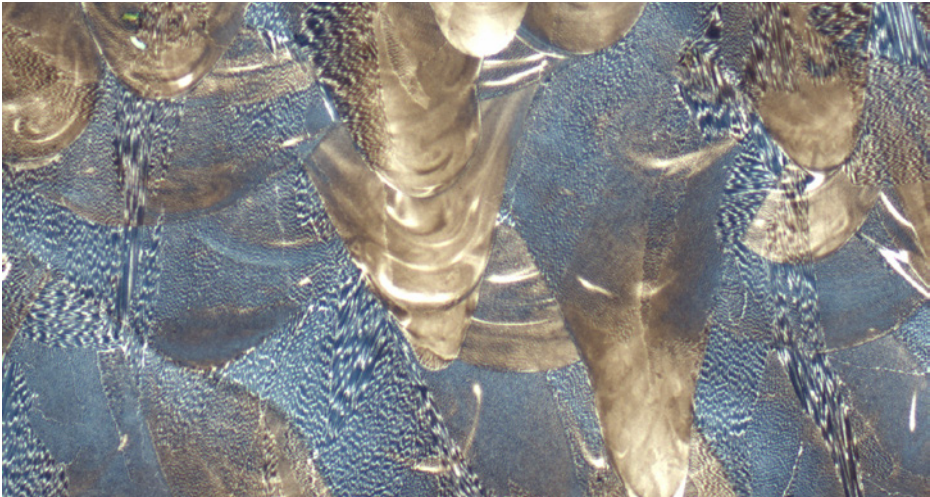


Fig. 27: Tool steel 1.2709 after etching with modified Klemm (10/3) and addition of HCl.

Table 8 - Grinding and polishing method for unmounted 30 mm samples of tool steel

		Grinding		Polishing		
STEP		PG	FG	DP 1	DP 2	OP
	Surface	MD-Piano 220	MD-Allegro	MD-Dac	MD-Nap	MD-Chem
	Abrasive	Type	Diamond	Diamond	Diamond	Alumina
		Size	#220	9 μm	3 μm	1 μm
	Suspension/ Lubricant	Water	DiaPro Allegro/Largo 9	DiaPro Dac 3	DiaPro Nap B 1	OP-A
	rpm	300	150	150	150	150
	Force [N]/ specimen	35	35	35	25	15
	Time (min)	As needed	9	6	1	1

TARGET PREPARATION

It is for additive manufactured materials often of special interest to investigate microscopic attributes in the microstructure. These targets might be pores, cracks, or inclusions from a laser sintering process, as well as impurities from different feedstock materials, like powders (see Fig. 29).

If porosity and cracks open to the surface should be visualized, the use of epoxies with fluorescent powders mounted under vacuum are a useful solution, see Fig. 28. Heating up the epoxy to 50-60°C to increase viscosity and using active cooling during curing gives very good impregnation and very low shrinkage and gap formation. The use of fluorescent dye requires a special feature in the microscope.

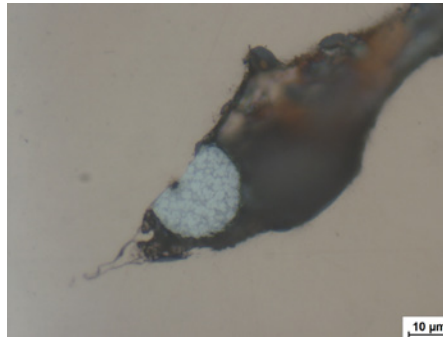


Fig. 29: Contamination of ALSi powder particle into CuCrZr1 sample. Bright field.

The special specimen holder, Accustop (Fig. 30), uses controlled material removal to hit a specific location in the specimen. Consumables with consistent removal are a prerequisite for this technique.

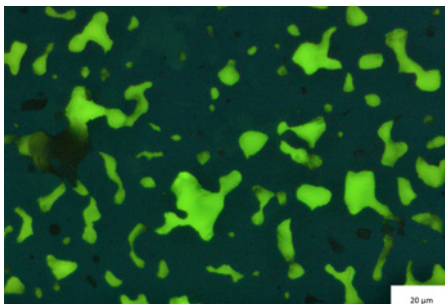
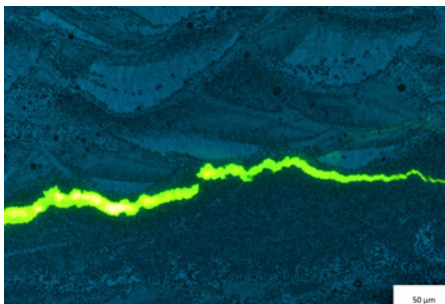


Fig. 28: Filling of a crack in an aluminium alloy with fluorescent resin (top). Open porosity in austenitic steel (bottom).

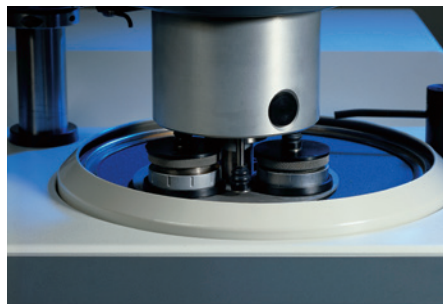


Fig. 30: Struers Accustop for controlled material removal

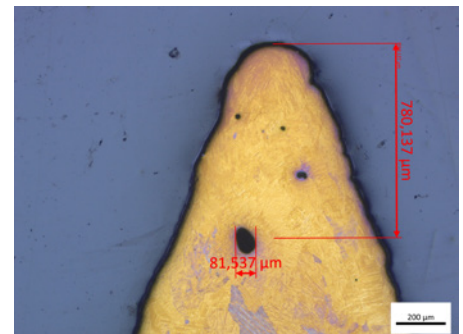


Fig. 31: Light microscope image of the largest pore in a titanium alloy, prepared with the Target system. The sample was etched with an etchant of 100 ml water, 10 gr. NaOH and 10 ml H₂O₂.



APPLICATION NOTE

Metallography of additive manufactures material

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3. All microstructure images are provided by Dr. Holger Schnarr, Struers GmbH