VERDER scientific

ENABLING PROGRESS IN

ADDITIVE MANUFACTURING

SOLUTIONS FOR ADDITIVE MANUFACTURING & POWDER INJECTION MOLDING

For a number of years Additive Manufacturing has been recognized as a key technology for Rapid Prototyping. New product iterations can be produced in a timely fashion, enabling initial functional tests which allow customers to ascertain the potential thanks to a functioning rapid prototype. This technology is advancing rapidly beyond mere prototyping as today, highly integrated parts are conceived, designed and produced using Additive Manufacturing techniques. This allows the manufacturing of highly sophisticated, often miniaturized, light parts which could not be produced by traditional methods, f. e. hydraulic parts for aircraft engines. For cost reasons, the process of AM is not yet effective for high-volume mass production of parts. In these cases traditional manufacturing methods like Powder Injection Molding are still superior.

VERDER SCIENTIFIC: YOUR SOLUTION PROVIDER FOR THE ADDITIVE MANUFACTURING & POWDER INJECTION MOLDING PROCESS

Particle size and shape analysis, elemental analysis, heat treatment, microstructural analysis and hardness testing: the Verder Scientific companies offer innovative, efficient solutions for your additive manufacturing or powder injection molding process – combined with expert advice and support service worldwide.

CONTENT

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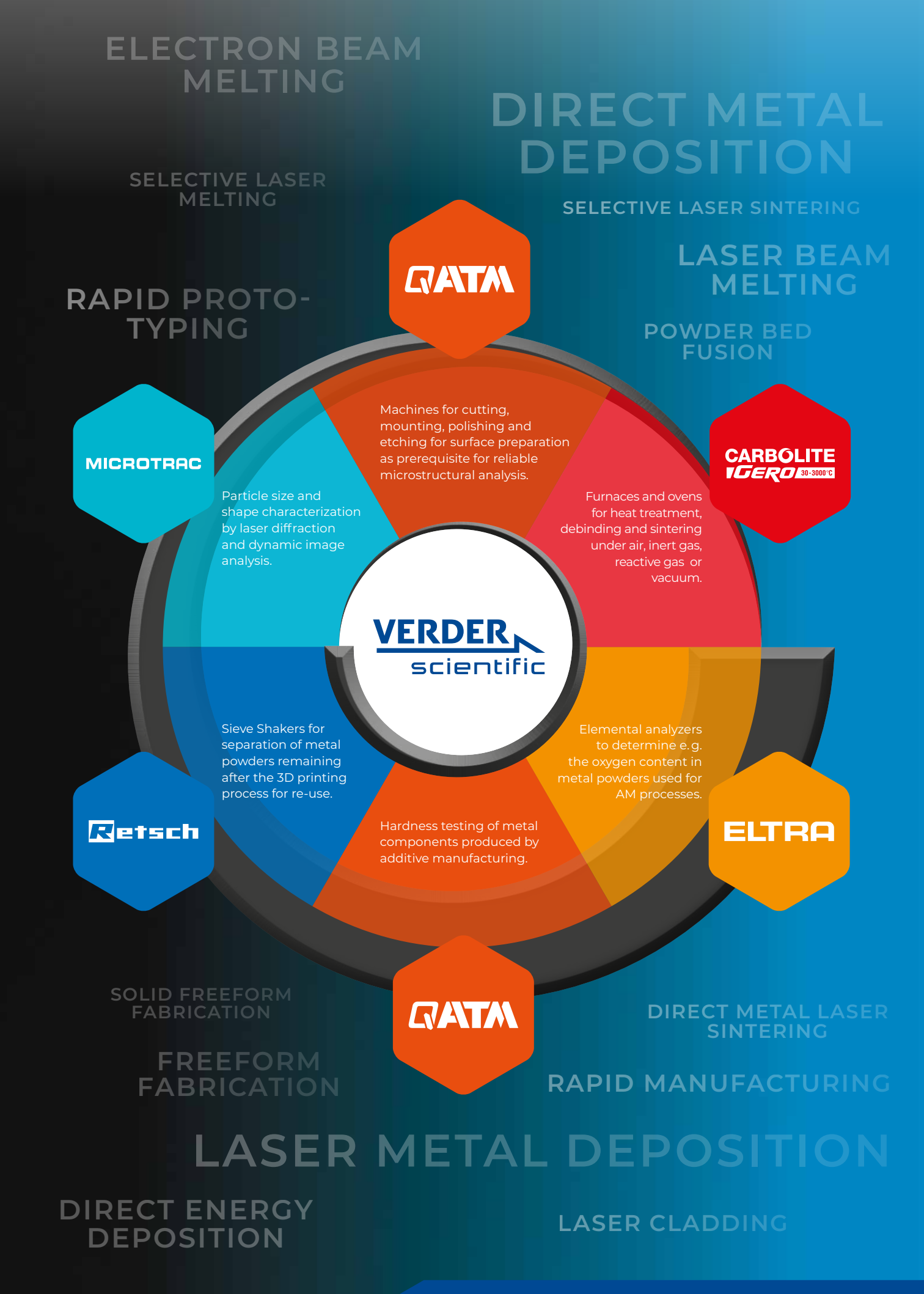
ELECTRON BEAM MELTING
DIRECT METAL DEPOSITION
SELECTIVE LASER MELTING
SELECTIVE LASER SINTERING
LASER BEAM MELTING
RAPID PROTO-TYPING
POWDER BED FUSION
QATM
MICROTRAC
CARBOLITE GERO 30-3000°C
Machines for cutting, mounting, polishing and etching for surface preparation as prerequisite for reliable microstructural analysis.
Particle size and shape characterization by laser diffraction and dynamic image analysis.
Furnaces and ovens for heat treatment, debinding and sintering under air, inert gas, reactive gas or vacuum.
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Sieve Shakers for separation of metal powders remaining after the 3D printing process for re-use.
Elemental analyzers to determine e. g. the oxygen content in metal powders used for AM processes.
Retsch
ELTRA
Hardness testing of metal components produced by additive manufacturing.
QATM
SOLID FREEFORM FABRICATION
DIRECT METAL LASER SINTERING
FREEFORM FABRICATION
RAPID MANUFACTURING
LASER METAL DEPOSITION
DIRECT ENERGY DEPOSITION
LASER CLADDING

PARTICLE CHARACTERIZATION OF METAL POWDERS

In this article, we present several examples of how the size and shape of typical metal powders and metal alloys can be characterized by Dynamic Image Analysis (DIA) and Laser Diffraction (LD) technologies, using the Microtrac CAMSIZER X2 and SYNC analyzers. The advantages of these instruments are short analysis times, excellent repeatability, and "infinite" resolution. Many different size and shape parameters are measured and reported, for each individual particle, and all data is available as soon as the measurement ends. Shape parameters are calculated as ratios of various size measurements and are reported on a scale from 0 to 1. The data for each parameter can be reported in both frequency and cumulative distributions, in volume and number format, and the complete parameter data set can be reported for each individual particle.

IMAGE ANALYSIS: WHAT YOU SEE IS WHAT YOU GET

Image analysis techniques provide a direct approach to particle size analysis. The basic idea is simple: "What you see is what you get". Automatic software algorithms determine size and morphology based on digital photographs of individual particles.

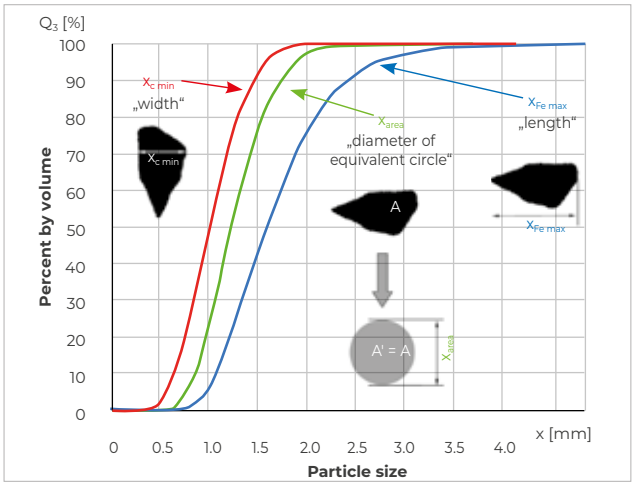


Fig. 1: Selection of CAMSIZER X2 basic size parameters used in image analysis. The size distributions are based on width (red), length (blue) or equal area diameter (green).

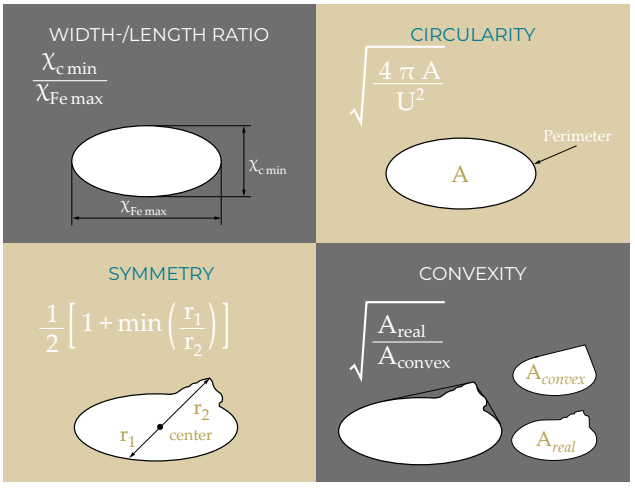


Fig. 2: Selection of basic shape parameters (CAMSIZER X2).

THE CAMSIZER X2: DIA ANALYZER

The CAMSIZER X2, with the widest dynamic range in the industry, 0.8 µm to 8 mm, can measure both suspensions, or dry samples, using one of three different sample dispersion accessories. Below in Fig 1. particle length, width and equivalent area diameter information from the CAMSIZER X2 are reported. A selection of shape parameters is explained in Fig. 2.

In the measurement set-up of the CAMSIZER X2 (DIA), particles move in front of a camera system, either transported by single pass air flow or recirculating in liquid. Thus, it is possible to obtain data from up to several millions of particles within a few minutes, especially when measuring dry. The results are based on a representative amount of sample material for both methods and are therefore statistically sound.

Fig. 3 displays the principal set-up of the optics for the CAMSIZER X2 Dynamic Image Analyzer. As the particles pass through the field of view a light source illuminates the particles from one direction while a camera system takes pictures from the opposite side. A software evaluates the shadow projections of the particles to determine the size distribution of the sample with a high acquisition rate. A unique feature of the CAMSIZER X2 is the dual camera technology: Two cameras with different magnifications cover a wide measuring range. One camera with high magnification is optimized for the analysis of small particles, a second camera with a lower magnification but wide field of view allows simultaneous analysis of the larger particles with high detection efficiency. The CAMSIZER X2 records more than 300 frames per second with one single frame easily containing several hundreds of particles, depending on the sample size range.

DIA measures the particle size distribution and quantitative particle shape (percentage of round versus irregular shaped particles, agglomerates etc.). Very small amounts of oversized, undersized, or irregular shaped particles can be detected, to a percentage as low as 0.002 %. DIA enables the user to obtain a comprehensive and thorough understanding of size- and morphology-related sample properties. DIA is the ideal method for both R & D applications and quality control because it is accurate, robust, sensitive and easy to use.

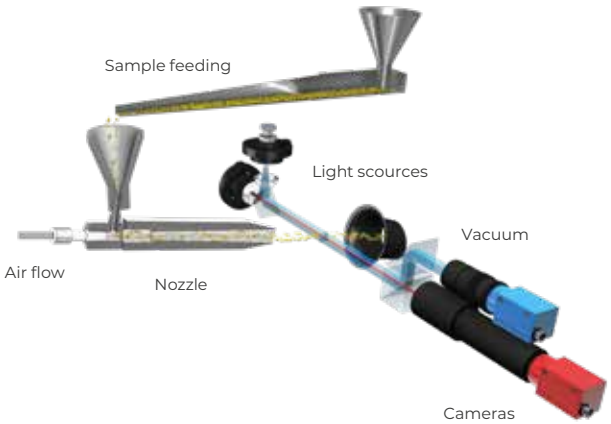


Fig. 3: Unique measurement principle of CAMSIZER X2 for analysis of dry powders.

WIDE RANGE OF MATERIALS, PARTICLE SIZES AND PARTICLE SHAPES

In the following, a selection of application examples demonstrates the suitability of DIA to comprehensively characterize metal powders. Fig. 4 shows the results of the size analysis of ten different metal powders which are typical for powder metallurgical applications. Irrespective of the difference in chemistry, density, size and shape, all samples can be analyzed with the CAMSIZER X2, using one instrument setup. An automatic feeding chute transports the sample to the analyzer where the particles are captured by an air flow. In this case 50 kPa have been found sufficient to achieve thorough dispersion, i. e. separation of individual particles.

The samples show a variety of mean particle sizes between 10 and 50 µm, with different widths of distribution (Fig. 4). In this example, the iron powder (Fe) is the coarsest whereas the steel powder (316) is the finest. The titanium powder is characterized by a very narrow size distribution.

The shape diagram (Fig. 5) shows that the iron powder has the lowest aspect ratio (breadth/length), whereas the titanium powder has the largest share of spherical particles.

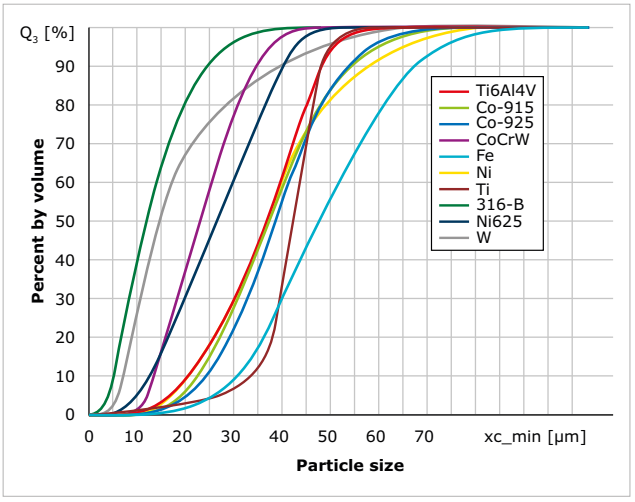
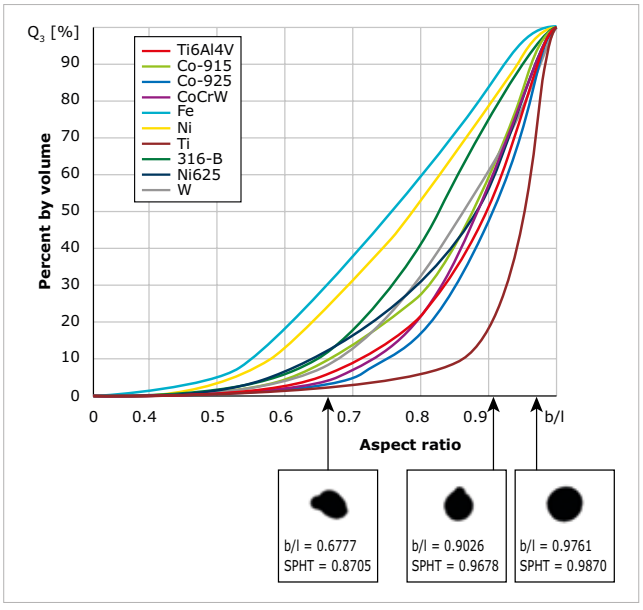


Fig. 4: Particle size analysis of ten different metal powders with the CAMSIZER X2. The direct measurement ensures accurate results.



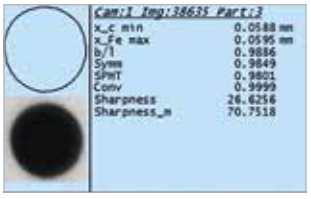
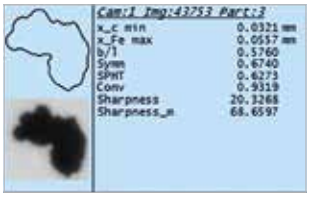


Image of a spherical metal powder particle



Irregular particles are reliably detected

Fig. 5: Analysis of particle shape of 10 different metal powders with Dynamic Image Analysis (CAMSIZER X2). Beside the quantitative results, the recorded images allow an intuitive understanding of morphology and size differences. More spherical particles with higher aspect ratio plot on the right side of the diagram. Detecting the smallest amounts of irregular particles in a large quantity of predominantly spherical particles is a great advantage of DIA.

Powder metallurgical processes usually require a wide size distribution to make packing the powder into the die easier by filling the void spaces between large particles with smaller ones. An irregular shape is often beneficial for the sintering process because it increases the contact between particles. However, the particles must not be too irregular as this will make compaction more difficult. For additive manufacturing, a spherical shape with a broad size distribution provides a smooth layer of powder to ensure proper sintering and good metal parts.

The average particle size is usually between 10 – 50 µm, hence the titanium powder in the above example is suitable for additive manufacturing. Oversized particles or very irregular particles need to be detected with great accuracy since these are likely to cause defects in the finished workpiece.

DIA reliably detects even small amounts of these undesired particles. Fig. 6 shows clearly how easily DIA can identify defective particles.

THE SYNC: HYBRID DIA & LASER DIFFRACTION (LD) ANALYZER

The novel SYNC analyzer is a revolutionary hybrid instrument which combines LD and DIA technologies in one unit, measuring the same sample in the same sample cell simultaneously. LD (a type of light scattering) technology has been used by the metal powder industries for decades as the de facto standard for measuring size distributions in outgoing QC certification by metal powder suppliers and incoming QC verification by powder metallurgy parts producers.

The optical bench of the SYNC is shown in Fig. 6. Three lasers, available in blue or red, in combination with two linear diode detector arrays allow the scattered light from the passing particles to be collected over a range of 163 degrees. Smaller particles scatter light at higher angles and lower intensities than do larger particles.

The SYNC algorithm for LD back-calculates the particle size distribution that created the light flux distribution measured. Using a modified Mie theory, the algorithm compensates for non-spherical and translucent particles.

Simultaneously, a rapid LED strobe lamp illuminates the particles and a set of optics focusses the transmitted light for a digital camera to photograph the complete video file of the particle images, as the CAMSIZER X2 does, except that the SYNC uses one camera.

The display shown in Fig. 7 is the LD size distribution report used by the metal powders industries.

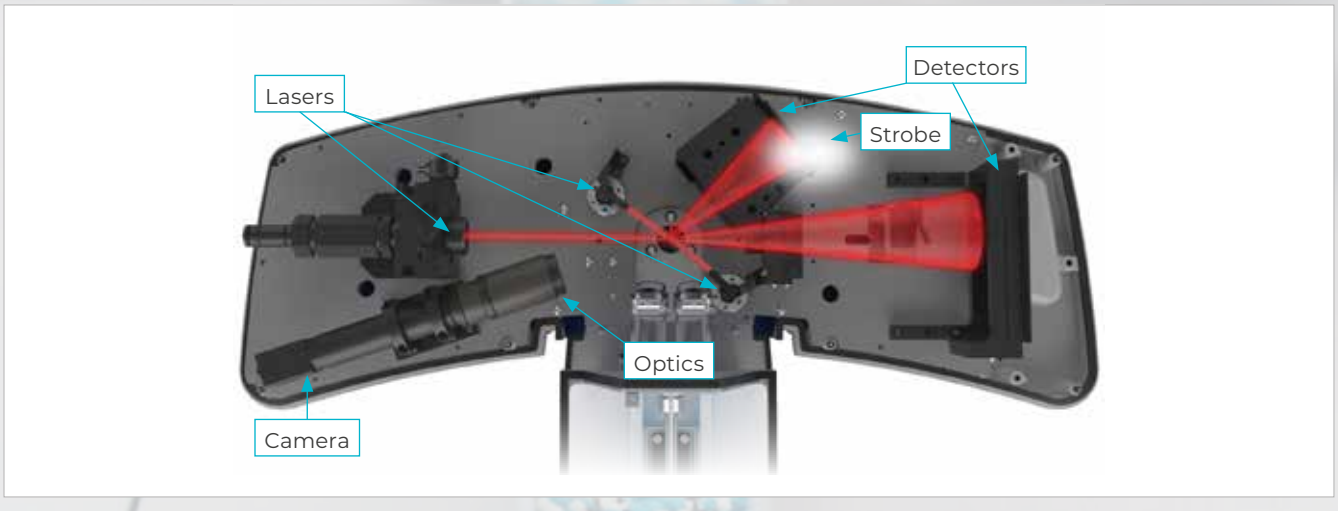


Fig. 6: Layout of the SYNC optical bench, combined LD and DIA components

The DIA post-measurement software features a Particle Viewer display (GUI) and a Scatter Diagram display. These can be used to identify and quantify the percentage of agglomerates in the metal powder batch. This allows metal powder suppliers to recycle a bad batch (too many agglomerates) before shipping to a customer, and it allows parts producers to reject a bad incoming batch and avoid wasting time and money processing it.

Both the CAMSIZER X2 and the SYNC can be relied on to quantify the agglomerates in a metal powder sample using two shape parameters. Size can't be used, because these agglomerates exist throughout the size distribution. The shape parameters consist of a parameter that measures aspect ratio and one that measures how convex the outer boundary of the particle is.

An example using the SYNC will be explained here. The parameters used will be the width to length aspect ratio (W/L Aspect Ratio) and Solidity. A particle with a solidity of 1 is a particle with a completely convex outer boundary with no concave indentations. A particle with a W/L Aspect Ratio of 1 would be a perfect sphere. In the CAMSIZER X2, the parameters are convexity (Conv) and breadth divided by length (b/l) respectively.

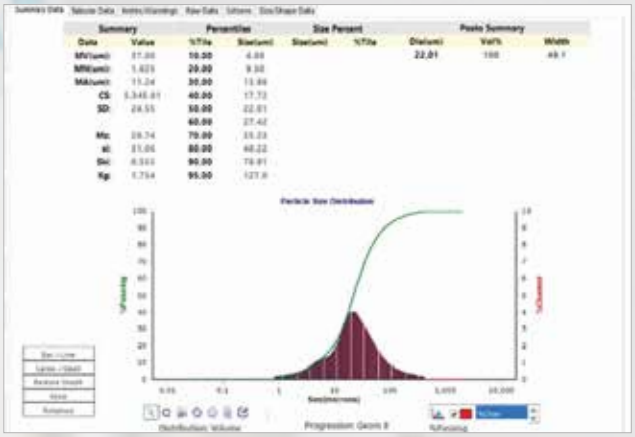


Fig. 7: LD particle size report from SYNC, including percentiles, summary statistics data and frequency / cumulative distribution graphs

The Search feature of the SYNC software was used to isolate and quantify the agglomerates, which include all particles not within the red rectangle in Fig. 9. The agglomerates were found to make up 23 % by volume and 12 % by number of the total sample. This is key QC information for the metal powder and parts industries.

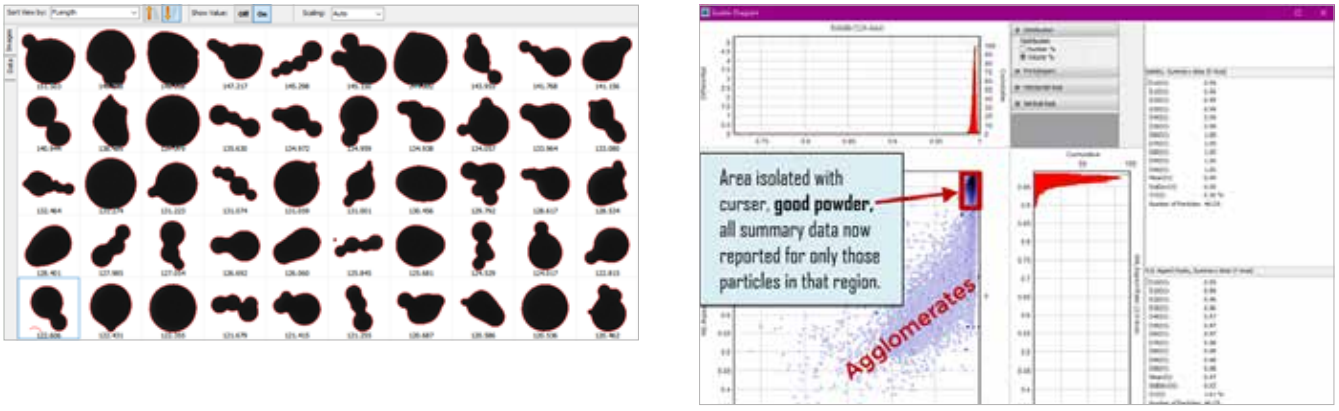


Fig. 8: Example of dynamic image analysis of a metal powder sample with the SYNC. Particles with specific properties or combinations of properties can be displayed (left) and quantified accordingly using scattergrams or distribution curves (right). However, the number of images and particles recorded is lower than with the CAMSIZER X2.

ADVANTAGES OF DIA AND LD OVER OTHER PARTICLE SIZING TECHNIQUES

For metal powders, mechanical sieve analysis is still used by some companies for particle size analysis. The absolute lower size limit for sieve analysis is defined by the smallest practically usable mesh size of 20 µm (air jet sieving), which is well above the average particle size of many samples for AM or MIM. As a consequence, air jet sieving is not suited for the precise and reliable analysis of the whole size distribution of fine powders. It is often used for detecting the amounts of oversized particles with one sieve only, for example with 45 µm or 63 µm aperture size. Another drawback is that sieve analysis does not deliver any information on particle morphology.

The metal powder industries began replacing sieves with Laser Diffraction in the 1970's. Since then, Laser Diffraction became, and still is, widely used throughout these industries and is expected to remain their standard method for certifying and verifying particle size distributions. Laser diffraction analyzers are easy to operate, and provide fast, robust results, and their technology and characteristics are well understood.

Dynamic Image Analysis is finding rapidly growing use by these industries for morphological analysis to set QC specifications on shapes to control properties that can't be identified by size analysis alone.

COMPARISON & CONCLUSION

With metal injection molding and additive manufacturing becoming increasingly prevalent techniques, there is an increased demand for specially designed metal powders with specific characteristics. Not only chemical composition, but also particle size & shape are of vital importance for the processability of powders. Depending on the application, the powder must meet a variety of specifications. Laser Diffraction size analysis for metal powders is embedded in these industries and expected to remain that way. Laser Diffraction (LD) plus Dynamic Image Analysis (DIA) with the SYNC, and DIA with the CAMSIZER X2 provide all relevant data on particle size & shape for metal powders. DIA, compared to (electron / optical) microscopy, measures a much larger number of particles and is therefore statistically more relevant and offers better reproducibility.

One measurement only takes 1 to 3 minutes which allows for a high sample throughput and continuous quality control. For both powder producers and manufactures of metal parts the SYNC and CAMSIZER X2 are precise, efficient tools which greatly improve the quality control process.

Performance Feature	DIA (CAMSIZER X2 & SYNC)	Laser Diffraction	Sieve Analysis
Wide dynamic range	++	+++	++
Reproducibility	+++	+++	++
High resolution for narrow distributions	+++	++	−
Particle shape analysis	+++	−	−
Compatibility of results with other techniques	+++	++	−
Reliable detection of oversize	+++	++	+++
Robust, easy operation	+++	+++	−
Measurement speed, sample throughput	++	+++	−
Analysis of individual particles	+++	−	++

CHARACTERIZATION OF METAL POWDER AND ITS AM PRODUCTS – DENSITY, SURFACE AND POROSITY

MEASUREMENT OF THE DENSITY OF FINELY DIVIDED METAL POWDER AND POROUS METAL BODIES WITH A GAS PYCNOMETER (ISO 12154)

Knowledge of the density, in particular the true density, is of fundamental importance for the characterization of all materials. Density as a quotient of mass and volume is usually determined by determining these two quantities separately, and weighing the measuring is relatively simple. In the case of metallic materials, the volume is often measured on a specially manufactured cuboid test specimen or determined according to Archimedes' principle by displacement of a liquid. This is not possible with powdered metals. Therefore, gas pycnometry using an inert measuring gas such as helium is recommended here. In a measuring chamber of known volume, the gas displacement caused by an inserted specimen is determined by measuring the changed pressure. The fully automatic helium multi-volume gas pycnometer BELPYCNO L is the measuring instrument for determining the volume and density of powders, granulates and porous solids, as well as pastes and liquids. Due to an integrated temperature control (Peltier elements) it is possible to measure in the range of 14 °C to 40 °C ± 0.01 °C (at 20 °C) without influence of the room temperature. Thus, for a given temperature and sample vessel size, only a single calibration is sufficient to measure permanently with an accuracy and reproducibility of 0.01%. Due to the enormous savings in calibration time, the effective sample throughput can be significantly increased.

The multi-volume concept allows the best possible characterization of the materials to be investigated through the optimal combination of sample vessel sizes and three corresponding reference chambers. The use of a high-precision absolute pressure sensor (+/- 0.002 kPa) allows permanent correction of atmospheric pressure variations during a measurement.

Depressurizing the test gas from the reference chamber into the sample chamber (DIN 66137) reliably prevents contamination of the reference chamber by fine metal powders.

A bayonet lock ensures reproducible and easy closing of the sample chamber. Convenient operation is ensured by using computer control or as standalone system via an alphanumeric keypad and a display. These features make the BELPYCNO L perfectly suitable for very rough environments.

An integrated microprocessor controls the complete measurement and performs the calculation of the results. Interfaces allow for connection of a PC, a balance, as well as a printer. The BELPYCNO L can be equipped with a vacuum pump and optionally with a humidity sensor.

The possible separation of the measuring chamber and control unit enables use of the instrument in a glove box, e.g. for applications in nuclear technology or oxygen-free handling for pure metal applications. Furthermore, the current pressure is displayed and recorded, an essential point to determine adequate pressure equilibrium times for volume determination of very fine-pored materials. Complete measurement protocols are also stored.

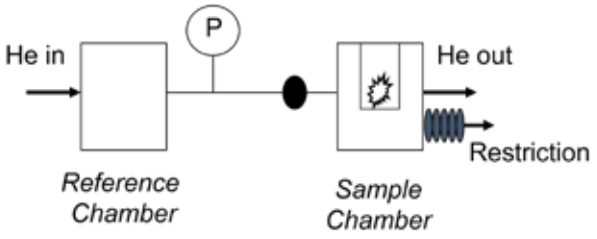


Fig. 1 Principle of the temperature-controlled helium pycnometer BELPYCNO L (DIN 66137), with absolute pressure sensor P.

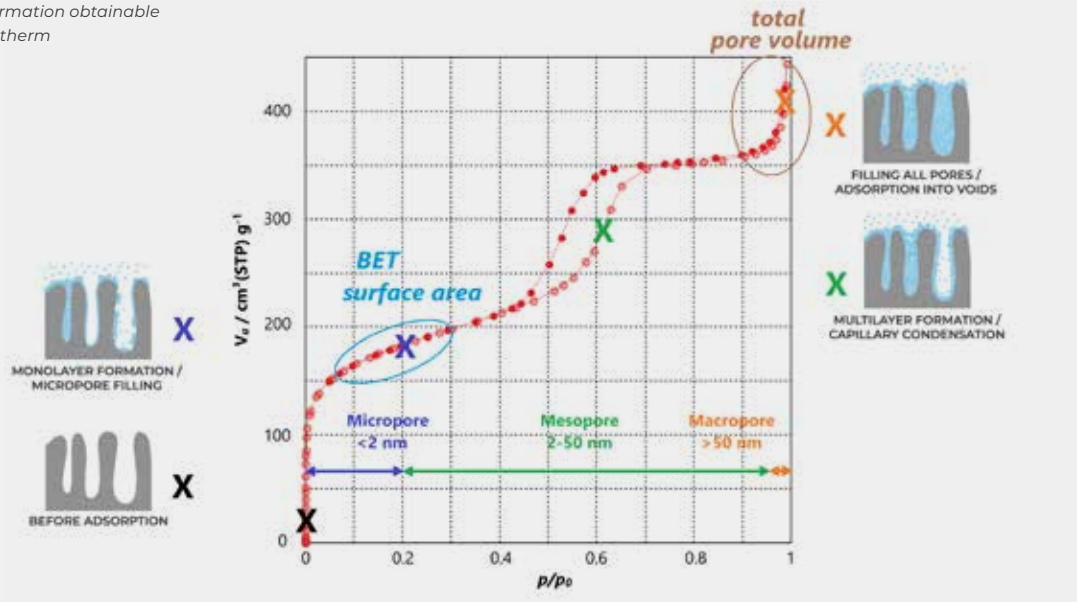
BELSORP MINI X – DETERMINE BET SURFACES OF METAL POWDERS QUICKLY, EASILY, ACCURATELY, AND ECONOMICALLY (ISO 15901-2, ISO 9277)

The surface area related to the mass as specific surface area (m^2/g) is an important parameter in the characterization and evaluation of metal powders and other materials. For example, the surface area is relevant for the achievable effect in catalysts, carrier materials (pharmaceuticals, electronics) and filters. Another aspect is the optimum application of coatings or, in the case of metallic powders, as a quality characteristic for processability. Sorption techniques are used in which an inert measuring gas adsorbs on the particle surface at a constant temperature near the condensation point. With increasing concentration or relative pressure, the thickness of this adsorbed layer will grow.

There are methods to calculate the number of adsorbed molecules in a monomolecular layer from such an experiment, the best known being the BET method. Knowing the surface area of a single molecule, it is easy to calculate the surface area of the whole sample. Furthermore, in case of porous materials the pore size can be determined from the measurement curve (isotherm) in the area of capillary condensation or pore filling using various models (e.g. BJH, NLDFT). As a rule, nitrogen is used as the sample gas for sorption measurements at a temperature of 77K (liquid nitrogen). Alternatively, measurements with argon gas at 87K are possible.

The BELSORP Mini X is a sorption measuring instrument which works according to the static volumetric principle. For this purpose, the sample vessel is first evacuated by a vacuum pump at liquid gas temperature, and then the sample gas is successively intruded, and the resulting increasing pressure is measured.

Fig. 2 Graphic: Information obtainable from a sorption isotherm



The adsorbed molecules generate a pressure difference from the expected theoretical pressure according to the ideal gas law. From this, the adsorbed amount and thus the adsorption isotherm are determined. After a given final pressure or saturation pressure has been reached, it is then possible to evacuate successively again and thus determine the desorption curve. Precise sorption measurements require a very accurate temperature measurement and control as well as precise pressure measurements, as technically implemented in the BELSORP Mini X.

By means of a control and evaluation software, the BELSORP Mini X can be operated from a PC to store and analyze measurement results.

In addition to pore size determinations, which typically take several hours, the purely rapid determination of BET surfaces is possible with up to 12 BET analyses per hour on the 3-port instrument - perfect for quality control of fine powders with high sample throughput.

The sum of these features results in a sorption device that meets today's quality control requirements with its accuracy and high sample throughput but can also be used in research.

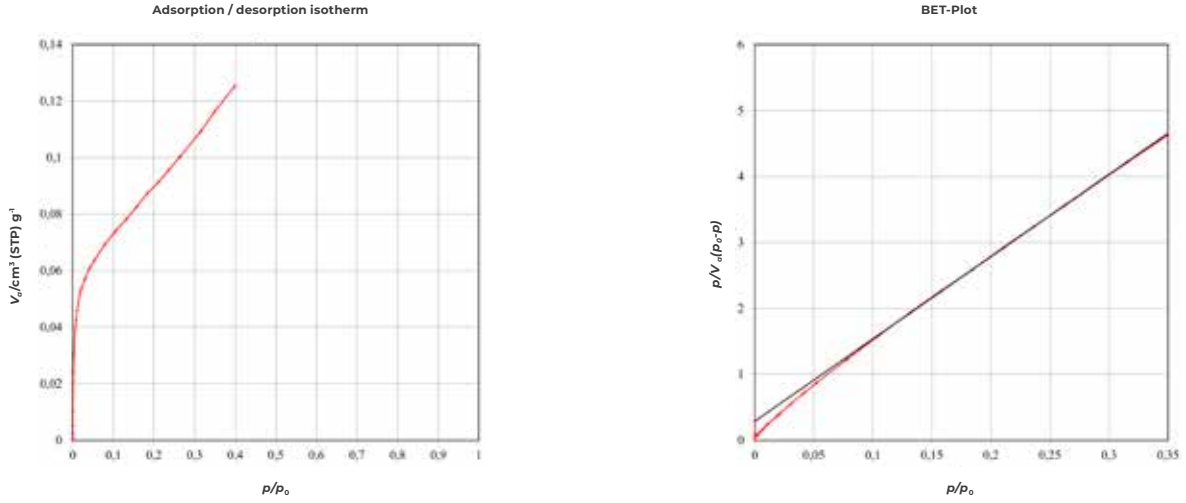


Fig. 3 BET Specific Surface Area (SSA) of Metal-powder - Lithium-Nickel-Mangan-Cobalt-Oxide with 0.34m²/g

PORE SIZE MEASUREMENT OF AM MANUFACTURED POROUS METALS WITH A MERCURY POROSIMETER (ISO 15901-1)

Porous materials include e.g. building materials, ceramics but also metal screens and metal foams which are produced from metal powders by additive manufacturing. Porous metals can be used for filtering particles (diesel soot), as catalyst supports or heat exchangers. On the other hand, porous metals can be interesting as components with lower density and due to their potential weight saving.

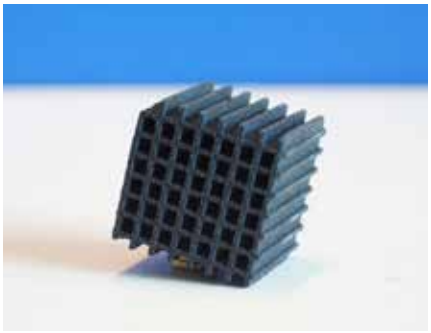
Knowledge of porosity, pore sizes and pore volume is fundamental to characterize porous materials. A widely used technique is mercury porosimetry, in which the non-wetting mercury is forced into the pores at room temperature with pressure up to 414 MPa. Any undesirable reaction of the porous metal with mercury can be prevented by passivation, e.g. by oxidation. The pore volume corresponds to the amount of mercury introduced. According to the Washburn equation, the pore size (radius, diameter) is inversely proportional to the applied pressure.

$$r = \frac{2\gamma \cos \theta}{p}$$

With pore radius r [m], surface tension of mercury γ **= 0.48 N/m**, intrusion pressure p [Pa]

With a contact angle θ **= 143.3°** a simple estimation is obtained ***r ≈ 0.7500/p***

The BELPORE mercury porosimeters measure pore diameters from 3.6 nanometers to 1 millimeter. BELPORE mercury porosimeters work according the P.A.S.C.A.L. principle which is not only a pressure unit. In the BELPORE porosimeter it means the equilibrium-controlled and optimized control of the pressure build-up by "Pressurization by Automatic Speed-up and Continuous Adjustment Logic".



MICROTRAC – PARTICLE CHARACTERIZATION

MICROTRAC is a technology leader, committed to innovation, with an extensive global network and unrivalled offering in particle characterization.

GAS ADSORPTION MEASUREMENT
- Surface Area & Pore Size Distribution
- Gas & Vapor Adsorption
- Catalyst Evaluation
- Density Measurement
- Breakthrough curve measurement
- High Pressure Gas Adsorption
- Mercury Porosimetry

PARTICLE SIZE & SHAPE ANALYSIS
- Dynamic Image Analysis (DIA)
- Laser Diffraction (LD)

STABILITY & DISPERSIBILITY ANALYSIS
- Dynamic Light Scattering (DLS)
- Static Multiple Light Scattering (SMLS)
- Zeta Potential (ZP) - Charge Titration

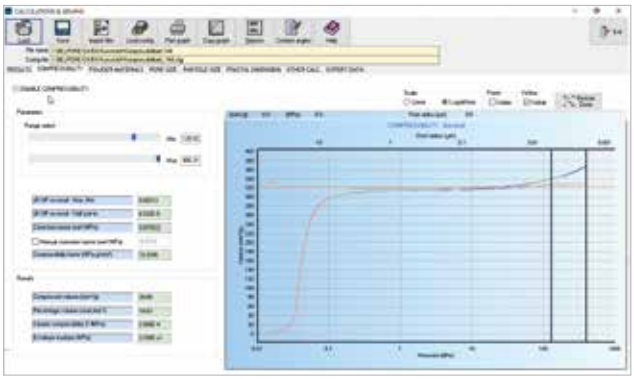


Fig. 4 Pore size distribution results

This automatic control is driven by the real pore system and allows shorter measuring times with guaranteed equilibrium conditions and detection of all pores within the specification, up to 20000 measuring points per analysis. Pressure tables are thus superfluous. Since only two types of dilatometers (sample vessels for powder or solids) are sufficient for all measuring tasks, and furthermore no gases or liquid nitrogen are required, operating costs can be kept significantly low.

Other innovations include simplified operation of the BELPORE LP low-pressure porosimeter, as well as an extended measuring range down to pore sizes of 1 millimeter! Vertical degassing and filling with mercury on the BELPORE LP allows the degassing pressure to be adjusted, and thus makes it possible to measure moist samples without changing the material moisture. The 3D evaluation software PoreXpert is optionally available. With this software, e.g. tortuousness, diffusion, percolation and much more can be derived from the porosimetry data.

BELPORE porosimeters are available in pressure ranges from 0.01 kPa to 450 kPa (and mercury filling), 0.1MPa to 228 MPa or up to 414MPa.

ELEMENTAL ANALYSIS
OF METAL POWDERS AND METAL PARTS PRODUCED BY ADDITIVE MANUFACTURING

Additive manufacturing (AM) has the big advantage that complex structures can be created in one single step. The DIN EN ISO/ASTM 52900:2018 standard (Additive manufacturing- General principles) defines additive manufacturing as a general term of those technologies that, based on a geometrical representation, create physical objects by successive addition of material.

Although AM is a much younger production technique than welding or founding, standard quality control procedures still have to be applied. One important part of quality control of metal powders (feedstock) and manufactured parts is the determination of the C/S and O/N/H content because these elements influence important mechanical parameters like hardness, ductility, corrosion and brittleness.

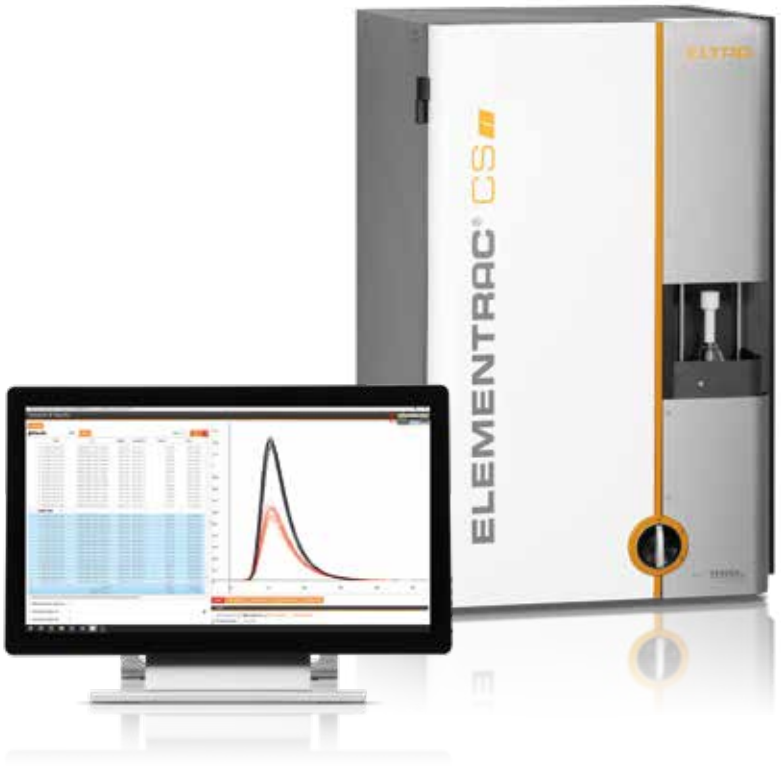

ELTRA
ON

INTRODUCTION OF C/S AND O/NH ANALYSIS

For covering the whole range of C/S and O/N/H analysis two different types of elemental analyzers are required which are described e. g. in the ASTM E 1019 or E 1447. The relevant standard for additive manufacturing DINENISO/ASTM 52907 (AM – Feedstock materials- Methods to characterize the quality of metal powders) refers to the above mentioned and other standards for quality control.

Combustion analyzers differ in the integrated furnace type (induction or electrode), the applied carrier gas and the used sample carrier (crucible). The common principle is the melting of the sample in a gas stream and measurement of the released gases in infrared (IR) or thermal conductivity cells (TCD). The table illustrates some basic features of a C/S and O/N/H combustion analyzers.

Elements	Technique	Carrier gas	Sample carrier	Sample weight (mg)	Suitable analyzer (ELTRA)
C/S	Combustion with induction furnace	Oxygen	Ceramic crucible	50-1000	ELEMENTARC CS-i
O/N/H	Inert gas fusion via electrode (impulse) furnace	Helium / Nitrogen / (Argon)	Graphite crucible	50-3000	ELEMENTRAC ONH-p 2



Placing sample with accelerator on the pedestal of the ELEMENTRAC CS-i

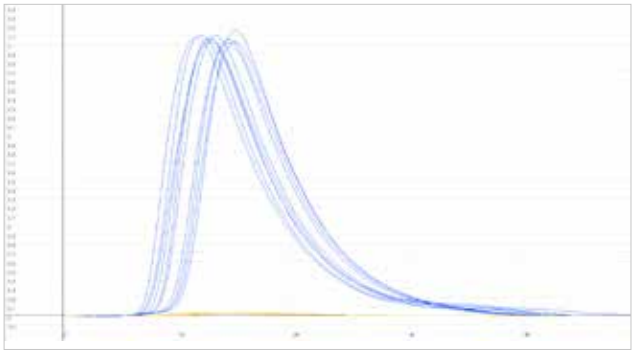
MEASUREMENT OF C/S IN METAL OR METAL POWDER SAMPLES

The utilization of a combustion analyzer like the ELEMENTRAC CS-i for carbon and sulfur analysis is fast and easy. After weighing the sample into a ceramic crucible some accelerator has to be applied (e.g. 1.5 g of tungsten) which assures a smooth and complete combustion of the sample and, consequently, complete release of the embedded carbon and sulfur as CO_2 and SO_2.

Due to the complete combustion of the applied sample, metal powders and metal components can be processed in the same way and the sample shape is not relevant for a correct analysis. The IR detection of the released CO_2 and SO_2 allows for safe and reliable measurement over a wide concentration range. A typical working range of a C/S analyzer like the ELEMENTRAC CS-i is about 1 ppm up to 7 % for a nominal weight of 1000 mg. The analysis cycle time usually is below 120 seconds and could optionally be automated with a 36 or 130 position autoloader.

Beside nickel, many other metals or alloys can be analyzed for their carbon or sulfur content. Typcial samples include steel, iron, nickel, copper, titanium and, of course, every alloy. Like for nickel the samples can be applied in the shape of powder, drillings, granules or solid samples.

Nickel powder ASTM AMPM 2010		
Weight [mg]	**Carbon [ppm]**	**Sulfur [ppm]**
499.4	250.1	11.2
499.8	250.9	10.4
499.5	248.8	9.1
500.1	250.9	12.5
500.6	248.3	11.8
500.8	242.9	9.9
500.8	248.5	9.9
500.5	246.9	9.0
499.4	250.8	11.8
500.9	250.3	11.1
Mean value	**248.8**	**10.7**
Deviation / relative deviation [%]	**2.5 / 1.0**	**1.2 / 11.2**



C/S ANALYSIS IN METAL POWDER

Analysis	Carbon and Sulfur with ELEMENTRAC CS-i
Sample	Nickel powder from ASTM Cycle AMPM 2010
Sample preparation	None
Settings	Accelerator 1.7 g tungsten Cycle time: 90 seconds/ analysis

MEASUREMENT OF O/N/H IN METAL OR METAL POWDER SAMPLES

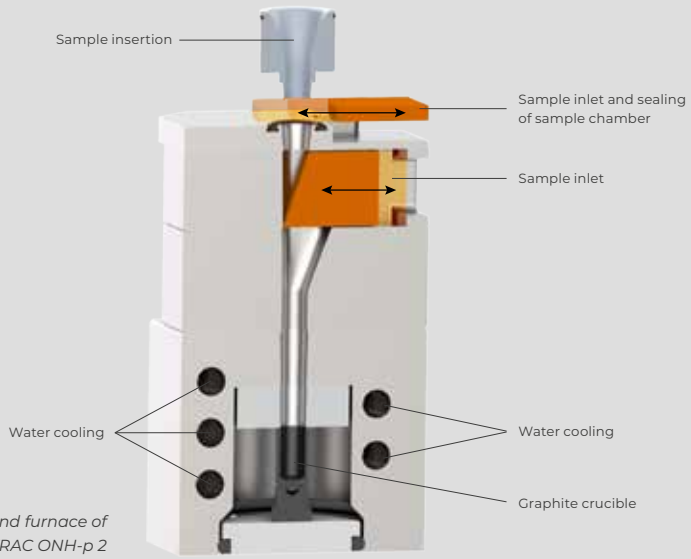
For a safe, fast and standard compliant determination of oxygen, nitrogen and hydrogen several details need to be considered. Sample shape, chemical composition and requested element to be analyzed affects the applied settings in the ELEMENTRAC ONH-p 2 as well as the applied flux or sample weight. ELTRA provides for the inert gas fusion analyzer ELEMENTRAC ONH-p 2 several application notes to make gas analysis as reliable and repeatable as possible.

After application of the sample to the furnace, the sample chamber is flushed with inert gas to remove atmospheric gases. Subsequently, the sample falls down into a hot graphite crucible where hydrogen and nitrogen are released in their elemental form, whereas oxygen reacts with the graphite crucible and forms CO. This CO is oxidized by a catalyst and measured as CO_2 in IR cells, whereas hydrogen and nitrogen are measured with a TCD cell.

This arrangement allows a safe analysis of oxygen, nitrogen and hydrogen from the lower ppm range up to the high percentage range.

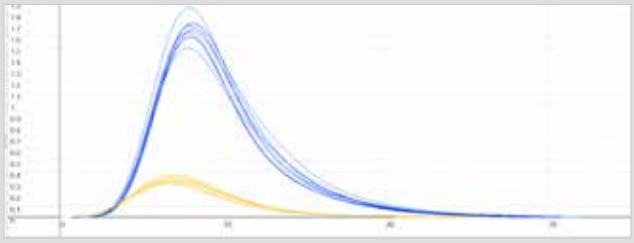
Like the ELEMENTRAC CS-i the ELEMENTRAC ONH-p 2 can be equipped with an Autoloader to enable automated analysis of up to 32 samples.

ELEMENTRAC ONH-p 2 with optional autocleaner



Sample port and furnace of the ELEMENTRAC ONH-p 2

Nickel powder ASTM AMPM 2010		
Weight [mg]	**Oxygen [ppm]**	**Nitrogen [ppm]**
260	198	11.2
250	199	10.4
225	202	9.1
243	199	12.5
227	203	11.8
287	202	9.9
233	203	9.9
291	203	9.0
270	202	11.8
255	199	11.1
Mean value	**201**	**107**
Deviation / relative deviation [%]	**2.0 / 1.0**	**1.7 / 1.6**



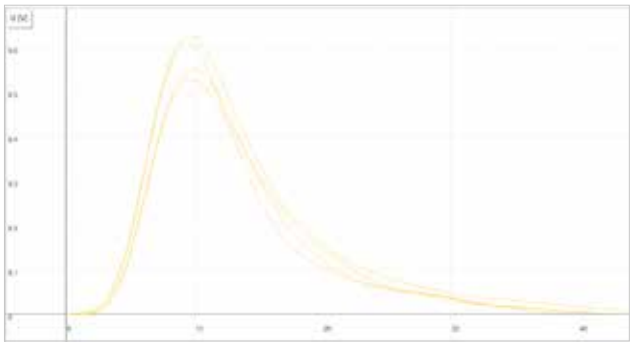
O/N ANALYSIS IN NICKEL POWDER

Analysis	Oxygen and nitrogen with ELEMENTRAC ONH-p 2
Sample	Nickel powder from ASTM Cycle AMPM 2010
Sample preparation	Sample filled in nickel capsule
Settings	Standard steel analysis with 4500 W; 2,5 -3 min. cycle time per sample

RELIABLE HYDROGEN ANALYSIS

Fe/Ni powder (customer sample)	
Weight [mg]	**Hydrogen [ppm]**
399	11.8
400	11.9
402	13.5
399	11.78
Mean value	**12.28**
Deviation / relative deviation [%]	**0.83 / 6.8**

For a precise measurement in the low and high concetration range the ELEMENTRAC ONH-p 2 utilizes a TCD cell with a senstive and robust measuring channel. This enures best repatability from the lower ppm range up to high percentages.



H ANALYSIS IN IRON/NICKEL POWDER

Analysis	Hydrogen analysis with the ELEMENTRAC ONH-p 2
Sample	Iron/ nickel feedstock (customer sample)
Sample preparation	Sample filled in nickel capsule
Settings	Standard H Steel analysis with 3500 W; 2.5-3 cycle time per sample

ELTRA – ELEMENTAL ANALYSIS

ELTRA is one of the leading manufacturers of elemental analyzers for rapid, precise and flexible C/S & O/N/H analysis of solid samples. Our instruments provide reliable results for a huge variety of sample materials and measuring ranges.

ELEMENTAL ANALYZERS

- C|H|S-Analyzers
- O|N|H-Analyzers
- Thermogravimetric analyzers
- Standards and Consumables

ELTRA®
ELEMENTAL ANALYZERS

SOLUTIONS FOR ADDITIVE MANUFACTURING

ELEMENTRAC ONH-p 2

- Inert gas fusion analyzer for O/N/H analysis.
- Easy and fast analysis of pins, powders and granulates
- Optional autocleaner

ELEMENTRAC CS-i

- Simultaneous carbon and sulfur determination with minimum sample preparation
- Induction furnace for temperatures above 2000 °C
- Freely selectable configuration of each IR cell

Find out more at **www.eltra.com**

SUMMARY

C/S and O/N/H analysis via combustion or inert gas fusion analysis are globally established standard-compliant methods which are suitable for metal powder as well as solid sample analysis.

Operation is easy and ELTRA provides a range of application notes for a great variety of sample materials. When further support is needed the ELTRA application laboratory in Haan is ready to process your sample and to provide reliable procedures for a precise measurement.

HEAT TREATMENT
POWDER INJECTION MOULDED & ADDITIVE MANUFACTURED PARTS

Carbolite Gero provides a range of furnaces that are suitable for different stages of the powder injection moulding and additive manufacturing processes for metal and ceramic parts. These furnaces can be used for processes such as thermal or catalytic debinding, drying of parts (e.g. after solvent debinding), stress relieving, and sintering under protective gas, hydrogen, vacuum, or partial pressure.

Additive manufacturing involving metals can be categorized into direct and indirect processes. Carbolite Gero has designed their product lines, including the GPCMA and V-L for direct and the EBO, GLO and HTK for indirect 3D additive manufacturing and powder injection moulding, to meet the highest standards. These two products are just a small part of the additive manufacturing oven & furnace portfolio offered by Carbolite Gero.

STRESS RELIEVING IN DIRECT AM PROCESSES

The direct process involves selectively melting and solidifying the starting powder layer by layer to directly produce a complex three-dimensional part.

When metal powders are melted using a laser (selective laser melting SLM – standard designation: Laser Powder Bed Fusion L-PBF), subsequent heat treatment of manufactured parts is required.

The SLM process is digitally driven, direct from 3D CAD data. For each slice of CAD data a thin even layer of fine sieved metal powder (titanium alloy Ti_6Al_4V, cobalt chromium, stainless steel, nickel alloys Inconel 625 and Inconel 718 and aluminium alloy $AlSi_{10}Mg$) is deposited on the build plate, before the selected areas of the powder are precisely melted by the laser. This precision process is repeated building up, layer by layer, until the finished part is complete.

With SLM it is possible to produce very small parts and features, including geometries that are impossible to machine, such as enclosed spaces.

With layers as thin as 20 microns and tolerances as small as ±50 microns on small features, SLM is a highly precise manufacturing method. At present build rates for parts using a SLM process are relative slow. Costs are also high as raw metallic powders must be produced using a ball-mill/grinder and then sieved and tested prior to usage. Current SLM machinery requires a substantial investment.

Nevertheless, for companies seeking rapid prototyping or small quantities of complex or otherwise "impossible" parts that can later be machine drilled, slotted, milled, reamed, powder coated, painted, polished, or anodized, the process may be ideal if the required part has dimensions up to 250 mm x 250 mm x 350 mm.

Fig.1: ***V-L Furnace*** *for Vacuum and High-Vacuum applications up to 10^{-6} mbar and oxygen levels lower 10 ppm*

Fig. 2: ***GPCMA Modified Atmosphere Furnaces*** *for stress relieving of parts manufactured by SLM at temperatures up to 1150 °C with an oxygen content below 30 ppm.*

Parts manufactured using the direct additive manufacturing method SLM exhibit high residual stresses due to the locally concentrated input of high energy and the formation of a high temperature gradient below the melt pool.

The reduction of the residual stresses requires subsequent heat treatment with precise temperature uniformity. For this purpose, the component is kept at a certain temperature for a specified period of time.

The heat treatment stage must be precisely controlled in order to set the mechanical parameters of the selected metal alloy in a targeted manner by relieving the residual stresses effectively.

In addition, the heat treatment is carried out in an inert atmosphere to ensure the sintered part is not contaminated by oxygen molecules which can alter the chemical and physical properties of the final part.

With the **General-Purpose Chamber furnace with Modified Atmosphere (GPCMA) & Vacuum furnace (V-L),** CARBOLITE GERO offers products for stress relieving of additive manufactured components, which minimizes the daily operating costs, avoids unwanted oxidation, and ensures “best in class” temperature uniformity.

Various sizes are available (GPCMA/37, GPCMA/56, GPCMA/117, GPCMA/174, GPCMA/208 & GPCMA/245) with capacities for between 1 to 4 build plates to fully utilize the chamber volume even with small samples. The V-L range is available in 2 sizes: 180 Ø x 300 mm heigth and 450 Ø x 600 mm height.

The GPCMA range can be optionally specified for compliance to AMS2750G or Nadcap for aerospace applications when used with an

Inconel or Haynes 230 retort (Fig.3). The heat treatment usually occurs in an inert (typically Nitrogen or Argon) atmosphere. Oxygen levels can be reduced to 30 ppm depending on the application and material.

For highly sensitive materials like Titanium and its alloys the vacuum furnace V-L features oxygen levels below 10 ppm. These furnace ranges have 360° heating to improve temperature uniformity inside the retort where temperature thermocouples are located.

The positioning of the Cascade Controls inside the retort enables accurate temperatures at the sample, faster heating times which can substantially reduce customer cycle times when used in conjunction with optional forced cooling. The furnaces have a temperature interlocked double-pivot door facilitating quick, safe, and easy access for loading / unloading with a water-cooled silicon rubber or Viton® seal which maintains, a modified atmosphere or vacuum inside the chamber throughout the entire heat treatment process.

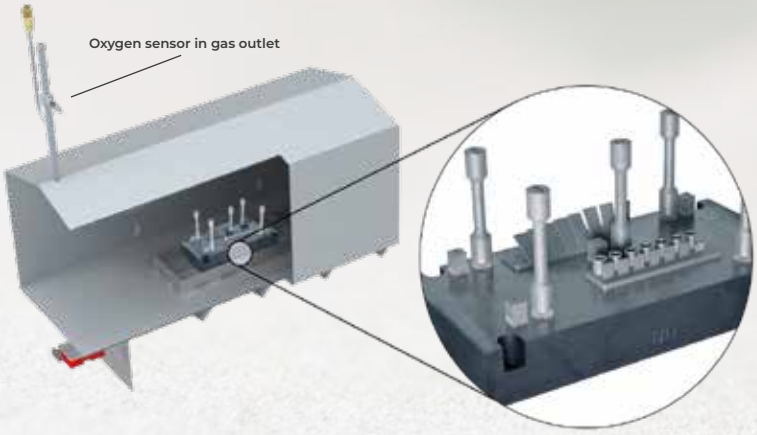


Fig.3: Retort of a GPCMA with SLM printed part. The gas outlet of the retort is equipped with an oxygen sensor to monitor the oxygen levels online. This ensures a great part quality with very little discoloring.

CATALYTIC AND THERMAL DEBINDING IN INDIRECT AM PROCESSES

Binder Jetting, Cold Metal Fusion, and Fused Deposition Modeling have become well established and rising AM technologies providing highest flexibility and versatility for 3D-printed parts.

Organic additives are used in the production of metallic, ceramic, or glass AM components to facilitate shaping and give a certain strength to the printed or pressed green body.

Before sintering, the binder must be removed from the green part. The gaseous reaction products that arise during this thermal debinding may not be able to dissipate quickly enough under certain circumstances, such as with large components or rapid heating rates. This creates excess pressure in the pores that can destroy the component.

Defined temperature profiles, excellent temperature control, and targeted gas guidance are key elements to ensure the best result when transitioning from the green to the brown part. For over 30 years, Carbolite Gero has provided high-quality heat treatment solutions for any questions in the field of additive manufacturing.

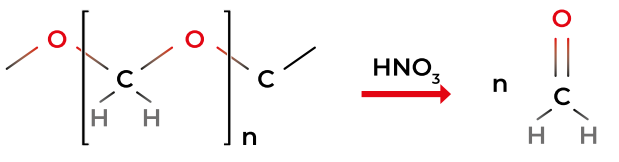
The EBO (Fig.4) was specially developed to meet the strict requirements of catalytic debinding of MIM / AM parts containing POM. This oven is an ideal solution for debinding green parts from Catamold® - made by BASF feedstock at low temperatures.

Nitric acid (HNO_3) is vaporized at 120 °C and introduced into the furnace with the carrier gas nitrogen, where a recirculation fan delivers the acid around the green parts. The nitric acid cracks the main binder, creating formaldehyde (CH_2O) which is gaseous and explosive in concentrations between 7 % - 73 %.

The gas flow directs the formaldehyde towards the furnace gas outlet where it is then safely combusted using an active torch afterburner. For AM technologies with different binder materials the thermal debinding process should guarantee the binder decomposition, safe removal of volatile substances and protection of metallic powder from oxidation. All requirements can be fulfilled by our GLO furnaces (Fig.5). Each furnace is equipped with a gas-tight retort and a continuous gas flow guides the volatiles to the exhaust system. No oxygen gets into the furnace, which means the sample is protected, does not oxidize and no dangerous atmosphere is created.

The main binder removal occurs below 600 °C. However, these furnaces are designed to enable the use of a controlled atmosphere up to 1100 °C, allowing the pre-sintering process to take place as well.

Next to inert gas, hydrogen, and vacuum any desired atmosphere for the debinding process is possible such as partial pressure debinding under nitrogen.



Polyoxymethylene (POM) **Formaldehyde**

*Fig.4: **EBO Oven** for catalytic debinding with nitric acid HNO_3 and equipped safety system*

*Fig.5: **GLO Furnace** for thermal debinding under inert gas, hydrogen, or partial pressure with fully equipped safety system*

BACKBONE DEBINDING AND SINTERING IN PIM AND INDIRECT AM PROCESSES

In the indirect additive manufacturing and the powder injection moulding process, which is suitable for metals and ceramics, the starting powder is mixed with a binder. The binder, which is still present after the shaping of the Green Part, will be removed in a next step thermally, catalytically or with solvents, which leads to a shrinkage of the part. The resulting Brown Part can then be sintered, giving the part its final shape and properties.

First, the main binder will be removed, e.g. thermally. After this process step, the powder is only held together by a backbone binder, which makes the part very sensitive. In a further step, the backbone binder is thermally removed and the part sintered directly in the same furnace. The debinding steps require the removal of the volatile products and a precise temperature distribution in order to specifically adapt the material properties of the sintered part.

Debinding can take place under vacuum, air, inert gas, hydrogen, or partial pressure. The latter ones are often used as carrier gases to improve the gas flow, to “sweep away” the binder offgassing and to shorten the debinding time. The sintering step requires furnaces with specific atmospheres, which are available in the CARBOLITE GERO product portfolio. To avoid oxidation of most metals and non-oxide ceramics, the sintering step is performed under inert gas (Ar or N_2), or reducing gas (H_2 for stainless steel); for high-purity applications, such as titanium sintering, even operation under high vacuum is required. Oxide or nitride-based ceramics such as alumina, zirconia and aluminum nitride can be sintered in air. CARBOLITE GERO’s HTK suits perfectly for backbone debinding and sintering of additive manufactured or powder injection moulded parts.

The high temperature uniformity allows precise debinding and sintering all over the total chamber volume. The possibility to work under inert or reactive gases, high vacuum or even ultra-high vacuum enables sintering of very sensitive materials.

*Fig. 6: **HTK Metallic Chamber Furnace** for debinding and sintering of injection moulded and additive manufactured parts up to 1450 °C.*

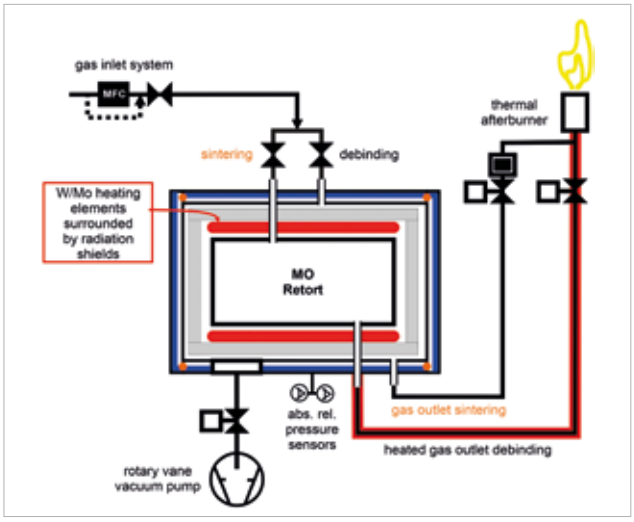


Fig. 7: Schematical drawing of HTK molybdenum with gas guidance for debinding or sintering mode

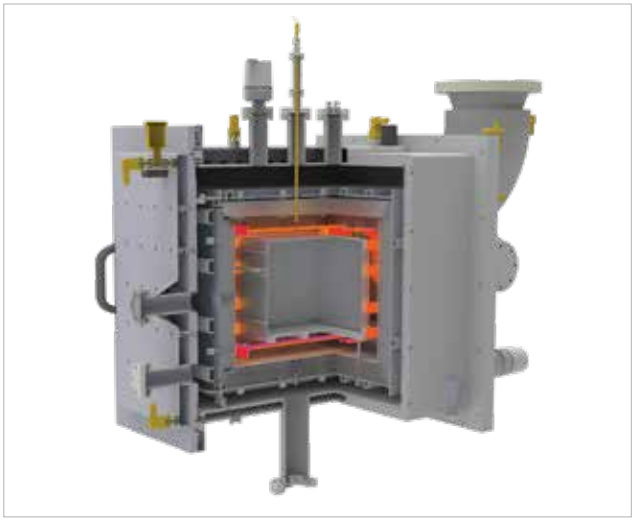


Fig. 8: Cross-section of HTK molybdenum for highest possible purity of atmosphere and very low vacuum levels

The rectangular design with a front door allows for easy loading and unloading of the fragile parts placed on a rack containing shelves. The HTK range is available in various sizes between 8 and 320 liters.

The metallic HTK furnaces constructed of tungsten (HTK W) or molybdenum (HTK MO) permit the greatest possible purity of inert atmosphere and vacuum levels in the high vacuum region (5 x 10^{-6} mbar). Even an ultrahigh vacuum can be requested. Common gases that are typically used include: Nitrogen, Argon (titanium), Hydrogen (stainless steel) or its mixtures.

The heating elements are made from the same metallic material as the insulation. The heating insulation is constructed of several radiation shields made from tungsten or molybdenum with respect to the furnace type selected. With a retort the gas flow can be guided and the temperature uniformity is improved. The maximum temperature for the HTK W is 2200 °C and 1600 °C for the HTK MO.

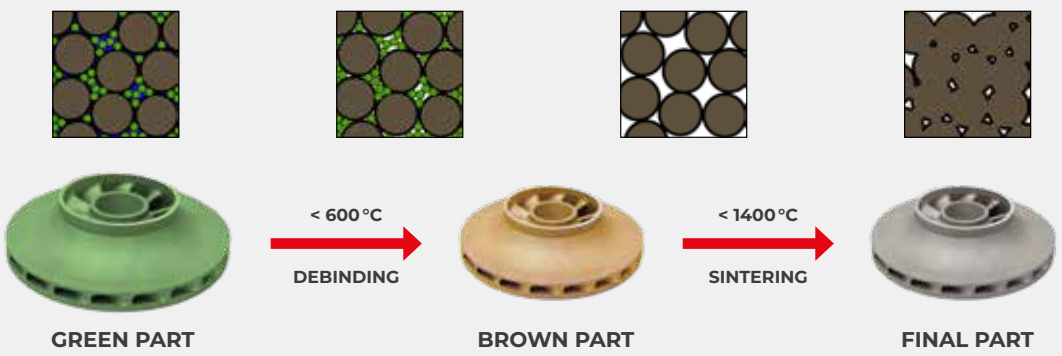
The gaseous waste products generated during debinding are passed through a heated gas outlet and converted in the gas fired afterburner. CARBOLITE GERO enables contamination-free sintering of highly sensitive materials through a switchable gas flow. This is shown in Fig. 7.

During debinding, the gas flows from the top through the right inlet ("debinding") above the retort. Since the retort is not fully sealed and the pressure outside is slightly higher than inside the retort, the gas flows into the retort. By flowing through the retort, the carrier gas takes the gaseous binder into the gas outlet at the bottom of the retort. Those gases are then directed through the heated outlet to the thermal afterburner.

After the debinding step, the gas flow can be changed to provide the purest atmosphere for the parts. The gas flows through the upper left inlet ("sintering") directly into the retort and from there to the outside of the retort, where it passes through the lower right gas outlet into the afterburner. Since there is no more binder present, the outlet no longer needs to be heated.

The improved gas flow prevents binder residues that might be outside the retort from getting back onto the samples during sintering resulting into cleaner samples and higher quality.

The heating elements are positioned 360° around the retort allowing for an improved temperature uniformity. For larger working volumes, the back wall and front door of the furnaces are equipped with heating elements as well to maintain excellent temperature uniformity.



Model	Dimensions: Internal retort H x W x D [mm]	Atmosphere**
Stress relieving up to 1150 °C under inert gas		
GPCMA/37	205 x 337 x 538	Nitrogen, Argon, Forming Gas
GPCMA/56	229 x 400 x 610	
GPCMA/117	279 x 500 x 840	
GPCMA/174	428 x 500 x 815	
GPCMA/208	428 x 500 x 970	
GPCMA/245	650 x 700 x 1050	
Stress relieving up to 1150 °C under vacuum		
V-L 180-300	300 X 125 x 125	High Vacuum, Nitrogen, Argon, Forming Gas
V-L 450-600	600 X 315 x 315	
Thermal debinding and pre-sintering up to 1100 °C		
GLO 8	125 x 125 x 300 (1300°Cversion!)	Vacuum, Nitrogen, Argon, Forming Gas, Hydrogen, Partial Pressure
GLO 40	200 x 200 x 600	
GLO 120	300 x 300 x 700	
GLO 260*	400 x 400 x 800	
Catalytic debinding up to 130 °C		
EBO 120	380 x 400 x 770	Nitrogen, Nitric Acid
EBO 250	500 x 500 x 1000	
Rest-debinding and sintering up to 1600 °C		
HTK 8	170 x 190 x 190	Vacuum, Nitrogen, Argon, Forming Gas, Hydrogen, Partial Pressure
HTK 25	250 x 250 x 410	
HTK 80	400 x 420 x 520	
HTK 120*	400 x 420 x 790	

*Larger sizes on request **other atmospheres on request

Fig. 9: Once the binder-containing parts are 3D-printed, they require debinding either with nitric acid, inert gas, hydrogen, or vacuum means to extract the organic material from the sample. This process results in a shrinkage of the part. Nevertheless, the backbone binder continues to stabilize the structure of the brown part until it is sintered into the final product. The shrinkage can be as much as 20 %, and accurately predicting this transformation is crucial in achieving parts with precise dimensions and high quality.

CARBOLITE GERO 30-3000°C

SOLUTIONS FOR ADDITIVE MANUFACTURING & POWDER INJECTION MOULDING

SAFETY is one of the most important aspects in our daily life. CARBOLITE GERO not only protects its furnaces but also the load and application of our customers.

Many furnaces are equipped with a so-called safety system whereas prior to each run, the furnace checks its health conditions and gas tightness to securely run the chosen program. This is a huge advantage over other products so that failures are minimzed, damages to the load prevented, and money saved in the long term. Our intelligent furnaces are supplied with a PLC observing each step in the process.

By incorporating hydrogen, we achieve SIL2 compliance. Our safety system is TÜV certified, and our products undergo weeks of rigorous testing prior to release and delivery, ensuring consistent, high-quality results.

As your reliable partner in HEAT TREATMENT, we offer solutions for the whole range of materials within the AM & MIM market, both now and in the future.

HTK Metallic Chamber Furnace for Powder Injection Moulding and Additive Manufacturing

- Debinding & Sintering under H_2, Ar, N_2, Partial pressure
- Switchable gas flow for processing of sensitive materials
- Fully automatic control system
- Low energy consumption

Find out more at **www.carbolite-gero.com**

SIEVING AND PULVERIZATION OF METAL POWDERS AND PARTS

Re-using raw materials is an important factor in powder metallurgical processes. RETSCH offers a range of instruments which are suitable for sieving powders and pulverizing metal parts both of which are re-introduced into the production process. The following examples demonstrate the suitability of RETSCH instruments for these applications.

SEPARATION OF SIZE FRACTIONS BY SIEVING TO RECOVER METAL POWDER RESIDUES AFTER 3D PRINTING USING LASER TECHNOLOGY

RETSCH sieve shakers, like the **Vibratory Sieve Shaker AS 200 basic**, are well suited to sieve agglomerated metal powder before it is used for 3D printing, or to separate the unused metal powder after the printing process into fractions with the objective to recover the fine particles for re-use.

Concept Laser, a manufacturer of machines for 3D printing of metal components, uses the AS 200 basic for this purpose. It is the economical model of the AS 200 series with familiar RETSCH quality and reliability. 1 to 17 fractions may be obtained after short sieving times. The shaker features digital setting and display of performance and time ensuring comfortable sieving of ferrous and non-ferrous metals like gold, tungsten carbide, or precious metals.

The most common **test sieves** used for this application are RETSCH test sieves with 200 or 203 mm diameter and a height of 25 mm or 50 mm according to ISO 3310-1 or ASTM E11. Aperture sizes of 32 µm–150 µm are best suited to separate the non-agglomerated metal powder after the printing process for recovery. Very common is the use of the following aperture sizes: 32 µm, 40 µm, 50 µm, 63 µm, 100 µm and 150 µm.

The well-proven RETSCH sieves consist of a high-stability stainless steel frame to ensure reliable sieving results. Paying close attention to mesh-specific requirements, the sieve fabric is precisely joined into the frame and tautened. The individual laser engraving of each RETSCH test sieve provides a clear and accurate labeling with full traceability.

RETSCH – MILLING & SIEVING

RETSCH is the leading solution provider for neutral-to-analysis sample preparation and characterization of solids. Based on a century of experience RETSCH develops size reduction and sieving equipment which is characterized by excellent performance, operating convenience, safety and a long lifetime.

MILLS AND CRUSHERS
- Jaw Crushers
- Rotor Mills
- Cutting & Knife Mills
- Mortar Grinders & Disc Mills
- Ball Mills

SIEVE SHAKERS & TEST SIEVES
- Analytical Sieving Machines
- Test Sieves (ISO, ASTM)

RECYCLING OF GREEN BODIES OR HARD METAL PARTS PRODUCED BY METAL INJECTION MOLDING

Metal Injection Molding is used to produce metal parts of complex geometrical shapes. Metal powders and binders are mixed to a feedstock and injected into a mold using plastic injection molding machines (MIM) to form so-called green parts in the first step, followed by partial removal of the binder to form fragile brown parts, and finally the sintering process to produce stable new metal parts of a defined complex shape. At each stage, intermediate parts with undesired properties may be produced. **These are crushed and pulverized to recover the raw material for re-use.**

Jaw Crushers like RETSCH's **BB 500** pulverize defective green parts, brown parts, or hard metal parts within minutes.

APPLICATION EXAMPLE

10 kg of green parts < 100 mm were crushed in two batches with closed gap (i.e. direct contact between fixed and moving crushing arm) in the Jaw Crusher BB 500 XL. Each batch was pulverized to a final fineness of 85 % < 250 µm after only 1 minute.

Retsch®
MILLING SIEVING ASSISTING

SOLUTIONS FOR ADDITIVE MANUFACTURING

Jaw Crusher BB 500

- Feed material: medium-hard, hard, brittle, tough
- High crushing ratio 50:1
- Continuous gap width setting

Vibratory Sieve Shaker AS 200 basic

- Measuring range*: 20 µm – 25 mm
- Digital display of performance and time
- Suitable for dry and wet sieving

Find out more at **www.retsch.com**

MATERIALOGRAPHIC PREPARATION
OF SPECIMENS PRODUCED BY 3D-PRINTING TECHNOLOGIES

One of the various 3D printing methods is **additive laser powder build-up welding**. This technique is characterized by coating materials in powder form with the help of laser welding. The desired shape of the specific product is formed by following trajectories which are predefined prior to manufacturing. The energy of the laser melts the used metal powder forming a welding bead. The final geometry is given its three-dimensional contour by the overlapping of the welding beads based on the paths of the predefined trajectories. Optimization of the additive laser powder build-up welding focuses on economical processing with high quality and accuracy. Another focus lies on scalability: large scale on the one hand and implementing microstructures less than 100 µm on the other.[1]

The materials used for additive laser powder build-up welding are mainly:

- Light metal
- Nickel super alloys
- Steel
- Intermetallic materials
- Hard materials (carbides)

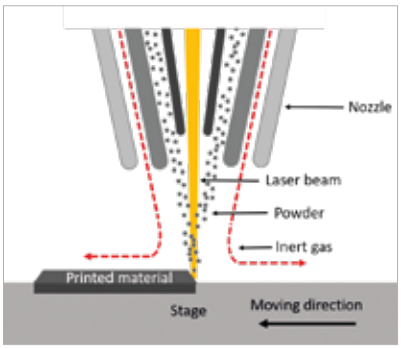
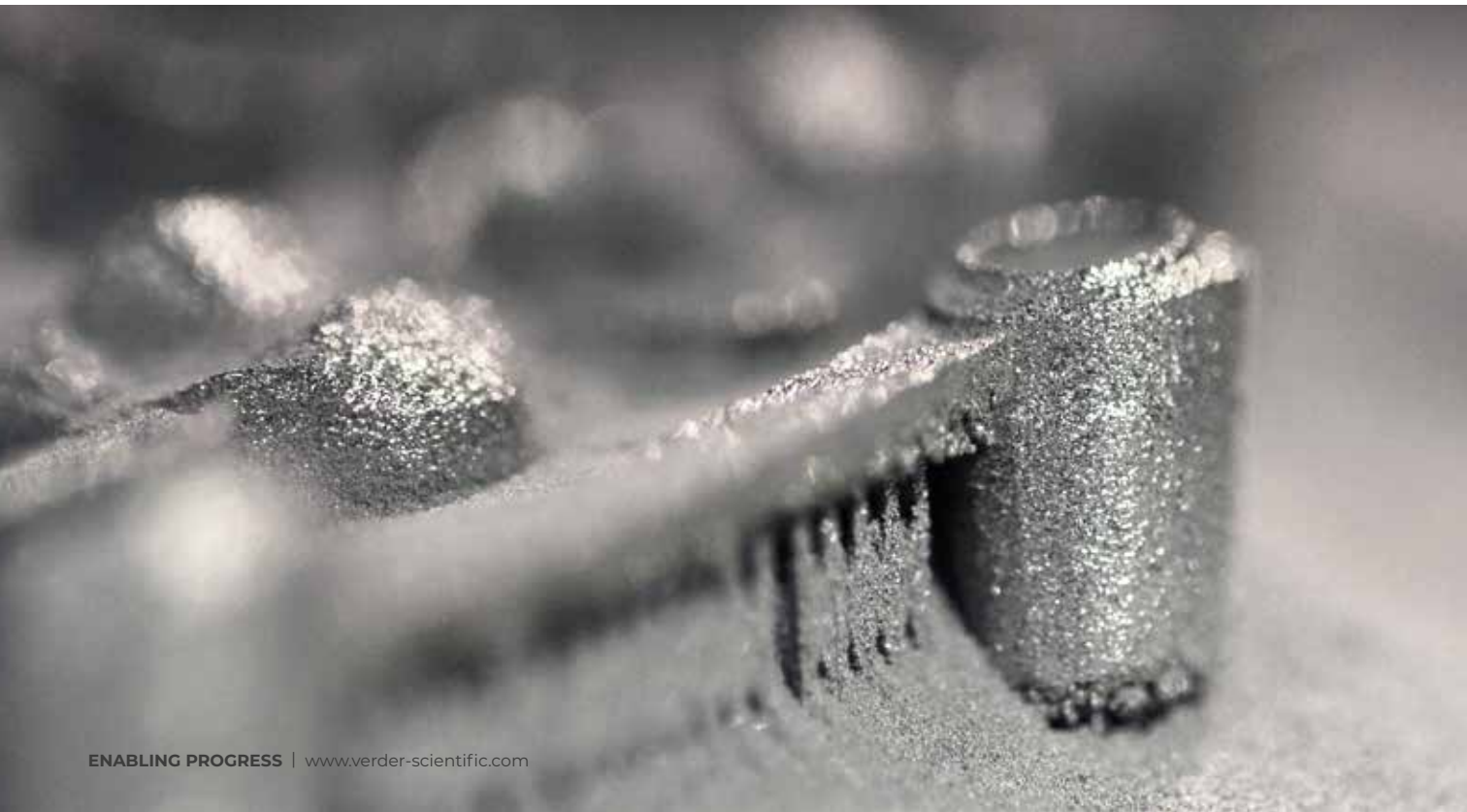


Fig. 1: process of additive laser powder build-up welding.

[1] Fraunhofer IWS, Additive Manufacturing, 2016, www.isam.network



MATERIALOGRAPHIC PREPARATION PROCESS

In the following, we will demonstrate the materialographic preparation process of a sample produced by additive manufacturing. In materialography, a sample taken from a work piece is called specimen.

A typical materialographic examination includes the following steps:

- Sectioning e.g. with an abrasive cutter
- Mounting which offers several advantages for further preparation
- Grinding/polishing for the preparation of the microstructure
- Examination by
  - Image analysis
  - Hardness testing

For this article a steel sample (X6Cr17, material number: 1.4016) manufactured by additive laser powder build-up welding was investigated. The first step was to obtain a smaller sample piece (=specimen) which is representative of the complete workpiece. This was achieved by using **QATM's Qcut 200 A precision cutter** with a thin CBN (cubic boron nitride) blade (wheel thickness: 0.65 mm, wheel diameter: 153 mm) as shown in Fig. 3.

Fig. 2: Cut-Off Machine Qcut 200 A

Fig. 3: Qcut 200 A machine setup. Detail: clamped sample (clamping tool: Qtool 40 S)

The cutting was effected with a pulsed direct cut (0.2 mm forwards and 0.2 mm backwards) with a feed speed of 1 mm/s and a rotational speed of 4500 rpm.

After cutting, the specimen was mounted in a hot mounting material (Epo black) with an **QATM Qpress 50 hot mounting press** to obtain a specimen which is easier to handle. Mounting was carried out at a pressure of 200 bar for 6 minutes at 180 °C, followed by a cooling cycle of 6 minutes. Another advantage is the high degree of parallelism of the mounted specimens of 51 µm ±1 µm (the tolerances are based on the caliper used for height measurements of the specimens). The mounted specimens were ground (individual force) and polished (individual force) afterwards with a **semi-automated grinding and polishing machine, QATM's Qpol 300 A1 ECO+**. The grinding process was divided into two steps.

The first one was plane grinding using a silicon carbide (SiC) grinding paper with grit size P240 to remove all deformations caused by the cutting process. This was followed by grinding with a SiC paper with grit size P600 to smoothen the surface for subsequent polishing steps. First, the specimen was pre-polished with the hard **Galaxy BETA polishing cloth** and 9 µm polycrystalline diamond suspension, followed by a **medium-hard cloth made of silk** (QATM: GAMMA) and 3 µm poly diamond suspension. The last step, called final polishing, was done on a **soft synthetic polishing cloth** (QATM: OMEGA) and Eposil M. The detailed preparation parameters are indicated in Table 1.

Fig. 4: Hot Mounting Press Qpress 50

Fig. 5: Automatic Grinding and Polishing Machine Qpol 300 A1 ECO+

TABLE 1: GRINDING AND POLISHING PARAMETERS

Step	Medium	Lubricant/ suspension	Speed platen [rpm]	direction sample holder	Single load [N]	Time [min]
Grinding	SiC, P240	Water	150	Clockwise	30	1:00
Grinding	SiC P600	Water	150	Clockwise	30	1:00
Polishing	BETA	Alcohol, diamond 9 µm (poly)	150	Counter-clockwise	35	4:30
Polishing	GAMMA	Alcohol, diamond 3 µm (poly)	150	Counter-clockwise	35	4:00
Polishing	OMEGA	Water, Eposil M	100	Clockwise	30	1:30

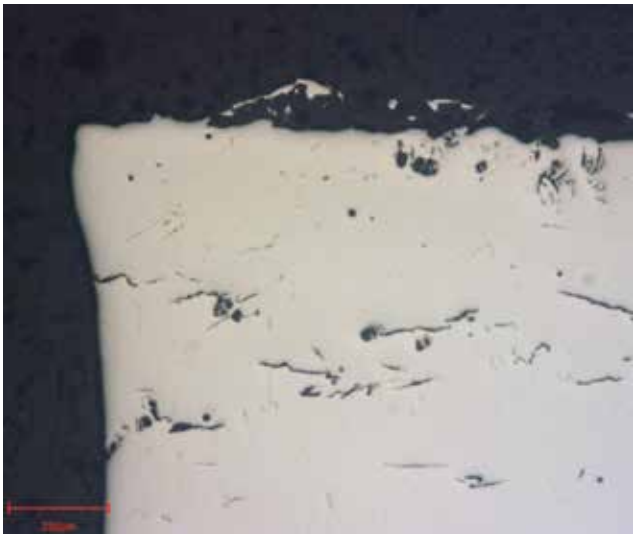


Fig. 6: Image of the prepared specimen surface. Due to the polished surface the light is reflected almost equally and the microstructure is not discernible.

Fig. 7: Etched specimen using "V2A Beize" (for 45 s). Edge section. The microstructure is clearly discernible.

Based on this preparation sequence, a finely polished specimen surface was obtained. Fig. 6 shows an image taken with an incident optical microscope (incident light) at a magnification of 100.

As the light is reflected almost equally over the whole specimen surface, the microstructure remains invisible. Due to the nature of the human eye, a minimum difference in contrast of 10% is needed to make the contrast visible on any surface. This contrasting is achieved by etching. In our example, the etchant "V2A Beize" for pickling was used to contrast the surface by selective etching of the different phases of the investigated X6Cr17 steel. Etching was done for 45 s and the microstructure is very well discernible as can be seen in Fig. 7.

The microstructure was also contrasted well in the middle of the specimen surface indicating that the whole prepared surface was successfully contrasted as shown in Fig. 8.

Further examinations, like **hardness testing**, require a plane and smooth surface to provide reliable and meaningful results. The materialographic preparation process described above ensures that the specimen is ideally suited for hardness testing. QATM offers the **Qness 60 M EVO** for this purpose, a powerful instrument for micro-hardness testing and optical evaluation.

The polished surface in Fig. 6 shows several cracks. The straight edge on the left was achieved by milling. The contour of the welded seams is not visible. For a more detailed examination, the contrast was enhanced by etching. The etched surface is shown in Fig. 7. It has more cracks and the colored spots indicate over-etched areas close to several cracks due to etchant residues. The welded seams, which have different dimensions, are well visible. The layer-by-layer deposition technique effectuates heat treatment of the subjacent layer. A heat affected zone (HAZ) is formed and causes a change in the

Fig. 8: Contrasted specimen. The welded-based microstructure of the manufactured workpiece is clearly visible.

microstructure, affecting the specimen's properties. For example, the hardness may be reduced, resulting in mechanical stress. As layers of different hardness are deposited one on top of the other, the mechanical stress continuously increases and may lead to so-called secondary cracks.

A reason for the formation of primary cracks are cooling gradients during deposition. Fig. 8 shows a magnification of single welding beads and their corresponding heat affected zones. Hardness testing can reveal the differences in hardness of the deposited layers.

HARDNESS TESTING
IN POWDER METALLURGY

Hardness testing in powder metallurgy requires completely different parameters and procedures compared to classic hardness testing applications. Samples have to be prepared well to enable the hardness test. Powder has to be embedded in resin, e. g. with a hot mounting press, and afterwards the materialographic specimen has to be polished to obtain a clean surface for hardness testing.

SELECTIVE LASER MELTING (SLM)

Selective laser melting is used to produce aluminum alloys which have a much higher strength than the pure metal. In SLM, powdered aluminum is deposited in a thin layer (usually between 15 and 500 µm) on a base plate and then melted.

The components produced in this way have a hardness of 115 to 130 HV, which subsequently requires test forces >100 g. For the aluminum powder in our example, a hardness of 125 to 130 HV is to be expected, so that results with test forces >100g (HV0.1) comply with the Vickers DIN EN ISO and ASTM standard (standard requirement: Vickers indentation diagonal >20 µm).

If lower test forces are used on the hardness tester, testing of finer particles is also possible, but not in conformity with the standard.

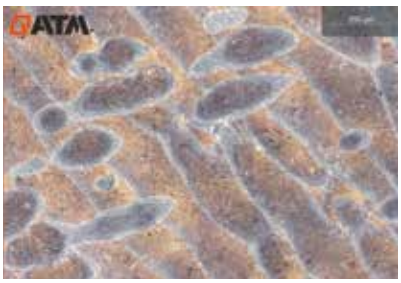


Fig. 1: The microstructure of the additively manufactured aluminium alloy - 5 x

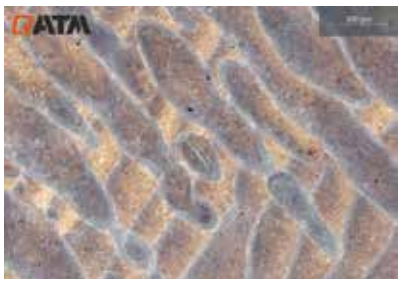


Fig. 2: Thickness of an additively manufactured layer, measured with the hardness testing software - 5x

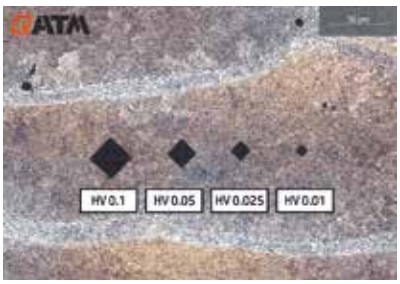


Fig. 3: Comparison of Vickers indentation sizes - 20x

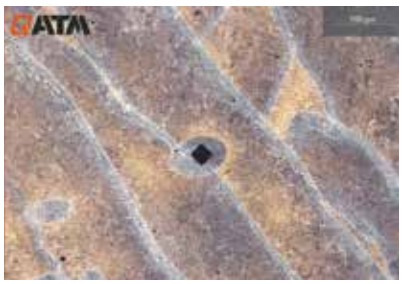


Fig. 4: Hardness: 129 HV0.1, tested in the center of the cross-section - 10x

QATM – HARDNESS TESTING

QATM is focused on the development and manufacturing of innovative high-end products for hardness testing.
In addition to the wide range of versatile standard machines, QATM is also specialized in the planning and realization of customer-specific solutions.

- Micro Hardness Testers
- Rockwell Hardness Testers
- Universal Hardness Testers
- Clamping Fixtures
- Customized Hardness Testers
- Fully Automatic Hardness Testing Plants

REQUIREMENTS FOR HARDNESS TESTERS IN POWDER METALLURGY

- Low Vickers test forces
- High accuracy in slide and turret movement
- Optical measurement system with high contrast at large magnification
- Simple operation
- Structured result management and reporting

CONCLUSION

For proving the quality of powder materials a powerful Vickers micro hardness tester like the QATM Qness 60 is needed. Depending on the amount of tested samples either the simple semi-automatic “M” version or the professional fully automated “A+” models are the perfect choice for powder material applications.

Depending on the test force and the surface preparation, the hardness testers are even able to use the integrated automatic image evaluation next to automatic brightness and focus adjustment. Reporting tool and export functions permit the creation of test protocols or data export to data management systems.

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