

SOLUTIONS FOR ADDITIVE MANUFACTURING & POWDER INJECTION MOLDING

VERDER
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Particle Characterization | Elemental Analysis | Heat Treatment | Sieving | Microstructural Analysis | Hardness Testing

CARBOLITE
IGERO 30-3000°C

ELTRA

Retsch

QATM

MICROTRAC
MEB

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.

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ELECTRON BEAM MELTING

DIRECT METAL DEPOSITION

SELECTIVE LASER MELTING

SELECTIVE LASER SINTERING

LASER BEAM MELTING

POWDER BED FUSION

RAPID PROTOTYPING



Particle size and shape characterization by Dynamic Image Analysis.



Machines for cutting, mounting, polishing and etching for surface preparation as prerequisite for reliable microstructural 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.



Hardness testing of metal components produced by additive manufacturing.



DIRECT METAL LASER SINTERING

SOLID FREEFORM FABRICATION

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 MRB CAMSIZER X2 and SYNC analyzers. The advantages of these instruments are short analysis times, excellent repeatability, and "infinite" resolution as because individual particles are measured and reported. 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.

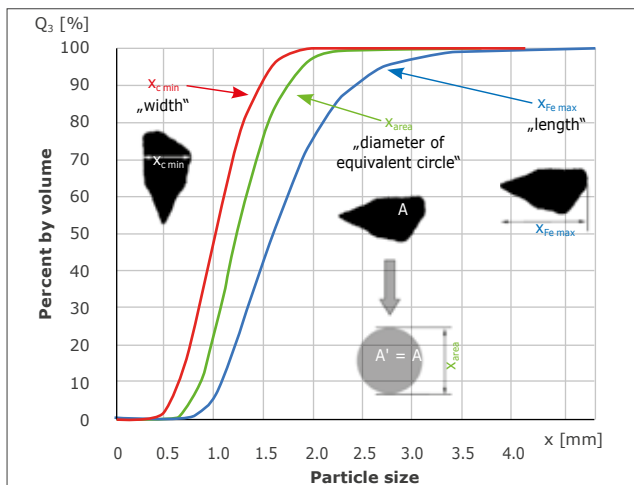


Fig. 1: Selection of CAMSIZER X2 basic size parameters used in image analysis. The size distributions are based on width (red), length (blue) or equal area diameter (green).

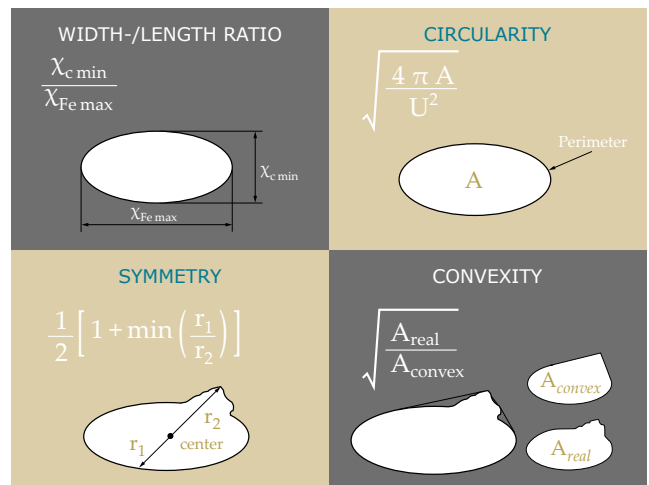


Fig. 2: Selection of basic shape parameters (CAMSIZER X2).



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. 3.

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.

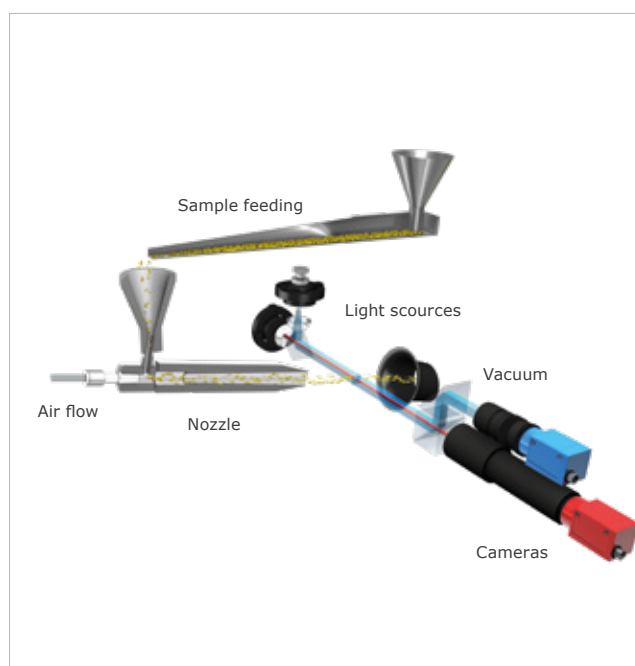


Fig. 3: Unique measurement principle of CAMSIZER X2 for analysis of dry powders.

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. 5 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. 5). 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. 6) shows that the iron powder has the lowest aspect ratio (breadth/length), whereas the titanium powder has the largest share of spherical particles.

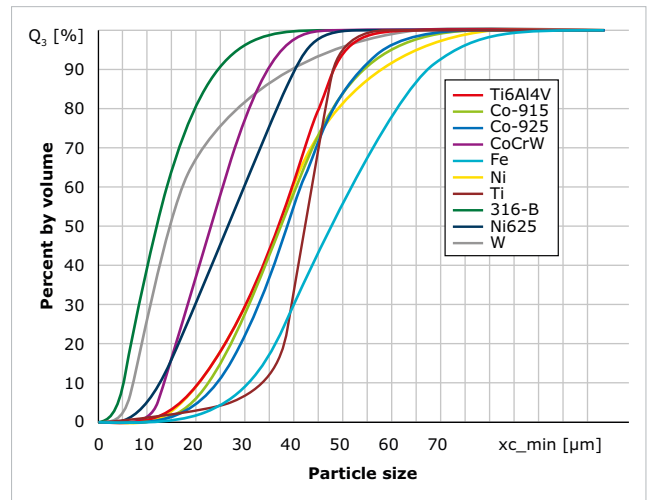


Fig. 5: Particle size analysis of ten different metal powders with the CAMSIZER X2. The direct measurement ensures accurate results.

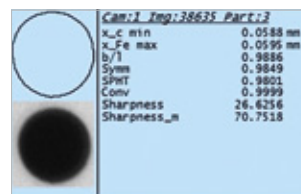
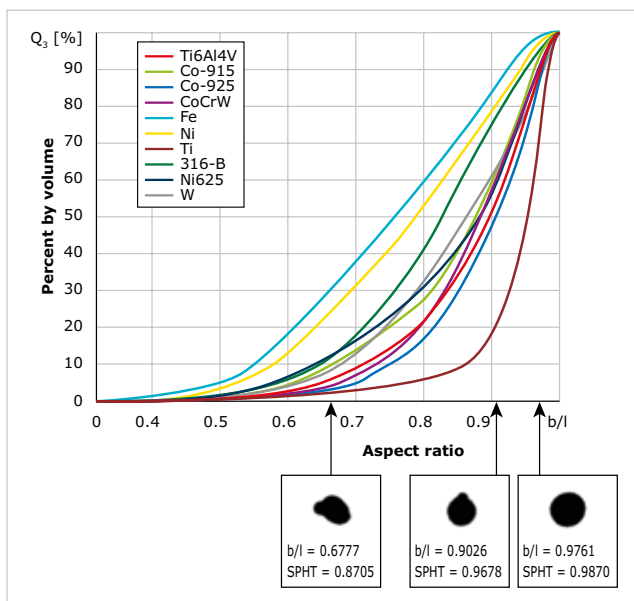
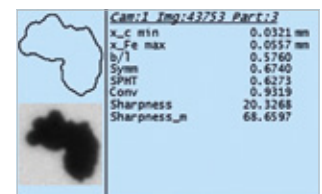


Image of a spherical metal powder particle



Irregular particles are reliably detected

Fig. 6: 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 en-

sure 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. 7. 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. 8 is the LD size distribution report used by the metal powders industries.

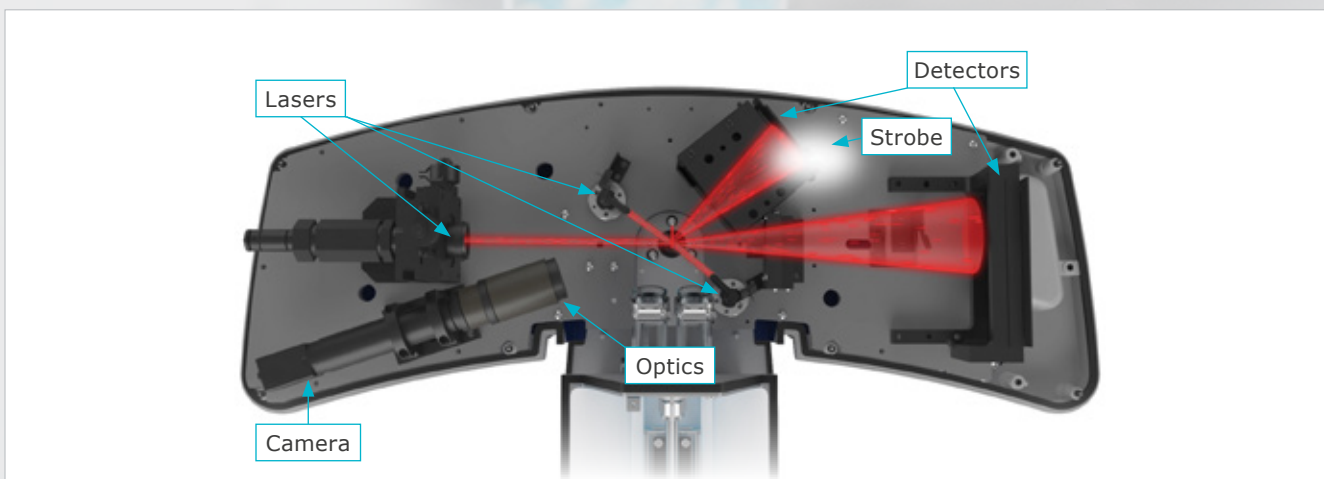


Fig. 7: Layout of the SYNC optical bench, combined LD and DIA components

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.

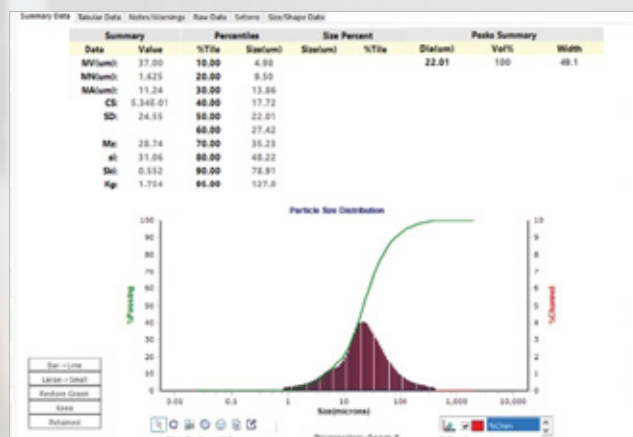


Fig. 8: LD particle size report from SYNC, including percentiles, summary statistics data and frequency / cumulative distribution graphs

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.

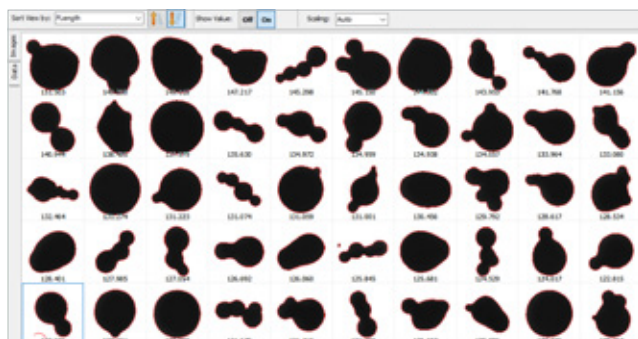


Fig. 6: Analysis of particle shape of 10 different metal powders with Dynamic Image Analysis (CAMSIZER X2). Beside the quantitative results, the recorded images allow an intuitive understanding of morphology and size differences. More spherical particles with higher aspect ratio plot on the right side of the diagram. Detecting the smallest amounts of irregular particles in a large quantity of predominantly spherical particles is a great advantage of DIA.

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.

MICROTRAC MRB – PARTICLE CHARACTERIZATION

CAMSIZERS

Dynamic Image Analyzers

- CAMSIZER P4
- CAMSIZER X2

SYNC

- Laser Diffraction / Dynamic Image Analysis

Measuring Range

- CAMSIZER P4: 20 μm – 30 mm
- CAMSIZER X2: 0.8 μm – 8 mm

- 0.01 μm – 4,000 μm

Find out more at www.microtrac.com

Comparison & Conclusion

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	+++	-	++

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.



MICROTRAC MRB SOLUTIONS FOR ADDITIVE MANUFACTURING



CAMSIZER X2

- Particle size and particle shape analysis from 0.8 μm to 8 mm with Dynamic Image Analysis (ISO 13322-2)
- Precise analysis of wide size distributions
- Excellent resolution of narrow or multimodal size distributions
- Reliable detection of smallest amounts of undersize and oversize
- Measurement results are 100% compatible to sieve analysis if required

www.microtrac.com

ELEMENTAL ANALYSIS

OF METAL POWDERS AND METAL PARTS PRODUCED BY ADDITIVE MANUFACTURING

Additive manufacturing is becoming an increasingly established production technology. However, as it is still new, the required process steps have not been uniformly defined yet. There are, for example, no industry-wide standards describing the quality control process. An established parameter is the particle shape of the powder used for AM. Particle size, however, should not be the only characteristic used for quality control.

Among the metal powders used for additive manufacturing are different types of Steel, Ti64, Al, Ni, Cr, W, as well as of alloys. To check the quality and purity of these raw materials, suitable processes need to be implemented. The content of various "foreign" elements, for example, should be closely monitored to ensure a high-quality end product.¹



¹ Berumen, S.; Bechmann, F.; et al, Quality Control of laser and powder bed-based Additive Manufacturing (AM) technologies, Physics procedia, 5, 617-622, LANE 2010



Elements which have an influence on the material properties

The determination of the element concentrations described below should be carried out before and after the additive manufacturing process to ensure that both the raw materials and the final product possess the required quality.

Titanium

The quality of titanium and its alloys e.g. Ti-6Al-4V (Grade 5) is influenced by these elements:

Hydrogen [H]

Has the same effect on titanium as on steel. Hydrogen may influence the formation of mixed phases in titanium alloys.

Nitrogen [N]

Nitrogen increases the brittleness of titanium.

Oxygen [O]

Even smallest amounts of oxygen have a considerable effect on the toughness or hardness of titanium. The Specification Book shows that even minor differences in the oxygen content may determine the difference between high-quality (grade 1: 0.18% O) and low-quality titanium (grade 3: 0.35%). Oxygen changes the mechanical and physical properties of titanium significantly. Titanium with an oxygen concentration of 0.1% is approximately 3 times more stable than with a concentration of 0.3%.

Sulfur [S] / Carbon [C]

These elements only have a very slight effect on titanium.

Steel

There are many elements which influence the properties of steel with carbon at the top of the list. Steel is classified into different quality grades and application fields, depending on the type and concentration of these alloy elements (C, Si, Mn, P, S, Cr etc.). In the following the most important non-metallic elements and their effects are described.

Carbon [C]: The carbon content affects various physical parameters of steel. This ferrous alloy contains between 0.0002% and 2.06% of carbon. The higher the carbon content, the lower the melting point. Moreover, brittleness and hardness increase with the carbon content.

Sulfur [S]: If the alloy contains sulfur, this increases the machinability of the steel, i.e. the material's suitability for being treated by methods like drilling or milling. The higher the sulfur content, the lower the ductility.

Nitrogen [N]: The nitrogen content may be divided into desired and undesired. There are some special applications which permit a high nitrogen concentration. In these cases its chemical form has to be taken into account. Nitrogen in its elemental form is localized along the grain boundaries

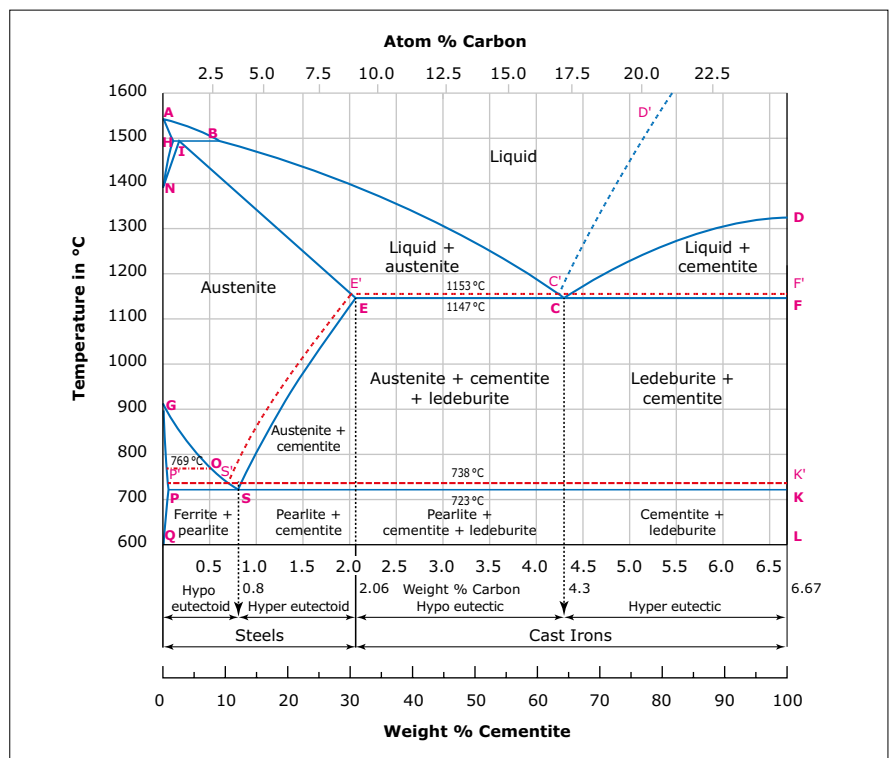


Fig. 1: Iron-carbon phase diagram

and influences the ductility of steel in a significant way. The nitrogen content which is bound to other elements is usually not considered important.

Oxygen [O]: Oxygen is a so-called steel parasite because it makes the steel brittle and causes ageing brittleness.

Hydrogen [H]: Hydrogen in steel makes the mechanical stability degrade. Hydrogen embrittlement is widely feared because it may cause considerable technical and economic damages. It means that the protons attach themselves to the metal matrix which may lead to cracks in the steel.

Combustion Analysis

There are different ways of measuring element concentrations and impurities, most of which require destruction of the sample. This is done to ensure that all relevant components of the analyzed sample are released.

Combustion analysis offers a number of advantages. The samples can be measured in solid form which means direct measurement without previous treatment. The average particle size required for metal powders used for additive manufacturing processes lies between 5 μm and 150 μm .

This is determined by particle size analysis, e.g. by Dynamic Image Analysis. If the powder has the right size distribution it can be analyzed for elemental concentrations by combustion analysis.

The measurement of H/C/N/O/S cannot be carried out in one single analysis. Oxygen, nitrogen and hydrogen are analyzed in one step and carbon and sulfur in another. This is because of the physical and chemical properties due to the elements to be analyzed.

O/N/H Analysis

In ELTRA's ELEMENTRAC ONH-p analyzer the sample is dropped into a graphite crucible and melts due to a defined high temperature. Consequently, oxygen, nitrogen and hydrogen are released. Oxygen converts to CO on the surface of the hot crucible. The inert carrier gas removes the gases from the crucible.

A copper oxide catalyst converts CO to CO_2 which is detected in the infrared cells (Fig. 2). An infrared ray with a specific wave length is used to excite the carbon dioxide molecules. The loss of energy, which was transferred to kinetic energy, is used to determine the exact oxygen concentration of the sample. The nitrogen and hydrogen content are measured in a thermal conductivity cell (Fig. 3).

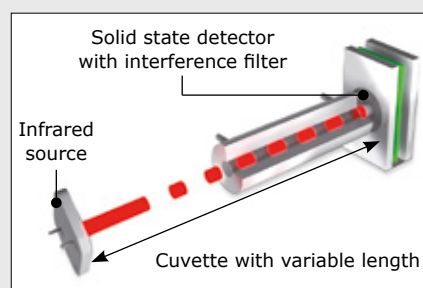


Fig. 2: Infrared cell

heated chamber,
60°C

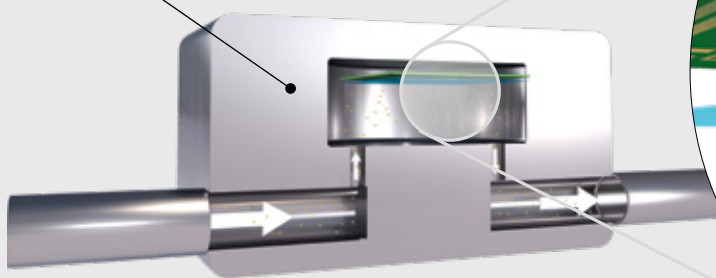


Fig. 3: Thermal conductivity cell

The ELEMENTRAC thermal conductivity cell is based on a micromechanical silicon chip which is coupled to a membrane and works independently of a reference gas flow. If the thermal conductivity of the gas changes, for example through nitrogen released from the sample, the

heating capacity required for heating the membrane changes as well. This is indicated by a measuring signal. The method is robust and sensitive and it guarantees stable measuring results over a wide concentration range.

ELTRA 91205-1003#1116B ¹		
Weight [mg]	Oxygen [ppm]	Nitrogen [ppm]
102.7	893.2	100.8
103.4	917.2	101.8
102.7	892.0	105.1
101.9	878.9	98.4
103.5	886.7	93.9
103.2	904.5	97.8
102.3	908.8	96.5
103.8	882.9	103.3
103.4	860.7	99.7
103.4	877.8	94.0
Mean value	890.3	99.1
Deviation / relative deviation	16.7 / 1.9 %	3.7 / 3.8 %

¹ certified values: O: 890 ppm ± 50 ppm; N: 99 ppm ± 10 ppm

Table 1 shows typical results for a simultaneous oxygen and nitrogen analysis of a titanium sample.



C/S Analysis

In the induction furnace of the ELEMENTRAC CS-i analyzer the sample is melted in a pure oxygen atmosphere, causing sulfur to react to sulfur dioxide (SO₂) and carbon to react to a mixture of carbon monoxide (CO) and carbon dioxide (CO₂).

The combustion gases pass through a dust filter and moisture absorber for purification. In the next step the sulfur dioxide is detected in infrared cells. In ELTRA's CS-i infrared cells with different sensitivities (high/low) can be adapted according to

the user's requirements. Oxidation of both, carbon monoxide to carbon dioxide and sulfur dioxide to sulfur trioxide follow the sulfur measurement. The SO₃ gas is removed with cellulose wool; the carbon content is detected by infrared cells which can be individually customized. ELTRA analyzers can be equipped with up to 4 independent infrared cells.

Conclusion

Non-metallic elements like carbon, sulfur, hydrogen, oxygen, and nitrogen influence the physical properties of metallic materials. These elements may be found in the powdered raw materials used for additive manufacturing, or may be introduced during the production process. Therefore, thorough quality control should always comprise analysis of the raw material and the final product. Combustion analysis offers convenient and reliable solutions to reproducibly measure element concentrations in a range from a few ppm to percentages.

ELTRA – ELEMENTAL ANALYSIS

ELTRA is one of the leading manufacturers of combustion analyzers for rapid, precise and flexible CHNOS 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
- Ash fusibility and biomass testing
- Standards and Consumables

Find out more at www.eltra.com



ELTRA SOLUTIONS FOR ADDITIVE MANUFACTURING



ELEMENTRAC CS-i

- Simultaneous carbon and sulfur determination with minimum sample preparation
- Induction furnace for temperatures above 2,000 °C
- Freely selectable configuration of each IR cell



ELEMENTRAC CS-i

- Simultaneous carbon and sulfur determination with minimum sample preparation
- Induction furnace for temperatures above 2,000 °C
- Freely selectable configuration of each IR cell

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AR 875 (LOT 1216F) ¹		
Weight [mg]	Carbon [%]	Sulfur [%]
1003.4	0.8005	0.0128
1001.9	0.8003	0.0125
1002.6	0.8012	0.0126
1003.2	0.8007	0.0126
1001.8	0.7971	0.0125
1004.2	0.7952	0.0125
1003.6	0.7962	0.0124
1003.1	0.7976	0.0123
1003.2	0.8020	0.0124
1002.9	0.8024	0.0123
Average values	0.7993	0.0125
Deviation / relative deviation	0.0026 / 0.32 %	0.0002 / 1.20 %

¹ certified value: C: 0.799 % ± 0.017, S: 0.0125 % ± 0.0034

Table 2 shows a typical result for a steel sample.



HEAT TREATING POWDER INJECTION MOLDED & ADDITIVE MANUFACTURED PARTS

CARBOLITE GERO offers suitable furnaces for the various process steps in powder injection molding and additive manufacturing of metal and ceramic parts, such as thermal or catalytic debinding, drying of parts e.g. after solvent debinding, stress relieving, as well as sintering under protective gas, hydrogen or vacuum.

Additive Manufacturing (AM) involving metals can be divided into direct and indirect processes. CARBOLITE GERO has purposely designed their product ranges to the highest specifications; with the GPCMA for direct, and the HTK for indirect 3D Additive Manufacturing and Powder Injection Molding (PIM) processes. These are just two products from the comprehensive additive manufacturing portfolio offered by CARBOLITE GERO.

Stress relieving in direct AM processes

In the direct process, the starting powder is selectively melted and solidified on top of each other so that the complex three-dimensional part is directly produced layer by layer.

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 Ti6Al4V, cobalt chromium, stainless steel, nickel alloys Inconel 625 and Inconel 718 and aluminium alloy AlSi10Mg) 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.

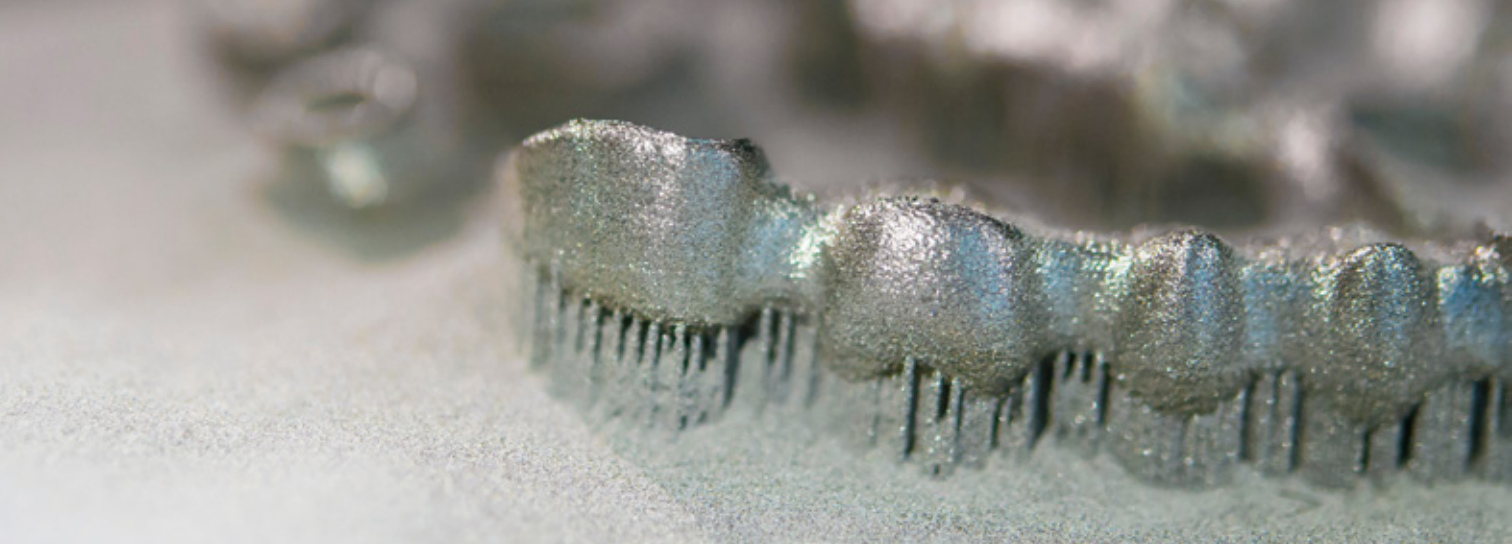
SLM can be used for very small parts and features. It can reproduce geometries that would otherwise be impossible to machine such as enclosed spaces. Layers can be as thin as 20 microns and tolerances on small features can be as small as ± 50 microns.

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.

However, if the required part has dimensions up to 250 mm x 250 mm x 350 mm the process could well be perfect for organisations who require rapid prototyping or small quantities of complex or 'impossible' parts that can subsequently be machine drilled, slotted, milled, reamed, powder coated, painted, polished or anodised.



Fig. 1: GPCMA Modified Atmosphere Furnaces for stress relieving of parts manufactured by SLM at temperatures up to 1200°C with a possible oxygen content < 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 Modified Atmosphere (GPCMA)**, CARBOLITE GERO offers a product 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 and 4 build plates to fully utilize the chamber volume even with small sample sizes. This range of furnaces can be optionally specified for compliance to AMS2750E Nadcap class 1 for aerospace applications when used with an Inconel or Haynes 230 retort.

The heat treatment stage occurs in an inert (typically Nitrogen and Argon [for Titanium]) atmosphere. Oxygen levels can be reduced to 30 ppm depending on the application.

The GPCMA range has under hearth heating combined with heat from the top and sides to improve temperature uniformity inside the retort where temperature thermocouples are located. The positioning of the Cascade Controls inside the retort enables faster heating times which can substantially reduce customer cycle times when used in conjunction with optional forced cooling.

To further shorten cycle times, the GPCMA/174 furnace has a temperature interlocked double-pivot door facilitating quick, safe and easy access for loading / unloading with a water-cooled silicon rubber door seal which maintains, a modified atmosphere inside the chamber throughout the entire heat treatment process.

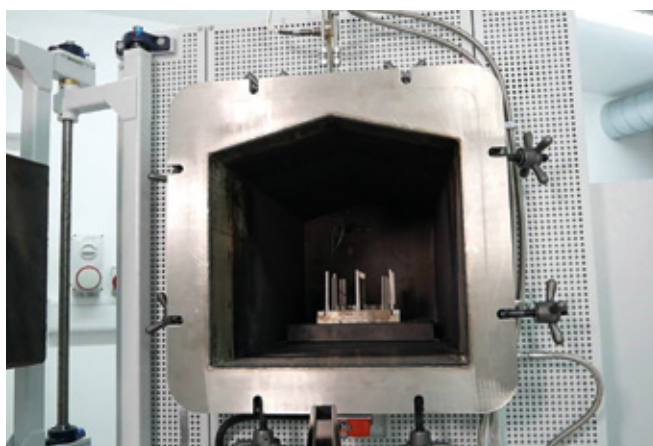


Fig. 2: View into metallic GPCMA/174 retort with additive manufactured sample for stress relieving contained therein.



Fig. 3: HTK Metallic Chamber Furnace for debinding and sintering of injection molded and additive manufactured parts up to 1450°C.

Backbone debinding and sintering in PIM and indirect AM processes

In the indirect additive manufacturing process and the powder injection molding 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 then thermally removed and the part sintered directly in the same furnace. The debinding steps require the removal of the gaseous waste 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 or inert gas. 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 N₂), or reducing gas (H₂ 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 is perfectly suited for backbone debinding and sintering of additive manufactured or powder injection molded 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.

The rectangular design with a front door allows for easy loading and unloading of the fragile parts that only contain the backbone binder – main binder was removed before. The HTK range is available in four different sizes, 8 litres, 25 litres, 80 litres and 200 litres.

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

The gaseous waste products generated during debinding are passed through a heated gas outlet and burnt in the afterburner. CARBOLITE GERO enables contamination-free sintering of highly sensitive materials through a switchable gas flow. This can be seen in Fig. 4.

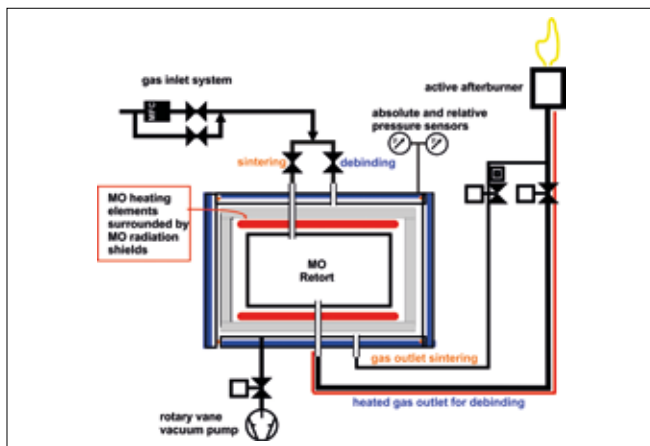


Fig. 4: Gas guidance during debinding or sintering through the retort.

During debinding, the gas flows from the top through the right inlet behind the retort. Since this 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 with it into the gas outlet at the bottom of the retort. Those gases are then directed through the heated outlet to the afterburner.

After the debinding step, the gas flow can be changed to provide the purest retort atmosphere. The gas now flows through the upper left inlet 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. Due to the lack of gaseous binder parts, the outlet no longer needs to be heated.

This changed gas flow prevents binder residues that might be outside the retort from getting back onto the samples during sintering resulting into clean samples.

Inside the chamber, heating elements are positioned at the bottom, left, right, and top sides of the furnace chamber allowing for improved temperature uniformity. For larger volumes, the back wall and front are equipped with heating elements to maintain excellent temperature uniformity. The HTK furnaces are surrounded by a water cooled vessel; thus classifying, the HTK systems as a cold wall furnace. The cooling water is guided through the double walled vessel.

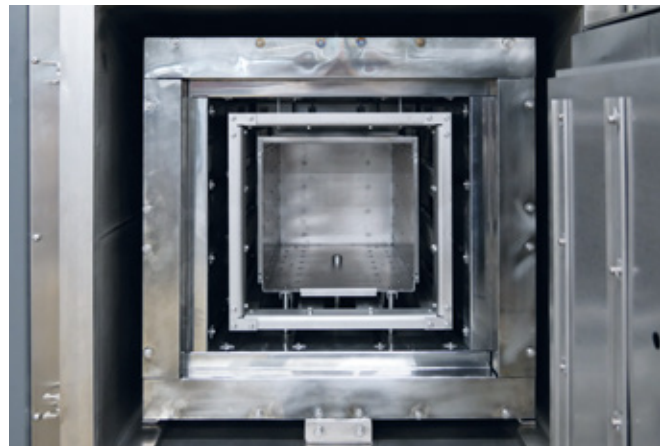


Fig. 5: Mo retort of the HTK for highest possible purity of atmosphere and vacuum level.

CARBOLITE GERO – HEAT TREATMENT

CARBOLITE GERO is a leading manufacturer of high temperature furnaces and ovens from 30°C to 3000°C with a focus on vacuum and special atmosphere technology. With more than 80 years of experience in thermal engineering our products are used in research laboratories, pilot plants and manufacturing sites worldwide.

- Ovens
- Chamber Furnaces
- Tube Furnaces
- Vacuum Furnaces
- Special Applications
- Custom Designed

Find out more at www.carbolite-gero.com

Conclusion

With the GPCMA, CARBOLITE GERO offers a product for stress relieving of additive manufactured parts, which minimizes the daily operating costs for our customers, avoids unwanted oxidation and ensures "best in class" temperature uniformity. Most importantly, production cycle times are minimized thanks to heating on all sides, optional forced cooling and simple loading & unloading through the unique water-cooled, silicon sealed double-pivot door.

CARBOLITE GERO's HTK is perfectly suited for backbone debinding and sintering of powder injection molded or additive manufactured parts. The high temperature uniformity allows precise debinding and sintering all over the total chamber volume. The greatest possible purity of inert atmospheres, final vacuum level in the high vacuum region and even the possibility of ultra-high vacuum enables sintering of very sensitive materials such as titanium.

On request, CARBOLITE GERO offers customer trials to validate a heat treatment process for their additive manufactured parts.

Model	Dimensions: Internal retort H x W x D [mm]
GPCMA/37	205 x 337 x 538
GPCMA/56	229 x 400x 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
HTK 8	190 x 170 x 200
HTK 25	250 x 250 x 400
HTK 80	400 x 400 x 500

CARBOLITE GERO SOLUTIONS FOR ADDITIVE MANUFACTURING & POWDER INJECTION MOLDING



GPCMA Modified Atmosphere Furnace for Additive Manufacturing

- Stress relieving under N₂, Ar
- O₂ level below 30 ppm
- Precise temperature uniformity



HTK Metallic Chamber Furnace for Powder Injection Molding and Additive Manufacturing

- Debinding and Sintering under H₂, Ar, N₂
- Switchable gas flow for processing of sensitive materials
- Fully automatic control system

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)

Find out more at www.retsch.com

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 XL** pulverize defective green parts, brown parts, or hard metal parts within minutes.



Application example:

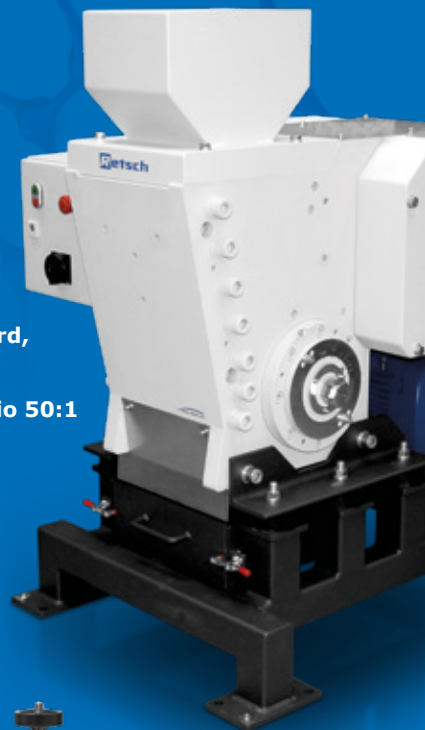
10 kg of green parts < 100 mm were crushed in two batches with closed gap (i. e. direct contact between fixed and moving crushing arm) in the Jaw Crusher BB 500 XL. Each batch was pulverized to a final fineness of 85% < 250 µm after only 1 minute.



RETSCH SOLUTIONS FOR ADDITIVE MANUFACTURING

Jaw Crusher BB 500 XL

- 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

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 pow-

der 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)

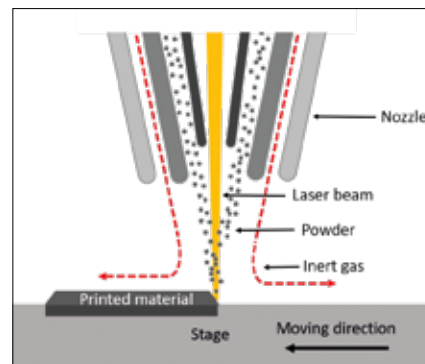
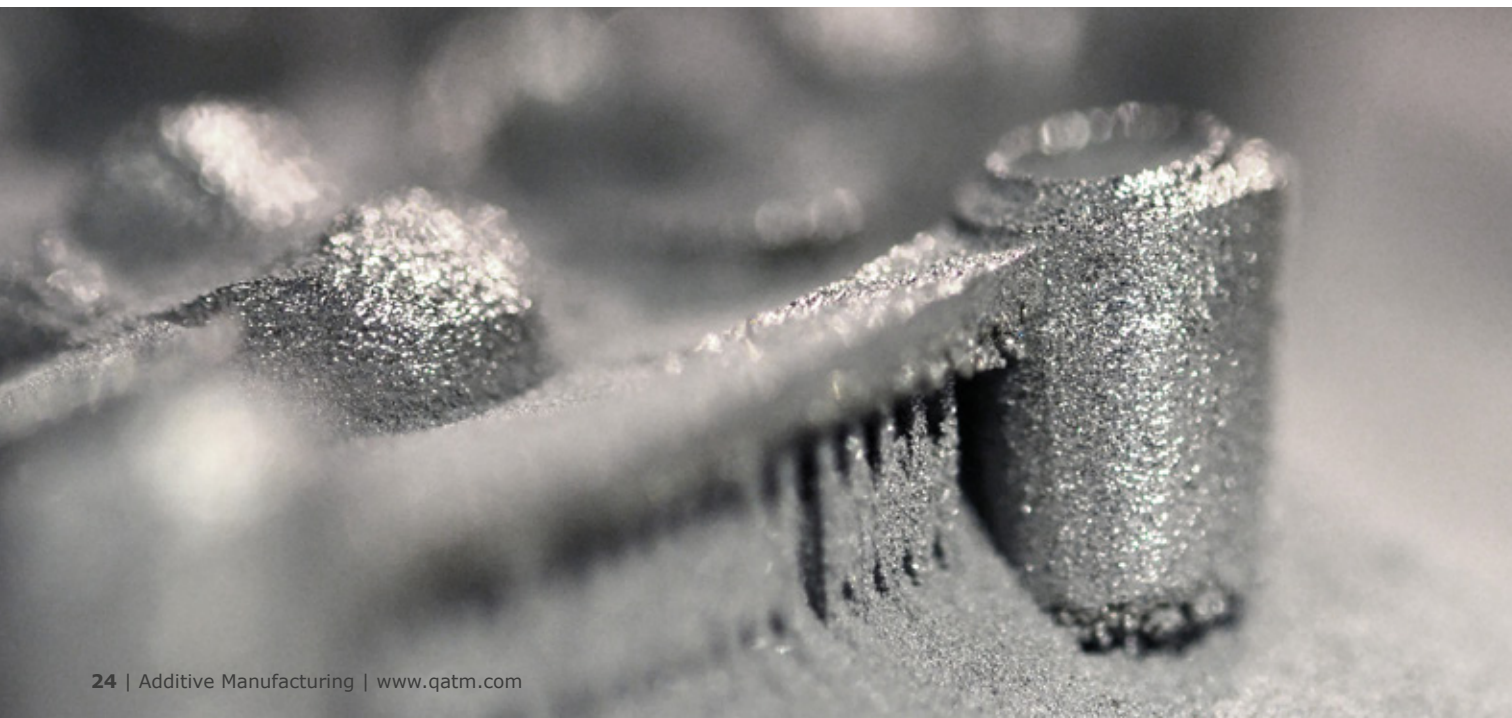


Fig. 1: process of additive laser powder build-up welding.

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



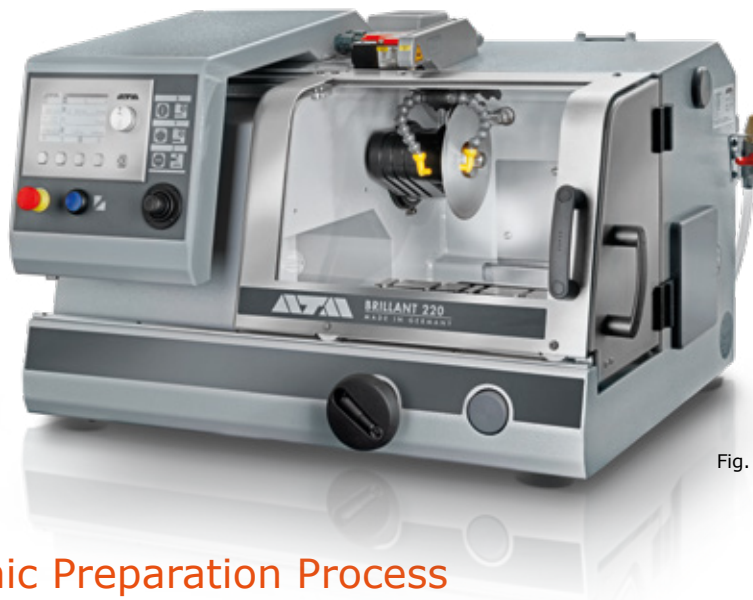


Fig. 2: Cut-Off Machine Brilliant 220

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 Brilliant 220 precision cutter** with a thin CBN (cubic boron nitride) blade (wheel thickness: 0.65 mm, wheel diameter: 153 mm) as shown in Fig. 3.

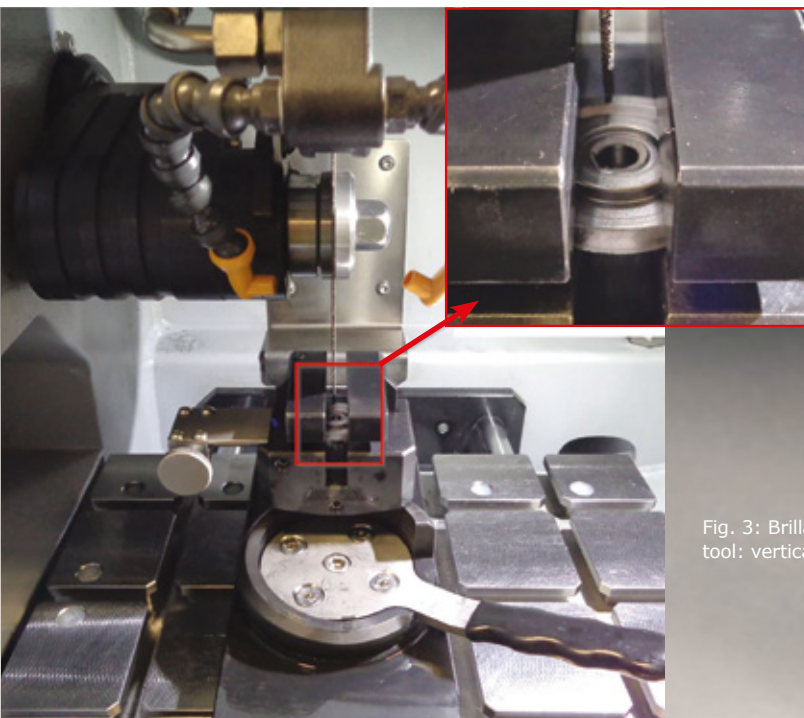


Fig. 3: Brilliant 220 machine setup. Detail: clamped sample (clamping tool: vertical vice single).

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 Opal X-Press 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 $51\ \mu\text{m} \pm 1\ \mu\text{m}$ (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 Saphir 550**. 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 Opal X-Press



Fig. 5: Automatic Grinding and Polishing Machine Saphir 550



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



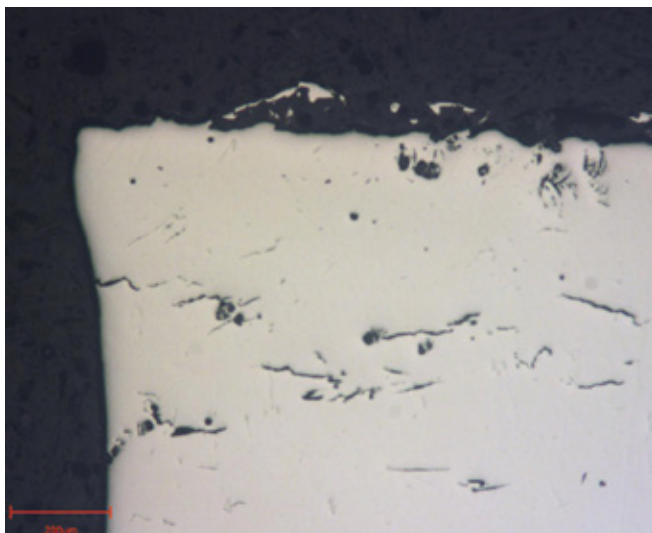


Fig. 6: Image of the prepared specimen surface. Due to the polished surface the light is reflected almost equally and the microstructure is not discernible.

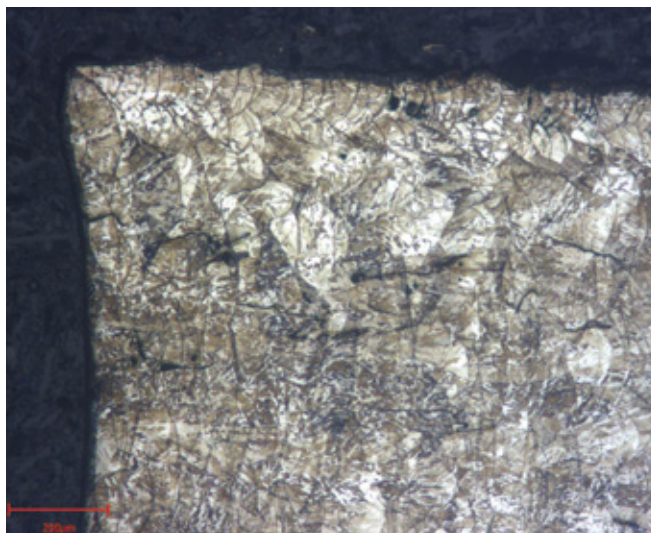


Fig. 7: Etched specimen using "V2A Beize" (for 45 s). Edge section. The microstructure is clearly discernible.

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 **Carat 930** 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

QATM – ADVANCED MATERIALOGRAPHY

QATM is a technology leader in the development and construction of machines for materialography (metallography). QATM equipment is successfully used in areas like quality control, damage analysis, production control as well as research and development.

- Wet Abrasive Cut-off Machines
- Hot Mounting Presses
- Grinders, Polishers, Etchers
- Microscopes
- System Laboratories
- Consumables

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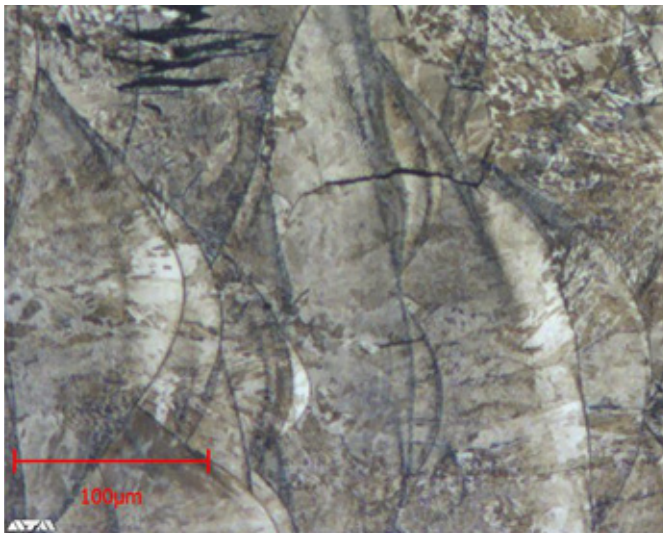


Fig. 8: Contrasted specimen. The welded-based microstructure of the manufactured workpiece is clearly visible.

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 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.



QATM SOLUTIONS FOR ADDITIVE MANUFACTURING



Cut-Off Machine Brilliant 220

- Precision cutting machine
- Spacious cutting chamber with large table
- Openings on left and right for continuous long parts



Hot Mounting Press Opal X-Press

- Easy to handle closure system
- Fully automatic, electronic controlled
- Simple operation via large LC display and optimized user interface



Automatic Grinding and Polishing Machine Saphir 520

- Single wheel grinder/polisher with Rubin 520
- Single and central pressure
- Variable speed of working wheel and polishing head

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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.

Test procedure and test methods

Non-ferrous material is usually tested with Brinell or Vickers test methods – depending on the work piece and application with test forces between 2.5 and 1000 kg. The requirements for testing of powder materials are very different: the small particle size (<0,1 mm particle size) requires very low test forces and small indent diagonals which are possible with Vickers test methods only. For the Aluminum powder in

our example we expect an estimated hardness of 25 to 35 HV which means results of test forces higher than 15 g (HV0.015) may already correspond to Vickers DIN EN ISO and ASTM standards (standard requirement: Vickers indent diagonal >20 µm). If the hardness tester is able to execute even lower test forces, the testing is also possible on smaller particles (but not according to standard).

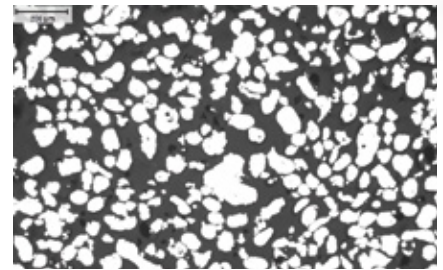


Fig. 1: Embedded Aluminum powder / polished surface / 4x microscope lens
Powder particles polished down to half of the particle size or big particles are suited best for hardness testing with meaningful results.

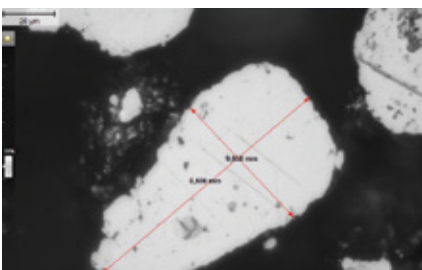


Fig. 2: Size of an aluminum powder particle measured in hardness testing software (40x lens)

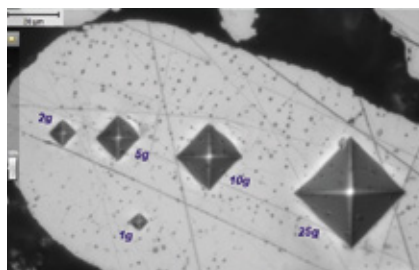


Fig. 3: Comparison of Vickers indent sizes: HV0.001, HV0.002, HV0.005, HV0.01 and HV0.025 – Test forces between 1 g and 25 g

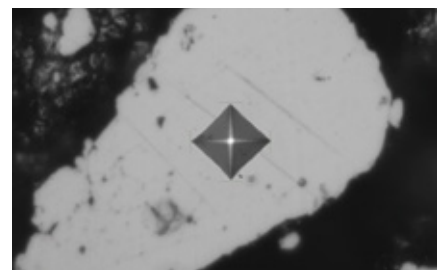


Fig. 4: Hardness result: 30.3 HV0.005 tested in the middle of the cross section of the aluminum particle

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

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QATM SOLUTIONS FOR ADDITIVE MANUFACTURING

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 Q10/30/60 is needed. Depending on the amount of tested samples either the simple semi-automatic "M" version or the professional fully automated "A" or "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.

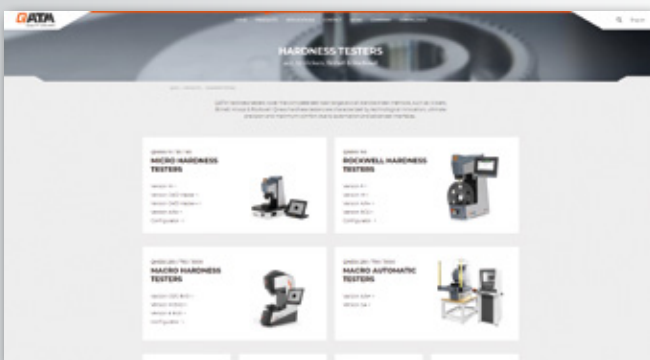
Micro Hardness Tester Q10 M



Semi-automatic Q10 M Vickers hardness tester for effective manual hardness testing at powder materials. Possible test forces between 0.25 g and 10 kg

- Exact positioning and large test room
- 6-fold measurement turret
- Dynamic height adjustment

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