SOLUTIONS FOR ADDITIVE MANUFACTURING & POWDER INJECTION MOLDING

VERDER scientific

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

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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PARTICLE CHARACTERIZATION OF METAL POWDERS WITH DYNAMIC IMAGE ANALYSIS

In this article, we present several examples of how the size and shape of typical metal powders like Ti64, Al, Ni, Cr, W, as well as of alloys can be characterized by Dynamic Image Analysis using the CAMSIZER X2. The advantages of this method are short analysis times, high resolution and excellent repeatability. In addition, a wealth of material data is provided, giving the user a detailed understanding of the powder quality.

Image Analysis: What you see is what you get

Imaging 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 pictures of individual particles. Particle length and width information are directly available as shown in Fig. 2. DIA offers great versatility by simultaneously measuring particle size and shape. A selection of shape parameters is explained in Fig. 3.

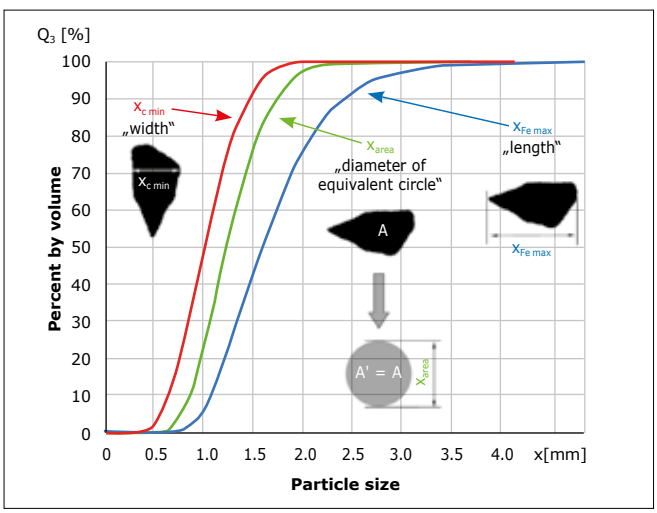


Fig. 2: Selection of basic size parameters used in image analysis. The size distributions are based on width (red), length (blue) or equal area diameter (green).

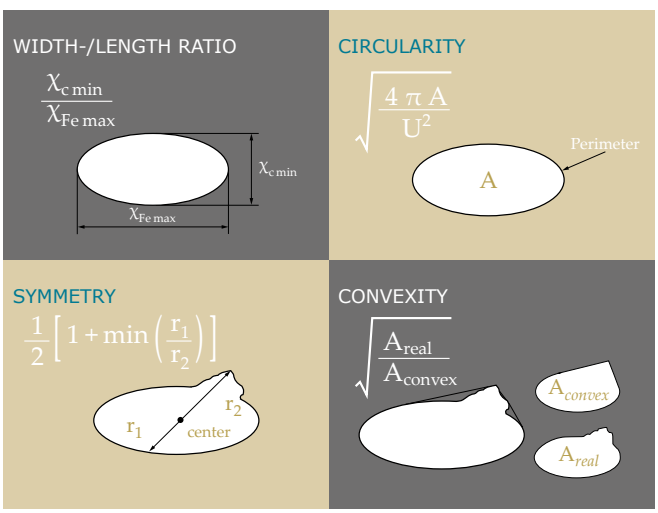


Fig. 3: Selection of basic shape parameters used in image analysis.

Two imaging techniques are available, **Static and Dynamic Image Analysis** (SIA and DIA, ISO 13322-1 and 2). The static optical microscopy (SIA) has commonly been used to obtain a qualitative impression of the shape of the particles. However, the insufficient dispersion of the particles on the microscope slide and the small amount of material prevent reliable quantitative analysis. The same drawbacks are associated with Scanning Electron Microscopy, plus this method is even more difficult, expensive and time consuming.

In the measurement set-up of **Dynamic Image Analysis**, particles, typically in a size range from 0.8 micron to several millimetres, move in front of a camera system, either transported by air flow or in liquid. Thus, it is possible to obtain data **from hundreds of thousands up to several millions of particles** within a few minutes. The results are based on a representative amount of sample material and are therefore statistically sound.

Fig. 4 displays the principal set-up of the optics for Dynamic Image Analysis. 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 RETSCH TECHNOLOGY's 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 to simultaneously analyze the larger particles with high detection efficiency. The CAMSIZER X2 records more than 300 pictures per second with one single image easily containing several hundreds of particles.

DIA allows to measure particle size distribution and quantitative particle shape (percentage of round versus irregular shaped particles, satellites, agglomerates etc.). Smallest amounts of oversized, undersized, or irregular shaped particles can be detected, even with a percentage as low as 0.01%. 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 provides accuracy and sensitivity as well as robustness and easy handling.

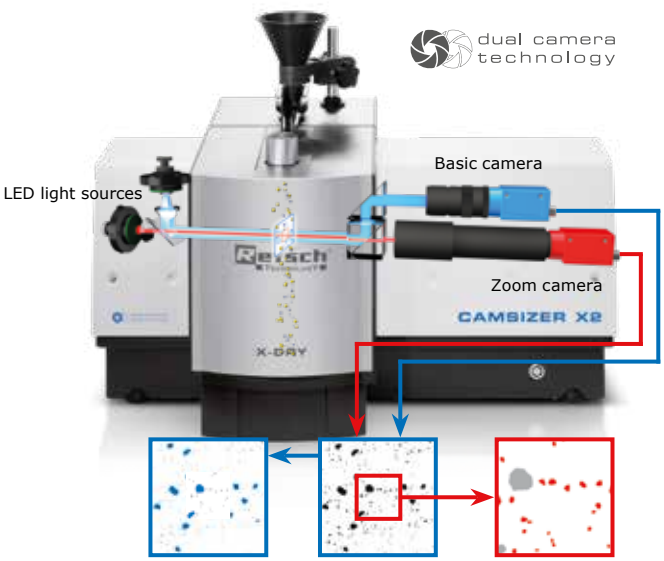


Fig. 4: Unique measurement principle of CAMSZER X2

In the following, a selection of application examples demonstrates the suitability of DIA to comprehensively characterize metal powders.

Wide range of materials, particle sizes and particle shapes

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. The air pressure is adjustable from 5 kPa to 460 kPa. 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.

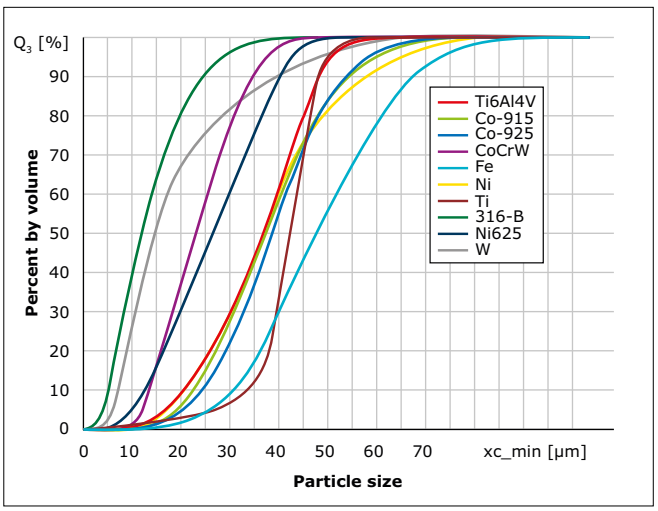


Fig. 5: Particle size analysis of ten different metal powders with the CAMSIZER X2. The direct measurement ensures accurate results.

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.

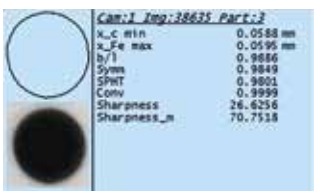
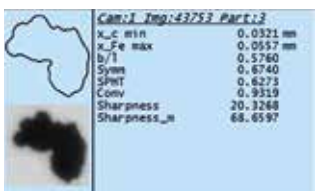


Image of a spherical metal powder particle



Irregular particles are reliably detected

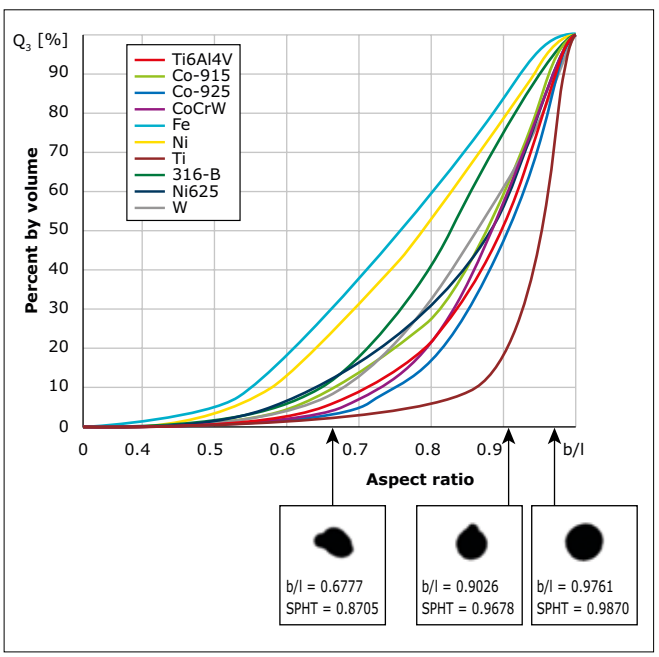


Fig. 6: Analysis of particle shape of 10 different metal powders with Dynamic Image Analysis (CAMSIZER X2). Beside the quantitative results, the recorded images allow an intuitive understanding of morphology and size differences. More spherical particles with higher aspect ratio plot on the right side of the diagram. Detecting 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 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 and a narrow, uniform particle size distribution are required to create a smooth, homogenous layer of powder to ensure accurate sintering. 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.

Fine metal powders for Metal Injection Molding

For MIM applications metal powders consisting of very fine spherical particles are required, usually with a median size below 10 µm. The example in Fig. 7 shows the measurement results of two different types of metal powder as they are used for MIM. The analyses have been made with the CAMSIZER X2 in dry mode at 50 kPa dispersion pressure. Note that the CAMSIZER X2 is able to detect even smallest differences between the two materials and accurately characterizes the distribution width.

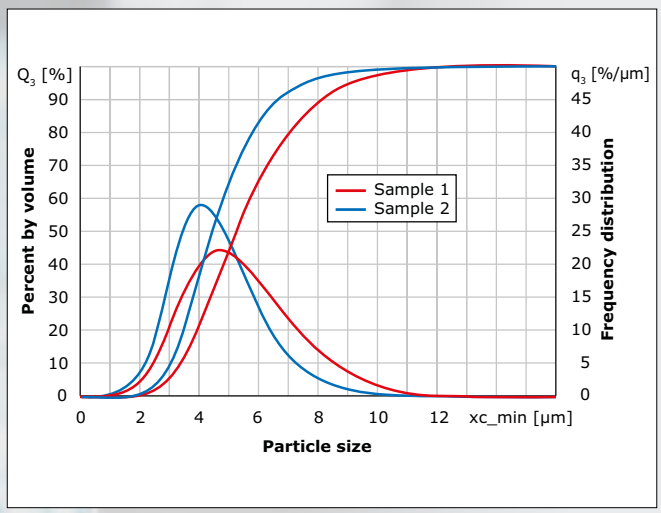


Fig. 7: Two measurements of two different metal powders with a median (d_{50}) size of 4.5 µm and 5.2 µm. The CAMSIZER X2 detects particles as small as 0.8 µm.

Reproducibility Study with Solder Powders

Powder metallurgy is a major application area for metal powders but there are others such as solder powder for circuit boards. Different types of solder powder are available which need to be characterized accurately with regard to size and shape due to tight product specifications (Fig. 8).

A major criterion to evaluate the reliability of any measuring device is reproducibility. One of our customers who is a producer of solder powder has performed a reproducibility test by analyzing the same sample of solder powder with four different CAMSIZER units in two different plants. The test comprised 180 measurements in total and the results can be seen in Fig. 9. The median size of the test material was determined to be 27.3 µm with a standard deviation below 0.1 µm!

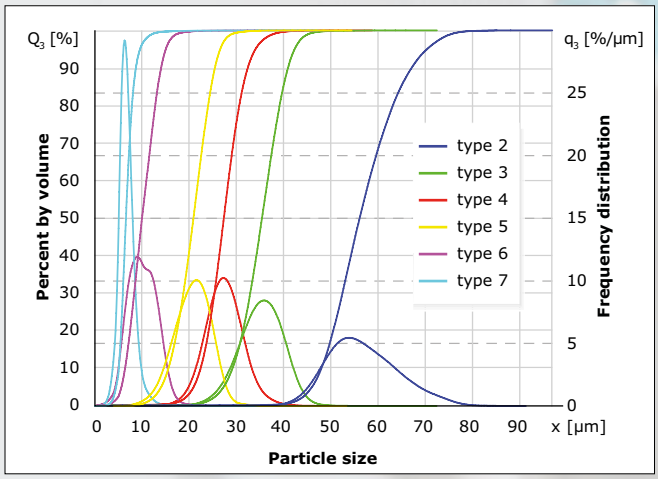


Fig. 8: Measurement results of 6 different solder powders obtained from different manufacturers. Displayed are the cumulative distributions (Q_3, left y-axis) and the corresponding frequency density distributions (q_3, right y-axis)

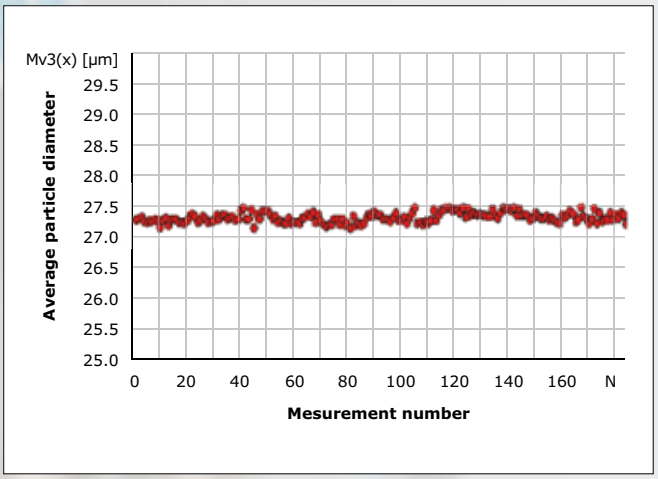


Fig. 9: 180 different measurements of the same sample type with 4 different CAMSIZER units at two different locations. The x-axis shows the measurement number, the y-axis the mean particle diameter. The average measured particle size varies by less than +/- 0.1 µm

Advantages of DIA over other particle sizing techniques

For metal powders, mechanical sieve analysis is traditionally the most common method for particle sizing. Standards ISO 4497 and ASTM B214 describe the most relevant procedures.

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 amount 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.

Laser diffraction is widely used to measure fine metal powders with particle sizes below 100 microns. This technology uses static light scattering, as described in ISO 13320. Laser diffraction analyzers are easy to operate and provide fast measuring results; however, this method calculates the particle size from the scattering angle and light intensity of a laser light beam interacting with the sample and is thus based on indirect measurement. Sophisticated software algorithms are necessary to calculate the particle size distribution based on assumptions and approximations. One basic assumption is, for example, that all particles are spherical. Consequently, no information on particle geometry is available and any deviation of the actual particle shape from the "ideal" shape causes discrepancies in the calculated particle size distribution. This leads to inaccurate results, especially when it comes to measuring the correct distribution width. Another major drawback is the very low sensitivity for detection of small amounts of over- and undersized particles.

Recently the German VDI released a guideline regarding the characterization of powders for AM. See guideline VDI 3405 Part 2.3. This guideline refers to Dynamic Image Analysis as best suited method for characterization of size and shape of metal powders.

Comparison sieve analysis, dynamic image analysis and laser diffraction

Performance Feature	CAMSIZER X2 Dynamic Image Analysis	Sieve Analysis	Laser Diffraction
Wide dynamic measurement range	+++	++	+++
Reproducibility and repeatability	+++	++	+++
High resolution for narrow distributions	+++	−	−
Particle shape analysis	+++	−	−
Direct measurement technique	+++	+++	−
Compatibility of results with other techniques	++	−	−
Reliable detection of oversized grains	++	+++	−
Robust hardware, easy operation for routine analysis	+++	+++	+++
Analysis of individual particles	++	−	−
High measurement speed, short measurement times	+++	−	+++

Conclusion

With metal injection moulding and additive manufacturing becoming increasingly prevalent techniques, there is an increased demand for specially designed metal powders with very specific characteristics. Not only chemical composition, but also particle size and shape are of vital importance for the processability of the powders. Depending on the application, the powder must meet a variety of specifications. Dynamic Image Analysis with **the CAMSIZER X2 provides all relevant data on particle size and shape.** Compared to laser diffraction or (electron or optical) microscopy, the measurement data is based on a large number of analyzed 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 CAMSIZER X2 is a precise and efficient tool which helps to greatly improve the quality control process.

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


ELTRA

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.

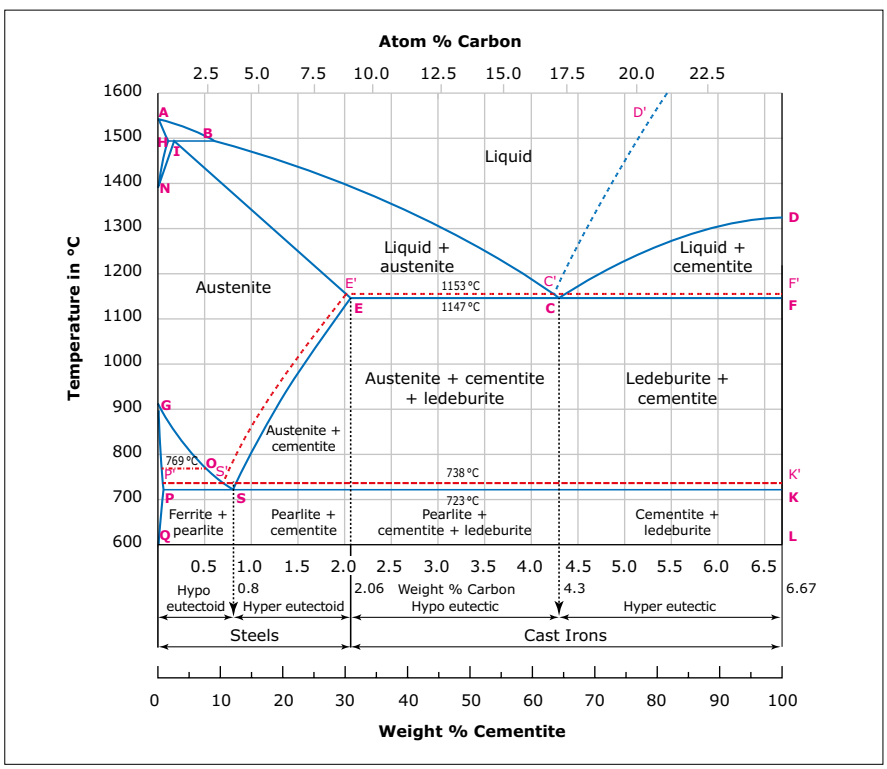


Fig. 1: Iron-carbon phase diagram

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 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).

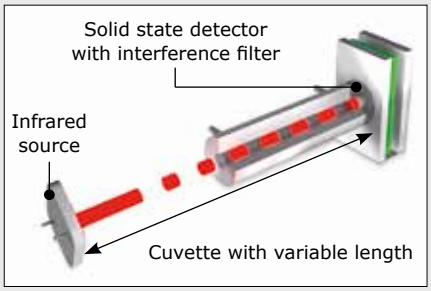


Fig. 2: Infrared cell

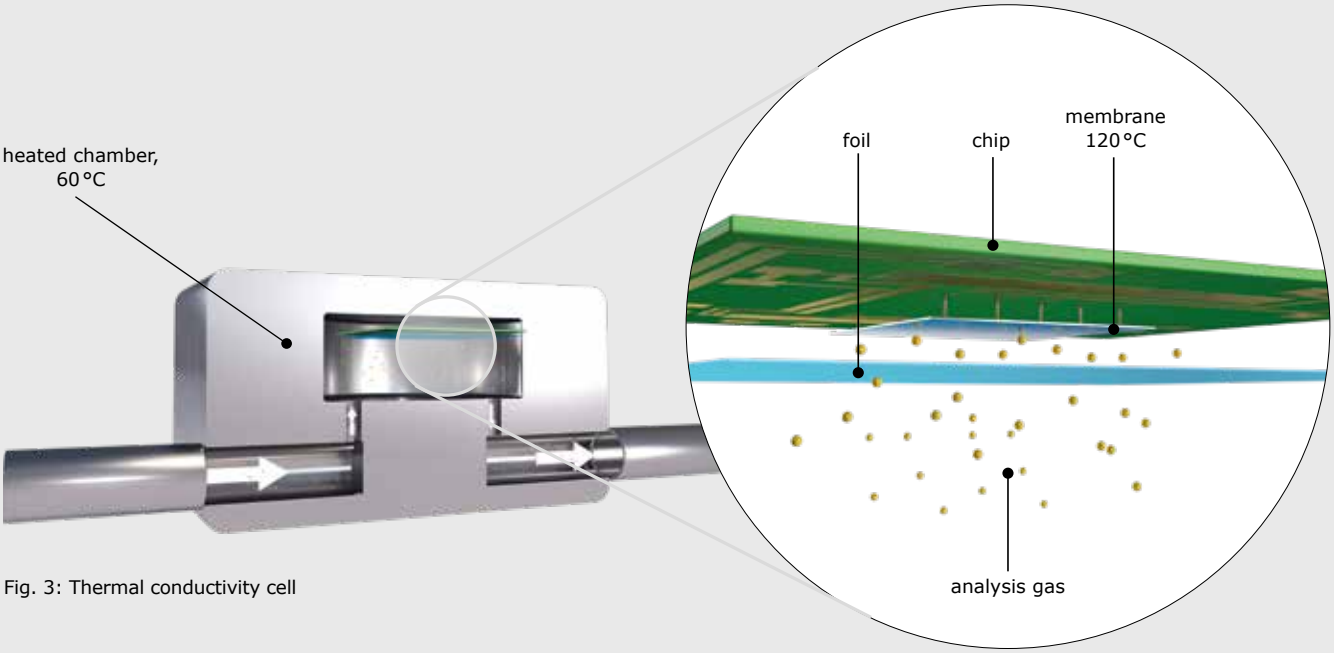


Fig. 3: Thermal conductivity cell

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 (SO_2) and carbon to react to a mixture of carbon monoxide (CO) and carbon dioxide (CO_2).

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 SO_3 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.

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.

ELTRA SOLUTIONS FOR ADDITIVE MANUFACTURING

ELEMENTRAC ONH-p

- **Simultaneous oxygen/nitrogen or oxygen/hydrogen determination with inert gas fusion technique**
- **Closed gas management and optimized gas circulation for sensitive ONH determination**
- **Use of cost efficient argon as carrier gas possible**

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**

www.eltra.com

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.

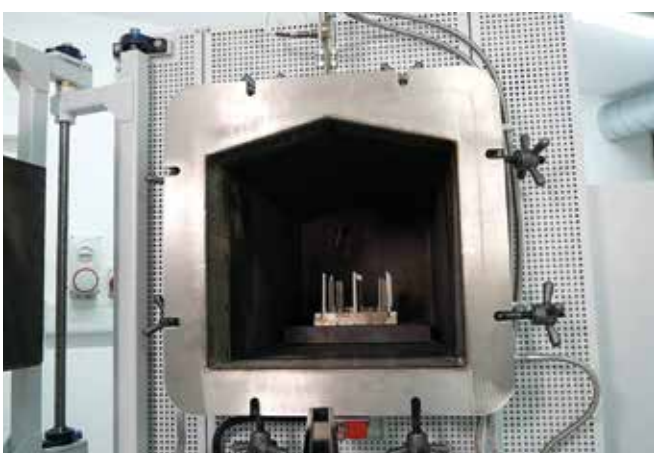


Fig. 2: View into metallic GPCMA/174 retort with additive manufactured sample for stress relieving contained therein.

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 letter 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 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 x 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.

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.

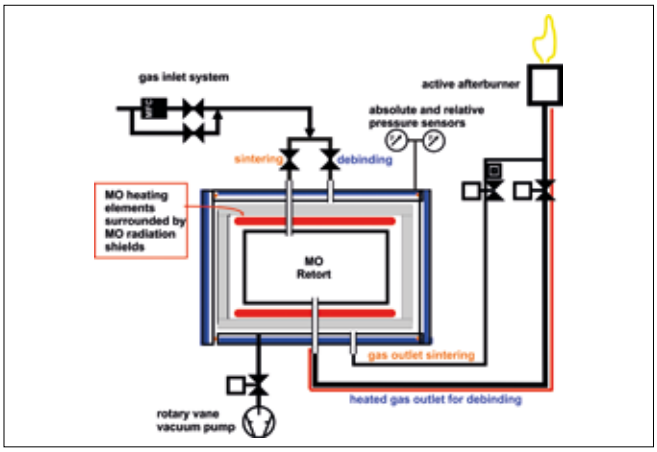


Fig. 4: Gas guidance during debinding or sintering through the retort.

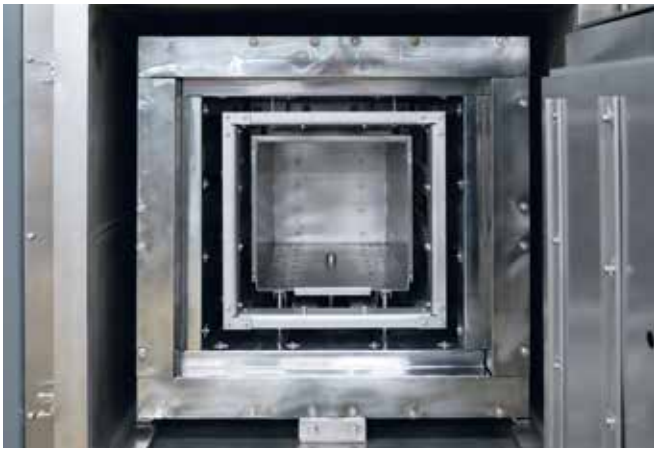


Fig. 5: Mo retort of the HTK for highest possible purity of atmosphere and vacuum level.

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 N_2, Ar**
- **O_2 level below 30 ppm**
- **Precise temperature uniformity**

HTK Metallic Chamber Furnace for Powder Injection Molding and Additive Manufacturing

- **Debinding and Sintering under H_2, Ar, N_2**
- **Switchable gas flow for processing of sensitive materials**
- **Fully automatic control system**

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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.

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.

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)

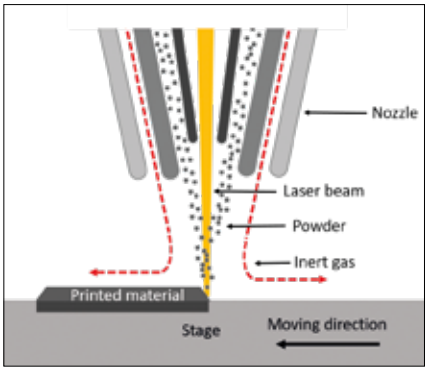

Fig. 1: process of additive laser powder build-up welding.

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

Fig. 2: Cut-Off Machine Brillant 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 **ATM's Brillant 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.

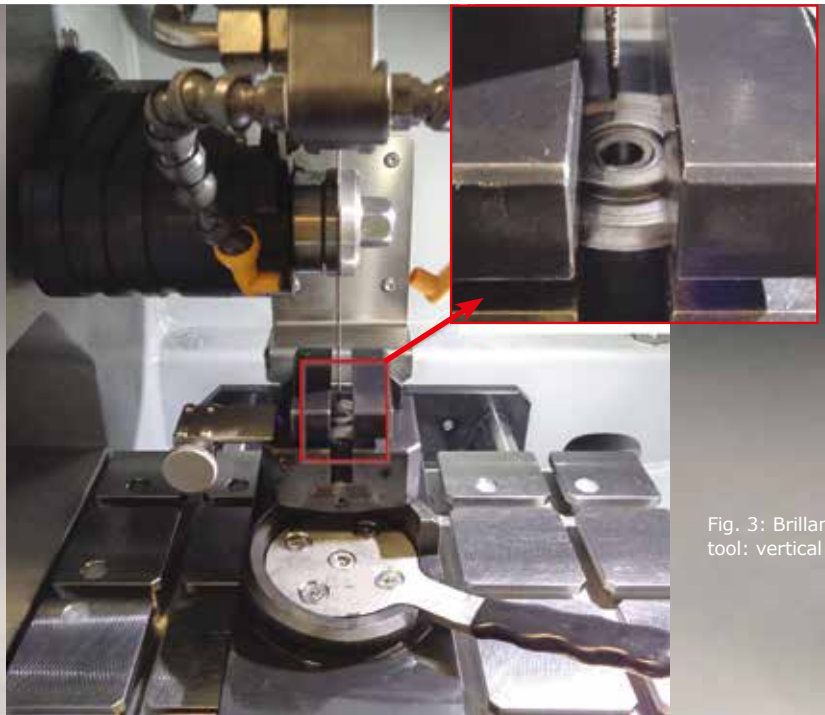

Fig. 3: Brillant 220 machine setup. Detail: clamped sample (clamping tool: vertical vice single).

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 **ATM 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 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, ATM'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** (ATM: GAMMA) and 3 µm poly diamond suspension. The last step, called final polishing, was done on a **soft synthetic polishing cloth** (ATM: 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

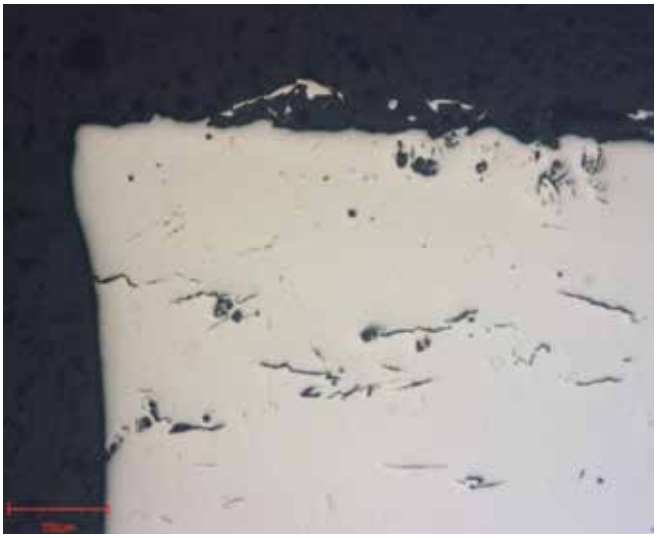

Fig. 6: Image of the prepared specimen surface. Due to the polished surface the light is reflected almost equally and the microstructure is not discernible.

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. ATM 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

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.

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).

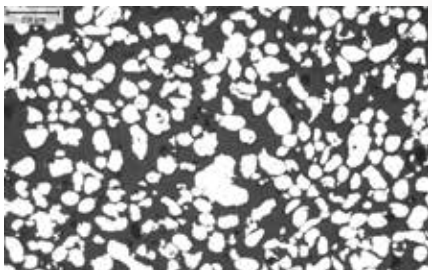

Fig. 1: Embedded Aluminum powder / polished surface / 4x microscope lens
Powder particles polished down to half of the particle size or big particles are suited best for hardness testing with meaningful results.

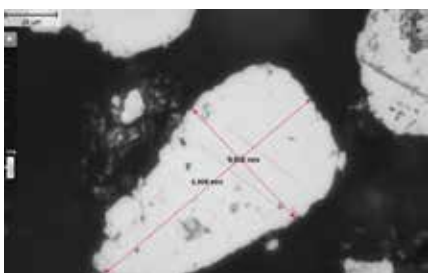

Fig. 2: Size of an aluminum powder particle measured in hardness testing software (40x lens)

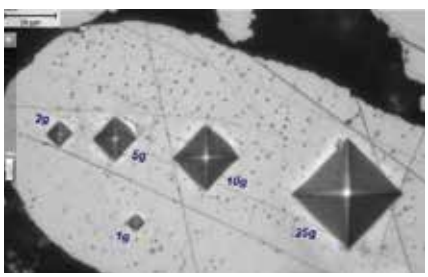

Fig. 3: Comparison of Vickers indent sizes: HV0.001, HV0.002, HV0.005, HV0.01 and HV0.025 – Test forces between 1 g and 25 g

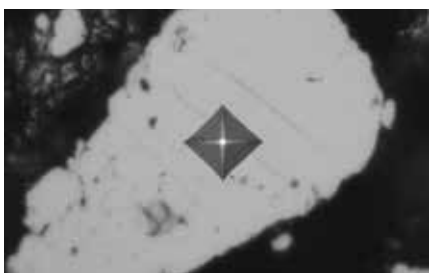

Fig. 4: Hardness result: 30.3 HV0.005 tested in the middle of the cross section of the aluminum particle

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 QNESS 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.

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