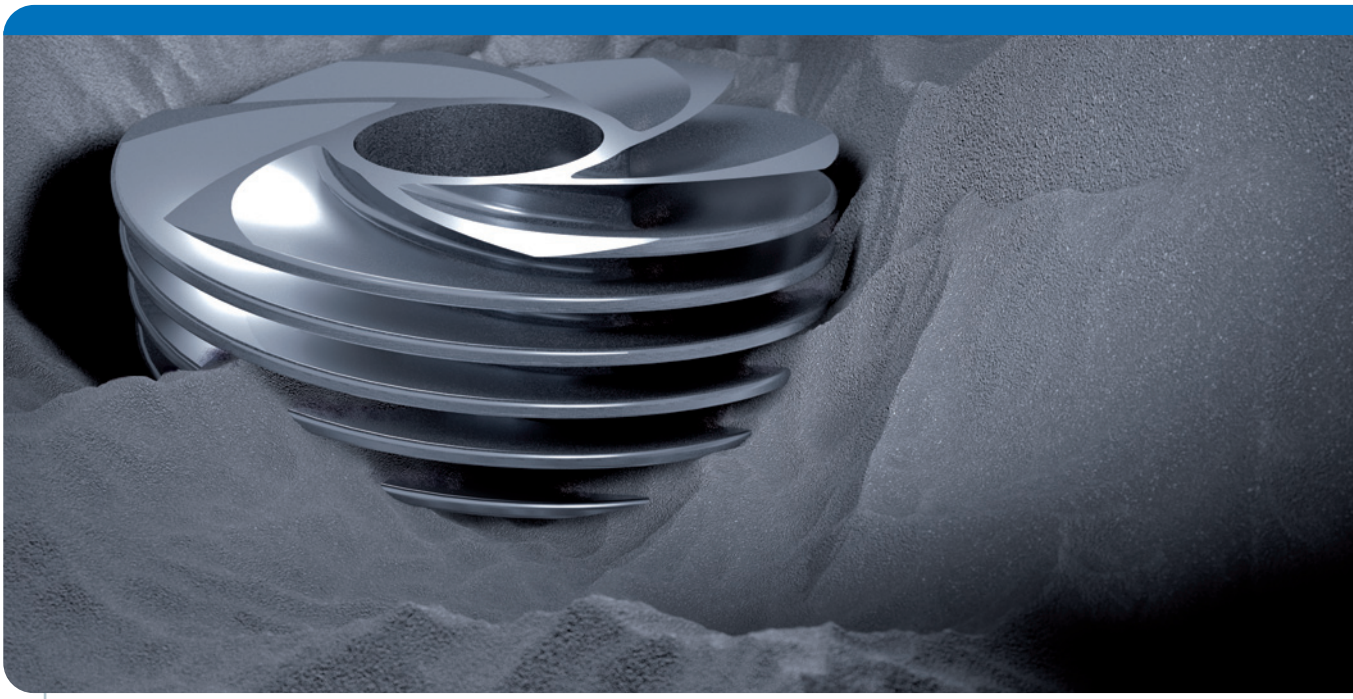


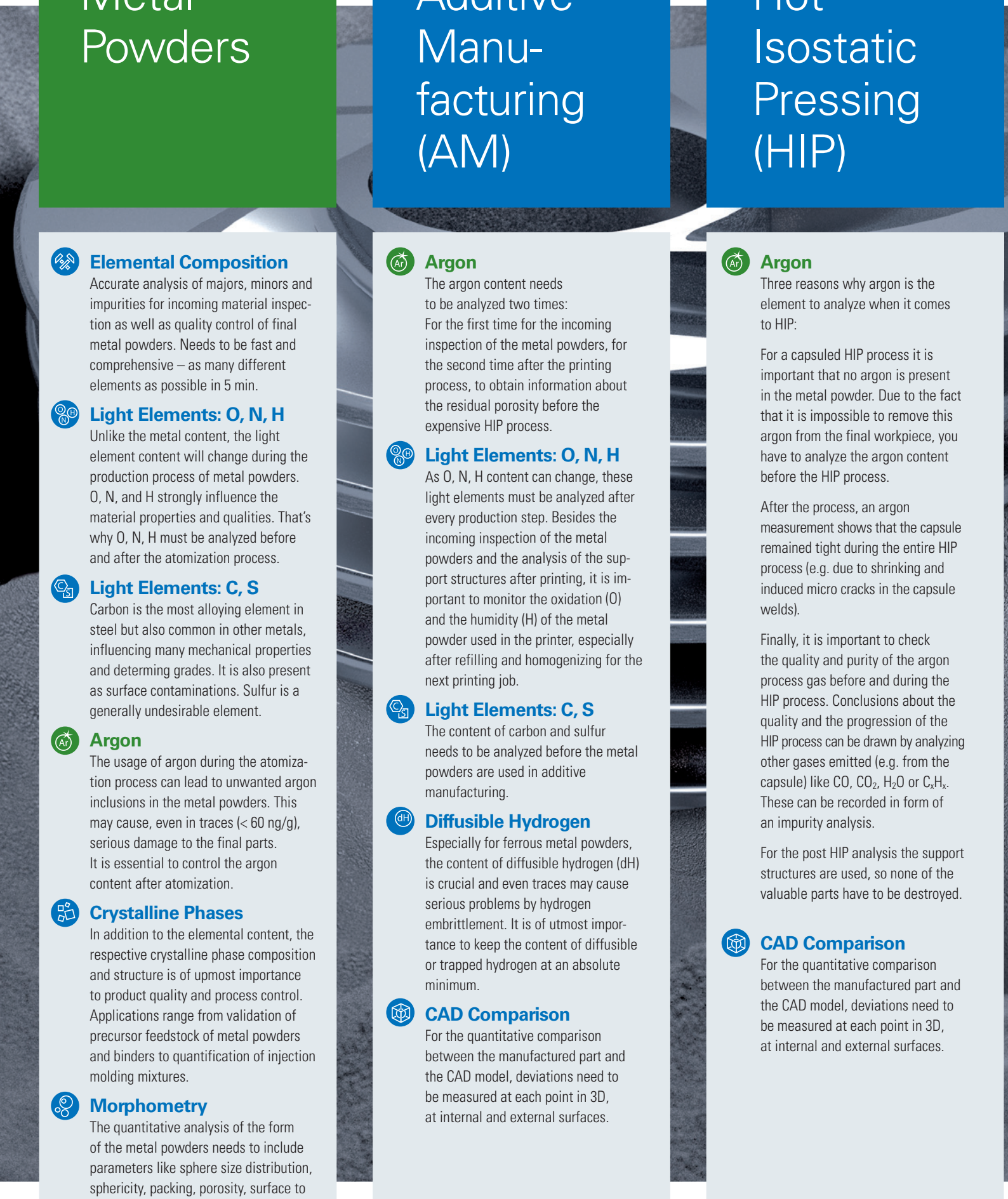
Elemental Analysis & Metrology for Powder Metallurgy

Analytical Solutions for Modern Production Processes

Additive manufacturing, hot isostatic pressing and metal injection molding all have one common component: metal powders.

Powder metallurgy enables today's most demanding production processes, and Bruker offers the most comprehensive range of elemental and compositional analysis of these advanced materials. Our instruments monitor light elements and argon content, quantify crystalline phases and determine the morphometry of powder particles.

The content of light elements like O, N, H and C, S can change. They need to be measured at every process step. Argon and diffusible hydrogen are especially sensitive because even traces can cause fatal damage to the final workpiece. To ensure product quality internal and external surfaces should be compared to their underlying CAD models. Element mapping, residual stress and texture analysis, and final part inspection are also all required. Only Bruker offers all these analytical solutions.



Metal Powders

Elemental Composition
Accurate analysis of majors, minors and impurities for incoming material inspection as well as quality control of final metal powders. Needs to be fast and comprehensive – as many different elements as possible in 5 min.

Light Elements: O, N, H
Unlike the metal content, the light element content will change during the production process of metal powders. O, N, and H strongly influence the material properties and qualities. That's why O, N, H must be analyzed before and after the atomization process.

Light Elements: C, S
Carbon is the most alloying element in steel but also common in other metals, influencing many mechanical properties and determing grades. It is also present as surface contaminations. Sulfur is a generally undesirable element.

Argon
The usage of argon during the atomization process can lead to unwanted argon inclusions in the metal powders. This may cause, even in traces (< 60 ng/g), serious damage to the final parts.
It is essential to control the argon content after atomization.

Crystalline Phases
In addition to the elemental content, the respective crystalline phase composition and structure is of upmost importance to product quality and process control. Applications range from validation of precursor feedstock of metal powders and binders to quantification of injection molding mixtures.

Morphometry
The quantitative analysis of the form of the metal powders needs to include parameters like sphere size distribution, sphericity, packing, porosity, surface to volume ratio, surface roughness, convexity, and form factor.

Additive Manufacturing (AM)

Argon
The argon content needs to be analyzed two times:
For the first time for the incoming inspection of the metal powders, for the second time after the printing process, to obtain information about the residual porosity before the expensive HIP process.

Light Elements: O, N, H
As O, N, H content can change, these light elements must be analyzed after every production step. Besides the incoming inspection of the metal powders and the analysis of the support structures after printing, it is important to monitor the oxidation (O) and the humidity (H) of the metal powder used in the printer, especially after refilling and homogenizing for the next printing job.

Light Elements: C, S
The content of carbon and sulfur needs to be analyzed before the metal powders are used in additive manufacturing.

Diffusible Hydrogen
Especially for ferrous metal powders, the content of diffusible hydrogen (dH) is crucial and even traces may cause serious problems by hydrogen embrittlement. It is of utmost importance to keep the content of diffusible or trapped hydrogen at an absolute minimum.

CAD Comparison
For the quantitative comparison between the manufactured part and the CAD model, deviations need to be measured at each point in 3D, at internal and external surfaces.

Hot Isostatic Pressing (HIP)

Argon
Three reasons why argon is the element to analyze when it comes to HIP:

For a capsuled HIP process it is important that no argon is present in the metal powder. Due to the fact that it is impossible to remove this argon from the final workpiece, you have to analyze the argon content before the HIP process.

After the process, an argon measurement shows that the capsule remained tight during the entire HIP process (e.g. due to shrinking and induced micro cracks in the capsule welds).

Finally, it is important to check the quality and purity of the argon process gas before and during the HIP process. Conclusions about the quality and the progression of the HIP process can be drawn by analyzing other gases emitted (e.g. from the capsule) like CO, CO_2, H_2O or C_xH_x. These can be recorded in form of an impurity analysis.

For the post HIP analysis the support structures are used, so none of the valuable parts have to be destroyed.

CAD Comparison
For the quantitative comparison between the manufactured part and the CAD model, deviations need to be measured at each point in 3D, at internal and external surfaces.

Metal Injection Molding (MIM)

Diffusible Hydrogen
Especially for ferrous metal powders, the content of diffusible hydrogen (dH) is crucial and even traces may cause serious problems by hydrogen embrittlement. It is of utmost importance to keep the content of diffusible or trapped hydrogen at an absolute minimum. That's the reason why the dH level must be analyzed before and after the molding and after the debinding process.

Light Elements: O, N, H
For incoming inspection and after every process step, molding, cleaning and debinding, the content of O, N and H must be checked. Because unlike the amount of metals, the content of light elements will change during the production process.

Light Elements: C, S
The content of carbon and sulfur needs to be analyzed before the metal powders are used. The carbon level in the intermediate and final products are important to monitor the debinding process after the MIM process.

CAD Comparison
For the quantitative comparison between the manufactured part and the CAD model, deviations need to be measured at each point in 3D, at internal and external surfaces.

Failure Analysis

Element Mapping
Analysis of inhomogeneity, inclusions and foreign particles in manufactured parts with 300 µm resolution by high performance XRF[2].

Residual Stress Analysis
Understanding the stress state that remains in a component after processing is essential to maximizing lifetime of manufactured components. Components with compressive surface stress traditionally exhibit prolonged lifespan, while a tensile surface stress can lead to premature failure.

Texture Analysis
As materials are pushed to their limits it is important to ensure that every crystallite is in proper alignment. Texture analysis is used to quantify the magnitude and direction of crystallite orientation in a component.

Crystalline Phases
Issues with dimensional stability are not only the consequence of macroscopic issues, but may be also due to an improper phase composition and structure. Austenitic phase transformations are a classic example of this in ferrous alloys.

Argon
To discover the reasons of argon induced cracks and voids you have to measure its amount.

Light Elements: O, N, H
Oxygen is measured to find the reasons for corrosion and defects, total hydrogen to detect the cause of pores in aluminum.

Diffusible Hydrogen
Hydrogen embrittlement – the name says it all.

Parts Inspection
Failed products must be inspected and analyzed for cracks and voids. The contact area at the interface between different materials should be analyzed for defects.

X-Ray Fluoresence (XRF)
- Elemental Composition: Ti, Cr, Mn, Fe …
- Element Mapping

Inert Gas Fusion Analysis (IGF)
- Light Elements: O, N, H
- Diffusible Hydrogen: dH
- Nobel Gas: Ar

Combustion Gas Analysis (CGA)
- Light Elements: C, S

X-ray Microtomography (µCT)
- Morphometry: Sphericity, Size, Porosity …
- CAD Comparison
- Parts Inspection

X-Ray Diffraction (XRD)
- Residual Stress Analysis
- Texture Analysis
- Crystalline Phase Identification and Quantification

X-ray Fluorescence

S8 TIGER

The S8 TIGER is the most versatile tool available for advanced elemental analysis in both industry production and research. This high-performance wavelength dispersive X-ray fluorescence spectrometer is analyzing almost the entire periodic table.

Element Composition

- Turn-key solution METAL-QUANT to cover more than 23 elements in less than 5 minutes.
- Elements of interest: Ti, Cr, Mn, Fe, Co, Ni, Cu, Nb, Mo, W …
- Traces, down to ppm: Si, P, S, Cl, Ta, Pb …

Element Mapping

- XRF² small spot mapping provides detailed maps of the elemental distribution in materials.
- 300 µm spot and 100 µm step size based on HighSense™ beam path for optimal precision and detection power.

Inert Gas Fusion Analysis

G8 GALILEO MS

The G8 GALILEO MS is a high-end instrument designed for fast determination of O, N, H, and Ar. This analyzer is based on the inert gas fusion principle, coupled to a mass spectrometer (MS).

Argon

- Detection of Ar in raw materials and final products down to a few ng/g (ppb).
- Discovers reliably Ar inclusions, which may lead to complete part failures.

Light Elements, gaseous: O, N, H

- Reliable detection from traces up to high concentrations.

Diffusible Hydrogen: dH

- Detection of diffusible and trapped H.
- Thermal desorption mass spectroscopy for analyzing the kinetic bonds of H in the material.

Combustion Analysis

G4 ICARUS

The combustion analyzer G4 ICARUS is the ideal instrument for rapid and precise C and S analysis in solids, especially with metallic materials and other inorganic materials.

Light Elements, solid: C, S

- Especially suited for the detection of surface contaminations, occured from dust or hydrocarbons, in powder metallurgy.
- Ideal for analyzing the bulk content of C and S.
- Monitor the debinding process after the MIM process.
- Monitor the C level before and after carburization during case hardening.

X-ray Microtomography

SKYSCAN 1173
SKYSCAN 1272

Desktop µCT is used for fast, non-destructive characterization of samples from the µm to cm scale with a resolution down to a few microns within minutes.

Morphometry

- Quantitative determination of porosity & inclusions, volume fractions, orientation, dimensions, surface, and angles.

CAD Comparison

- Automatic, quantitative comparison of external and internal surfaces.
- Pass/fail monitoring with defined tolerances at pre-defined inspection points.

Parts inspection

- 3D µCT scan, reconstruction and quantitative analysis of defects.
- Reaction of parts to mechanical stress and temperature changes using in-situ stages.

X-ray Diffraction

D8 DISCOVER
D8 ADVANCE
D2 PHASER

X-ray diffraction is the only method to determine the phase composition, the texture and the residual stress in a non-destructive way.

Crystalline Phases

- Comprehensive phase identification, quantification and microstructural characterization of metal powders with a LYNXEYE XE-T 1D detector and TOPAS software.
- Small spot measurements of manufactured components with a microfocus source, large sample stage and a 2D detector (XRD² setup).

Residual Stress

- Fast and reliable results with XRD² setup and LEPTOS S for analysis.

Texture Analysis

- XRD² setup and DIFFRAC.TEXTURE software, the best choice for texture analysis.

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