Powder metallurgy and additive manufacturing

Material characterization solutions

Shaping the future

Powder metallurgy is an umbrella term that covers a range of processes for manufacturing metallic components from metal powders usually by first forming a dimensionally stable compact and then sintering it. The main processes include;

- Press and Sinter
- Metal injection molding
- Hot/Cold Isostatic pressing
- Additive manufacturing

Traditional powder metallurgy processes such as press and sinter have been used to produce metallic components since the 1920s while processes such as Hot Isostatic Pressing (HIP) and Metal Injection Molding (MIM) have been employed since the 1960s and 70s respectively.

Additive Manufacturing is a relatively new technique that permits local fusing of metal powders using a laser, electron beam, or by post-sintering adhesive bonded powders.

There are a number of reasons for using a powder metallurgy process instead of a traditional process such as machining. These include one or more of the following:

- Significant cost savings
- High dimensional accuracy
- Minimal post processing
- Good part to part reproducibility
- Greater dimensional freedom and compositional complexity
- Reduced waste

Powder metallurgy refers to the production and working of metals as fine powders which can be pressed and sintered to form objects.

Metal powder manufacture

Metal powders are common to all powder metallurgy processes hence the powder manufacturing process and resultant powder properties are critically important and intrinsically linked.

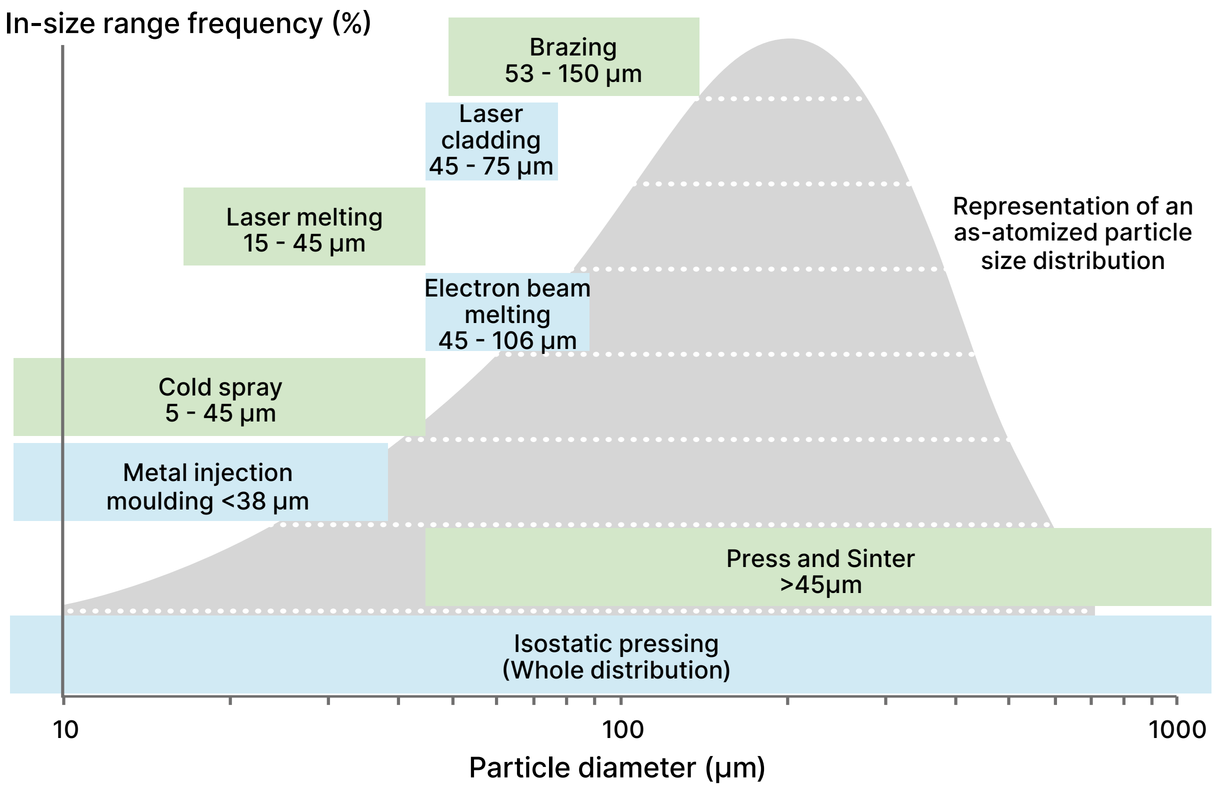
The main routes for manufacturing metal powders are:

- Comminution of solid metal
- Precipitation of a salt from solution
- Thermal decomposition of metal carbonyl (Carbonyl process)
- Solid state reduction of metal oxide
- Electrodeposition
- Atomization of molten metal

The choice of powder manufacturing route depends on the metal / alloy, the powder metallurgy process, and the powder properties required for that process.

Key powder characteristics are:

- Particle size distribution
- Particle shape
- Micro-structure
- Surface condition
- Elemental composition
- Flowability



Press and sinter

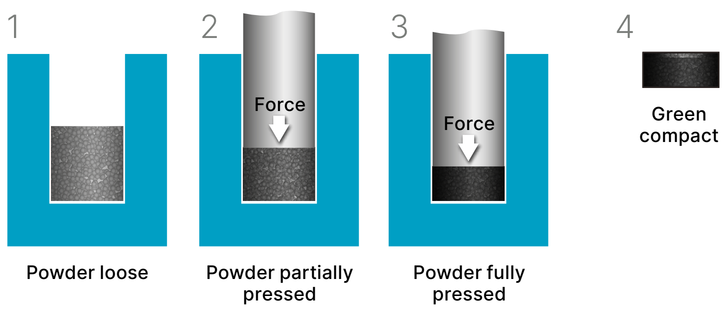
Powder characteristics are important for press and sinter, as for other powder metallurgy processes, with particle packing and apparent density of the powder blend critical.

A lower apparent density gives greater compaction and cold-welding of particles on pressing, resulting in a stronger green body. Interparticle friction is also important and facilitates contact, deformation and densification of the structure during pressing.

Metal powder hardness also affects pressing performance and can depend on phase composition and grain size. Malvern Panalytical's and NETZSCH's characterization solutions for Press and Sinter can be used to:

- Predict and control powder packing to give the required volume reduction and degree of cold-welding
- Comprehensive determination of sintering behavior
- Measure and control batch-to-batch variability in the metallic powder
- Ensure optimum flowability and packing in the die to prevent defects in the sintered component
- Ensure alloys have the correct elemental composition and phase structure
- Determine residual stress, strain and texture of sintered parts

1. Powder blending - metal powder is mixed with a suitable lubricant
2. Die compaction - the powder blend is pressed in a die to form a compact 'green body'
3. Sintering – the 'green body' is heated to below its melting point to form the sintered component



Additive manufacturing

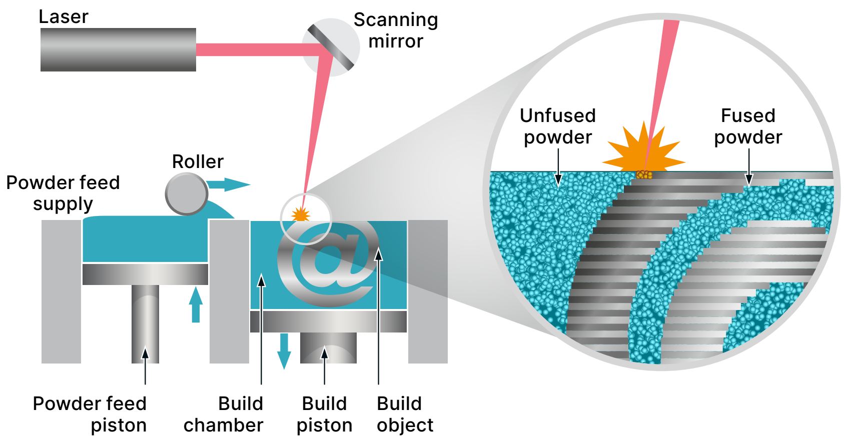
In powder bed fusion processes a metal powder layer is applied to a building platform and a laser or electron beam is used to selectively melt or fuse the powder. After melting the platform is then lowered and the process repeated continually until the build is complete. The unfused powder is removed and either reused or recycled depending on its condition.

The efficiency of powder bed additive manufacturing processes and the quality of finished components is largely dependent on the flow behavior and packing density of the powders.

The particle size directly influences these properties and is a key specification for this process, with the optimum particle size being in the range 15-45 μm for SLM and 45-106 μm for EBM for example.

Malvern Panalytical's and NETZSCH's characterization solutions for Additive Manufacturing can be used to:

- Ensure a consistent powder supply and prevent variations in product quality
- Identify suitable powders for machines with different spreader or rake designs
- Optimize atomization conditions to achieve desired powder properties
- Predict and optimize powder packing density and flow characteristics
- Ensure powders have the correct elemental composition and phase structure
- Determine residual stress, strain and texture of manufactured components
- Optimize melting and crystallization behavior



Metal injection molding

The rheological properties of the feedstock are of major importance for MIM applications since they influence the homogeneity of the molten feedstock, how well it flows through the die into the mold cavity, and the mechanical properties of the green part on cooling.

Rheological properties are influenced by a number of factors including; binder molecular weight and structure; solids loading; particle size and shape; temperature and flow rate.

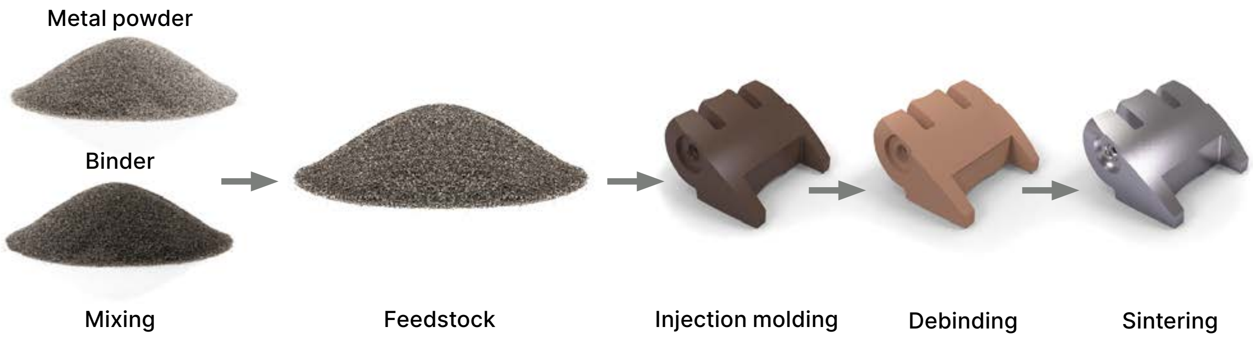
Most metals can be used in MIM if they have the correct powder properties, including particle size and shape.

High packing densities are required, so spherical particles with the correct size distribution are preferable, as are smaller particles (< 38μm) that sinter more readily.

Slight particle irregularity can be beneficial to give mechanical strength to green and brown parts.

Malvern Panalytical's and NETZSCH's characterization solutions for Metal Injection Molding can be used to:

- Optimize particle loading to minimize part shrinkage and void formation
- Ensure appropriate feedstock rheology during molding, and dimensional stability post-extrusion
- Optimize binder properties including molecular weight and structure
- Ensure consistent metal powder supply
- Characterize debindering and sintering behavior of green/brown parts



Isostatic pressing

Isostatic pressing is a forming process where pressure is applied uniformly (using gas or liquid) to a hermetically sealed container filled with compacted metal powder. The benefits of isostatic pressing over press and sinter are equal compaction in all directions and more uniform density in the final component.

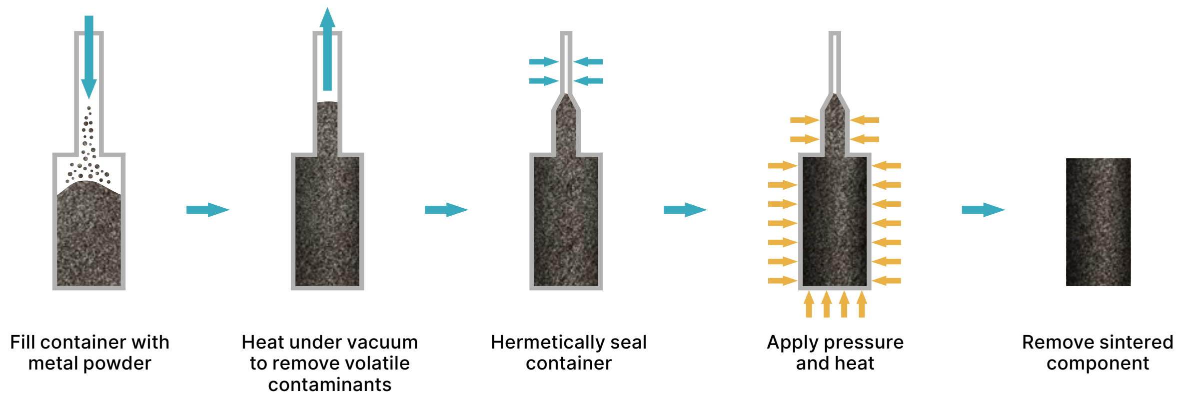
Isostatic pressing can be performed at elevated temperatures (Hot Isostatic Pressing (HIP)) or at ambient temperature (Cold Isostatic Pressing (CIP)). Although HIP is a direct manufacturing route for metal components it can also be used for densifying parts from other powder metallurgy processes.

Spherical powders with a relatively wide but consistent particle size distribution are preferred for HIP as they give higher fill densities. For CIP, some particle irregularity may help increase cold-welding, ideally without compromising powder flow and packing.

Phase composition and crystallite size are also important since these can affect powder hardness and melt properties which impact pressing efficiency and sintering behavior.

Malvern Panalytical's and NETZSCH's characterization solutions for Isostatic Pressing can be used to:

- Predict and control packing density and minimize void formation in the sintered part
- Specify and control metal powder quality
- Optimize powder flow in the mold and maintain process efficiency
- Ensure powders have the appropriate phase structure and elemental composition

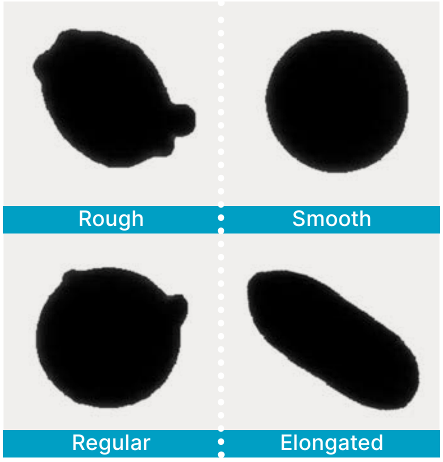


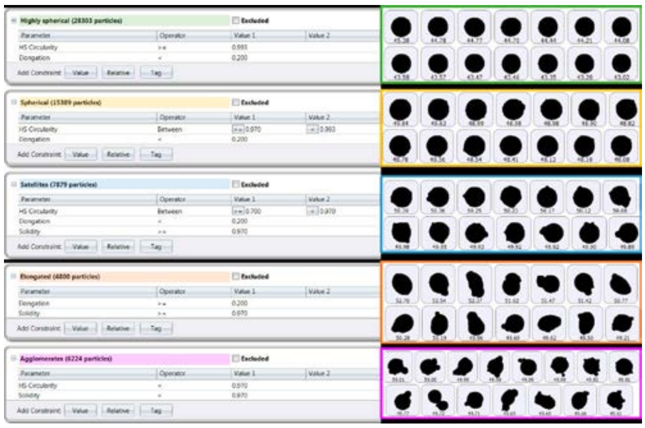
Morphologi 4

Direct measurement of metal powder size and shape

The Morphologi 4 is an advanced yet easy-to-use, particle characterization tool for measuring particle size and shape from 0.5 microns to several millimeters. The instrument offers the flexibility required for R&D and troubleshooting applications, as well as the user-independent results and validation required for automated QC analysis.

For many powder metallurgy applications, particle shape can be just as important as particle size. The Morphologi 4 reports shape information using parameters such as elongation, circularity, convexity to quantify particle irregularity and surface roughness. More efficient than manual microscopy and electron microscopy, automated imaging provides statistics on tens of thousands of particles.





Features	Benefits
Particle size range from 0.5 µm to 1000 µm	Make size measurements of powders for all powder metallurgy applications
Measurement of non-spherical particles in terms of their length and width	Provides a more relevant size measurement of irregular particles
Automated image analysis that reports a range of shape parameters	Quantify particles in terms of the degree of irregularity or surface roughness
Automation of manual methods such as microscopy	Perform microscopy measurements faster and less subjectively, while freeing up resource
Integrated dry powder dispersion unit	Easy, reproducible sample dispersion which is critical to achieving meaningful results
Optimized microscope optics and high signal to noise CMOS camera	Generation of high quality particle images and image analysis data
Automated SOP control from sample dispersion to results analysis	Simple, intuitive operation and robust, repeatable measurement protocols

What do your customers say?

Wall Colmonoy invest in Morphologi 4 to improve their metal powder products and manufacturing processes

Wall Colmonoy is a leading global materials engineering group of companies engaged in the manufacturing of Colmonoy® and Wallex® surfacing and Nicrobraz® brazing products, precision castings, coatings, and engineered components across aerospace, automotive, oil & gas, mining, energy and other industrial sectors. Headquartered in Madison Heights, Michigan, Wall Colmonoy have been an established name in metallurgy since 1938, with several sites in the US and European headquarters in Swansea, UK.

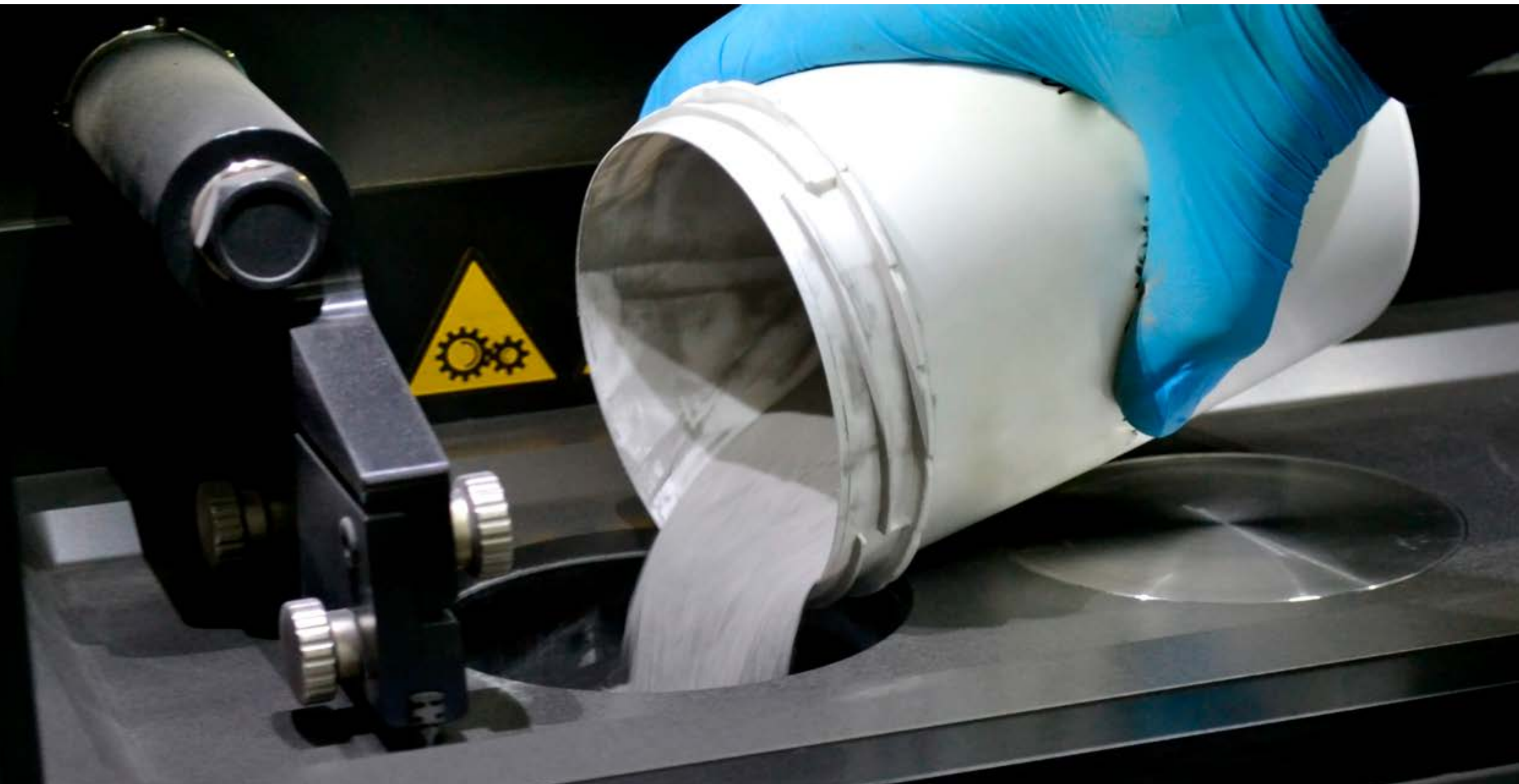
Wall Colmonoy manufactures over 500 different metal powder products at their Pontardawe facility in Swansea and have a comprehensive suite of analytical equipment for characterizing their powders: including laser diffraction, chemical analysis, rheometry, optical microscopy and electron microscopy. With a recent move into Additive Layer Manufacturing, where powder properties such as particle size and shape are often more critical, there was a need to expand their analytical capabilities.

“We know that particle shape directly influences Additive Layer Manufacturing performance and our customers expect a high-quality product with the correct size distribution and a spherical morphology” said Tom Roblin, Process Engineering Manager. “We have manual microscopy and electron microscopy which allow us to qualitatively check the products we manufacture but we also needed a technique that could provide quantitative data on a statistically representative sample.”

The technical team recognized that an automated optical imaging platform capable of analyzing a large number of particles was required. They also needed image analysis capabilities that could automatically quantify the size and shape distribution of those particles. “We identified three platforms that we thought could meet our requirements. This included two Dynamic Imaging systems and the Morphologi 4 Static Imaging system from Malvern Panalytical” said Roblin. “While Dynamic Imaging was able to analyze a larger sample volume, the superior image clarity provided by the Morphologi 4 was of greater importance to us and also gave more consistent results”.

The Morphologi 4 has found multiple applications within the laboratory, not just for Additive Layer Manufacturing powders, but for many of their other products also.

“Morphologi 4 is used to quantitatively measure the particle shape of our atomized alloy powders. By investing in this measurement equipment, we can benchmark and improve our current processes and products.” said Roblin. “It will also aid in the development of a new range of Wall Colmonoy powders for Additive Layer Manufacturing. For us, it is a valuable tool that provides clear images, quantitative analysis and consistent results.”



Mastersizer 3000

Accurate particle size with minimum effort

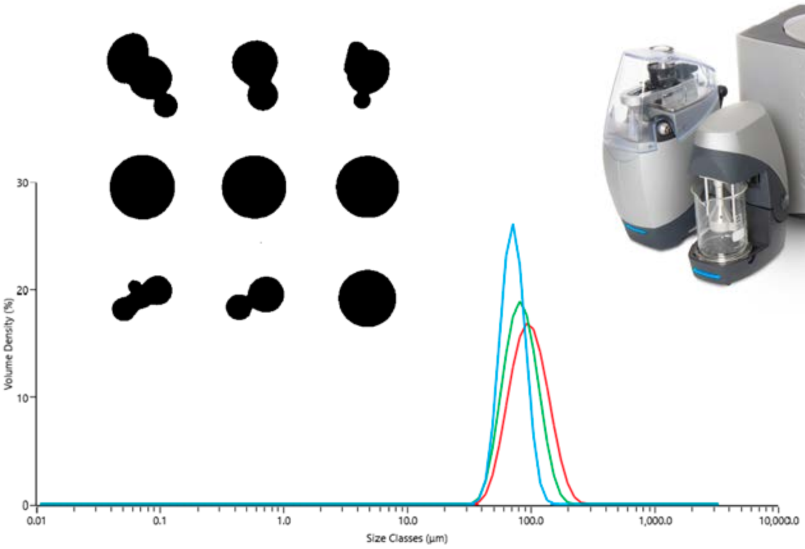
The Mastersizer 3000 is the latest generation of the world's most widespread particle sizing instrument, used by many thousands of companies and research institutes across a wide range of industries.

Malvern Panalytical's considerable experience and applications know-how has gone into every stage of the design of the Mastersizer 3000 instrument, from fundamental particle sizing performance right through to user ergonomics and method advice.

The Mastersizer 3000 also has an optional dynamic imaging module called Hydro Insight allowing simultaneous particle images and particle shape information to be generated.

Many of the disadvantages of traditional sieve analysis are addressed by the Mastersizer 3000:

- Faster and simpler analysis
- Increased measurement range including very fine particles
- Better measurement resolution for improved product quality
- Trouble free maintenance
- Particle images and shape information
- Sieve correlation algorithm



Features	Benefits
Measure particles in the size range 0.01 µm – 3.5 mm	Enable accurate measurement of almost any metal powder fineness and formulation
High accuracy, repeatability and reproducibility (< 1% variation)	Verifiable instrument-to-instrument performance that you can rely on
Easy sample loading and cleaning	Reproducible dispersion with minimal sample to-sample contamination
Rapid measurements (<20 sec)	Increased throughput/productivity and more efficient process control
TCP/IP or Autolab driver remote control via Malvern Link™ II	Feed results directly to the plant process control system
Modern and intuitive software interface	Streamlined method development and built-in expert advice
Meets all the recommendations for ISO13320	Guarantees quality and compliance

OMNISEC

The most advanced multi-detector GPC/SEC system

OMNISEC is a Gel Permeation Chromatography (GPC) / Size Exclusion Chromatography (SEC) system for the measurement of absolute molecular weight, molecular size, intrinsic viscosity, branching and other parameters.

The system includes OMNISEC RESOLVE, the integrated GPC/SEC module, OMNISEC REVEAL, the integrated multi-detector module and OMNISEC software, for the characterization of synthetic and natural polymers.

OMNISEC can accurately measure the most important characterization parameters, including:

- Absolute molecular weight and molecular weight distribution
- Intrinsic viscosity and molecular structure

Features	Benefits
Highly sensitive light scattering (LS) detector	Measure molecular weights of polymers, oligomers and waxes down to 200 g/mol
Integrated differential viscometer	Measure intrinsic viscosity (IV) to investigate molecular structure and branching
Temperature controlled detectors	Achieve better baseline stability for improved accuracy and sensitivity
Workflow oriented software	Makes GPC/SEC measurements and analysis as easy and intuitive as possible
Integrated column oven	Improve separation quality and resolution
Refractive Index (RI) detector	Measure the concentration of almost any solute
Triple detection (RI, IV and LS)	Combine data to determine as hydrodynamic radius (Rh), radius of gyration (Rg) and Mark-Houwink parameters

Epsilon

Benchtop XRF spectrometers for simple elemental analysis

X-ray fluorescence (XRF) is routinely used by metal powder producers and component manufacturers for determining the elemental composition of metal alloys and for detecting the presence of contaminants. Elemental composition is particularly important for alloys since small variations in the concentration of alloying elements can affect its chemical and physical properties, including strength, hardness, fatigue life and chemical resistance.

The Epsilon range of Energy Dispersive X-ray fluorescence (EDXRF) analyzers are capable of simple element identification and quantification from sodium (Na) to americium (Am). They are easy to operate, compact and X-ray safe instruments without the need for additional chemicals. Considerable savings in time and cost are two of the many benefits EDXRF can bring compared to alternative analytical techniques such as ICP.

Features	Benefits
Analysis with/without reference standards	Can be calibrated with reference materials for most accurate data or without for samples with unknown composition
Versatile sample presentation	Can analyze sample types including liquids and slurries, pressed powders, loose powders, fused beads and irregularly shaped objects
Both systems have compact design with Epsilon 1 having a built-in computer and touchscreen.	Requires less bench space and allows for easy and direct instrument operation in the lab or at-line
Epsilon 4 has optional SDD^{Ultra} detector for ultra light element analysis	Analysis of ultra light elements such as carbon, nitrogen and oxygen down to 1 wt%
Epsilon 4 has a 10-position removable sample changer with spinner	Automatic processing of sample batches and minimal errors caused by non-homogeneity of sample
Epsilon 1 has optional small spot capability (1 mm spot size) with camera for easy positioning	Quantify inclusions and contaminants to determine their origin

Zetium

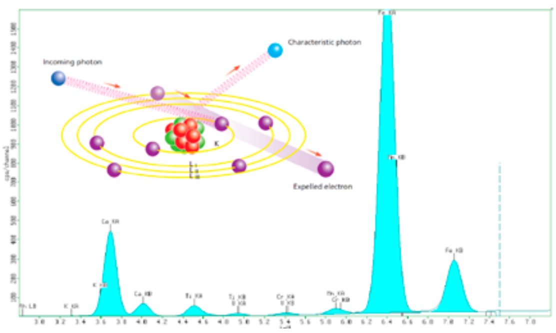
Floor-standing XRF for elemental excellence

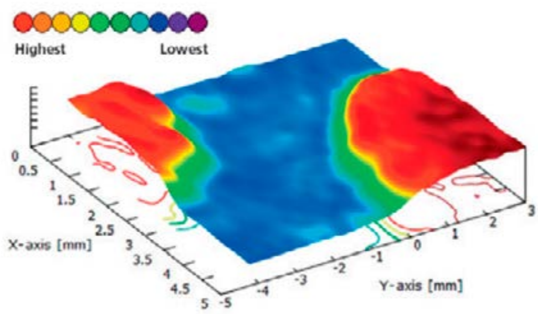
Designed to meet the most demanding process control and R&D applications, the Zetium spectrometer leads the market in high-quality design and innovative features for quantitative analysis of elements ranging from beryllium (Be) to americum (Am), and is the preferred choice when light element analysis and analytical precision is important. The modular design also allows for various task-oriented enhancements to meet specific application requirements.

Zetium uses SumXcore technology which brings together the benefits of WDXRF (Wavelength Dispersive) and EDXRF (Energy Dispersive) XRF with unique advantages for the analysis of metals, including:

- reduction in analysis time and improved precision
- identification of unexpected elements in samples

For inclusion or contaminant detection, small spot mapping and elemental distribution analysis can be performed.





Features	Benefits
Analysis with/without reference standards	Can be calibrated with reference materials for most accurate data or without for samples with unknown composition
Versatile sample presentation	Can handle sample types including pressed powders, loose powders, fused beads and irregularly shaped objects
High-capacity sample changer	High-throughput analysis of up to 240 samples per 8-hour shift
System enhancement packages	Can be easily adapted and upgraded to meet future testing requirement
SuperQ software with Virtual Analyst	Simple and intuitive task-oriented workflow with expert guidance for ease-of-use
Small spot analysis with elemental mapping	Determine spatial distribution of elements and presence of inclusions or contaminants
SumXcore - Combined WD/ED XRF	Reduced measurement times with improved precision and sensitivity

Empyrean

The multi-purpose X-ray diffractometer

Empyrean is a multipurpose X-ray diffractometer for determining the micro-structural characteristics of metal powders and their fabricated components, including;

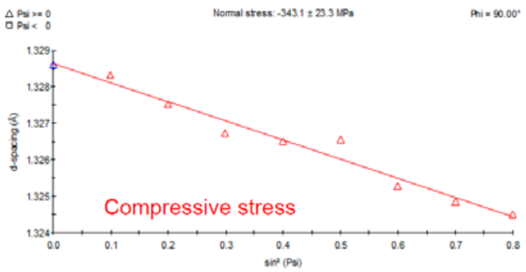
- phase composition
- crystallite size
- texture (crystallite orientation)
- residual stress/strain

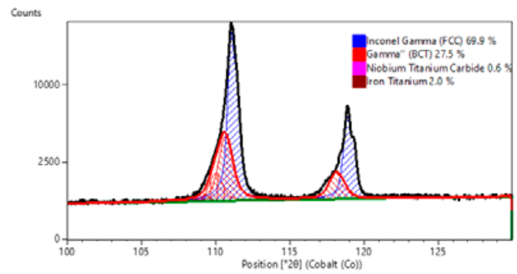
These characteristics can affect properties such as hardness, strength and fatigue life, and are influenced by thermal and mechanical processes such as atomization, laser melting and powder pressing.

Empyrean features a MultiCore Optics system that enables a large variety of different measurements to be performed automatically without manual intervention.

Empyrean has the unique ability to measure a variety of sample types including powders, thin films, nanomaterials and solid objects, over a wide range of temperatures.

It has an unrivaled collection of sample stages and optical components, including a tensile stage for sample analysis under stress, that can be easily interchanged to allow a wide range of diffraction, scattering and imaging applications, including computed tomography, to be performed.





Features	Benefits
MultiCore optics (iCore and dCore)	Automates measurements including multiple measurement geometries to maximize instrument utilization and obtain a more complete understanding of your materials
Pre-aligned fast interchangeable X-ray modules (PreFIX)	Enables the diffractometer to be reconfigured in a few minutes without module re-alignment, saving time and effort
Wide range of sample types	Can measure all sample types - from powders to thin films, from nanomaterials to solid objects - even during tensile testing
Full suite of configurable modules and measurements including a tensile stage	Provides access to a wide range of diffraction, scattering and imaging applications on a single platform
A range of non-ambient chambers for analysis at temperatures ranging from -260°C to 2000°C and under different atmospheric conditions	Study the effect of temperature, pressure, gas atmosphere and mechanical stress on phase transformation, chemical reactions, recrystallization, etc.
HighScore/HighScore Plus Software	Provides comprehensive phase identification, profile fitting, Rietveld, crystallographic and extended cluster analysis

Aeris

Phase analysis made simple

Aeris is a user-friendly compact X-ray diffractometer that provides fast, reliable and accurate phase identification and quantification of your metal powders. Aeris makes X-ray diffraction simple and accessible to everyone.

The unique touch screen user interface lets you proceed effortlessly through the measurement process and results can also be analyzed in the HighScore software to provide a wealth of crystallographic information.

Aeris' low cost of ownership delivers maximum return on investment since it has limited infrastructure requirements. For example, it has a small footprint and does not require compressed air or water cooling. It also has a much lower power consumption compared to floor standing systems and its X-ray tube has a virtually unlimited lifetime.

Aeris is also the first compact diffractometer to be fully automatable and can be easily incorporated into a relevant process.

Features	Benefits
Simple touch-screen user interface	Intuitive operation that make measurements quick and easy even for the novice
Minimal infrastructure requirements	No cooling water, no chiller, no compressed air - the only thing you need is a single-phase power socket
HighScore/HighScore Plus Software	Provides comprehensive phase identification, profile fitting, Rietveld, crystallographic and extended cluster analysis
Powerful search-match algorithm for multiple reference databases	Optimizes search criteria across a wide range of reference databases making it easier to identify minor phases in the diffractogram
Automatable bench-top that can connect to a belt	Automate a process or perform high-throughput analysis

Sample preparation equipment

What is fusion and why should I use it in my laboratory?

Fusion is a sample preparation method developed in the mid 50s. It involves dissolving a fully oxidized sample (at high temperature) in a suitable solvent (a flux) in a platinum, zirconium or graphite crucible. The melted mixture is agitated and poured into a mold to create a glass disk for XRF analysis. It can also be poured into a beaker to create a solution for AA or ICP analysis.

This universal technique has numerous benefits when you compare it with other sample preparation methods such as pressed pellets or acid digestion. One of the main advantages of using fusion in the preparation of metals is that the technique is not affected by mineralogical or particle size effects, thus enhancing the precision and accuracy of XRF results.

Why should I use fusion in my lab?

This universal technique has numerous benefits when you compare it with other sample preparation methods such as pressed pellets or acid digestion

	Fusion	Pressed pellets
Affected by mineralogy	No	Yes
Affected by particle size	No	Yes
Desirable size of powder (microns)	50 - 100 (Easy)	5 - 30 (Difficult)
Accuracy	≤1%	≤10%
Easy clibration with synthetic standards	Yes	No
Application of matrix correction	Yes	No

LeNeo fusion instrument prepares glass disks for XRF analysis as well as borate and peroxide solutions for AA and ICP analysis. This automatic electric instrument has one fusion position and is easy to use. It guarantees safety for the operator as well as superior analytical performance in the laboratory.

TheOx Advanced fusion instrument has been designed by our experts to suit our customers' ever-changing needs. This instrument is powered by electricity and has six fusion positions. It is used to prepare glass disks for XRF analysis as well as borate and peroxide solutions for AA and ICP analysis. Its extra features enhance analytical performance and safety, benefiting users at all levels.

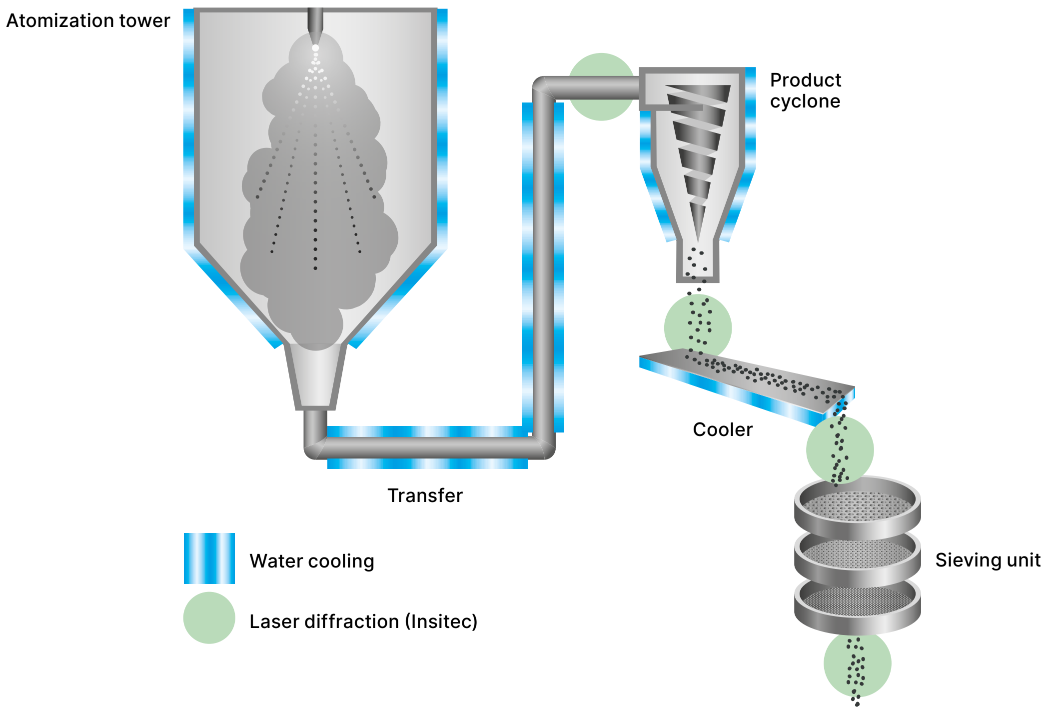
The Eagon 2 is a fully automatic fusion instrument that prepares glass disks for XRF analysis. Its innovative patented design ensures performance, operator safety and ease of use. The Eagon 2 instrument makes fusion and the consequent benefits for accurate XRF analysis easily achievable.

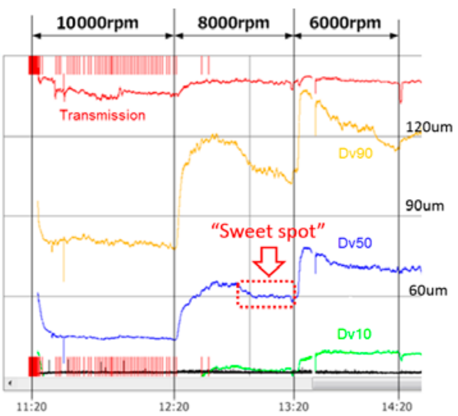
Real-time particle sizing

A watchful eye to optimize your process

Manufacturing high quality metal powders economically demands exemplary process control. Established in-line and on-line particle sizing technologies provide continuous process monitoring and a data stream that supports complete process automation. Such technology therefore makes it easier to make highly consistent products to a tight particle size specification at the lowest possible variable cost.

At the same time, continuous particle sizing has a role to play in managing the re-use of high-value AM powders, a critical issue for economic viability. With a proven track record and strong reliability, in-line and on-line particle sizing technology holds considerable value for metal powder manufacturers establishing robust supply chains for the PM industry, and for AM users looking to optimize their powder management processes.





Real-time measurement system makes it possible to instantly observe the effect of decreasing the speed of rotation of the atomizing disk and the time taken for the process to re-establish a steady state. (Green Dv10, blue Dv50, orange Dv90, red is transmission).

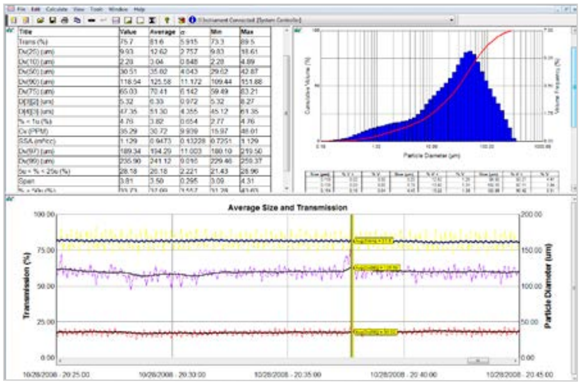
Insitec

Robust, reliable, real-time particle sizing for process control

Insitec analyzers are on-line laser diffraction systems that provide continuous particle size analysis, for efficient, cost-effective monitoring and control of industrial process streams. This includes dry powders, slurries, and sprays in the size range 0.1 µm to 2.5 mm.

Insitec provides real-time particle size distribution for automatic 'closed loop' process control, enabling the operator to optimize and control processes such as spray drying, atomization, milling and screening, and react in real-time to production fluctuations.

Malvern Link™ II software interface, enables full integration with your plant control system and unlocks the potential value of timely particle size measurement. The dedicated analysis software, automation and data reporting can be customized to meet individual requirements.



Features	Benefits
Measure particles in the size range 0.1 µm – 2500 µm.	Enable accurate measurement of metal powder from a range of processes
Optical head and sample path certified to Pressure Shock Resistance of 11bar(a) (PSR11)	Withstands extreme conditions of metal powder production
Ceramic linings protect critical, high-velocity areas of the sample pathway and purging protects windows from dust	Maintenance requirements are minimal
Provides continuous particle size data	Operators can react quickly and confidently when making control decisions
Meets all the recommendations for ISO13320	Guarantees quality and compliance
Measures representative sample volume	Statistical reliability for process control
Control system integration options – OPC, Modbus, Profibus, wired IO	Ease of reporting
Integrated software interface for plant automation	Deliver results in real-time to your control room and allow automation of routine tasks such as cleaning, maintenance and background checks

Parsum

Real-time size measurements for screening applications

The Parsum probe measures particle size distributions and velocities of solid particles in gas streams using a patented spatial filtering velocimetry technique. It has a size range of 50 µm to 6 mm and is widely used for monitoring and optimization of screening operations. Real-time particle distribution data enables the user to reduce variability during processing and detect screen breakages by monitoring for oversized particles.

The probe contains an array of light sensitive detectors which are illuminated by a laser. This array can detect single particles as they pass through the laser beam within the probe measurement zone. The shadow produced by each particle can be used to calculate the particle velocity and its chord length (particle size).

The detector signals are sampled very rapidly (up to 10,000 particles per second) and particle size distribution data is continuously updated during operation to produce a real time particle size trend. This provides direct insight into the performance of the process without the need for sample extraction.

Features	Benefits
Real-time measurement of particles in the size range 50 µm to 6 mm during processing	Continuous and uninterrupted monitoring of processes such as sieving, pneumatic transport and mixing
Measure up to 20,000 particles in real-time	Fast and statistically representative data for confident decision making and optimum process control
Wide range of accessories, such as inline dispersers and cleaning cells	Provides a so-called “process interface” for adapting the measurement probe to different conditions within the process.
Range of different probes for various processes and environments	Make real-time measurements inside mixing vessels, under sieve-decks and during conveying, even in ATEX zones
IPP measuring software	Flexible and adaptable system of programmes and interfaces that can control 4 probes simultaneously and gives real-time updates of particle size distributions and sieve fractions/throughputs

Rheology

Capillary and rotational rheometry for process-relevant material testing

Rosand capillary rheometers allow the characterization of polymer or suspension viscosities at shear rates and temperatures applicable for extrusion and injection processes. During MIM, shear rates in the range of 100 s^{-1} to 100,000 s^{-1} are created, which can be covered with the Rosand capillary rheometers.

Twin bore barrels enable absolute shear viscosity measurements and simultaneous measurement of extensional viscosities. Rosand Flowmaster software also provides a comprehensive data acquisition and analysis package for determining properties such as thermal stability, wall slip and melt fracture.

Kinexus Prime is an advanced rotational rheometer that can measure complex shear rheology, including viscoelasticity, on all material types from liquids through to solids.

Rotational rheometry is ideal for evaluating changes in thermal properties due to compositional or microstructural differences and can help to optimize product and process performance.

Kinexus Prime is capable of measuring time, temperature and low shear properties of materials in oscillatory shear using small sample volumes. It can measure temperature transitions of binders and the effect of particle composition on feedstock viscoelasticity. Other rheological properties such as yield stress, creep and stress relaxation can also be measured. Advanced Axial Capabilities allow Squeeze Flow tests to be performed as well as thermal expansion or shrinkage of the feedstock to be determined.

Thermal analysis

Relevant thermal analysis solutions for metals and alloys

The thermal behavior and thermophysical properties of metal powders is an important criterion for the quality of products manufactured by 3D printing. Heat transfer characteristics during, e.g., selective laser melting process, the temperature distribution and molten pool dimensions build the base of modeling.

Therefore, the thermal behavior (melting, crystallization, etc.) and thermophysical properties such as thermal diffusivity, thermal conductivity, specific heat capacity, and density should be known.

These properties influence the linear energy density, volume shrinkage, scanning track length, hatch spacing and time interval between two neighboring tracks on the temperature distribution and molten pool dimensions.

The results of thermoanalytical measurements support the improvement of temperature distribution and molten pool dimensions.

Shorter track, shorter hatch-spacing and less time interval can lower the temperature gradient and increase the temperature of each track. The thermophysical properties provide favorable values for the numerical simulation.

Method	Recordable Information	Temperature Range
Thermogravimetric Analysis (TGA)	Mass changes, thermal stability, decomposition	RT to 2400°C
Differential Scanning Calorimetry (DSC)	Phase transformation temperatures and enthalpies, specific heat capacity	-180 to 1650°C
Simultaneous Thermal Analysis (STA)	Phase transformation temperatures and enthalpies, specific heat capacity, mass changes, thermal stability	-150 to 2400°C
Gas Analysis (EGA) Coupled to Thermal Analysis	Characterization of gases emitted by means of MS, FT-IR, GC-MS	-180 to 2000°C
Dilatometry (DIL) / Thermomechanical Analysis (TMA)	Dimensional changes, CTE (Coefficient of linear thermal expansion), density changes	-180 to 2800°C
Laser / Light Flash Analysis (LFA)	Thermal conductivity and thermal diffusivity	-125 to 2800°C
Seebeck Coefficient (SBA)	Seebeck coefficient, electrical conductivity	-125 to 1100°C

Differential scanning calorimetry

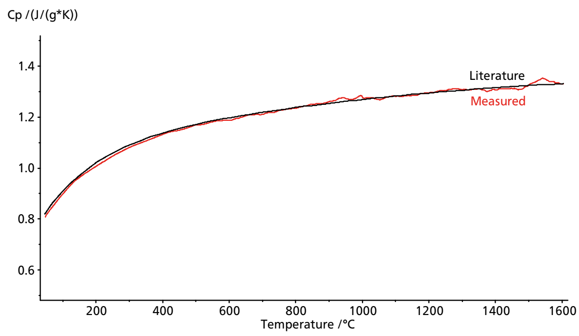
Unlike traditional metals manufacturing processes, metals manufactured by Additive Manufacturing (AM) undergo localized thermal cycles during fabrication. As a result, the liquid-solid phase transformation requires control. In addition, thermal cycles create a challenge with respect to solid-solid phase transformations. These thermal effects can be investigated by means of Differential Scanning Calorimetry (DSC). In accordance with ISO 11357-1, a sample and a reference are subjected to a controlled temperature program (heating, cooling or isothermal).

The actual measured properties are the temperature of the sample and the temperature difference between the sample and reference. From the raw data signals, the difference in heat flow between the sample and reference can be determined.

Measurement results by DSC

- Specific heat capacity (cp)
- Melting temperatures and enthalpies (heat of fusion)
- Crystallization temperatures and enthalpies
- Post-crystallization
- Reaction temperatures and enthalpies
- Phase transitions (solid-solid, solid-liquid, phase diagrams)
- Eutectic purity
- Liquid crystal transitions
- Oxidative stability
- Decomposition onset
- Kinetic studies

DSC 404 ***F3*** *Pegasus®*



Highly accurate determination of the specific heat capacity of a polycrystalline Al sample across a wide temperature range.

Features	Benefits
Wide temperature range from -150 to 2000°C	DSC and DTA measurements on a broad range of metals and alloys
Variety of furnaces and sensors easily interchangeable by the user	For each application the right furnace and sensor combination; future-proof for upcoming applications
Vacuum-tight design	Measurements under reduced pressures and pure atmospheres
Oxygen trap system (OTS®)	Avoiding oxidation of oxygen-sensitive materials
Determination of the specific heat capacity (cp)	Determination of a thermophysical property for developing performance data and optimum material design

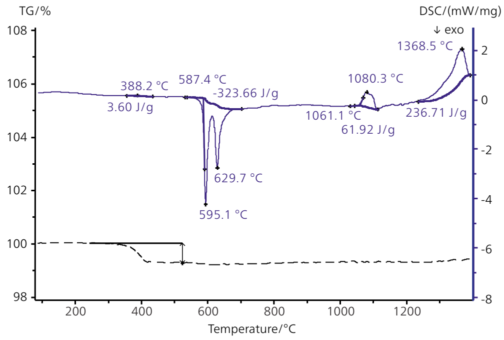
Simultaneous thermal analysis

Hyphenated techniques for more insight

Simultaneous Thermal Analysis (STA) generally refers to the simultaneous application of Thermogravimetry (TGA) and Differential Scanning Calorimetry (DSC) to one and the same sample in a single instrument. The advantages are obvious: The test conditions are identical for both the TGA and DSC signals (same atmosphere, gas flow rate, water vapor pressure, heating rate, thermal contact with the sample crucible and sensor, radiation effect, etc.). Furthermore, sample throughput is improved as more information is gathered from each test run.

Coupling Gas Analyzers to Thermal Analyzers

By coupling a Thermobalance (TG, TGA), Simultaneous Thermal Analyzer (STA) or Dilatometer (DIL) with a Quadrupole Mass Spectrometer (QMS), it is possible to detect and identify evolved gases in exact time.



STA measurement on amorphous Fe alloy; the DSC curve shows endo- and exothermal effects and the TGA curve exhibits the mass loss behavior.

Measurement results by TGA

- Mass changes
- Temperature stability
- Corrosion studies
- Oxidation/reduction behavior
- Decomposition
- Compositional analysis
- Kinetic studies
- Rate-controlled mass lossDecomposition onset
- Kinetic studies

Measurement results by hyphenation

- Decomposition stability
- Solid-Gas Reactions
- Compositional Analysis
- Identification
- Evaporation

STA 449 ***F1*** *Jupiter®* coupled to QMS 403 *Aëolos®*

Features (STA 449 ***F1/F3/F5 Jupiter®***)	**Benefits**
Wide temperature range from -150 to 2400°C	Combined DSC/DTA-TG measurements, two analytical methods applied to one and the same sample
Variety of furnaces and sensors easily interchangeable by the user	For each application the right furnace and sensor combination; future-proof for upcoming applications
Water-vapor furnace	Tests in humid atmospheres up to 1250°C (e.g., corrosion studies)
Vacuum-tight design	Measurements under reduced pressures and pure atmospheres
Oxygen trap system (OTS®)	Avoiding oxidation of oxygen-sensitive materials
Temperature-modulated DSC	Separation between reversible and non-reversible thermal effects for an improved resolution and sensitivity
Determination of the specific heat capacity (cp)	Determination of a thermophysical property for developing performance data and optimum material design
Coupling to gas analyzer systems	Deep insight and better understanding of thermal reactions
Wide temperature range from -150 to 2400°C	Combined DSC/DTA-TG measurements, two analytical methods applied to one and the same sample

Dilatometry

Each time a material is exposed to temperature changes – it shows a variation in its dimension. Whether it is in the course of its regular thermal expansion, by passing a phase transition or while it undergoes sintering, the substance will either be shrunk or elongated.

Dilatometry (DIL) is the method of choice to study length change phenomena, thus revealing information regarding their thermal behavior and about process parameters or sintering (and curing) kinetics.

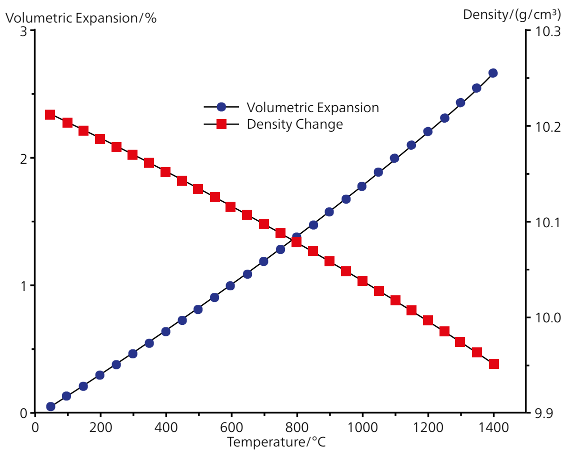
Due to the wide dynamic range of the measurement system of the NETZSCH DIL 402 *Expedis®* series, it is possible to measure both soft and hard samples without impairment of the properties in the temperature range from -180°C to 2000°C (depending on furnace). Additionally, it enables force modulation and builds a bridge to thermo-mechanical analysis (TMA).

All NETZSCH dilatometers are based on, e.g., DIN EN 821, DIN 51045, ASTM E831, ASTM E228.

Measurement Results by Dilatometery

- Linear thermal expansion and coefficient of thermal expansion (CTE)
- Volumetric expansion and density change
- Shrinkage steps
- Phase transitions
- Sintering temperatures and steps
- Influence of additives and raw materials
- Debinding
- Anisotropic behavior
- Rate-Controlled Sintering (RCS)
- Kinetic analysis (Kinetics Neo)

DIL 402 *Expedis® Supreme*



Density determination based on volumetric expansion

Features	Benefits
Friction-free construction	Small contact forces for green bodies and soft samples
High measuring range	Measurement of large length changes (expansion, shrinkage)
Highest temperature range from -180°C to 2800°C	Measurements on high-melting materials
Controlled contact force	Constant sample load during the entire measurement
Measurements in different atmospheres	Oxidizing reducing and inert atmospheres and under vacuum

Laser / light flash analysis

Researchers and engineers are interested in the best way to thermally characterize highly conductive materials at cryogenic and elevated temperatures. Reduced development times and costs, optimization of the manufacturing processes and lower masses in spite of increasingly higher demands on thermally stressed components are important goals in industry. Many challenges can only be met with precise knowledge of two fundamental thermal properties: thermal diffusivity and thermal conductivity.

Laser/Light Flash Analysis (LFA) allows for the meeting of challenges typically arising in heat transfer processes such as, e.g., determining how quickly an aluminum ingot solidifies, figuring the temperature gradient in a part during use, determining the thermophysical properties of parts, etc.

LFA 467 HT *HyperFlash*®

Over the past two decades, NETZSCH has led the way in this technology, extending our application range from -125°C to 2800°C.

In carrying out a LFA measurement, the lower surface of a plane-parallel sample is first heated by a short energy pulse. The resulting temperature change on the upper surface of the sample is then measured with an infrared detector. The thermal diffusivity (a) and finally the thermal conductivity (λ) can be calculated by means of the following formula:

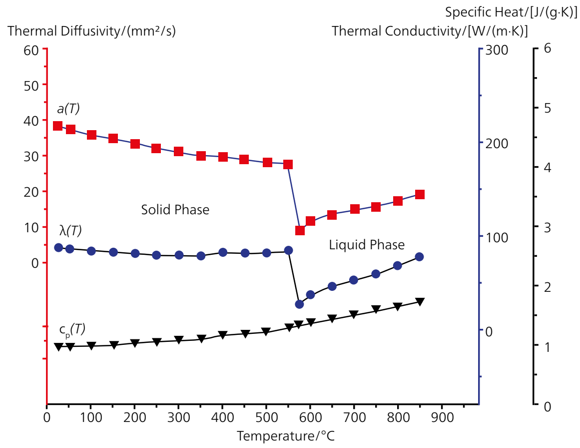
$$\lambda(T) = a(T) \cdot c_p(T) \cdot \rho(T)$$

λ = thermal conductivity [W/(m·K)]

a = thermal diffusivity [mm²/s]

c_p = specific heat capacity [J/(g·K)]

ρ = bulk density [g/cm^3]



Thermal diffusivity by means of LFA (red), specific heat capacity by means of DSC (corrected by subtracting the latent heat of melting, black) and the calculated thermal conductivity (blue)

Features	Benefits
Temperature range RT ... 2800°C	Various instruments for a broad application range
Adjustable flash pulse and maximum measurement time	Measurements on high-conducting and/or thin specimens up to thicker specimens becomes possible
Specimen holders	Measurements on small and large specimens up to 25,4 mm possible, different shapes and geometries are allowed
Variety of software models for the evaluation of the signals	Each model can be combined with different baseline corrections and with or without pulse correction, radial and facial heat loss, model wizard

About Malvern Panalytical

We draw on the power of our analytical instruments and services to make the invisible visible and the impossible possible.

Through the chemical, physical and structural analysis of materials, our high precision analytical systems and top-notch services support our customers in creating a better world. We help them improve everything from the energies that power us and the materials we build with, to the medicines that cure us and the foods we enjoy.

We partner with many of the world's biggest companies, universities and research organizations. They value us not only for the power of our solutions, but also for the depth of our expertise, collaboration and integrity.

We are committed to Net Zero in our own operations by 2030 and in our total value chain by 2040. This is woven into the fabric of our business, and we help our employees and customers think about their part in creating a healthier, cleaner, and more productive world.

With over 2300 employees, we serve the world, and we are part of Spectris plc, the world-leading precision measurement group.

Malvern Panalytical. We're BIG on small™

NETZSCH Analyzing and Testing

The NETZSCH Group is an owner-managed, international technology company with headquarters in Germany. The Business Units Analyzing & Testing, Grinding & Dispersing and Pumps & Systems represent customized solutions at the highest level. More than 4,000 employees in 36 countries and a worldwide sales and service network ensure customer proximity and competent service.

Our performance standards are high. We promise our customers Proven Excellence – exceptional performance in everything we do, proven time and again since 1873.

When it comes to Thermal Analysis, Accelerating Rate Calorimetry, the determination of Thermophysical Properties, Rheology and Fire Testing, NETZSCH has it covered.

Our 60 years of applications experience, broad state-of-the-art product line and comprehensive service offerings ensure that our solutions will not only meet your every requirement but also exceed your every expectation.

Elevate the understanding of materials

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