Malvern | Material relationships

- PARTICLE SIZE
- PARTICLE SHAPE
- MOLECULAR WEIGHT
- MOLECULAR SIZE
- MOLECULAR STRUCTURE
- SOLUTION VISCOSITY
- RHEOLOGICAL PROPERTIES

MATERIAL CHARACTERIZATION SOLUTIONS FOR POWDER METALLURGY

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 or electron beam.

There are a number of reasons for using a powder metallurgy processes 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.

Benefits of PM

By producing parts with a homogeneous structure the PM process enables manufacturers to make products that are more consistent and predictable in their behavior across a wide range of applications.

In addition the PM Process has a high degree of flexibility allowing the tailoring of the physical characteristics of a product to suit your specific property and performance requirements.

European Powder Metallurgy Association.

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:

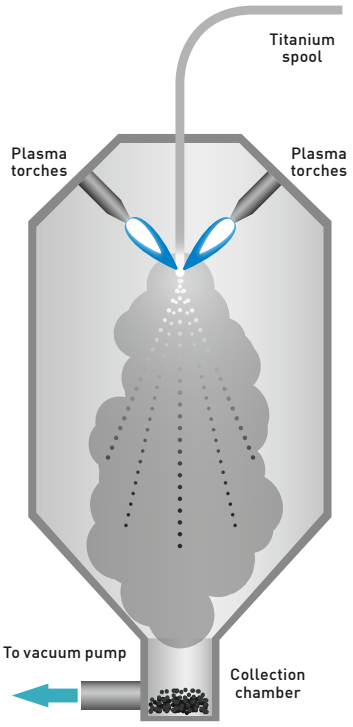
- Comminution of solid metal
- Precipitation of a salt from solution
- Thermal decomposition of metal carbonyl (Carbonyl process)
- Solid state reduction of metal oxide
- Electrodeposition
- Atomization of molten metal

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
- Material structure
- Surface condition

Which powder characteristics are most important depends on the particular powder metallurgy process.



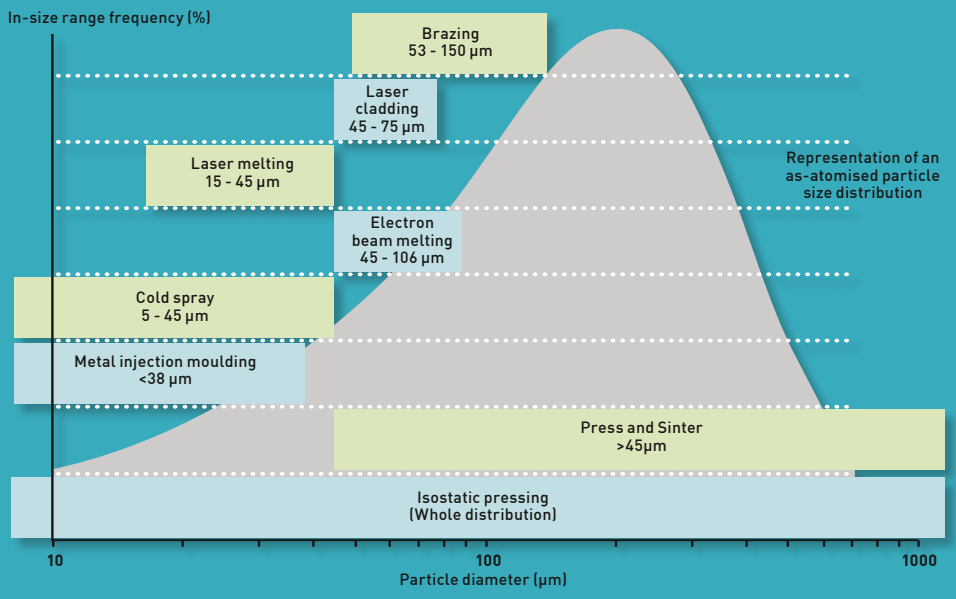
What's the optimal particle size distribution for powder metallurgy?

Every fraction of the particle size distribution plays a role in powder metallurgy processes but to different extents:

Fine particles flow less readily than larger particles so are less favored for dry powder processes where flow characteristics are important

Finer particles give better packing and are more readily sintered so are favored for extrusion processes such as MIM

Larger particles pack less efficiently but are more easily compressed hence are favored for pressing processes.



PRESS AND SINTER

Press and sinter is the most conventional manufacturing method associated with powder metallurgy and involves three basic steps

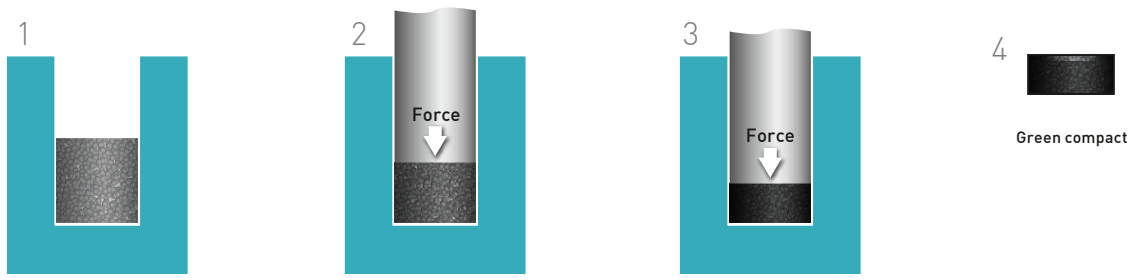
1. Powder blending - metal powder is mixed with a suitable lubricant
2. Die compaction - the powder blend is pressed in a die to form a compact 'green body'
3. Sintering – the 'green body' is heated to below its melting point to form the sintered component

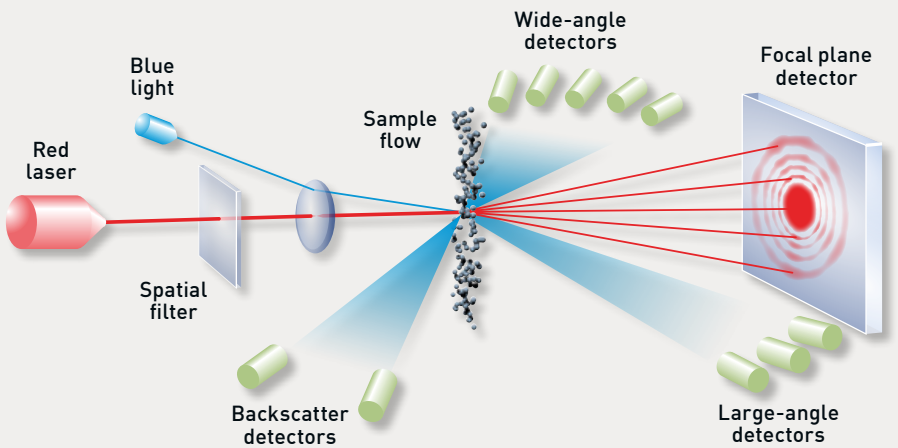
Powder characteristics are important for press and sinter, as for other powder metallurgy processes, with particle packing and apparent density of the powder blend critical.

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.

Malvern'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
- Ensure optimum contact between particles for more efficient sintering
- 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





Laser diffraction

In a laser diffraction measurement a laser beam passes through a dispersed particulate sample and the angular variation in intensity of the scattered light is measured. Large particles scatter light at small angles relative to the laser beam and small particles scatter light at large angles.

The angular scattering intensity data is then analyzed to calculate the size of the particles that created the scattering pattern using the Mie theory of light scattering.

ADDITIVE MANUFACTURING

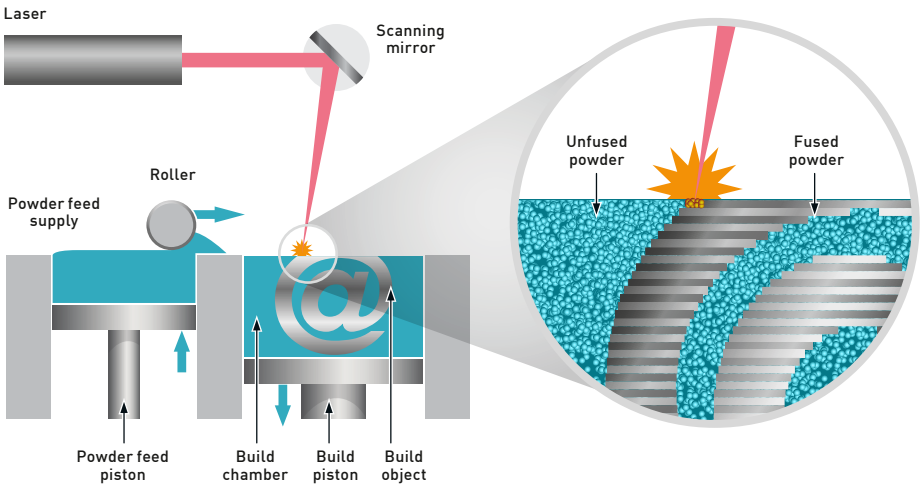
In powder bed fusion processes a metal powder layer is applied to a building platform and a laser or electron beam is used to selectively melt or fuse the powder. After melting, the platform is then lowered, and the process repeated continually until the build is complete. The unfused powder is removed 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 15-45 µm for SLM and 45-106 µm for EBM for example.

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

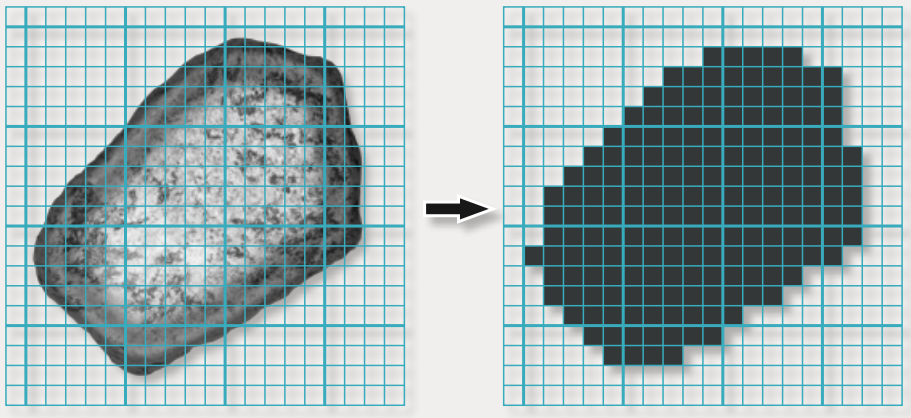
- Ensure a consistent powder supply and prevent variations in product quality
- Identify suitable powders for machines with different spreader or rake designs
- Optimize atomization conditions to achieve desired powder properties
- Predict and optimize powder packing density and flow characteristics
- Determine when a powder can no longer be reused or recycled



Automated imaging

Automated imaging techniques use a digital camera to capture 2D images of a dispersed particulate sample. Individual particle images are identified using digital thresholding techniques, and then analyzed to determine their size, shape and other physical properties such as transparency.

Automated imaging instruments typically measure tens to hundreds of thousands of particles in the same amount of time needed to measure a very small number by manual microscopy.



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.

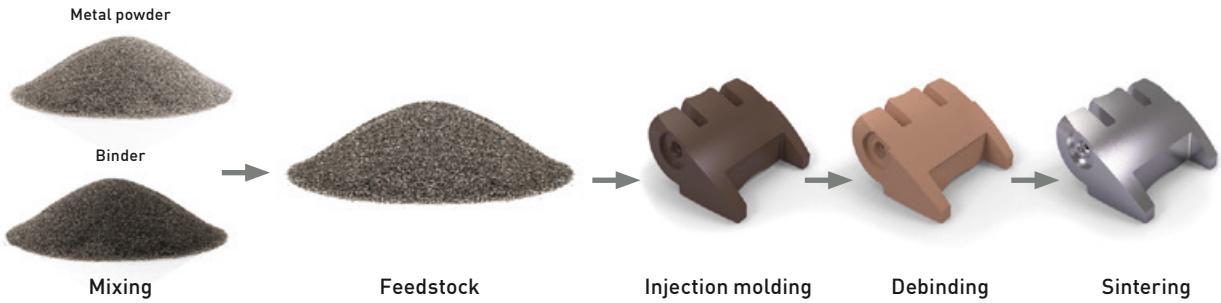
Rheological properties are influenced by a number of factors including binder and solids composition, particle size and shape, temperature and flow rate.

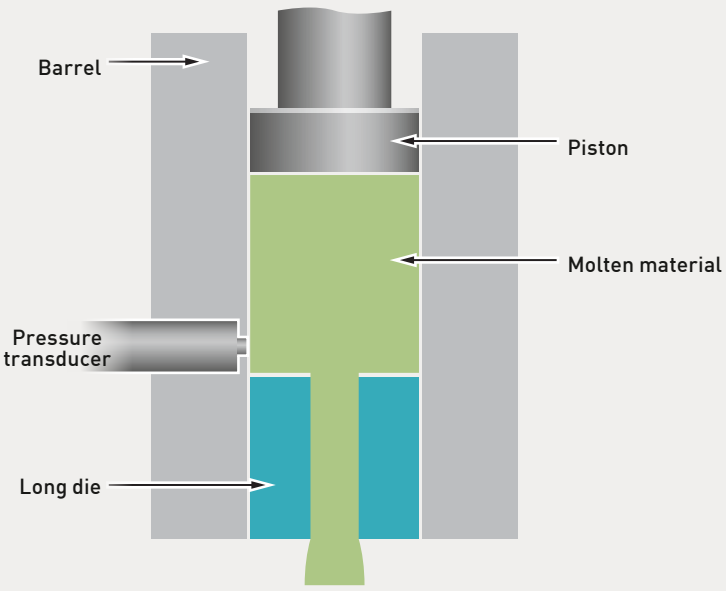
Most metals can be used in MIM if they have the correct powder properties, including particle size and shape.

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'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 for flow behavior and debinding performance
- Ensure consistent metal powder supply





Capillary rheometry

Based on controlled extrusion of a test material, capillary rheometry enables material flow and deformation properties to be characterized under process relevant conditions such as high force (or pressure), high shear rate and at elevated temperature.

The sample is extruded through a capillary die, and the resultant pressure is measured at the die entrance. Shear viscosity is calculated from knowledge of the capillary die dimensions, piston speed and pressure.

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.

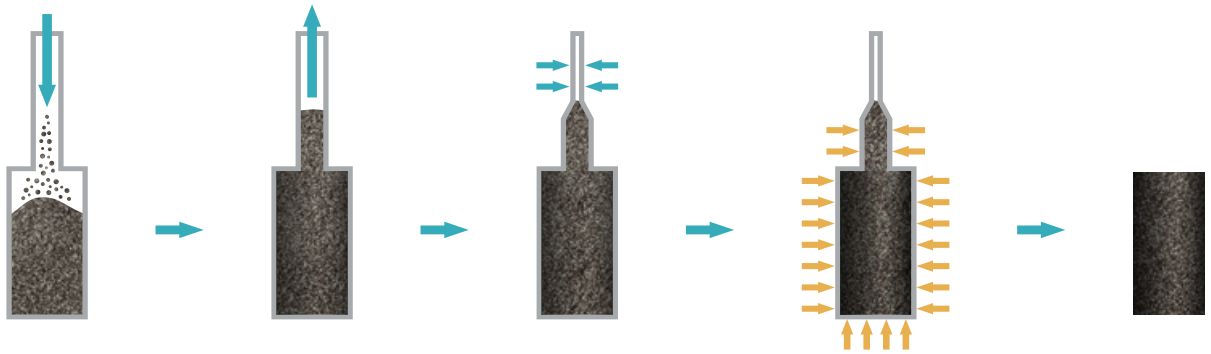
Isostatic pressing can be performed at elevated temperatures (Hot Isostatic Pressing HIP) or at ambient temperature (Cold Isostatic Pressing (CIP))

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.

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

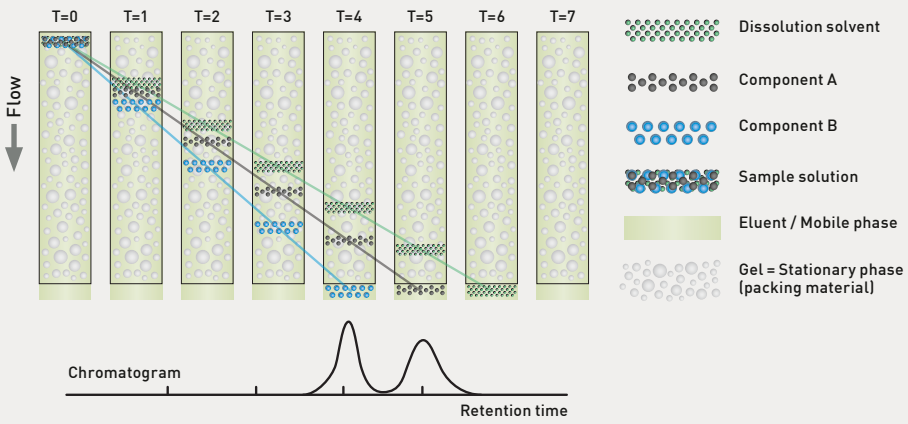
- Predict and control packing density and minimize void formation in the sintered part
- Specify and control metal powder quality
- Optimize powder flow in the mold and maintain process efficiency
- Reduce sintering time



Gel Permeation Chromatography

Gel Permeation Chromatography (GPC) is an analytical technique that separates dissolved macro-molecules by size based on their elution from columns filled with a porous gel.

When GPC is coupled with light scattering, viscometer and con-centration detectors (known as triple detection), it can measure absolute molecular weight, molecular size and intrinsic viscosity, and generate information on macromolecular structure, conformation, aggregation and branching of polymers and waxes.



MASTERSIZER **3000**

PARTICLE SIZE

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'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.

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

'The performance of the dry dispersion system allows us to measure powders in the dry state. This makes measurement and analysis fast, easy and efficient so we achieve excellent analytical productivity.'

Dr Robert Deffley
LPW Technology

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

MORPHOLOGI **G3**

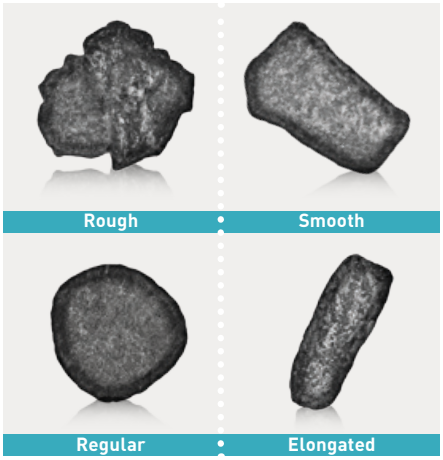
PARTICLE SHAPE

PARTICLE SIZE

Direct measurement of metal powder size and shape

The Morphologi G3 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 G3 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.



'We know that particle shape directly influences AM performance and so an efficient imaging system is an essential tool for us. In a comparative assessment the Morphologi G3 delivered the perfect balance between speed of measurement, image resolution and price.'

Dr Robert Deffley
LPW Technology

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 CCD 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

ROSAND

RHEOLOGICAL PROPERTIES

Capillary rheometers 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. The Rosand range includes a bench top instrument (RH2000) and floor standing instruments (RH7/RH10) with different drive force capabilities.

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 sample stability, wall slip and melt fracture.

A wide range of high precision tungsten carbide dies and interchangeable pressure transducers cover all materials and test types. Several accessories are available to enhance the testing capability of the base units, including;

- Nitrogen purge option
- Die swell measurement
- PVT test option

'The RH10 capillary rheometer has been reliable and repeatable for the classification of feedstock materials allowing us to further improve stability.'

Dr Ben Blackham
Sandvik PBT

Features	Benefits
Twin bore barrels supplied as standard	Simultaneous measurement of shear and extensional viscosity
Nitrogen purge option for testing in a dry, inert atmosphere	Minimize sample degradation/oxidation during testing
Wide dynamic speed range up to 1200 mm/min and up to 100 kN in drive force	Allows high viscosity and high shear rate measurements
Easy to use Flowmaster software supporting a range of test capabilities	Enables full hardware functionality, data analysis and export capabilities
Various accessories available for bench op and floor standing systems	Enhance the testing capability of the base units to meet application requirements
Range of dies available with different Length/Diameter ratios	Access range of shear rates and correct for wall slip and die entrance effects
PVT (Pressure, Volume and Temperature) measurement	Enables assessment of material compressibility
Selection of pressure transducers available covering different pressure ranges	Measure a range of shear stresses and hence range of viscosities

OMNISEC

The most advanced multi-detector GPC/SEC system

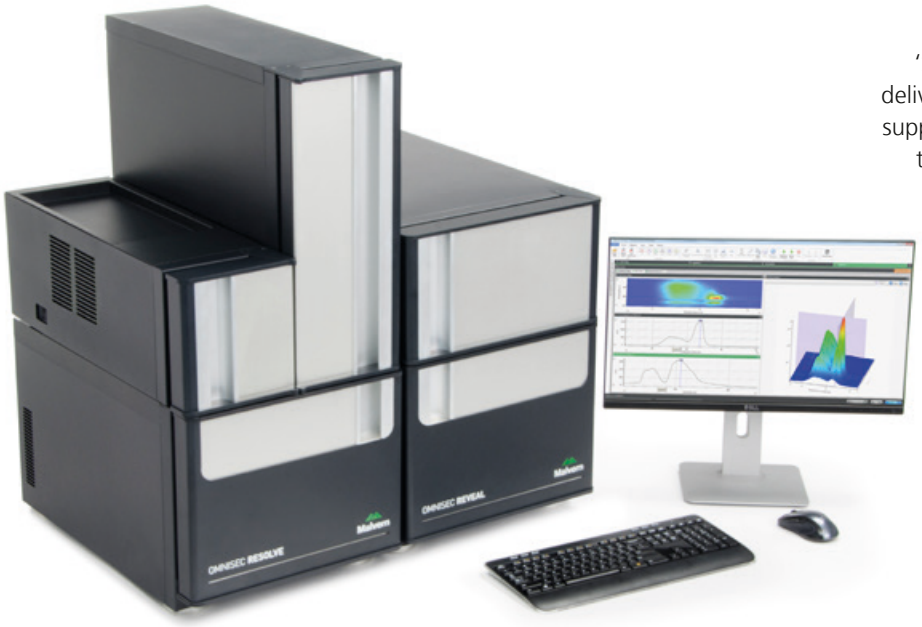
MOLECULAR WEIGHT
MOLECULAR SIZE
MOLECULAR STRUCTURE

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



'Since we purchased OMNISEC, we're able to deliver more sensitive GPC data, so our ability to support decisions around different suppliers and troubleshooting has been directly enhanced.'

Dr Kirt Durand
Syngenta

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

INSITEC

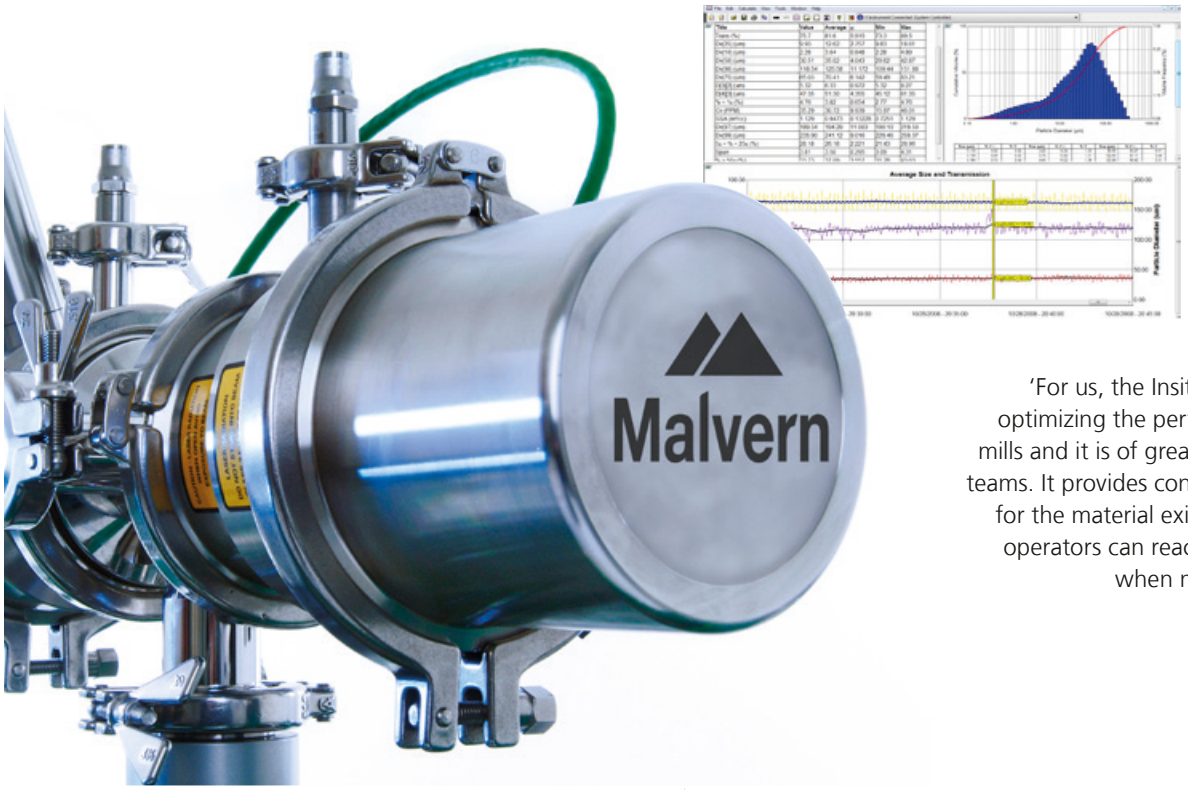
PARTICLE SIZE

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 including 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.



'For us, the Insitec is an essential tool for optimizing the performance of the finishing mills and it is of great value to the production teams. It provides continuous particle size data for the material exiting the mills, so that the operators can react quickly and confidently when making control decisions.'

Michel Berger
Holcim

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

KINEXUS

RHEOLOGICAL PROPERTIES

Viscosity and viscoelasticity of MIM feedstock materials

Kinexus is an advanced rotational rheometer that can measure complex shear rheology on all material types from liquids through to solids. Rotational rheometry is ideal for evaluating changes in feedstock properties due to compositional or microstructural differences and can therefore help to optimize product and process performance.

Kinexus is capable of measuring time, temperature and shear dependent properties of feedstock materials using small sample volumes. It can measure temperature transitions of binders and the effect of particle composition on feedstock viscosity. Measurements can be made quickly and easily making it a valuable formulation tool.

Common test modes are steady rotation to measure shear viscosity and oscillation to measure viscoelastic properties. Other rheological properties such as yield stress, thixotropy, creep and stress relaxation can also be measured. Advanced axial capabilities also allow properties such as tack and contraction to be determined.

'Kinexus helps us to study the rheological properties of our systems analytically and develop future formulations. It allows us to continually improve product quality, leading to a direct saving in time and costs.'

Dr. Daniel Kessler,
Steuler KCH Materials GmbH

Features	Benefits
Wide continuous torque range (0.5 nNm – 250 nNm)	Allows the measurement of low viscosity liquids through to stiff solids
Wide temperature range (- 40-300°C)	Measure temperature dependent material properties and transitions
Stress control, shear rate control and direct strain controlled oscillation	Allows a wide range of rheological test protocols to be performed
'Plug and play' accessories with auto-recognition	Quick accessory interchange and configuration with intelligent software feedback
Advanced software interface with SOP driven test protocols	Provides on screen guidance to users during a measurement
Integrated library of application specific test protocols	Help users to find and make relevant measurements easily
Advanced axial capabilities	Perform vertical testing such as penetration, tack and squeeze testing
Wide variety of measurement geometries and accessories available	Ability to use the full functionality of Kinexus for a wide range of axial and torsional testing

PARSUM

PARTICLE SIZE

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.

ABOUT MALVERN

Malvern Instruments is a leading provider of scientific instrumentation that delivers relevant and reliable information to enable our customers to address their business challenges. Underpinned by Malvern's extensive industry knowledge, technical expertise and applications understanding, Malvern's analytical characterization systems help customers to maximize productivity, develop better products and get them to market faster. They are applied across industry and academia, from fundamental research through to manufacturing and QC.

Malvern's analytical instruments address a wide variety of measurement needs, and they help users accelerate their research and better understand the materials they work with.

Understanding material properties is fundamental to predicting how products will behave during use, optimizing their performance and achieving manufacturing excellence.

Headquartered in Malvern, UK, Malvern Instruments has subsidiary organizations in all major European markets, North America, Mexico, China, Japan and Korea, a joint venture in India, a global distributor network and applications laboratories around the world.

VALIDATION AND SUPPORT

Purchasing a Malvern product is only the first stage of a collaborative relationship that lasts for the lifetime of the instrument. Malvern is dedicated to helping all customers maximize the return on their investment. We are committed to providing the highest quality support, from simply answering occasional questions and supplying software updates through to delivering a comprehensive range of support packages tailored to your needs.

Malvern has a wealth of scientific and technical expertise and experience, with applications and technical specialists available around the world. All are backed by truly comprehensive online resources that ensure every customer has access to information designed to help make their job easier.

Areas we work in:

- ACADEMIC BIOCHEMICAL RESEARCH
- BIOSCIENCE
- FOOD AND DRINK
- ASPHALT
- (BIO) PHARMACEUTICALS
- COSMETICS AND PERSONAL CARE
- CHEMICALS
- MINING AND MINERALS
- POWER GENERATION
- CEMENT
- METAL POWDERS
- PLASTICS AND POLYMERS
- SURFACE COATINGS
- ELECTRONICS
- CERAMICS
- ADHESIVES AND SEALANTS

Excellence through experience

Many Malvern systems are used in highly regulated environments, and product validation and R&D traceability are priorities for our customers. Operating to ISO9001: 2008 with TickIt accreditation for software development, Malvern is a major supplier to the highly demanding (bio) pharmaceutical and chemical industries. Malvern's products play pivotal roles in high quality research and manufacturing throughout the world.

As a global supplier, we understand our responsibility to minimize the impact we have on the environment, and operate to both ISO14001 and OHSA18001.

Validation

To help our customers comply with the requirements of the Regulatory Authorities, such as the US Food and Drugs Administration (FDA) and the Medicines and Healthcare products Regulatory Agency (MHRA), Malvern provides a comprehensive range of validation tools.

These aids follow a user's validation process through from Installation and Operational Qualification (IQ/OQ) to the maintenance phase with annual OQ renewals.

World-class service and support

Malvern offers professional support at all levels. Our intention is to increase your laboratory's productivity through the creation of a working relationship for the lifetime of your instrument, providing service, support, training and information.

- Global network of fully-trained service personnel
- Worldwide coordination for multinational companies
- Technical support from the Malvern Helpdesk via telephone or email
- Range of maintenance contracts and service agreements to cover all requirements
- Validation support
- Consultancy-based onsite training courses
- e-Learning training courses
- Classroom training courses
- Web seminars
- Sample and application consultancy

No other company offers more

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About Malvern Instruments

Malvern Instruments provides a range of complementary materials characterization tools that deliver inter-related measurements reflecting the complexities of particulates and disperse systems, nanomaterials and macromolecules. Analytical instruments from Malvern are used in the characterization of a wide variety of materials, from industrial bulk powders to the latest nanomaterials and delicate macromolecules. These systems deliver industrially relevant data enabling our customers to make the connection between micro (such as particle size) and macro (bulk) material properties (rheology) and chemical composition (chemical imaging).

Material Relationships - distributor details

Malvern Instruments is part of Spectris plc. Spectris is the parent company of 13 individual businesses with operations around the world that develop and market complementary precision instrumentation and controls. Like Malvern Instruments, PANalytical, the world's leading supplier of instrumentation and services for X-ray diffractometry (XRD) and X-ray fluorescence (XRF) spectrometry, is part of the Materials Analysis segment within Spectris. Spectris and the Spectris logo are trademarks of Spectris plc.

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