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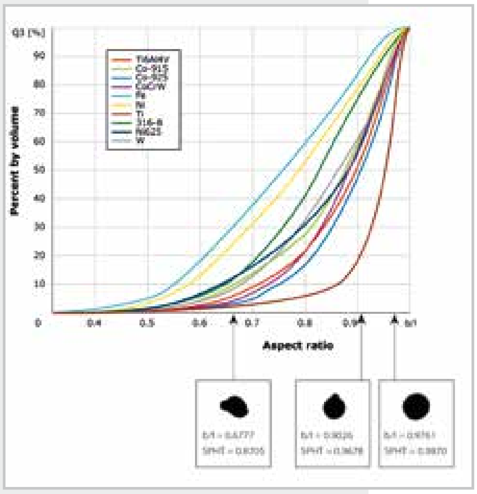
Ensuring High Quality in the Additive Manufacturing Process

Additive Manufacturing and Powder Injection Molding offer great potential - but to make sure that quality and cost of the manufactured parts are adequate, it is not only necessary to monitor the actual production process but also previous and subsequent steps.

The great potential of additive manufacturing (AM) consists in the ability to produce functional components directly from CAD data without any tools. Thus, prototype development is accelerated and becomes more cost-efficient. The complexity of the parts is virtually unlimited. This technology can even be used in the sense of industry 4.0 for the batch size 1 but also, in the case of Reverse Engineering, to manufacture spare parts for old machines at a reasonable cost. For serial production, however, AM is hardly suitable and clearly inferior to powder injection molding when metal is involved.

One should not make the mistake of considering additive manufacturing and powder injection molding as isolated processes. Both place **high demands on the raw material and further treatment**. Thanks to the pricey metal powders, **recycling** is also an important factor in the cost calculation.

Retsch Technology
PARTICLE CHARACTERIZATION



Particle shape analysis of 10 different metal powders with Dynamic Image Analysis (CAMSIZER X2). Even smallest percentages of irregularly shaped particles are reliably detected.

Focus on particle quality

The metal powders used for additive manufacturing should consist of perfectly spherical particles, preferably of identical size with an average between 10 and 50 microns. The particles required for metal injection molding (MIM) should also be spherical but not bigger than 10 microns in diameter. **Particles which are too big or irregular will with high probability cause defects in the finished components**. Hence, the raw material needs to be prepared and analyzed with great care.

The simplest method for particle sizing is mechanical sieve analysis which, however, is not suitable here as the smallest usable mesh size is 20 microns. Laser diffraction is better suited for measuring fine metal powders. It is easy to execute and provides quick results. The particle size distribution is calculated by a software which evaluates all particles as spherical.

There are two methods to analyze particle size and particle shape simultaneously: static and dynamic image analysis. Due to the static nature of the first, it is only possible to evaluate relatively small sample volumes with this method. With dynamic image analysis, however, the particles pass the camera transported by an air jet or a liquid. In this way, hundreds of thousands to millions of particles are automatically analyzed and thus provide a true image of the sample quality.

RETSCH Technology's CAMSIZER X2 uses Dynamic Image Analysis (DIA) for quick and accurate particle analysis of metal powders with excellent repeatability. In addition, the user obtains a wealth of material data, thus gaining deeper knowledge of the powder quality. The system works with two cameras with different magnifications, thus covering a wide measuring range from 0.8 µm to 8 mm.

A big advantage of DIA is the possibility to simultaneously measure particle size and **particle shape** (percentage of round/irregular particles, satellites, agglomerates, etc.). Even smallest percentages (~0.01%) of oversized or undersized grains or irregular particles are detected by the system within 1 to 3 minutes which allows for continuous quality control.

ELTRA
ELEMENTAL ANALYZERS

Carbon / Sulfur Analyzer
ELEMENTRAC CS-i

The purity of metal powders is key

The energy required by AM to melt metal powder not only depends on the size of the particles but also on the chemistry of the powder. With increasing carbon content, e. g. in steel (0.002 % - 2.06 %), hardness and brittleness also increase while the melting point drops. Sulfur improves the machinability of steel but reduces its ductility. Free nitrogen is localized along the grain boundaries and influences the ductility. Oxygen makes steel brittle and hydrogen reduces mechanical stability.

To analyze the purity with ELTRA's **ELEMENTRAC ONH-p** analyzer, a metal sample is melted, and the contained oxygen, nitrogen and hydrogen are released. **Oxygen** converts to CO and oxidizes to CO_2. The oxygen content is detected in an IR cell whereas **nitrogen** and **hydrogen** concentrations are measured in a thermal conductivity cell.

The thermal conductivity cell is based on a micromechanical silicon chip coupled to a membrane. If the thermal conductivity of the gas changes, the heating capacity required for heating the membrane changes as well. This is indicated by a measuring signal. This method is robust and **guarantees stable measuring results over a wide concentration range.**

In the induction furnace the sample is melted in a pure oxygen atmosphere; combustion gases are purified and sulfur dioxide is detected in the infrared measuring cells. After oxidation of CO to CO_2 and of sulfur dioxide to sulfur trioxide, the SO_3 gas is removed and the carbon content is reliably detected in the IR cells.

View into a metal retort of the GPCMA/174 with additive manufactured sample to be stress relieved

Heat treatment is essential

If the quality of the raw material is right, metal components can be additively manufactured in two ways: directly or indirectly. In the direct process, a laser beam melts a layer of metal powder (titanium alloy Ti6AI4V, stainless steel, etc.) which may be as thin as 20 microns. The high energy input and temperature gradients below the melt pool generate high residual stress in the manufactured part.

To relieve these stresses, heat treatment with precise temperature control is carried out. Due to the negative impact of oxygen, this process step happens under nitrogen or, in the case of titanium, under argon. With the **GPCMA Modified Atmosphere Chamber Furnace** and the **HTK Vacuum Furnace** CARBOLITE GERO offers perfectly suited solutions for this application.

For indirect AM, just like for powder injection molding with metals or ceramics, the starting powder is mixed with a binder and a Green Part is formed. In a next step, the binder is removed thermally, catalytically or with solvents. Now the powder is only held together by a backbone binder which makes the part very sensitive. The rest of the binder is then thermally removed under vacuum, air or inert gas and the part is sintered in the same furnace under the specifically required atmosphere. **The possibility to work under inert or reactive gases, high vacuum, or even ultra-high vacuum enables sintering of very sensitive materials.**

The vibratory sieve shaker
AS 200 basic is suitable for separating metal powders into size fractions

Recycling of valuable residues

Due to the way 3D printing with metal powders works, a considerable amount of excess powder is left after the printing process; this is partly agglomerated but the finer parts are usable for further printings. For this purpose, **the excess material is separated into fractions with vibratory sieve shakers like RETSCH's AS 200 basic**, to be re-introduced into the production process.

When, during powder injection molding, the binder is removed from the part and it is sintered, an intermediate product with undesired properties may be the result. These defective parts are pulverized, for example with **RETSCH's jaw crusher BB 500**, to recover the raw material for re-use.

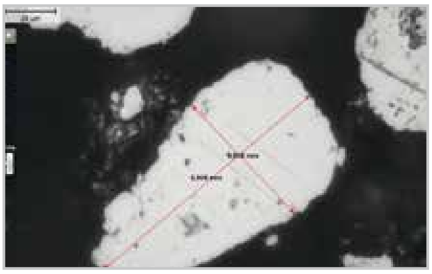
Cutt-off machine Brillant 220

Reliable detection of defects

In spite of meticulous analysis and preparation of the metal powder, **the additively manufactured parts may be defective**. Parts produced by additive laser powder build-up welding, for example, consist of overlapping welding beads; each round of welding influences the microstructure of the layer below by reducing the hardness and in some cases, this may result in fissures.

To **detect potential defects**, the part needs to be cut first. After that, the specimen is mounted in a hot mounting press like ATM's Opal X-Press with Epo black, and, in a next step, it is ground and polished. After subsequent etching, the **microstructure of the sample** is clearly discernible.

For the step of **microhardness testing** and optical evaluation, Qness offers the **Q10/30/60 series of hardness testers**. Powders <0.1 mm only require low test forces and small indent diagonals which makes Vickers the only suitable method.



Size of an aluminum particle,
measured with the hardness testing software
(40x lense)

Conclusion

It can be concluded that additive manufacturing, as well as powder injection molding, offer great potential if the raw materials employed are of optimum quality. But the process needs to be continuously monitored, and the finished parts need to be examined meticulously to ascertain the relevant parameters for a high-quality, cost-efficient production process. The companies of the Verder Scientific Division, introduced in this article, provide reliable and user-friendly solutions for these requirements.

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