

Making metal powders for additive manufacturing

By Stephen B. Harrison, sbh4 consulting

hen Michelangelo carved his masterpiece David from a massive marble block, he used a subtractive technique, chiselling fragments of stone away at the larger block to the form of his smaller creation. Additive manufacturing works in the opposite way. Large structures are formed when layers are successively added to a foundation. It is like a hard fan coral growing in the sea. Over time, the form emerges to its full glory.

Additive manufacturing can be performed using plastic wire or metal wire, which are molten in the precise location that they are required. As they cool and resolidify, the rigid structure under construction is formed. The process can also be performed by using the heat of a laser to melt and fuse metal powders. Additive manufacturing using this laser powder bed fusion technique could not happen without industrial gases. They are integral to the process at many touchpoints.

As successive metal powder layers are added to the piece being made, high purity argon is used to purge the chamber. This prevents oxygen, nitrogen, and moisture from ambient air reacting with the metal that is continuously molten by the laser. The elimination of these gases is essential to ensure the finished piece has the correct

metallurgical properties. During the production of the fine metal powders using the Inert Gas Atomisation (IGA) process, argon is also used for similar reasons.

Molten metal spraying

The metal powders used in additive manufacturing range from commodity metals such as aluminium to more specialised metals such as titanium.

Iron-based alloys and nickel-based super-alloys are also common. The metals are molten using electrical heating through induction or plasma technologies. After a period of degassing, the molten metal is processed to produce the powder. Vacuum induction melting (VIM) is commonly combined with inert gas atomisation, yielding the VIM-IGA process (also known as the VIGA process).

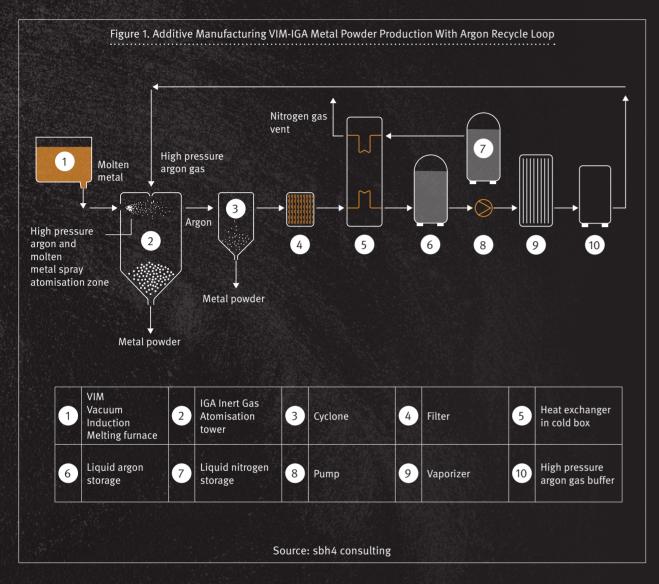
In the IGA process, molten metal is sprayed into a high-pressure argon jet. The liquid metal is atomised into tiny spheroid droplets, which cool and solidify to form the metal powder. Careful selection of the inert gas is essential. Air contains oxygen, nitrogen, and moisture – all of which would damage the atomised molten metal. Argon is super-inert and does not react with the atomised droplets.

"Using an inert gas is important because the metal powder must flow freely..."

Also, the high pressure of argon in the atomisation chamber prevents the ingress of air.

Using an inert gas is important because the metal powder must flow freely in the laser powder bed fusion additive manufacturing process. When smooth spheroids are formed, as is the case when inert argon is used, the powder will flow freely and consistently. If uneven shapes are formed, the powder properties are unpredictable, and production of high-precision components using additive manufacturing process cannot be achieved.

Each type of metal powder can subsequently be used in the additive manufacturing laser powder bed fusion process to produce components with specific properties. High value components using the most exotic alloys are manufactured for demanding applications such as aero engines or power generation turbines.



Argon recovery using liquid nitrogen

Argon is a high-value industrial gas. It is present in air at less than one percent and is only recovered from large cryogenic air separation units fitted with an argon extraction column and argon purification system. Oxygen and nitrogen are abundant in comparison to argon, and their costs of production are lower.

The use of nitrogen in the IGA process might sound like an economical alternative, but nitrogen forms nitrides with molten metals and changes their physical properties. There really is no

alternative to using argon.

To control the cost of using argon, equipment can be installed to recover argon from the atomisation chamber and reliquefy it for repeated use. The liquefaction of the argon is generally achieved in a cryogenic heat exchanger where the cold energy of liquid nitrogen is used to re-liquefy the gaseous argon. The nitrogen gas is vented to the atmosphere – it is sacrificed to recover the argon. Considering the different values of nitrogen and argon, conservation of argon is economically advantageous for the metal

powder producer.

There are parallels to this process from other industrial gases applications. In the distant past, the use of helium for leak testing of car radiators was a one-shot process. The helium was filled into the radiators, a leak check took place, and the helium was vented to the atmosphere.

Over the years, the price of helium rose, and helium shortages made operators acutely aware of the value of this gas. Investment in helium capture and recycling systems became common for large-scale testing operations where

• the equipment CAPEX could be justified by helium recovery and cost savings.

Liquefaction of small-scale biomethane production to form green LNG also applies the sacrificial vaporization of liquid nitrogen. The process economics are based on the situation that biomethane production is constant, but the money earned for injecting the biomethane into the gas grid fluctuates according to the demand on the gas grid at any point in time.

If the biogas can be produced and stored during periods of low price and then vaporized and released when required, the value of the biogas can be maximised. The increased revenue can be allocated to the CAPEX cost of the heat exchangers to liquify the biogas of the liquid nitrogen.

faster than the speed of sound

The turbine in an aero-engine operates at around 10,000 rotations per minute (rpm). The turbine tips are moving faster than the speed of sound at 343 metres per second (m/s). That's quite impressive.

But a small-sized turbine in a hydrogen liquefaction unit may operate at up to 300,000 rpm. For larger magnetic bearing turbines processing heavier gas molecules such as nitrogen, 100,000 rpm would be a reasonable maximum.

The tip speed of a turbine is limited by the strength of the material from which it is made. For conventional alloys, the range of 400 to 500 m/s is reasonable. For titanium, this may stretch to more than 550 m/s. Additive manufacturing is enabling turbine components to be made from extremely strong metals and alloys, which is, in turn, enabling tremendous advances in turbine technology. The result is better energy efficiency. Additive Manufacturing is helping to create cleaner, greener turbines.

With such extreme conditions, there is no room for compromise. The catastrophic failure of an aero engine at 10,000m elevation on an intercontinental passenger jet is simply unimaginable. There are multiple checks in place throughout the Additive Manufacturing value chain to ensure that the entire process is working at the required quality level.

For metal powders, X-Ray Fluorescence (XRF) is used to determine prior to the vacuum induction melting. The technique can detect parts per Quality control for turbine tips moving million levels of trace impurities such as silicon, phosphor, sulfur, and lead. A high concentration of these elements would weaken the metal.

> One the powder is created, the composition can be re-tested to ensure that oxygen, nitrogen, and moisture from ambient air have not contaminated the powder. For many materials, it is also essential to check the structure of the powder spheroids at this stage in the process. The absence of inclusions, foreign particles, or inhomogeneity in the metallic crystalline structure must be confirmed. Any one of these could lead to weak spots in the final structure. XRF analytical instrumentation is also used for these tests.

Operation of the XRF analytical instrument relies on the creation of X-Rays, for which the specialty gas

mixture of 10% methane in argon is commonly used. Users will refer to this as 'P10'. Alternatively, a 5% mixture is possible on some types of instruments. This gas mix is known as 'P5'.

Industrial gases are also used for the heat treatment of components that have been produced by additive manufacturing. The metal powder is molten by the laser powder bed fusion technique and it then re-solidifies. These phase changes can create stresses in the metal that must be relieved by heat treatment known as post-processing. Depending on the metal, this may either be done in an argon blanket or in a nitrogen-hydrogen annealing gas mixture.

The value chain for additive manufacturing is fragmented. A few global companies have developed the expertise and invested in the equipment to make the powders. Others have emerged to operate the Additive Manufacturing equipment and make the components. These parts are often transferred to specialist heat-treatment companies to leverage assets that have been in place for many years for other metallurgical treatment applications such as automotive engine and transmission components. Many diverse opportunities for industrial gases have been created by the emergence and growth of Additive Manufacturing. gw

ABOUT THE AUTHOR

Stephen B. Harrison is Managing Director of sbh4 consulting. Harrison has over 30 years' experience of the industrial and specialty gases