Test methods for Additive Manufacturing (AM) – New analytical possibilities for characterization and monitoring from powder to final product.

BRUKER

Werkstoffwoche in Dresden 2017, Beitrags-Nr. 04-6

Christian Zühlke, Dr. Istvan G. Molnar and Dr. Peter Paplewski
Bruker AXS GmbH, Östl. Rheinbrückenstr. 49, 76187 Karlsruhe, Germany

Introduction

According to ASTM F2792-10, additive manufacturing (AM) is defined as "the process of joining materials to make objects from 3D model data, usually layer upon layer, as opposed to subtractive manufacturing methodologies," such as machining. In the recent years AM technologies developed rapidly and techniques like Electron Beam Melting (EBM), Selective Laser Melting (SLM), Selective Laser Sintering (SLS) prepared the ground for AM technologies being increasingly established in the metal part production industry (Figure 1). Generally AM is complementing other powder metallurgy (PM) technologies like Hot Isostatic Pressing (HIP) or Metal Injection Molding (MIM). HIP is used to produce massive, near net shape parts of several 100 kilograms (with fine and full isotropic microstructure), but also as a densification step for parts produced by AM technologies. In contrast, MIM, like other press & sintering technologies, is widely used to produce large series of small near net shape parts. Although the choice of a suitable PM process depends on the type and size of the part to be produced as well as the requirements and possibilities of the user.
All techniques share one common element:
<the metal powder>

Objectives

One key aim of the AM process is to build parts without porosity. Thus, the monitoring of those non-metallic elements that can form gas inclusions during the 3D printing process is of high importance. This calls for suitable analytical techniques that are capable of characterizing not only the starting metal powder, but also the final product with high precision. The Bruker AXS division is able to offer a complete analytical package, including X-ray fluorescence (XRF), optical emission spectroscopy (OES) and combustion gas analysis (CGA) instruments, for the AM industry. Our main goal with this study is to give an overview on the determination of the critical non-metallic elements C, S, O, N, H and Ar in various ferrous and nonferrous metal powders, such as Inconel® 718, Ti_6Al_4V, and $AlSi_{10}Mg$, as well as in final products, using combustion and inert gas fusion (IGF) techniques. We demonstrate that the concentration of these non-metallic elements strongly depends on the type of the metal alloy as well as on the particle size. Furthermore, we show that the concentration of some critical elements, such as carbon and oxygen, can change significantly during AM processes, emphasizing the quality control in both powder and end product.

Analysis Methods

- The determination of carbon and sulfur was performed on a Bruker G4 Icarus HF (Figure 2, left) combustion analyzer equipped with a induction furnace and a non-dispersive infrared (NDIR) detection system for CO_2 and SO_2.
- The analysis of nitrogen, oxygen, and hydrogen was performed on a Bruker G8 Galileo IGF instrument (Figure 2, middle), equipped with an impulse furnace, a NDIR detection system for CO, and a thermal conductivity detector (TCD) for N_2 and H_2.
- The Bruker G8 Galileo ONH instrument has the unique feature to be coupled to an mass selective detector, enabling the determination of other specific elements, such as argon, in powder or compact pieces (Figure 2, right). The quadrupole detector is fine-tuned to the mass range of 2 to 100 m/z, and thus an ultra high sensitivity can be achieved with a detection limit of <10 ppb.

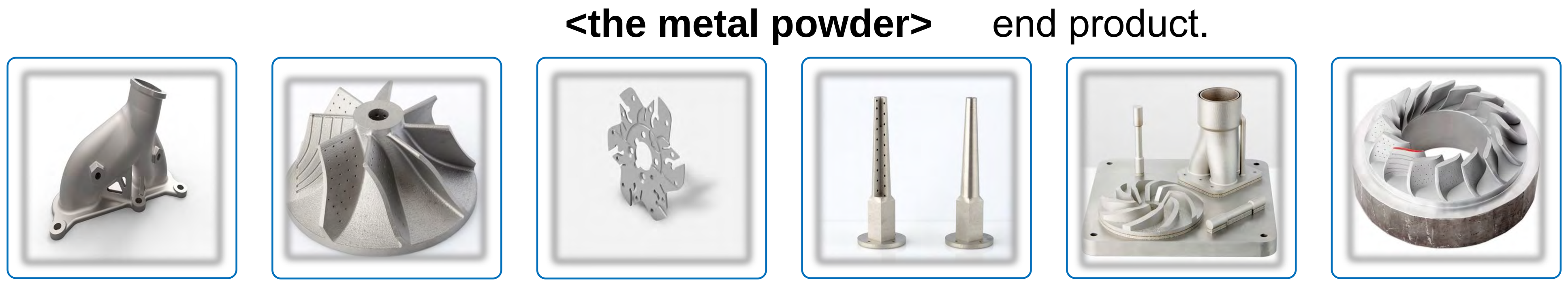


Figure 1. SLM 2800HL 3D printer products from 'Edelstahl Rosswag GmbH' in Pfinztal, such as racing car elbow, impeller or milling machine disc.

Figure 2. Bruker G4 Icarus HF CS analyzer (left) and Bruker G8 Galileo ONH analyzer (middle) coupled to a MS detector (right).

Results and Discussion

- Table 1 shows some typical values of C/S/O/N/H/Ar obtained for different metal powders frequently used for AM processes. Such analyses should be part of the quality control (QC) process for 3D products in order to ensure the highest level of quality that is of high importance in industries, such as automotive and aerospace.
- The results in Table 2 demonstrate that not only the starting powder, but also the final product could be analyzed with high precision, using the same instruments and methods.

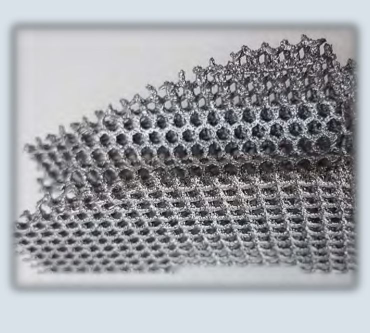
Table 1. Typical results of C/S/O/N/H/Ar-determination in various metal powders.

Sample Type	Carbon	Sulfur	Oxygen	Nitrogen	Hydrogen	Argon
SS 1.4404	185.4 ± 2.3 ppm	39.4 ± 1.3 ppm	412.4 ± 21.2 ppm	940.4 ± 8.6 ppm	6.8 ± 0.3 ppm	318 ± 242 ppb
Inc® 718	545.4 ± 4.6 ppm	37.6 ± 1.4 ppm	218.5 ± 6.9 ppm	219.1 ± 6.3 ppm	7.9 ± 0.6 ppm	1436 ± 207 ppb
$AlSi_{10}Mg$	30.9 ± 2.2 ppm	34.5 ± 6.3 ppm	456.0 ± 33.2 ppm	13.4 ± 0.2 ppm	29.9 ± 0.5 ppm	330 ± 93 ppb
Ti_6Al_4V	84.0 ± 3.8 ppm	19.1 ± 1.7 ppm	959.9 ± 16.6 ppm	150.1 ± 2.7 ppm	28.3 ± 2.3 ppm	280 ± 71 ppb

ON/H/Ar-determination: Samples were filled into capsules and analyzed in a graphite crucible. Typical sample weight was between 100 and 1000 mg.
CS-determination: Samples were filled into pre-baked ceramic crucibles, covered with tungsten accelerator, and analyzed. Typical sample weight was between 100 and 1000 mg.

Table 2. Results on $AlSi_{10}Mg$ final structures.

Element	Measured Content
Carbon	134.7 ± 9.9 ppm
Sulfur	16.8 ± 2.4 ppm
Oxygen	882.8 ± 88.4 ppm
Nitrogen	16.6 ± 1.4 ppm
Hydrogen	29.5 ± 3.5 ppm
Argon	344 ± 17 ppb



- The most critical elements in regard to surface contamination are oxygen and hydrogen. Their concentration can increase significantly during storage and/or reuse due to eldering. Furthermore, the concentration of these elements is significantly influenced by the particle size, which is demonstrated with the example of $AlSi_{10}Mg$ powder in Table 3.

Table 3. Correlation between O/H and particle size.

Particle Size	Oxygen Content	Hydrogen Content
10-53 µm	1640 ppm	68.2 ppm
20-63 µm	998 ppm	53.7 ppm
40-100 µm	722 ppm	38.1 ppm

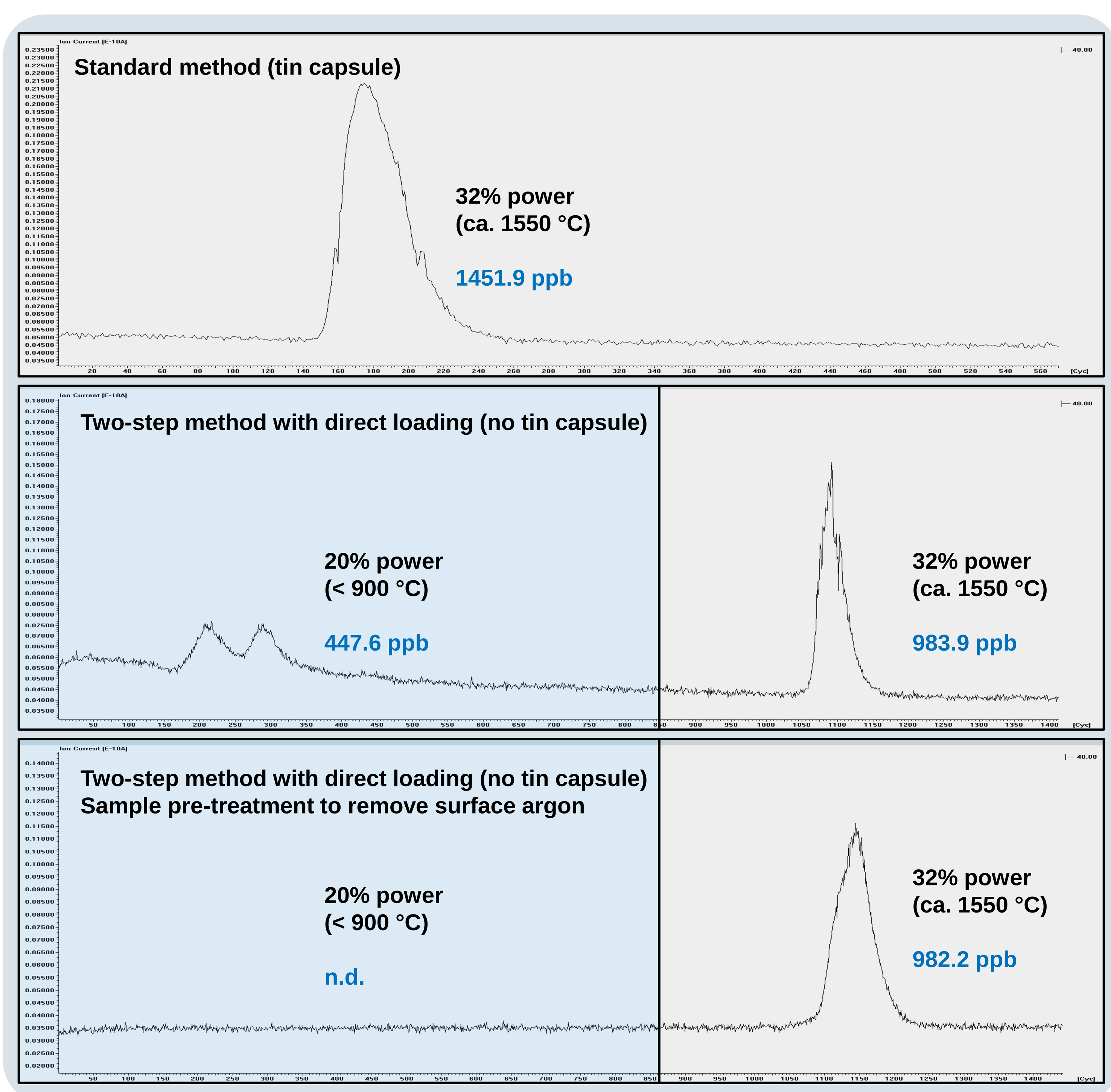


Figure 3. Surface vs bulk argon in Inconel® 718 powder.

- Argon causes similar embrittlement problems like hydrogen, but starting at significantly lower concentrations (< 100 ppb). Therefore, the argon content should be monitored before and after HIP and AM processes. Due to the high surface area of powders, the residual air can significantly influence the results of argon determination, which needs to be taken into consideration during the QC of metal powders (Figure 3).

Conclusions

- Complete solution package for the characterization of metal powders and final products in AM.
- Oxygen and hydrogen are the most critical elements, whose concentration depends on factors, such as particle size.
- During argon determination, the surface contamination by air should be also considered.

Technology