Application note | AN1584

Thermo Fisher SCIENTIFIC

Rapid chemical characterization of feedstock powder

The challenge

Understanding the chemical composition of any manufactured material is critical to predicting a part's performance. This is especially important for additive manufacturing because rapidly melting and solidifying materials can affect chemical properties and impact part functionality. Despite its importance, performing chemical characterization is difficult and time-consuming, requiring multiple analytical instruments, complicated operational and calibration procedures, and trained chemists.

The solution

A new mass spectrometry technique can quantify the metallic, trace, and low-mass constituents in a single analytical session with minimal sample preparation time. As a result, it reduces the time required for quality control of additive manufacturing powders and builds. This is especially impactful to prolonging the life of recycled powders. Often, additive manufacturing users have a "rule of thumb" for how many build cycles they can use powder before discarding it. If a user can quickly verify the chemical composition meets specifications, they can avoid disposing of powder after a predefined number of build cycles, saving the approximately $100,000 required to refill the printer's powder prematurely. For example, a team of materials engineers in a Department of Defense branch revealed their facility spends $80,000 and $180,000 to refill the titanium and cobalt chrome powder printers, respectively.

The motive

Using a single desktop instrument to quantify the metallic, trace, and low mass constituents of an additive manufacturing powder significantly reduces the time required for quality control (QC). Instead of using 4 different standard test methods and 3 different analytical instruments, the EXUM™ MASSBOX™ LALI-TOF-MS performed full chemical characterization on a titanium powder in minutes.

The result

Instead of using 4 different standard test methods and 3 different analytical instruments, the MASSBOX LALI-TOF-MS performed full chemical characterization on a titanium powder in minutes.

EXUM MASSBOX LALI-TOF-MS

MASSBOX LALI-TOF-MS

The MASSBOX LALI-TOF-MS ‘s Laser Ablation Laser Ionization Time of Flight Mass Spectrometry (LALI-TOF-MS) addresses many challenges associated with other analytical techniques to offer rapid, high-sensitivity quantification of nearly the entire periodic table. The ionization source, LALI, uses two lasers to first ablate, or release, material from a solid sample’s surface and then ionize neutrals present in the ablated material. The laser ablation process allows direct analysis of solid materials, like additive manufacturing powders or builds, without the complicated sample preparation procedures of other techniques that require liquid sample introduction.

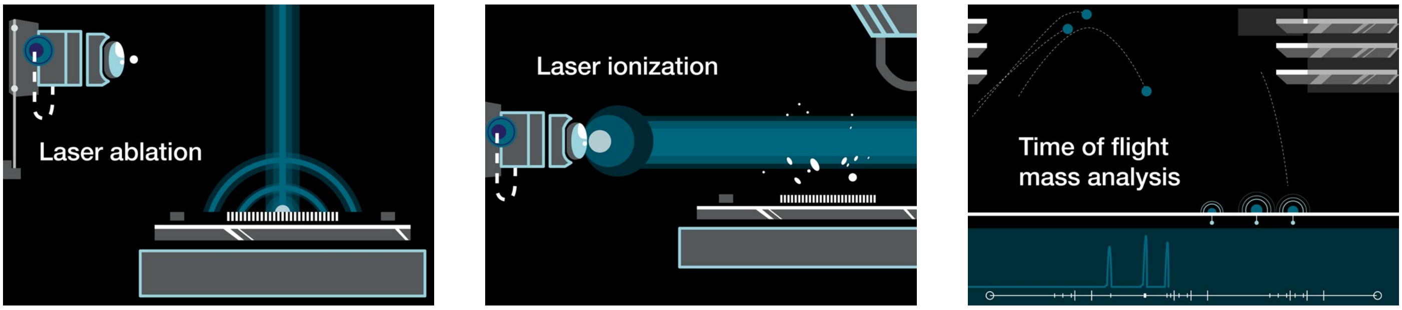

Figure 1: A) Ablation laser fires perpendicular to the sample’s surface. The laser spot size is adjustable from 5-150 micron;
B) Secondary laser performs multiphoton ionization of neutral particles created by ablation process;
C) Ions are separated by Time-of-Flight mass spectrometry and detected with a multichannel plate (MCP)

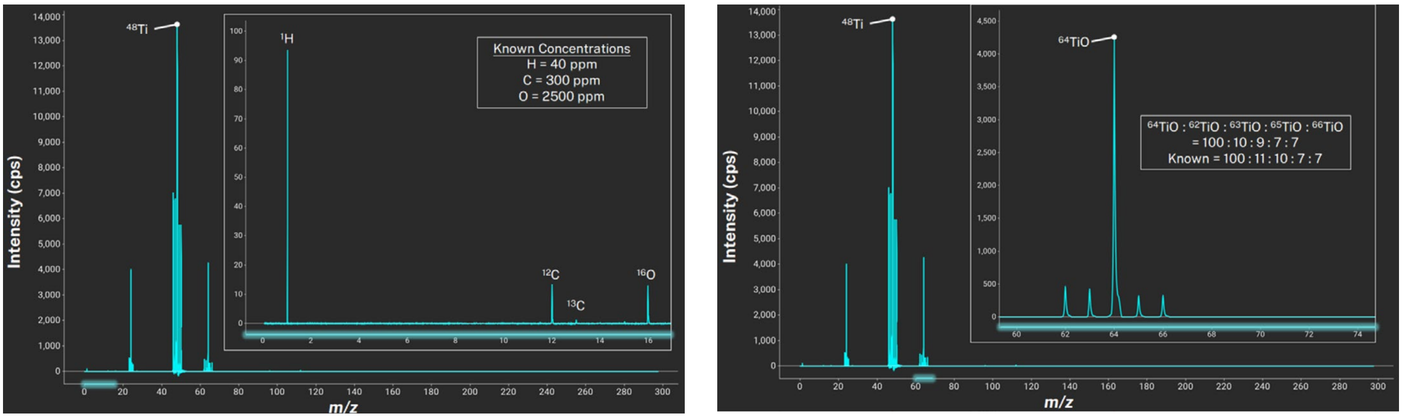

Figure 2: Mass spectrum from a titanium powder sample, analyzed by the MASSBOX LALI-TOF-MS. The zoomed inset in the top pane highlights the trace light elements hydrogen, carbon, and oxygen, and the bottom pane’s zoomed inset shows the naturally occurring isotope pattern of titanium oxide. The text box compares measured and theoretical (known) isotope ratios.

The ionization laser targets the neutral particles created by ablation, which are more representative of the sample’s constituents than plasma-generated ions. Overall, LALI results in more reliable elemental verification and reduces sample matrix effects. After ionization, the TOF mass analyzer creates a full mass spectrum at each laser spot, as shown above. Figure 2 displays an example titanium powder sample’s mass spectrum. Based on the naturally occurring isotope patterns, the software verifies each detected element. The top pane highlights trace low-mass elements that are difficult for many techniques to reliably quantify: hydrogen, carbon, and oxygen. The bottom pane shows the naturally occurring isotope pattern for titanium oxide. The text box compares the theoretical (known) isotope ratios with those quantified from the mass spectrum’s peak areas, which are within 1%. This demonstrates the MASSBOX LALI-TOF-MS ‘s isotopic accuracy.

To demonstrate the MASSBOX LALI-TOF-MS’s quantification capabilities, we analyzed two titanium powders: one blind sample (Sample #1) and one reference sample (Sample #2). The elemental composition acquired by other techniques on Sample #2 was used as a reference material to quantify the unknown sample’s constituents. For all quantified elements, results matched the powder’s certified values and those generated by an accredited testing laboratory using other methods. Once the pelletized powder samples were loaded into the instrument, results were acquired within minutes.

Quantification capabilities

This study involved analyzing a pair of titanium powder samples. One was a known reference standard, used to quantify the unknown sample's constituents using a linear single-point calibration. Because the MASSBOX LALI-TOF-MS requires a solid sample, powders are pressed into 1.3-cm (0.5-in) solid pellets using a pellet die set and hydraulic press.

Each sample was analyzed using three 0.2 mm areas. The areas are each rastered twice, removing any surface contaminants in the first pass and using the second pass for quantification. The system's sample tray can accommodate up to 16 pressed pellets, and the user can set up the instrument to analyze all samples within the same session. While the user is defining testing conditions, the instrument brings the closed sample chamber to vacuum. Once vacuum is achieved, the analysis requires only three minutes per sample.

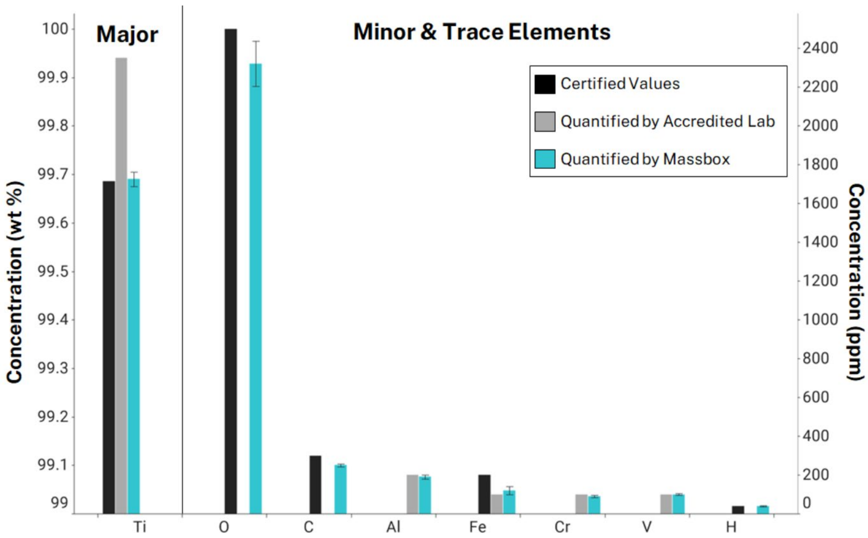

Figure 3: Results from quantifying the unknown titanium powder (Sample #1) through single-point calibration using the known titanium powder (Sample #2) as a reference. Bars compare results quantified by the MASSBOX LALI-TOF-MS with certified values and those acquired by an accredited lab.

Note certified values are included for elements that were not measured by the accredited lab. Error bars are 1σ standard deviation across triplicate raster areas.

For Sample #2, the known sample, a laboratory chemical test report was provided. To quantify the composition of nine elements, the lab used four standard test methods and three analytical instruments. This known information was then used to quantify Sample #1's elemental concentrations with the MASSBOX LALI-TOF-MS.

Figure 3 compares results quantified by the MASSBOX LALI-TOF-MS with certified values and those provided by an analytical testing laboratory. In this graph, values certified by the powder provider are black, results acquired by the lab are gray, and those quantified using the MASSBOX LALI-TOF-MS are blue. Titanium's concentration is presented in weight percent (wt. %) and those of minor and trace elements are in parts-per-million (ppm).

In this case, the accredited lab quantified four trace elements using only conventional Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES). Testing laboratories typically charge per element and the excluded low-mass elements can cost ~$100 each because they require additional analytical techniques. The graph includes certified values for the elements that were not measured by the lab. For results quantified by the MASSBOX LALI-TOF-MS, error bars show the internal precision. These values are 1σ standard deviation of the three different raster areas.

MASSBOX LALI-TOF-MS summary

This study highlights the MASSBOX LALI-TOF-MS as a promising alternative technique for chemical characterization of additive manufacturing powders. Using Sample #2 as a reference, the elemental constituents of Sample #1 were quantified within 8% of known values, on average. Internal precision was 6% relative standard deviation (RSD), calculated from the triplicate raster areas' variance. This low variance demonstrates the repeatability of the technique.

Because this innovative technology can quantify the metallic, trace, and light elemental constituents in the same analytical session, it has the potential to significantly reduce quality control time for additive manufacturing powders. Instead of using four different standard test methods and three different analytical instruments, the MASSBOX LALI-TOF-MS acquired the results presented in Figure 3 within minutes. Its simplified operations and desktop package bring high-sensitivity chemical characterization out of the traditional laboratory environment. For additive manufacturing users, this solution can support a procedure for reusing powder based on rapidly acquired data instead of a predetermined number of build cycles.

In addition to the quantitative capabilities presented in this study, the MASSBOX LALI-TOF-MS can also perform broad-scale elemental mapping and high-resolution depth profiling to determine how elements vary spatially.

Elemental mapping

Depth profiling

Rapid screening

Quantitative analysis

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Thermo Fisher Scientific is an authorized distributor of the EXUM MASSBOX LALI-TOF mass spectrometer in Europe, United Kingdom, and China. For all other regions, please contact us at marketing.ppa@thermofisher.com.

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