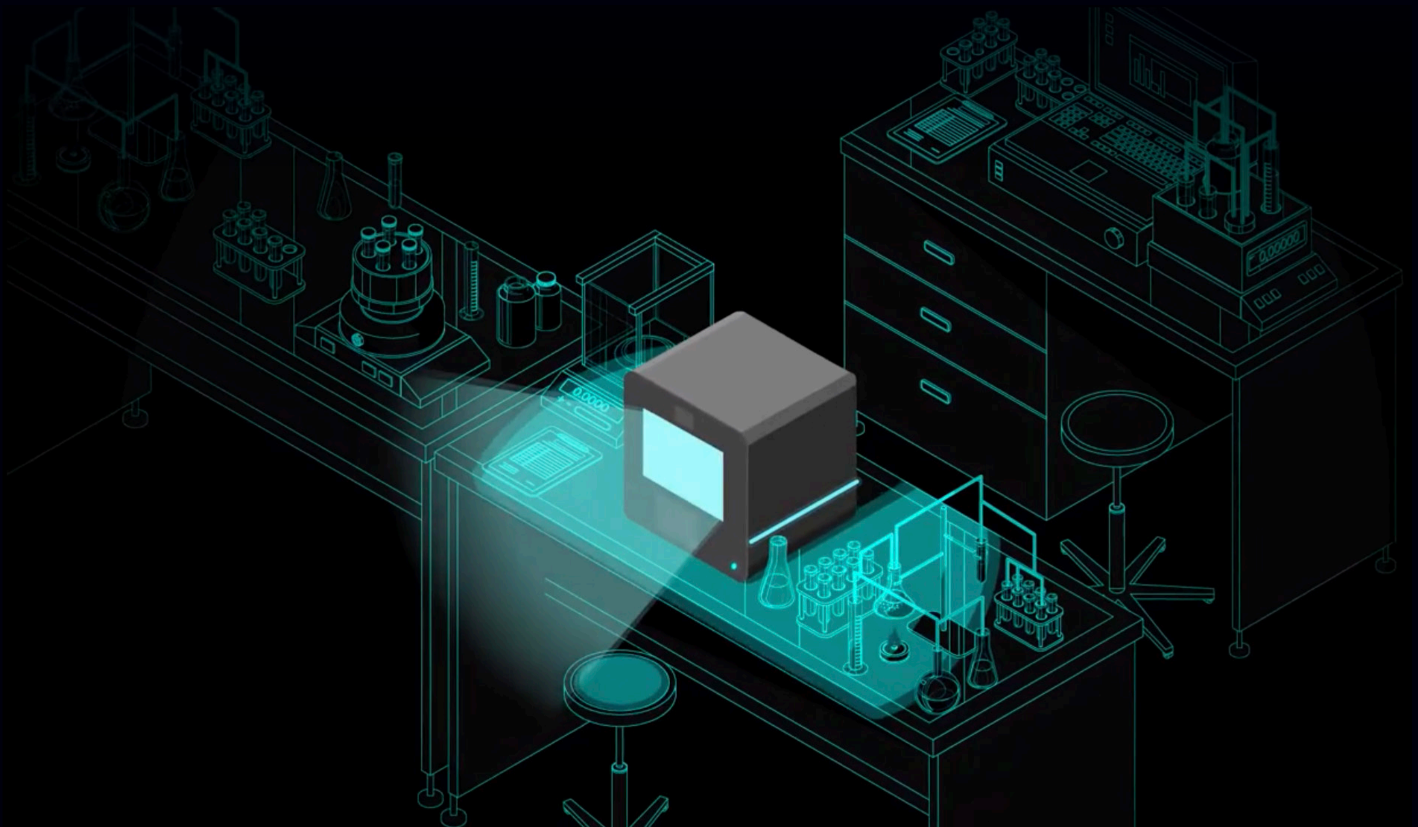
EXUM

SCIENCE | SIMPLIFIED

Quality Assurance for Manufacturing Materials Using the Massbox

The Massbox is the first commercial Laser Ablation Laser Ionization Time of Flight Mass Spectrometer (LALI-TOF-MS), which combines low detection limits with low-cost, uncomplicated operations. This work demonstrates the Massbox's capabilities for quality control in manufacturing and materials applications. We analyzed six metal alloy samples to identify concentrations of major and trace elements, generate a calibration curve, and calculate quality control limits. For sample powders of 2 vol% additive, results indicated a 2σ quality control limit of 2.00 +/-0.06 vol% additive.



Introduction

The physical properties of any manufactured material depend on its chemical composition. Ensuring the quality of a metal alloy requires characterizing its feedstock to confirm the various elemental concentrations are within the acceptable ranges. If a certain element is outside of the range, it could affect the material's performance. As a result, manufacturers need a reliable tool to quantify major and trace elements in both metal alloy feedstocks and finished products.

LALI-TOF-MS

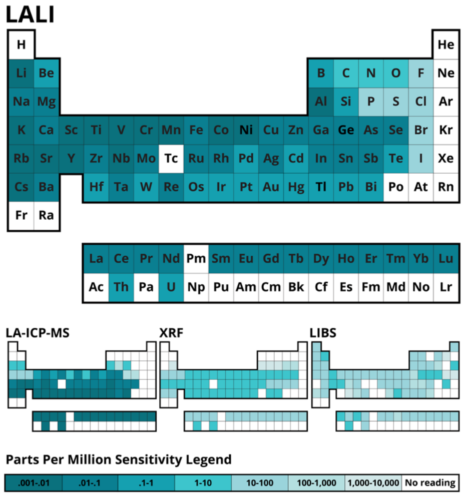
LALI incorporates two lasers to first ablate (or desorb, in the case of organics) material from the solid sample's surface and then ionize that material in a second step. By analyzing solid samples directly, LALI eliminates the intricate dissolution/digestion sample preparation procedures that complicate other methods. The initial ablation (or desorption) process creates both a temporal plasma and a neutral particle cloud, and the second laser ionizes the neutrals. Compared to other plasma-ionizing techniques, targeting neutral particles greatly reduces the matrix effects.

After ionization, the particles move through the optics system to the Time of Flight (TOF) mass analyzer, which measures the time required for ions of different masses to impact a detector. The resulting measurement creates a full mass spectrum, which facilitates multielement quantitation. Additionally, from ablation to mass analysis, the sample and its representative ions are under vacuum, which improves ion transport efficiency compared to other techniques.

METHOD AND MATERIALS

Elementum3D provided three different aluminum alloy base powders, two powders with 2 vol%, and a powder with 10 vol% additive. From the base alloy and 10 vol% additive powder samples, we mixed four additional samples with ~3, 5, 6, and 8 vol%. Prior to analysis, we pressed ~1.0-1.5 g of each sample into a pellet using a pellet die set and ~2 metric tons of force with a hydraulic press. Quantitative analysis was performed in triplicate on each pellet. Briefly, three areas (12,000 individual mass spectra each) were rastered twice and averaged. To exclude any potential surface contaminants, we used only the second passes of each area for the quantitation. Exum's software picked peaks from the average, baseline-subtracted, calibrated mass spectra, and verified assignments using known isotopic ratios. Peak areas were then normalized to ^{27}Al for quantitation.

EXUM
SCIENCE | SIMPLIFIED



Results

Figure 1 shows the resulting mass spectrum from the aluminum alloy with 10 vol% additive concentration. The figure contains two zoomed insets. The one from *m/z* 10-16 (left) shows light elements boron, carbon, and oxygen and the one from *m/z* 61-66 (right) shows titanium oxide's five isotopic peaks. These indicate the mass spectra generated by the Massbox are isotopically correct across the entire mass range, which is only possible because it eliminates virtually all polyatomic and isobaric interferences that plague other techniques. As a result of reliable isotopic verification, quantitation from major isotope peaks is possible.

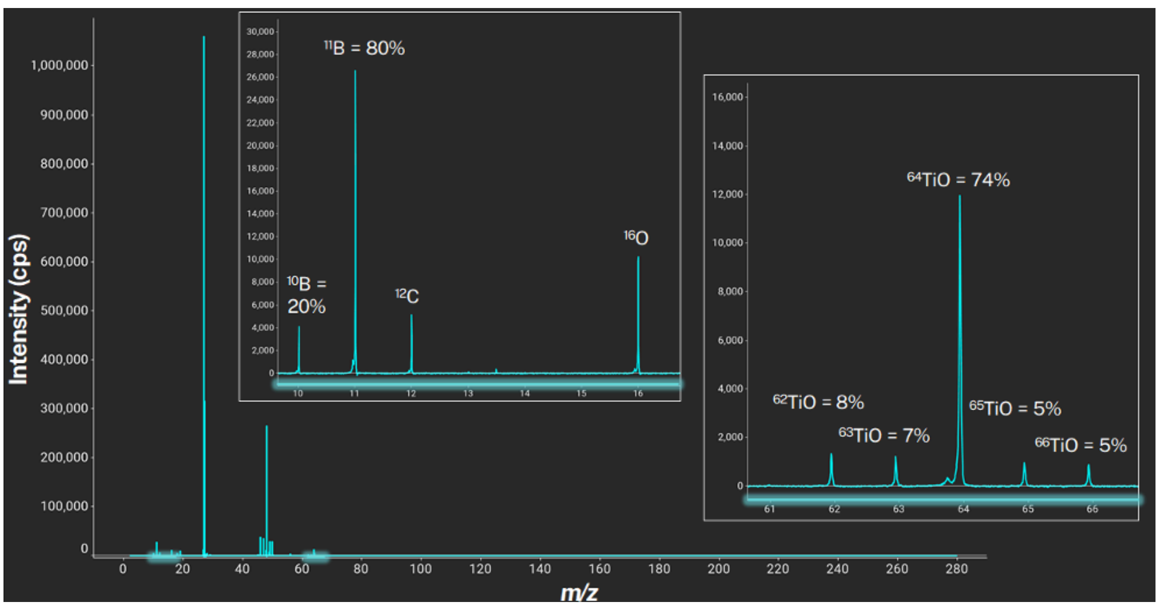


Figure 1: Mass spectrum and zoomed insets from the aluminum alloy sample with 10 vol% additives. The zoomed inset on the left, from m/z 10-16, highlights the light elements, B, C, and O. The zoomed inset on the right, from m/z 61-66, shows isotopically-correct mass spectral peaks for TiO. Text boxes include the theoretical (known) isotope ratios for each element.

Results (cont'd)

The calibration curve shown in Figure 2 plots the average aluminum-normalized boron signal against vol% additive with the error bars representing one standard deviation. With a nearly perfect correlation coefficient of 0.997, results from the hand-mixed samples indicate a 2_σ quality control limits of 2.0 +/-1.0 vol% when calculated from the boron-normalized signal and 2.00 +/-0.26 vol% when calculated from average standard deviation of the titanium-normalized signals. The standard deviation of the various sample pellets also indicates differences in homogeneity between samples. As expected, the hand-mixed samples show greater heterogeneity compared to the premixed samples provided. The hand-mixed samples had an average relative standard deviation of 6.5% for titanium-normalized signals, while the two provided samples with 2 vol% additives were much more homogeneous. The two pre-mixed samples had relative standard deviations of 2.6% and 4.3% for titanium-normalized signals, which correlates to an average 2_σ quality control limit of 2.00 +/-0.06 vol%.

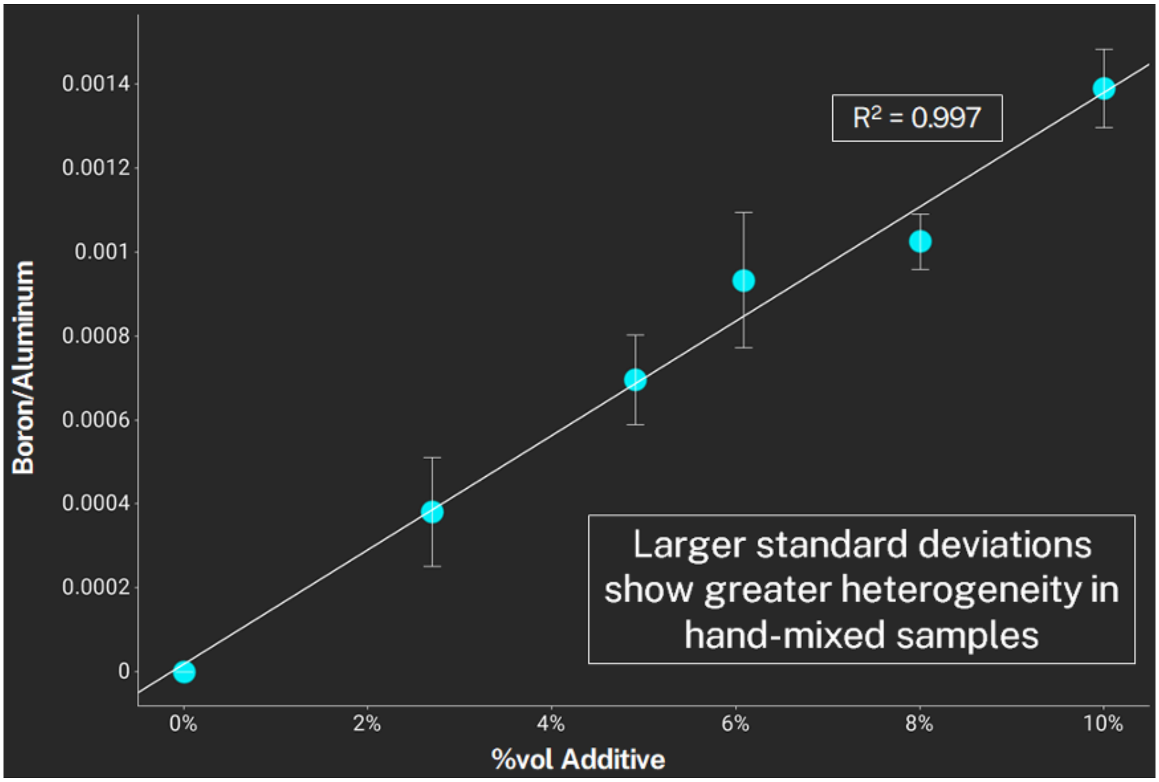


Figure 2: Calibration curve showing the aluminum-normalized boron signal. Error bars show the standard deviations from triplicate measurements for each sample.

Conclusion

With the intuitive operations of its all-in-one system, the Massbox is the ideal instrument for quality control and quality assurance applications. Its capabilities provide accurate quantification of major and trace elements, including light to heavy atomic masses, in each analytical session. With simple sample preparation procedures and a few minutes for analysis and quantitation, the Massbox offers rapid turnaround for precise quality control measurements.