Why choose spark optical emission spectroscopy for metal additive manufacturing

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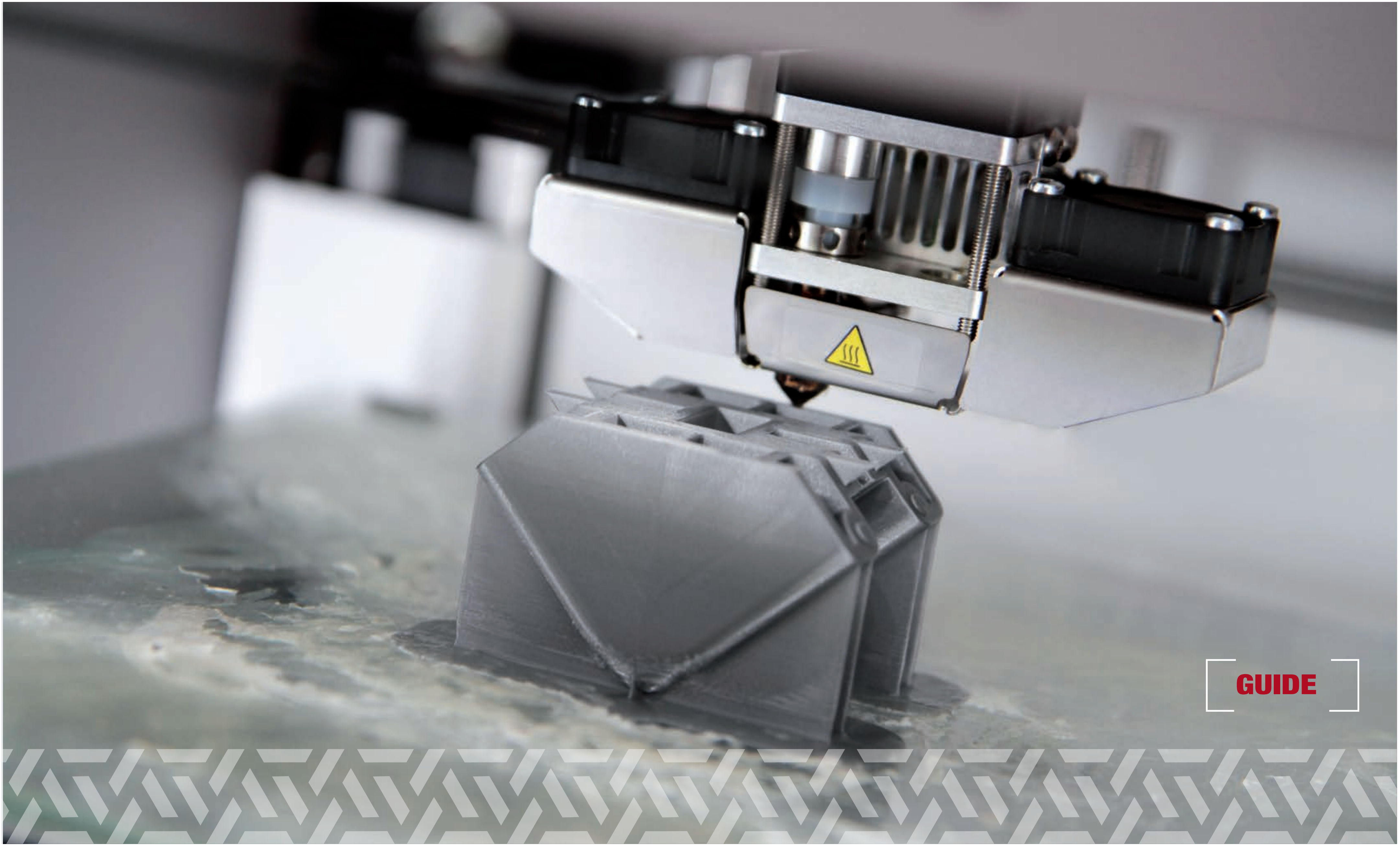


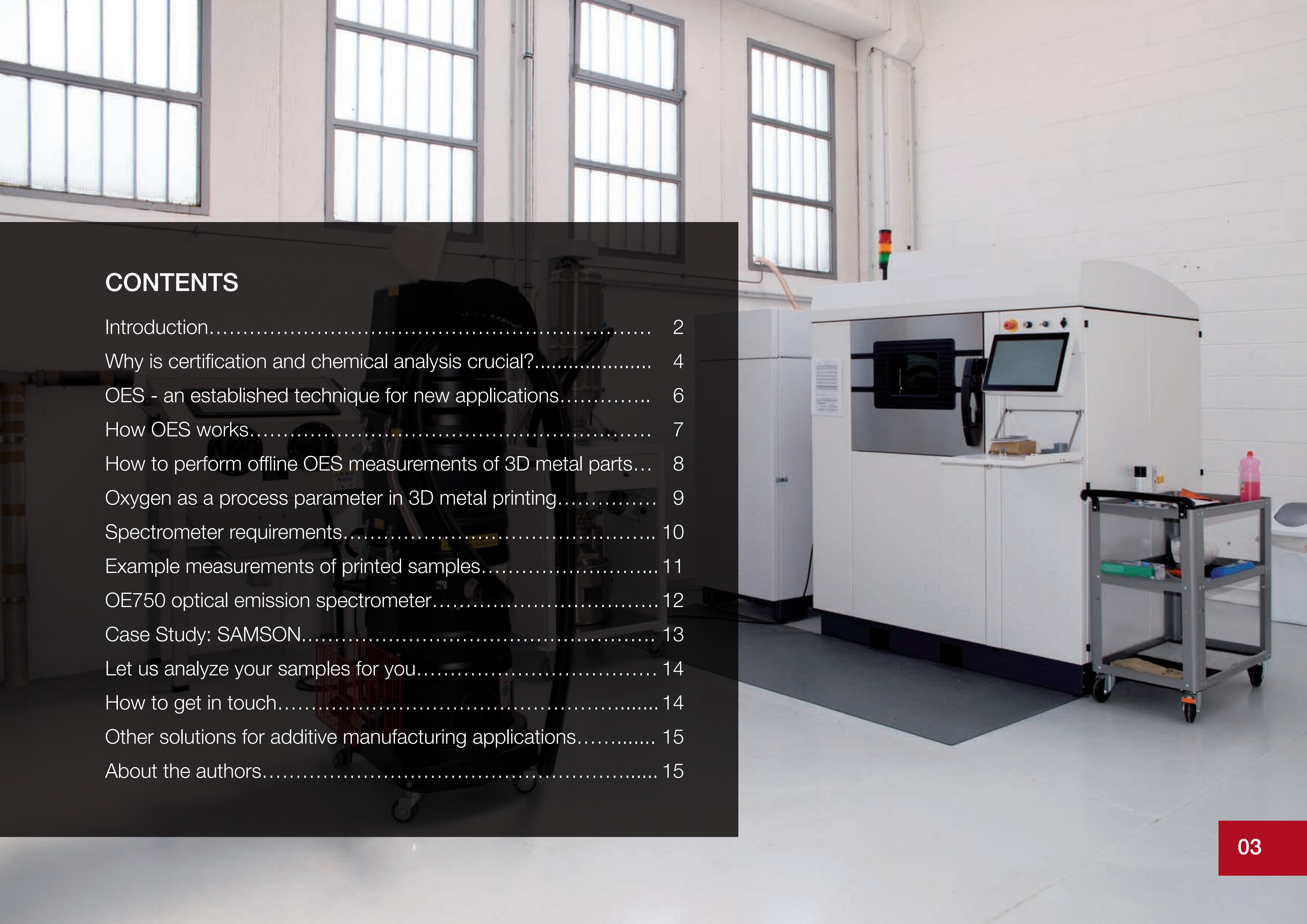
INTRODUCTION

Additive manufacturing – or 3D printing – has revolutionized component manufacture. The ability to form metal or plastic components layer by layer means that complex shapes with tight tolerances can be easily produced rather than using a subtractive method where material is removed from a larger part like carving an item from a block. Techniques like direct metal laser sintering (DMLS) and electron beam melting (EBM) are giving product engineers scope to design intricate components that weren't possible or prohibitively expensive with traditional subtractive techniques.

Another benefit of additive manufacturing is that it's easy and cheap to make a prototype. You don't have to create specific tools or set up a costly production run, delivering less waste and better cost ratio for one-off or small-batch production.

However, the move from prototyping tool to reliable manufacturing asset has thrown up several challenges for 3D printing, especially in the metal additive manufacturing sphere. Here, powder bed fusion is used to form complex shapes for sensitive applications, such as medical devices for implantation in the body, or aerospace components for use in flight. These are areas where components simply can't afford to fail.

In this guide, Hitachi's Maryam BeigMohamadi, Application Scientist – OES, and Michael Molderings, Product Manager – OES, provide insight on how we can help you to find an optimal solution for your additive manufacturing process quality control.

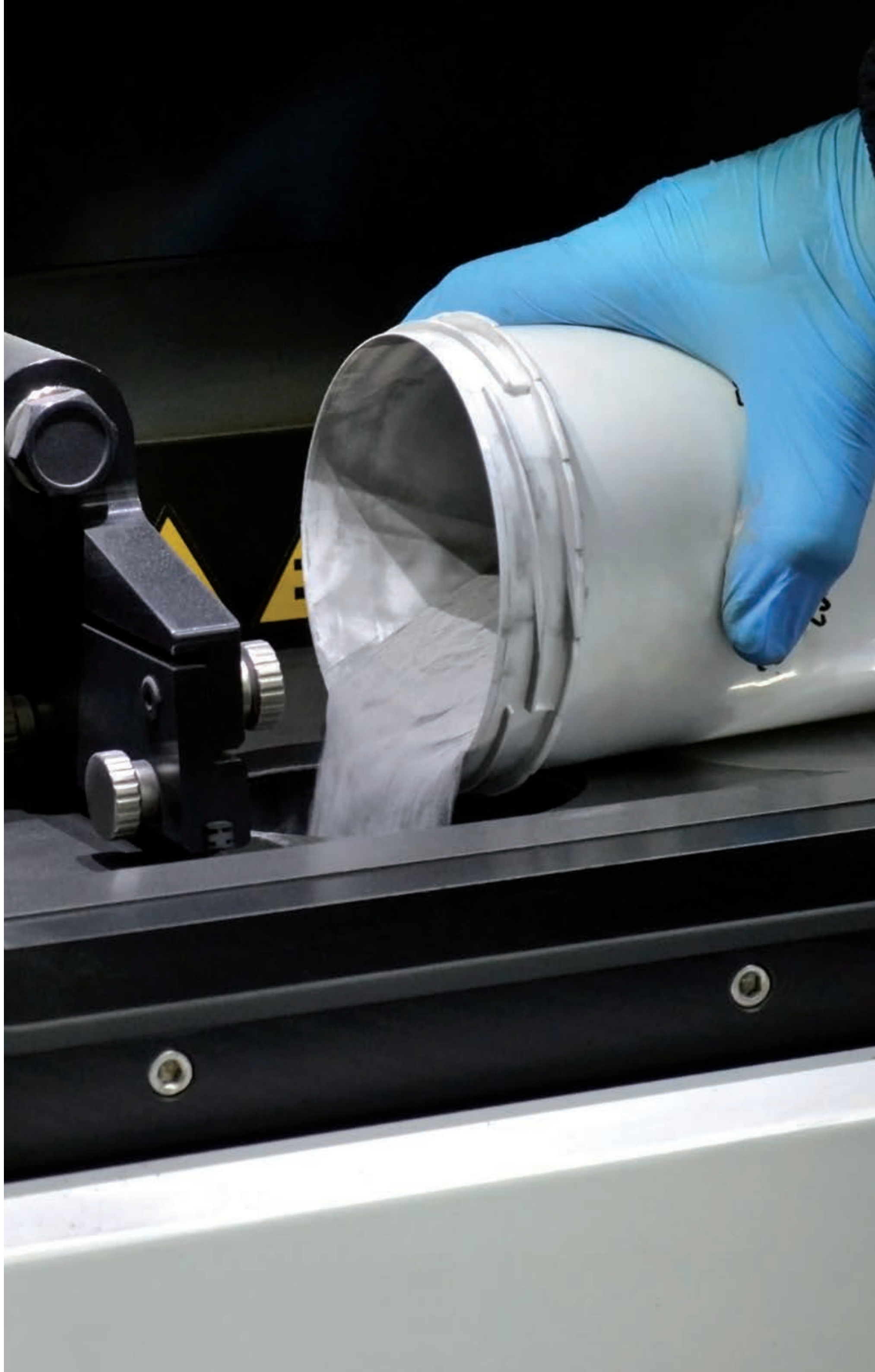


CONTENTS

WHY IS CERTIFICATION AND CHEMICAL ANALYSIS CRUCIAL?

As with every other metal forming activity, the composition of the metal powder needs to be right for the final product to have the correct characteristics to not only meet specification and avoid defects but also to comply with the applicable local and national statutory regulations. Understanding how to meet requirements, satisfy appropriate conformity assessment procedures and achieve the necessary certification isn't always easy to decipher though.

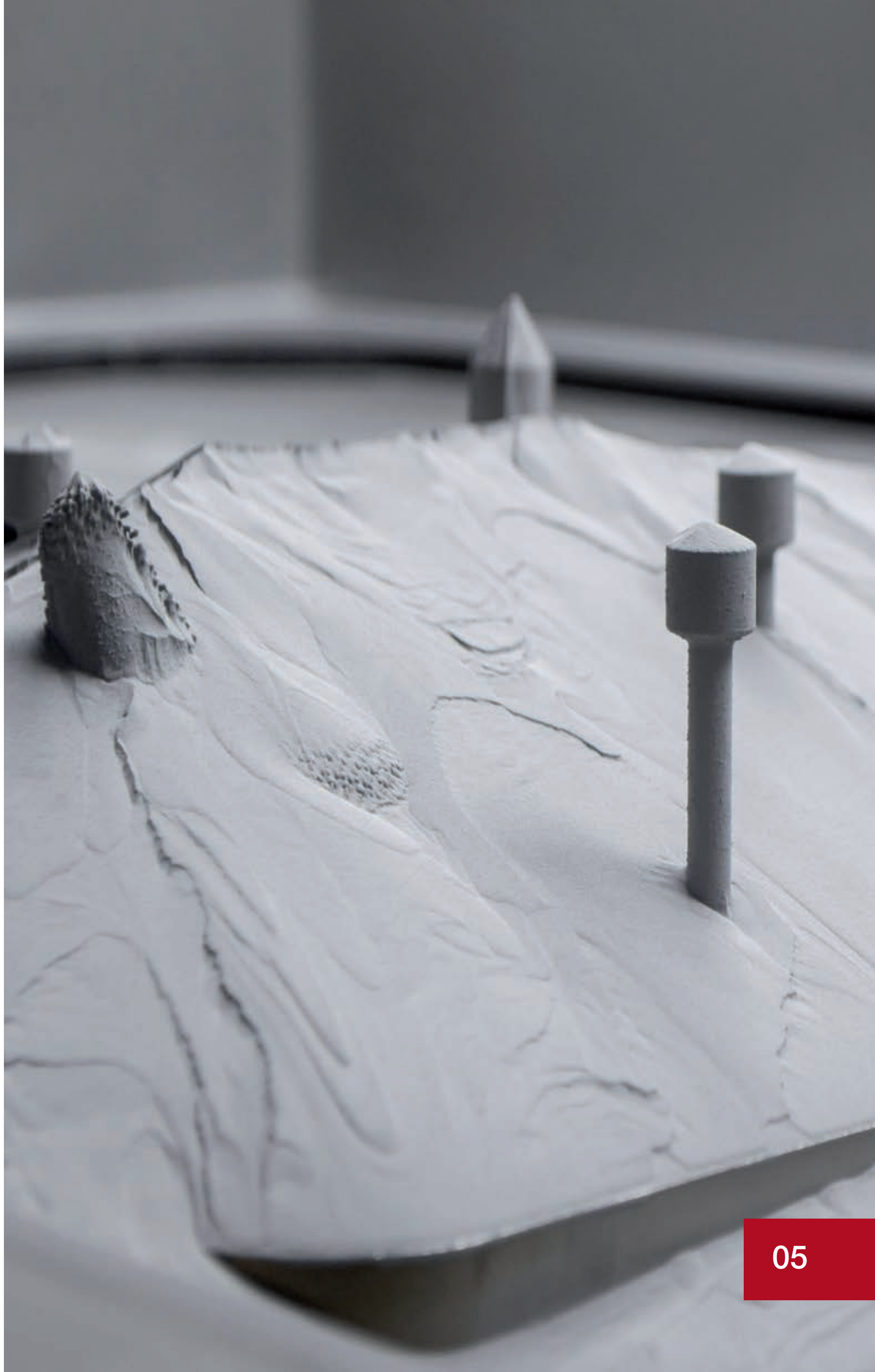
For example, the EU Pressure Equipment Directive (PED) applies to materials and semi-finished products that are additively manufactured and used in or as pressure equipment. For compliance and certification, different analytical methods are required for the precise control of the chemical composition of manufactured parts. Optical emission spectroscopy (OES) is one of these analytical techniques. In America, ASME has set standards for additive manufacturing and in China the Food and Drug Administration (CFDA) regulates the additive manufacturing standards businesses need to comply with.



Each batch of raw powder should be checked for chemical properties and uniform particle size distribution using various test procedures. Despite intensive cleaning of the printer, material mix also can't be ruled out for causing out of spec products. So, do you rely on the certificate of the powder you have purchased to ensure quality? How do you know if the powder complies with the specification after 3D-printing?

It's worth noting that the frequent recycling of powder through several printing runs leaves the process open to external contamination, especially when switching from one powder type to another. Another source of contamination is gasses such as oxygen which can accumulate in the powder and have an adverse effect on the chemical composition and material properties. The 3D metal printing process itself can also create defects within parts.

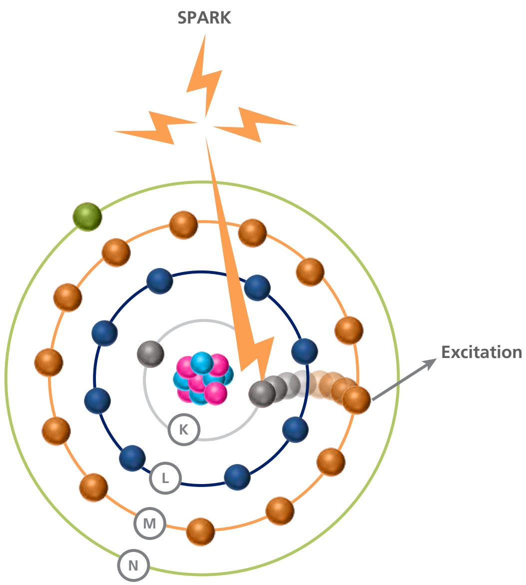
To prevent contamination of the finished part, it's important to verify the raw powder before printing, and check the composition of the finished part before shipping to reduce scrap rates, increase yield and really capitalize on the benefits of additive manufacturing. And this is where spark OES can be a key ally.

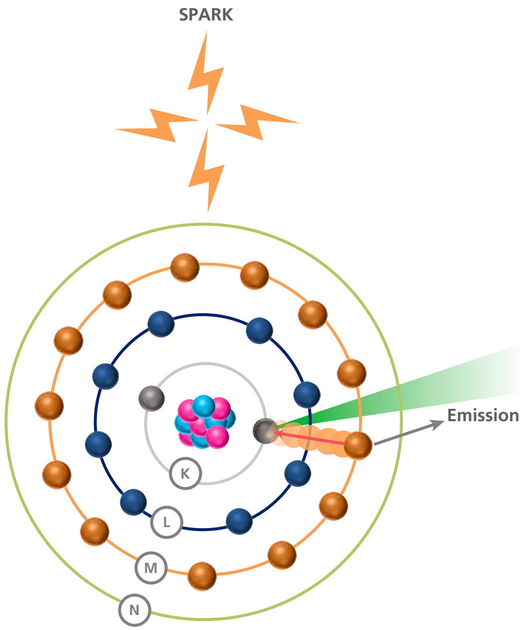


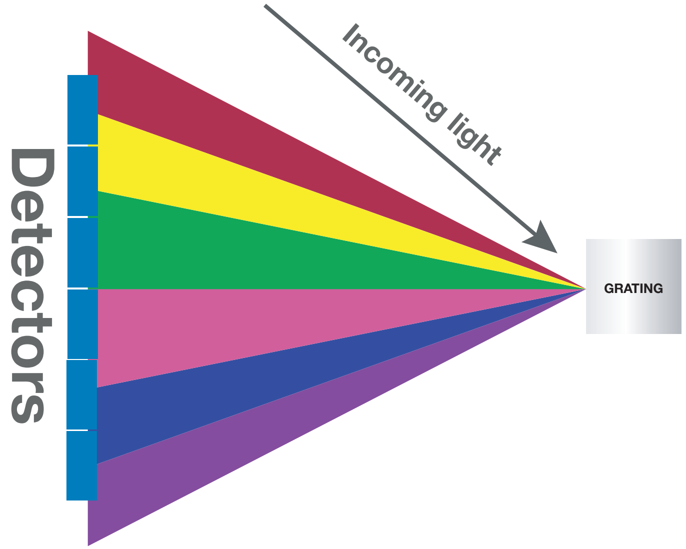
OES - AN ESTABLISHED TECHNIQUE FOR NEW APPLICATIONS

Optical emission spectroscopy is the ideal solution for measuring 3D printed parts. This method of elemental analysis has been used for decades and it's considered the most important method for the analysis of metals and alloys in the metallurgical industry. Spark spectrometers are used for seamless quality control in metal processing, starting with the analysis of the accompanying elements in scrap metals, the control of incoming materials, the melting process control, the outgoing goods to fabrication.

As one of the highest turnovers and most labor-intensive industries in the world, the iron and steel industry is of great importance. Our most recently developed optical emission spectrometer range, the OE series, is ideal for the analysis of steel and ferrous materials. With its new detector technology, the OE750 offers outstanding performance in metal analysis. It enables the analysis of ultra-low carbon steels, the monitoring of nitrogen content in steel and iron casting processes, and the determination of other trace elements for these applications. And, of course, it provides reliable results for the most important alloying elements.







HOW OES WORKS

A small-scale electrical spark is applied to a metal surface which elevates the temperature of the surface to thousands of degrees Celsius. This applied energy excites the atoms at the surface, causing their electrons to leave their usual orbits and 'jump' to orbits at a higher energy level. Almost immediately, these electrons fall back down to their original ground state energy levels (passing through several energy levels as they do so).

As they fall back down, their excess energy is released as light of a given wavelength (a spectral line). Each element will release many different wavelengths of light and the resulting spectral lines act as a kind of signature that's unique to that specific element. Detecting these specific sets of emitted light characteristics is what tells us what elements are present in the sample.

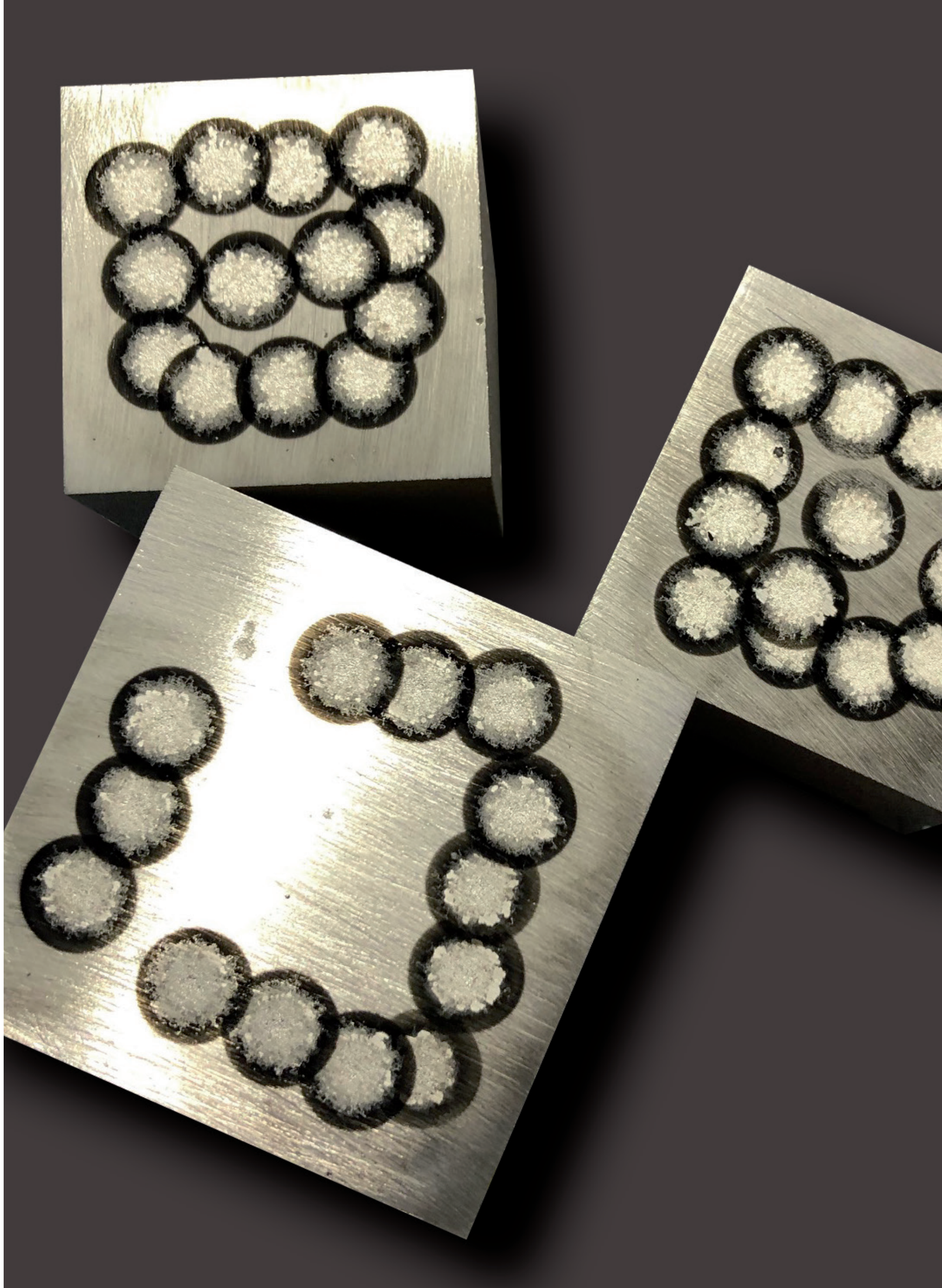
The technique is extremely accurate and precise, which is why OES is chosen for melt control, and tramp and trace element detection in sensitive components in metal fabrication facilities.

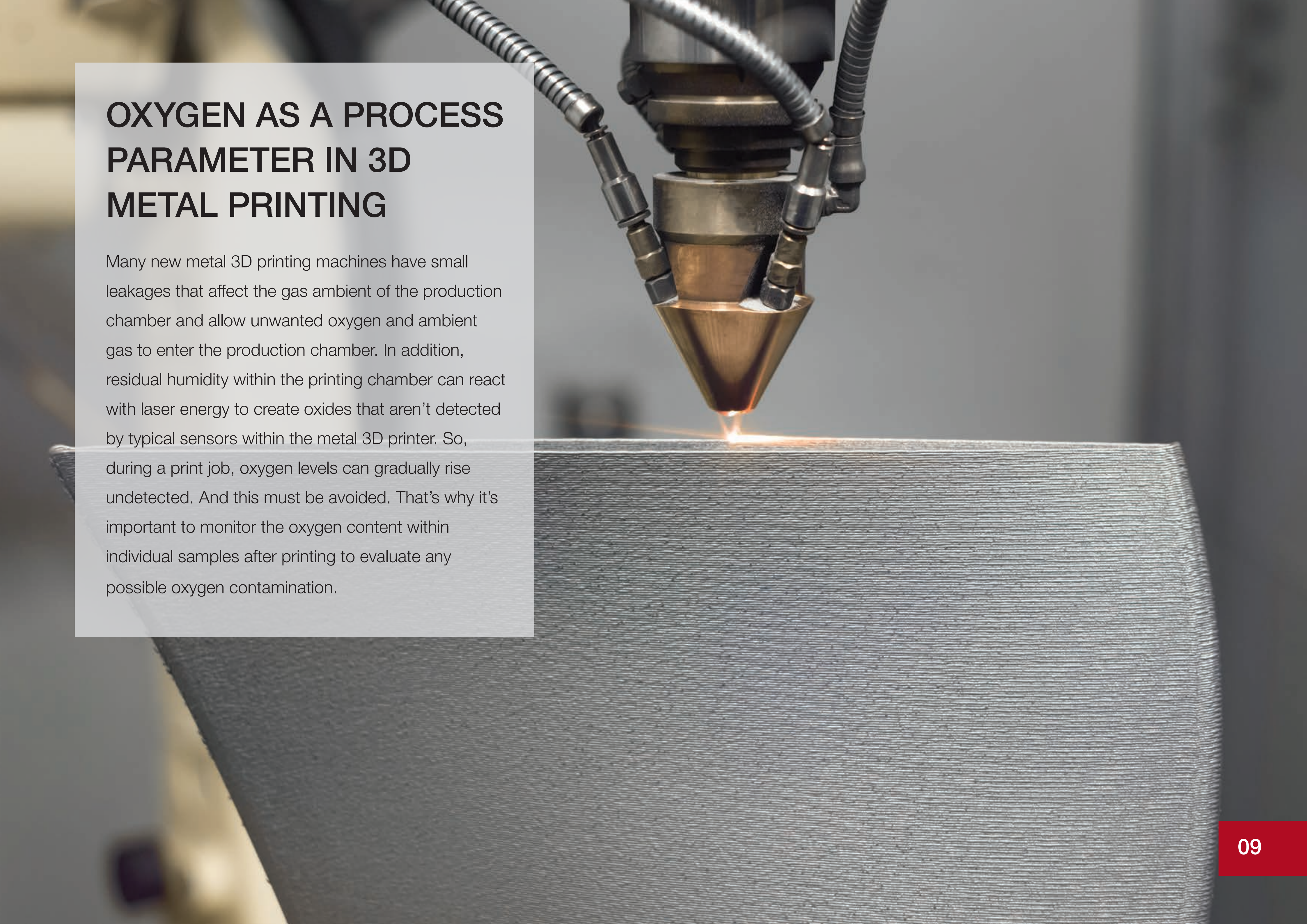
HOW TO PERFORM OFFLINE OES MEASUREMENTS OF 3D METAL PARTS

There are two ways you can use an OES analyzer within your additive manufacturing process: inline and offline. Inline is where the OES head is mounted right at the point where the powder is fused (i.e., next to the laser or electron beam). This allows you to monitor the composition of the component as it's being produced.

However, inline measurements are slightly more complex to set up and most of the time not as sensitive as offline measurements. Therefore, many facilities prefer to test using an offline setup.

With offline testing, a small control sample is produced alongside the main component and this control sample is tested within the OES instrument. The control sample can also be used for other quality control tests, as well as composition analysis. The sample then can be ground and tested with OES.





OXYGEN AS A PROCESS PARAMETER IN 3D METAL PRINTING

Many new metal 3D printing machines have small leakages that affect the gas ambient of the production chamber and allow unwanted oxygen and ambient gas to enter the production chamber. In addition, residual humidity within the printing chamber can react with laser energy to create oxides that aren’t detected by typical sensors within the metal 3D printer. So, during a print job, oxygen levels can gradually rise undetected. And this must be avoided. That’s why it’s important to monitor the oxygen content within individual samples after printing to evaluate any possible oxygen contamination.

SPECTROMETER REQUIREMENTS

Not all OES spectrometers are created equally; there's a huge variation in performance as you go from the cheapest instruments to the most expensive. When collecting OES data for additive manufacturing process monitoring, a spectrometer typically requires excellent spectral resolution to differentiate between similar atomic species.

For comprehensive metal 3D printing quality control, your spark spectrometer will need to be able to perform the following analysis:

TRAMP AND TRACE ELEMENTS

The detection of contaminants present within the raw material, or those accidentally introduced during the recycling process or storage.

POSITIVE MATERIAL IDENTIFICATION

Ensuring you're using the right metal powder for the final part and that is hasn't accidentally become mixed up with a different type within the printing chamber.

OXYGEN CONTENT IN SAMPLES

Control oxygen as a process parameter to ensure that oxygen content will not reach a critical level for printing in the 3D Feedstock.

Your OES analyzer must be capable of analyzing tramp and trace elements down to the ppm level to be confident of the impurity levels. If you need to perform carbon equivalence calculations, your instrument must reliably measure elements such as boron below the 5ppm level. Finally, not all OES instruments will be able to measure gasses or trace elements down to the level you need, so you should check the product specifications carefully before purchasing.

EXAMPLE MEASUREMENTS OF PRINTED SAMPLES

We analyzed several printed metallic samples with a gas-combustion technique to compare the gas content results with our newest spark spectrometer, OE750. The results showed quite similar values. However, using OES these results were obtained in a few seconds and not after few hours.

The following table shows typical results for a 316L printed sample with Hitachi's OE750. You see relatively good repeatability and accuracy. Nevertheless, the quality of these results should not be compared with the results of CRM (Certified Reference Material) measurements, as this is a printed process sample. The gas elements, N and O, have been double checked with combustion analysis.

The table shows only an excerpt of the analysis. In addition to the elements relevant to this alloy, the spark optical emission spectroscopy also offers a complete analysis of all feasible and relevant alloying elements for all common printed metal materials.

Burn	C	Si	Mn	P	S	Cr	Mo	Ni	N	O
1	0.0196	0.5102	1.369	0.0100	0.0086	17.39	2.515	12.61	0.0550	0.0550
2	0.0195	0.5026	1.352	0.0105	0.0087	17.41	2.509	12.57	0.0526	0.0526
3	0.0208	0.5144	1.360	0.0104	0.0077	17.31	2.527	12.73	0.0534	0.0534
4	0.0207	0.5029	1.357	0.0103	0.0077	17.32	2.522	12.44	0.0534	0.0534
5	0.0210	0.5067	1.356	0.0105	0.0074	17.41	2.521	12.66	0.0535	0.0535
6	0.0213	0.5052	1.343	0.0098	0.0077	17.30	2.506	12.65	0.0536	0.0536
7	0.0209	0.5046	1.263	0.0104	0.0078	17.41	2.552	12.60	0.0527	0.0527
8	0.0189	0.5107	1.257	0.0105	0.0075	17.26	2.550	12.80	0.0554	0.0554
9	0.0201	0.5128	1.309	0.0109	0.0072	17.37	2.535	12.74	0.0518	0.0518
10	0.0204	0.5099	1.357	0.0110	0.0080	17.35	2.540	12.69	0.0562	0.0562
AV	0.0203	0.5080	1.332	0.0104	0.0078	17.35	2.528	12.65	0.0537	0.0537
COMBUSTION									0.0550	0.0570
SD	0.0008	0.0042	0.041	0.0004	0.0005	0.04	0.016	0.10	0.0014	0.0061
RSD	3.8700	0.8200	3.440	3.4400	6.1600	0.33	0.630	0.75	2.5200	12.0200

AV = Average Value / **Combustion** = Analyzed by combustion methods / **SD** = Standard Derivation / **RSD** = Relative Standard Derivation

OE750 OPTICAL EMISSION SPECTROMETER

High performance at an affordable price

The ground-breaking OE750 has been designed with a brand-new optical concept that delivers results for all elements within metals, including gasses. This level of performance is usually only available from much more expensive instruments, yet innovations such as the use of dynamic CMOS detectors and direct coupling of the optics to the spark stand give the OE750 the optical resolution necessary for additive manufacturing applications.

In addition to the wide wavelength range of 119 nm to 670 nm, the OE750 keeps operating costs down thanks to low energy and argon consumption You can also extend the standard calibrations, making sure your analysis capability expands alongside your operations.

The analytical performance of the OE750 offers an easy and affordable solution for oxygen monitoring, making it ideally placed to support the growing need for quality control within additive manufacturing in metals.

If you've looked at OES instruments before and discounted them because of the cost, we highly recommend taking a look at the OE750

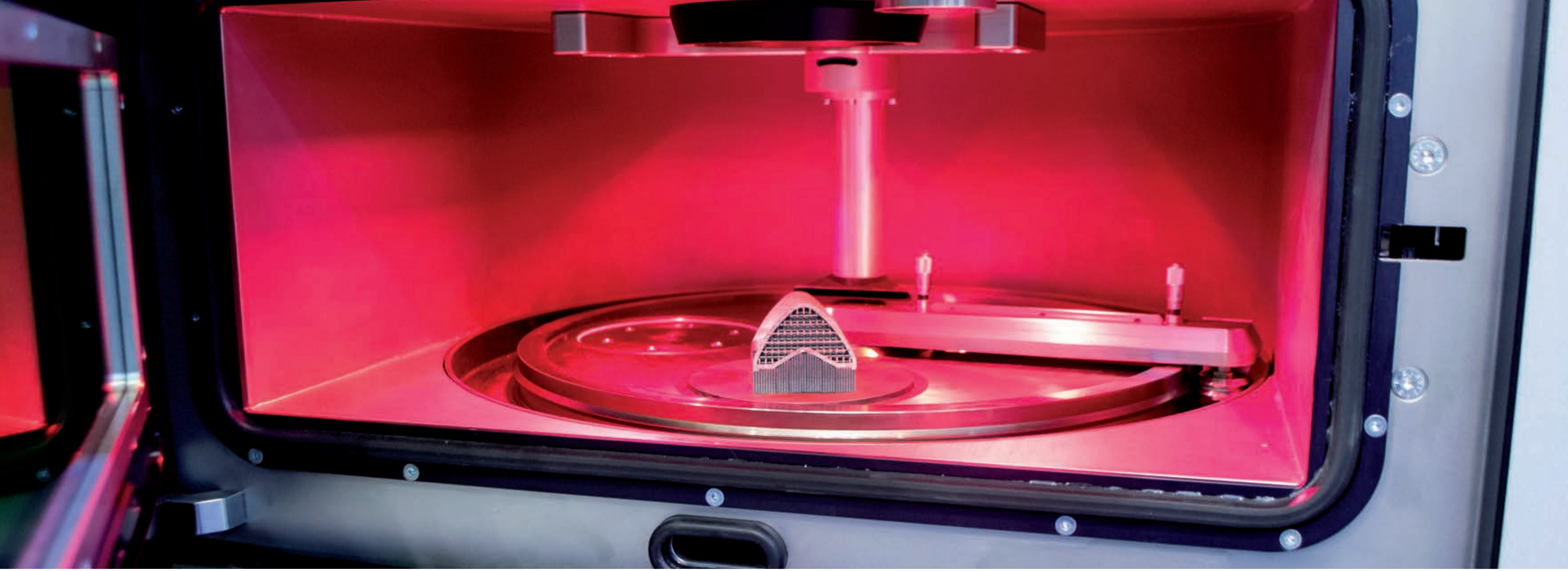
Case Study

SAMSON use the OE750 to support their quality control in metal additive manufacturing – including controlling oxygen levels

SAMSON currently manufactures valve parts from stainless steel 1.4404/316L. Eventually they intend to use other standard metal powders for components in process engineering. By applying the laser beam powder bed fusion method for metals (LB-PBF-M), SAMSON can make pressurized valve parts from the stainless steels 1.4401/316 and 1.4404/316L. Their 3D printer, a TruPrint 3000 by TRUMPF GmbH + Co. KG, produces valves in sizes up to DN 100/ NPS 4 that weigh up to 250kg.

SAMSON worked with Hitachi's application team to develop a method to monitor the oxygen content in their printed samples to ensure compliance with various norms and standards.

"The OE750 from Hitachi is the best way for us to perform analysis quickly and efficiently. We especially liked the robust design and sophisticated software, and these features convinced us from the beginning. Hitachi responded very quickly, and the applications team was very knowledgeable and helpful. It really is very easy to do business with Hitachi," says Dr. Martin Klein, the main user of the spectrometer.



LET US ANALYZE YOUR SAMPLES FOR YOU

We've been working alongside metal manufacturing and engineering companies for over 45 years, developing analytical techniques and solutions that support manufacturing processes, including quality and process control, as the industry specifications get more stringent. As a result, we've become experts in material analysis and today, we directly support our customers in finding the right analytical solution for their (often niche) processes.

If you have quality control issues with your printed products or are looking to avoid them, we want to help you. By working together with our application team, we'll be able to find the best solution for you.

How to get in touch

To arrange to send us your samples for analysis or to discuss your additive manufacturing requirements with an applications expert, get in touch with us at **contact@hitachi-hightech.com** or scan the QR code.

OTHER SOLUTIONS FOR ADDITIVE MANUFACTURING APPLICATIONS

X-ray fluorescence – or XRF – is a completely non-destructive analysis technique that can be used for metals and non-metals in solid, liquid and powder form. Like OES, it will deliver full compositional information on your samples and Hitachi offer both benchtop and handheld instruments for fast and easy analysis.

XRF instruments are used within electronics and general metal finishing to determine the thickness of coated layers. For example, our FT160 has an extremely small spot size and is ideal for analyzing the printed features that are less than 50 µm typically found in microelectronics applications. The X-MET8000 is a powerful handheld XRF instrument and is particularly useful for checking the composition of raw metal powders against the supplied certification.

X-MET8000 Handheld XRF

FT160 Benchtop XRF

About the authors:

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Dr. Maryam BeigMohamadi is responsible for OES application projects in Hitachi High-Tech Analytical Science. She has over 14 years' experience working with different analytical techniques such as optical emission spectrometry and high-resolution electron microscopy. She holds a PhD degree in physics from University RWTH Aachen in Germany.

Michael Molderings

Michael Molderings is responsible for the product management of Hitachi High-Tech Analytical Science's OES product portfolio. He has over 15 years' experience working with optical emission spectrometers and holds a master's degree in analytical science from Hochschule Niederrhein in Germany.

To explore the Hitachi High-Tech complete range of spectrometers for metals analysis, book a demo or to talk to a member of our team, contact us today.

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