Elemental Scientific LASERS

Rapid and Reliable LIBS Imaging with the ESLumen

ESLumen

LIBS (Laser-Induced Breakdown Spectroscopy) adds a whole new dimension to the analysis of solid samples. ESLumen expands your capabilities to include elements not normally measured by ICPMS (e.g. H, C, N, O, F).

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High Resolution Quantitative Imaging with ESLumen to Solve Catalyst Challenges

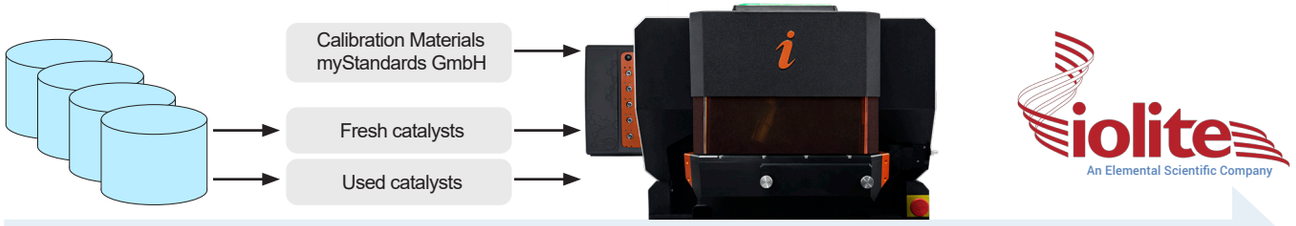
Synopsis

Johnson Matthey, a global leader in sustainable chemical technologies, produces catalysts that are used in advanced metals chemistry to create solutions for energy, chemical, and automotive companies. For certain applications, these catalysts are in pellet form and during the reaction some elements are lost due to sublimation. A clear understanding of this process is essential for JM to develop more robust catalysts that meet customer expectations.

At optimal conditions, a component in excess helps replenish vacancies left in the active sites of the catalyst. Eventually, this reservoir is depleted, altering the surface composition of the catalyst, while the inner part remains rich in the original material.

Acquiring a detailed understanding of catalyst surface interactions is vital for achieving optimal reaction conditions, thereby minimizing raw chemical usage and enhancing product yield.

Postmortem quantification of elemental loss is challenging as small, 4-6 mm diameter solid pellets are used, with changes occurring only at the catalyst surface. Acid digestion and analysis by ICPMS can provide accurate elemental data but offers no spatial information at the surface. An elemental surface imaging technique is required. Electron Probe Microanalysis (EPMA) was evaluated but found to be relatively slow, even for small maps. This note documents the ideal suitability of ESLumen for this application.



Analytical Workflow: JM catalyst samples, either fresh or used, in the production of formaldehyde. Samples mounted in TwoVol3 sample chamber within imageGEO193 laser ablation system. 5 x 5 mm maps created with 5 micron pixel resoution. Quantification using custom made calibration standards from myStandards GmbH. iolite 4 data processing software used to process the data including calibration and for image generation.

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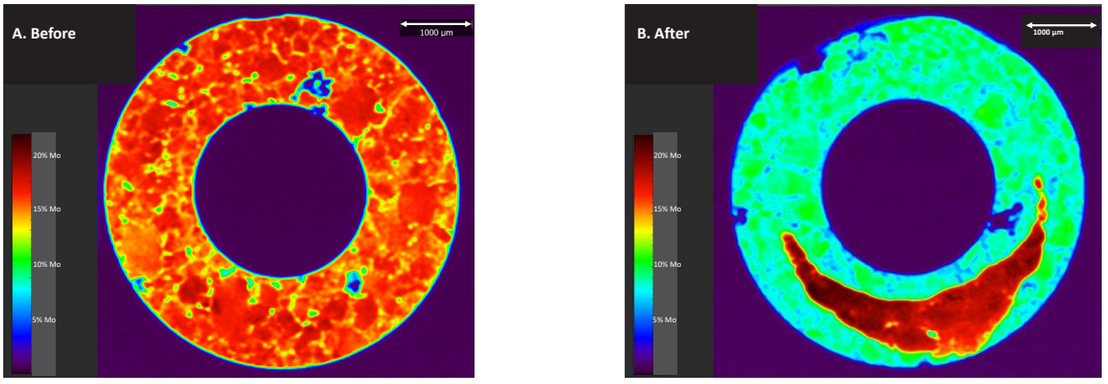
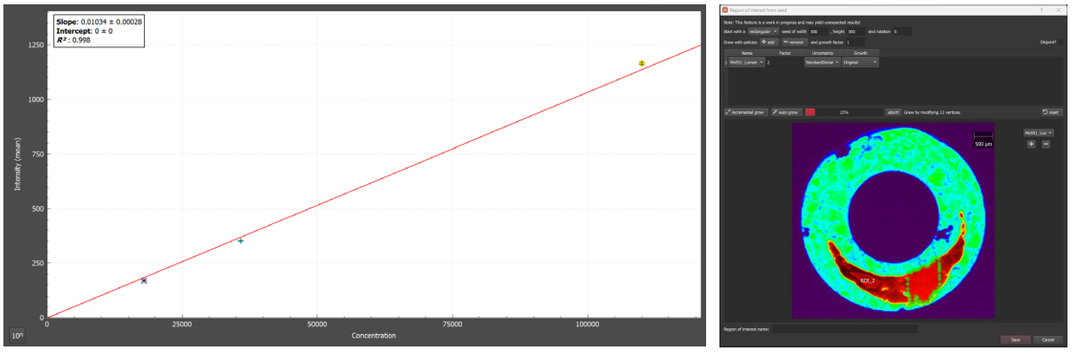
JM Catalyst Before and After Use



Element A, quantified at 551 nm, in JM catalyst before use (A) and after use (B). Both samples were ablated with a fluence of 10 J/cm^2, spot 50 µm, overlap 45 µm, raster spacing 5 µm, repetition rate 250 Hz, scan speed 1250 µm/s. Each image took <2 hours to obtain and clearly show Element A depletion on large portions of the catalyst surface.

iolite 4 Advanced Data Inspection Tools



A powerful software package for processing and visualizing data, iolite 4, allows a myriad of ways to pick out key findings from complex datasets. The Element A calibration on the left shows the three, tailor made, standards falling on a linear calibration line which has been fixed through the origin. This provides a high degree of certainty in the quality of the calibration routine for this application. The image on the right demonstrates iolite 4's ability to use highly advanced data inspection tools. In this case, a region of interest is being grown to highlight the high Element A area in the used JM catalyst. The inspection tool allows a small portion of the high Element A containing area to be seeded, with growth criteria based on user defined settings (2 SD was used for this example).

Conclusion

Johnson Matthey's processes are highly dependent on the quality and compositional stability of catalysts. Conventional methods such as EPMA lacks the throughput to allow the entire pellet to be analyzed. By contrast, ESLumen with iolite demonstrates superior suitability for postmortem analysis, enabling rapid, high-resolution elemental imaging of the entire pellet surface to obtain insights into the deactivation process on the surface of a catalyst.