CAMECA
SCIENCE & METROLOGY SOLUTIONS

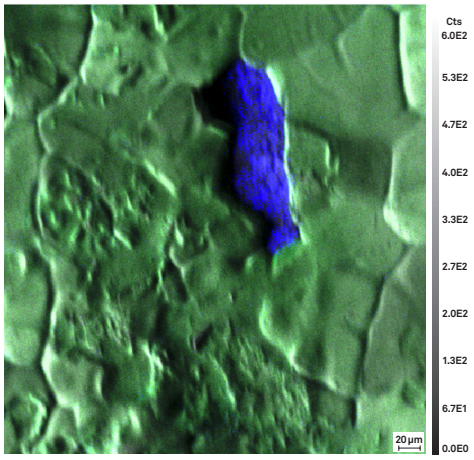
AMETEK®
MATERIALS ANALYSIS DIVISION

Building better steels with a CAMECA SIMS at ArcelorMittal

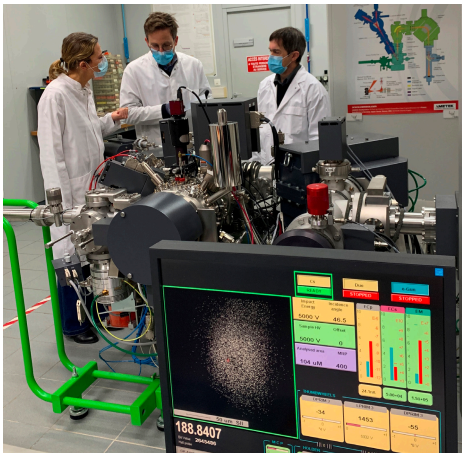
A SUCCESS STORY

CAMECA SIMS Advantages

- High-performance, universal magnetic sector secondary ion mass spectrometer
- Utmost-precision elemental & isotopic analysis
- Unparalleled depth profiling
- Extreme sensitivity
- Best detection limits on light & trace elements
- Wide versatility
- High reliability
- Materials science
- Metallurgy
- Semiconductors
- Environment



High-resolution SIMS mapping reveals an unwanted oxide (in blue) within a bare spot on aluminium silicium alloy coating.



At work on the CAMECA IMS 7f SIMS: ArcelorMittal researchers Valérie Champmartin, Sébastien Cremel, and Michel Pignon.

The Challenge

When is a sheet of steel just a sheet of steel? Practically never, nowadays. Modern steel coils produced to manufacture goods such as automotive parts, appliances, and packaging are instead formed from traditional iron (Fe) and carbon (C) plus a wide array of alloys; then often galvanized with corrosion protection metals and alloys — and finally covered by complex, ultrathin surface treatments containing elements such as manganese (Mn) or titanium (Ti), or organics.

Such sophisticated steels are key products for ArcelorMittal, the world's largest steel and mining company. Characterizing precisely which elements to include and what application process to recommend is a key responsibility for Dr. Sébastien Cremel and his team. He's the R&D Scientist in charge of the surface analysis platform — in the company's global research and development center at Maizières-lès-Metz, France.

As various coating formulations are proposed and tested on new steel chemistries in pilot studies, Dr. Cremel and the lab team apply a number of complementary techniques for quantification, profiling, and surface chemical mapping of the elements present on treated steel samples. Their analytical armament includes instrumentation for *X-ray photoelectron spectroscopy (XPS)*, *auger electron spectroscopy (AES)*, *glow discharge optical emission spectroscopy (GDOES)*, *scanning electron microscopy with energy-dispersive x-ray spectroscopy (SEM EDX)*, and *dynamic secondary ion mass spectrometry (D-SIMS)*. The team accesses even more instrument technologies internally or via research partners in other organizations — tools including an *electron probe microanalyzer (EPMA)* and an *atom probe tomography (APT)* microscope, both from CAMECA.

For many years, critical D-SIMS analyses were supplied by the lab's CAMECA 4f ion microprobe, installed in the mid-1980s. But the instrument's usefulness diminished as the decades passed, and newer instruments offered increased capabilities.

So in 2012, the team reviewed available options, and upgraded.

The Instrument

The ArcelorMittal lab team chose a CAMECA IMS 7f *secondary ion mass spectrometer (SIMS)* instrument (forerunner to the current IMS 7f-Auto model). This universal magnetic sector SIMS delivers advanced performance in trace element depth profiling and high-resolution imaging. Its unique optical system allows both direct ion microscopy and scanning microprobe mode.

The IMS 7f provides significantly higher transmission than with quadrupole analyzers. And it delivers much lower detection limits and a higher throughput than *time-of-flight (TOF)* SIMS instruments.

For the lab team, the new instrument represented a welcome improvement. "For sure, the gap of the CAMECA IMS 7f compared to our old 4f was big — especially in terms of electronic automation, informatics, and software," Dr. Cremel says. "Since the update, we've now got a powerful tool to answer a large range of questions in relation to product development and quality control — for high added-value coated steels, to be used in automotive and packaging applications."

The Work

In one series of regular studies, the lab performed defectology analyses on carefully selected samples from testing of new metallic coatings for automotive use. In the plant, a fast-moving band of alloyed steel would be galvanized by dipping in melted zinc (Zn) or other protective metals or alloys to form a galvanization layer.

A key concern highlighted by the studies: bare spots. Low-wettability zinc was failing to adhere to certain areas of the steel. If large enough to be visible, these defects would mar the aesthetic appearance of the material. And bare spots of any size — many of these were only 10-15 microns — would invite eventual corrosion in the finished product.

The team attacked the problem using a variety of cross-referenced analytical techniques (including SEM, GDOES, and SIMS). The IMS 7f SIMS was applied using its unique "checkerboard" option, which combines the advantages of both depth profiling and ion imaging modes.

"SIMS is very useful for this," explains Dr. Cremel. "The checkerboard option helps us analyze a very wide area — we used a 500-micron zone, to include a defect plus its bordering 'healthy' area. Then, in post-treatment, we could compare the depth profiles between the two."

The Results

The analyses revealed manganese oxide or mixed oxides of a given composition and thickness at the bottom of a number of sampled bare spots. The team concluded that these resulted from oxidation of a manganese alloying element in the steel during a continuous annealing step, prior to hot-dip galvanization.

Analyses of defects in other steels also showed the presence of thin oxides of boron (B) — another alloying element — in bare spots. In these cases, the IMS 7f's low detection limits for light elements such as boron proved especially invaluable, since technologies such as SEM EDX can match these capabilities only with difficulty.

The team uses the results of pilot studies like these to fine-tune recommendations for later plant production. During annealing, for example, these might include adjustments that optimize temperature, line speed, and dew point to prevent formation of the target defects. Thus the lab's SIMS instrument can help fulfill the plant's requirements for high-volume production and high-precision quality.

"At conferences, people will come up to me and say, 'We are amazed by the quality of the images you get.' And of course, SIMS is also excellent for depth profiling of low concentrations of trace elements," Dr. Cremel says.

But he feels the ArcelorMittal lab has not yet exhausted the full potential of the CAMECA IMS 7f SIMS. "With it, we can solve a lot of problems that we were not even *thinking* about before!"

About ArcelorMittal

ArcelorMittal is the world's leading steel and mining company. Around 168,000 employees in more than 60 countries produce steels for automotive, construction, household appliances, and packaging markets. Research and development are central to the company's strategy, with 15 research centers in Europe and the Americas holding 1,500 researchers. The ArcelorMittal Maizières research campus is the group's largest research site, with 600 people plus numerous students, apprentices, partners, and trainees accommodated each year. The facility's Products Center is home to the metallic coating and surfaces laboratory, which performs physical/chemical characterization studies to help develop safe and sustainable steels for the future.

About CAMECA

CAMECA is a world-leading supplier of microanalytical and metrology instrumentation for research and process control. Our instruments measure elemental and isotopic composition in materials down to atomic resolution. Advanced CAMECA technologies include secondary ion mass spectrometry (SIMS), electron probe microanalysis (EPMA), atom probe tomography (APT), and low-energy electron-induced X-ray emission spectrometry (LEXES). We address challenging characterization needs in diverse markets, including materials sciences, metallurgy, nuclear, life sciences, geology, cosmochemistry, environmental, and semiconductor research.

www.cameca.com

CAMECA SCIENCE & METROLOGY SOLUTIONS | AMETEK MATERIALS ANALYSIS DIVISION

Corporate Headquarters
29 quai des Gresillons – 92622 – Gennevilliers Cedex – FRANCE.
cameca.info@ametek.com – Tel + 33 1 43 34 62 00
www.cameca.com

Regional Contacts
www.cameca.com/contact/international-distributors