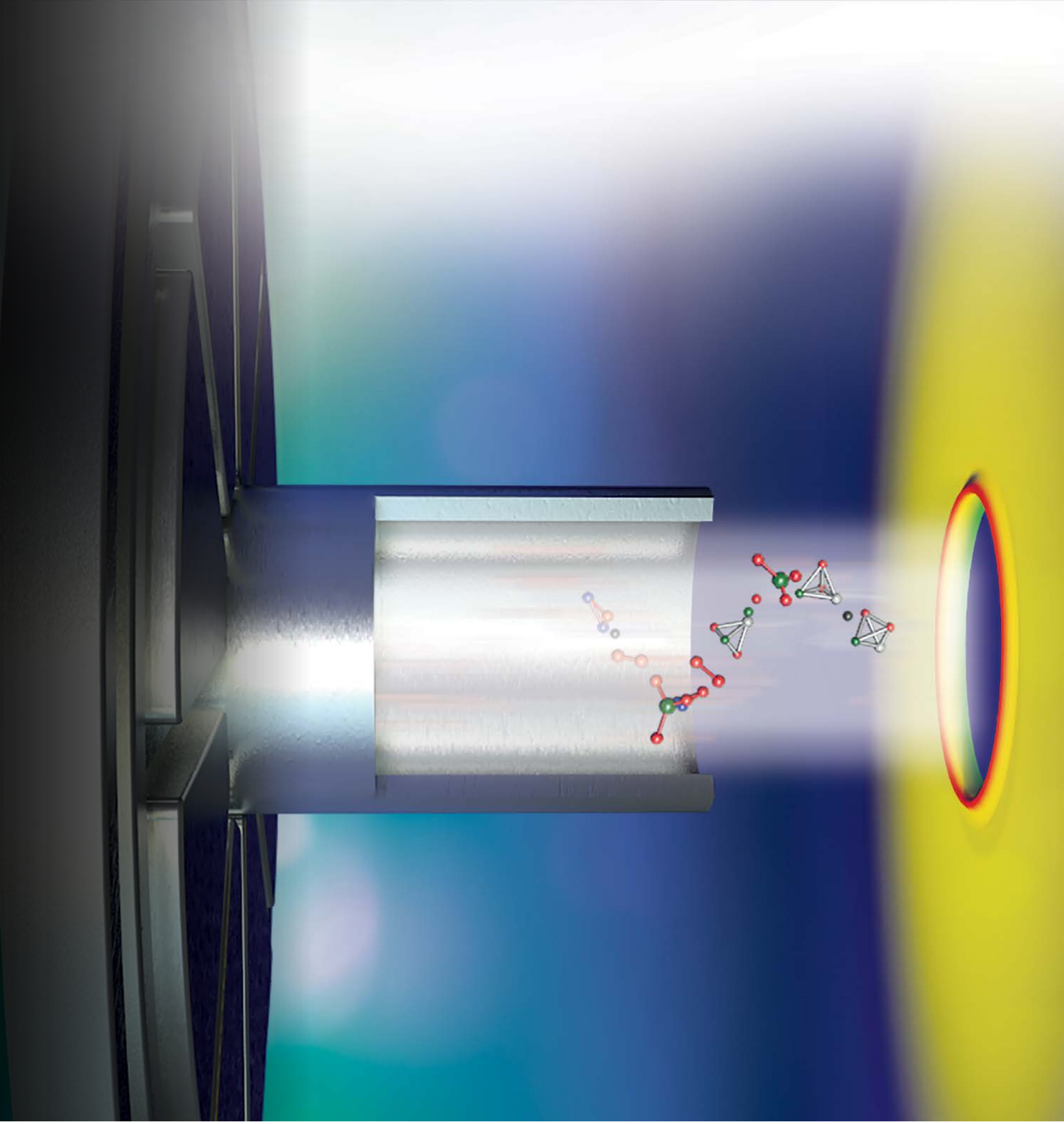
PULSED RF GLOW DISCHARGE
OPTICAL EMISSION SPECTROSCOPY

Ultra Fast Elemental Depth Profiling



ELEMENTAL ANALYSIS

HORIBA Scientific

What is Pulsed-RF GD-OES?

Until now GD-OES was not able to perform direct thickness measurement or to characterize complex materials involving organic/inorganic layer and temperature sensitive materials. But that was until now…

GD-OES is an elemental analysis technique.
A typical GD-OES instrument includes:

- A Glow Discharge source that creates a plasma used to sputter a sample and excites sputtered species
- An Optical Emission Spectrometer that collect the light, separate the wavelengths and measures the intensities

GD-OES can measure all elements from Hydrogen to Uranium, including Oxygen, Nitrogen, Carbon, Chlorine, Deuterium and is widely used for bulk analysis of metallic samples.

Under optimized conditions the crater shape on the sample, resulting from the sputtering, is flat allowing for depth profiling analysis with high depth resolution. Thanks to this feature, GD-OES can be used for the characterization of coatings.

The capability of a technique to perform depth profiling from the first nanometer down to 150 µm deep into the materials can be of utmost importance for many applications including most advanced materials: lithium battery electrodes, photovoltaic cells, metals involving inorganic and organic coatings, heat sensitive materials…

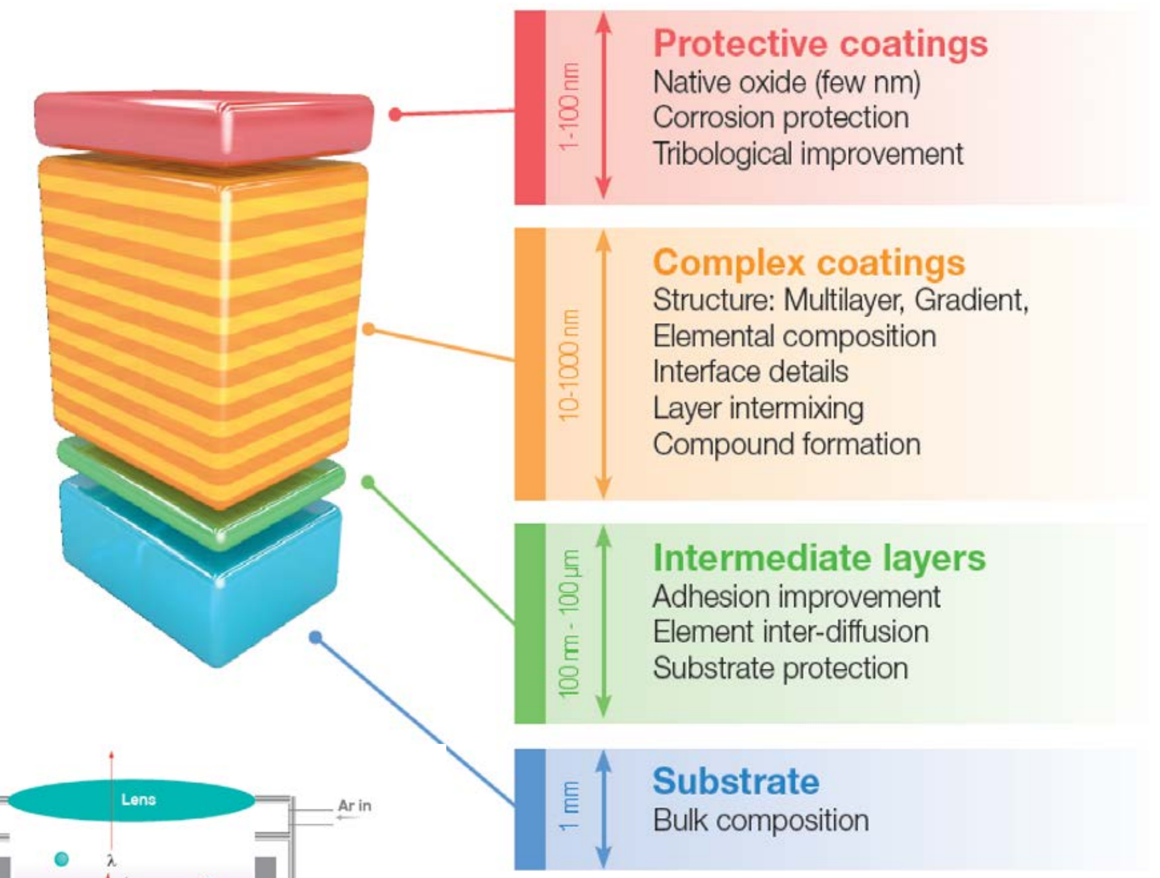
Standard GD-OES instruments are facing multiple challenges to handle such samples that may contain organic layers, may have element whose distribution will be affected by heat dissipation or whose structure will be damaged by the sputtering.

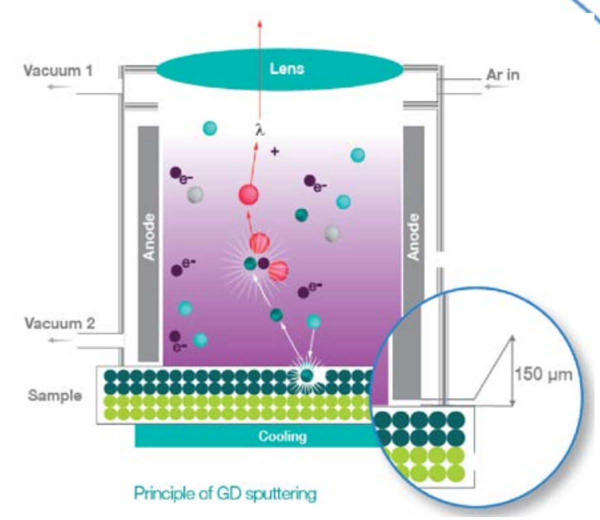
GD-Profiler 2

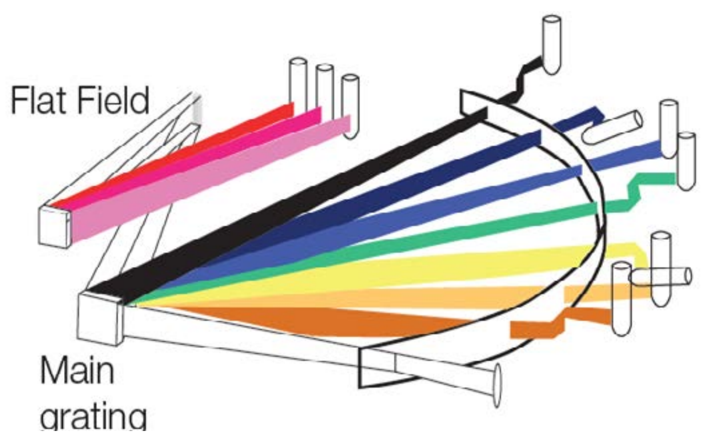
In 2005, HORIBA Scientific introduced GD-Profiler 2 as a standard Glow Discharge instrument. Over the past years, multiple new features were added to make it the most advanced Glow Discharge instrument on the market to characterize the most advanced materials.

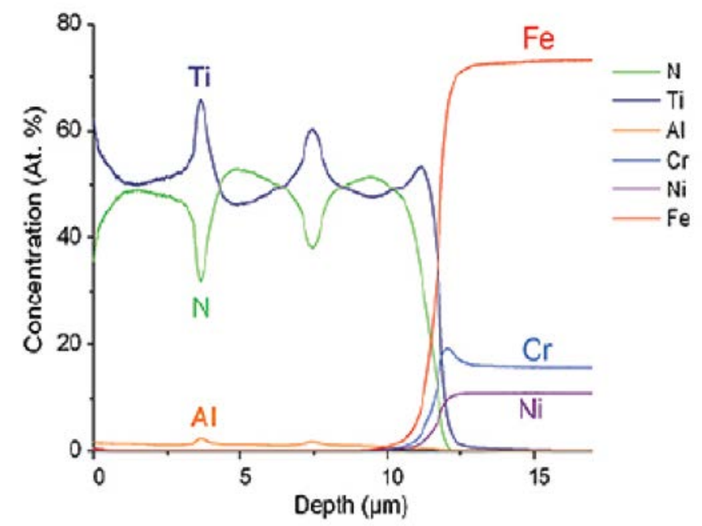
GD-Profiler 2 is now equipped with a RF generator able to work in Pulsed Mode to handle conductive, non-conductive and heat sensitive samples. It also features the Ultra-Fast Sputtering mode for fast sputtering of organic layers, DiP for direct depth and layer thickness measurement. It can also be equipped with unique accessories to manage air sensitive and odd-shape samples.

Learn more









Gas turbines are the worker bees behind any energy production. Increased efficiency and size reduction is a meaningful objective.

Making better gas turbines

Large, land-based gas turbines are the worker bees behind energy production. These devices convert the heat from nuclear fuel, concentrated solar power and fossil fuels like coal and gas, into electricity.

Thermal energy generated by these sources heat up a working fluid, which converts heat into rotary motion of the turbines. That motion generates electricity for our power grid.

Researchers are looking at Supercritical Carbon Dioxide (sCO_2) as a working fluid for a range of power generation applications. Supercritical CO_2 works as a heat transfer fluid at a low critical temperature and pressure. It is a friendly and fully recyclable solvent, stable, recoverable through pressure reduction and is a potentially more efficient working fluid.

The real benefit of Supercritical CO_2 is that it allows for much smaller turbo machinery..

Protecting components

But like other working fluids, Supercritical CO_2 interacts on a molecular level with the turbine blades. It acts like both a gas and a liquid. Supercritical CO_2 has many of the heat transfer properties of liquids. Corrosion and carbon ingress are two of them.

Michael Lance, Ph.D.

That's where Michael Lance, Ph.D. comes in. He is a senior research and development scientist in Oak Ridge National Laboratory's Materials Science and Technology Division.

His job is to analyze the compatibility of structural alloys used in these turbines submerged in Supercritical CO_2. He wants to document the corrosion and carbon ingress of the alloys as it is exposed to the working fluid. And he wants to build a model to predict these situations based on the alloy's composition under various environmental conditions.

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Making better gas turbines, cont.

GD-OES explained

The basis of GD-OES is the sputtering of the surface with an electric field-induced radio frequency plasma - or glow discharge - using low-pressure argon gas. The argon collides with the surface, sputtering atoms off the sample surface that then enter the plasma. The sputtered atoms collide with high-energy electrons and gas atoms and become excited.

These excited atoms de-excite by optical emission, releasing photons with energies that are characteristic of the atom. A grating disperses the optical emission that the spectrometer measures with photomultiplier tubes. The measured intensities are directly proportional to the number of sputtered atoms for each element.

The alloys are generally stainless steel compositions.

Lance said it's easy to measure the X-Y dimensions, that is the surface of the alloys after undergoing exposure to the Supercritical CO_2. But it's the Z axis, the depth on the material, that is harder to study.

Modeling depth behavior

"For our modeling, what we're interested in is where these elements go. How are they diffusing as we expose them," Lance said. "Most of the things are changing in the Z direction, in the depth. And that's what GD-OES allows us to measure. So it's very useful for looking at corrosion."

Lance exposes these compounds to high temperatures in different atmospheres – pressures - and measures the depth profile.

"We can measure the oxide scale composition, but also of interest is within the metal beneath the corrosion," Lance said. "The alloy can become depleted in elements that are forming the oxide scale at the surface."

GD-OES is very useful for measuring that depth profile over a very large spot.

"We can average a lot of material and see very tiny changes that would be hard to see using other techniques," he said.

Lance wants to quantify any potential alloy degradation. A specific concern for Supercritical CO_2 environments is the ingress of carbon into the alloy, which researchers have observed for more than 50 years in both iron and nickel-based alloys. That can affect the longevity and performance of the turbines.

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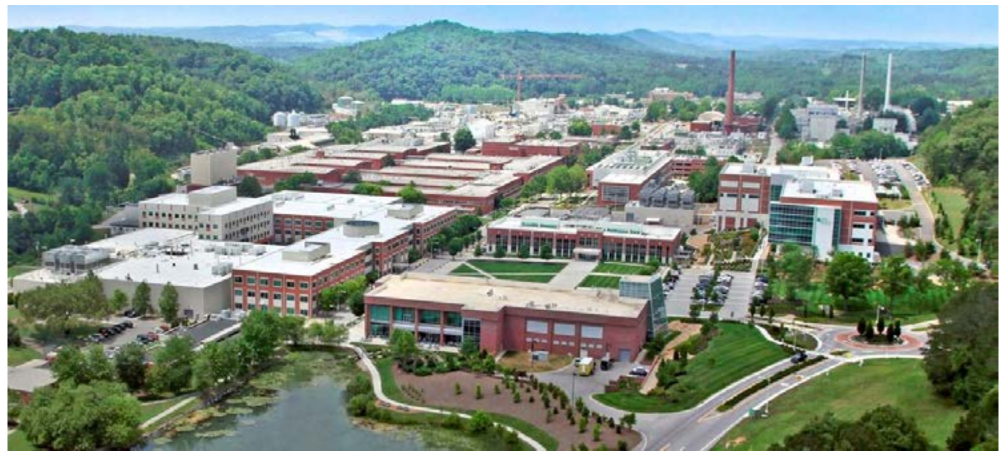
Making better gas turbines, cont.



Oak Ridge National Laboratory in Oak Ridge, Tennessee

Oak Ridge National Laboratory

Oak Ridge National Laboratory is Tennessee-based science and technology center sponsored by the U.S. Department of Energy. A contractor, UT–Battelle administers, manages and operates the lab as a federally funded research and development center.

The lab has produced research for a variety of scientific efforts, from creating better crops to studies on nuclear fusion.

A key focus of Lance's research is to measure the amount of carbon ingress as a function of time and temperature in order to predict long-term behavior. Long-term such as a 30-year lifetime for a concentrated solar power application.

"The problem is that the carbon dioxide (from the Supercritical CO_2) will breakup at the surface of the alloy and the carbon from that will actually go into the alloy," he said. "That can be very bad. It can embrittle the sample. So we are interested in understanding the carbon, what's called the carbon ingress."

Other technologies

Other methods to characterize the corrosion have been tried. The electron microscope will create x-rays characteristic of the elements present. But it doesn't work well with light elements like carbon.

That tends to cause the peaks to be weaker, which, which hurts the limit of detection. You can also get carbon contamination in the electron microscope. Researchers have used GD-OES to measure concentration depth profiles on corrosion samples for many years.

Shorter-term returns

Lance's work borders between basic and applied research, yielding results private entities incorporate into industrial designs. Funding for Lance's program is through an applied program. His group is in close communication with industry. "They're telling us what they're interested in, and where we're trying to fit their needs. We are considered to operate within the Department of Energy as A HORIBA GD Profiler-2 at the Oak Ridge National Lab in Oak Ridge, Tennessee. applied science.

However, for most people, for politicians, they will think of us as basic. So it depends on your perspective."

The government funds other researchers at the lab through a line called basic energy sciences.

"They're really looking at pie-in-the sky things that won't work for certain," Lance said. "Maybe somebody will use it 30-years from now. So we're much closer to what industry is currently interested in. Nevertheless, we're still more basic from industry's perspective."

A HORIBA GD Profiler-2 at the Oak Ridge National Lab in Oak Ridge, Tennessee.

Lance works with a HORIBA GD-Profiler 2™ in his lab to perform depth analysis using GD-OES. It provides fast, simultaneous analysis and is an ideal tool for thin and thick films characterization and process studies.

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Stories

Nuclear energy is an attractive energy source with no emission of greenhouse gases. New technologies should provide more efficient, cheaper, and longer lasting nuclear power plants…

Designing a new breed of nuclear reactors

To most scientists, climate change is real. The challenge is to find more and better energy sources to generate electricity that do not emit greenhouse gases into the atmosphere.

Nuclear energy has been an attractive option, and accounts for about 20 percent of the U.S. electricity supply. The rate is a little more than worldwide - about 14%. In France, it's as high as 75 percent.

Yet the current U.S fleet of nuclear reactors are aging. Engineers built most of them in the 70s and the 80s. Some have reached the end of their licensed life and their owners face either closing those down or working towards extending their licenses.

Scientists are developing radical, next-generation technologies to produce more efficient, cheaper, and longer lasting nuclear power plants. Designs will include passive safety, where reactors will shut themselves down automatically in emergency instances.

Adriean Couet, Ph.D., an Assistant Professor at the University of Wisconsin-Madison's Department of Engineering Physics

Adriean Couet, Ph.D., an Assistant Professor at the University of Wisconsin-Madison's Department of Engineering Physics is working on these advances.

His job is to understand the aging of nuclear materials and how that can affect the design of these advanced nuclear reactors for longer lifetime and efficiencies.

"I do work to find materials that can sustain the environmental conditions of this new generation of nuclear reactors," he said. "We want to go to harsher environments such as higher temperatures."

Water coolant, the traditional cooling method, won't do in the next generation of nuclear reactors with higher design temperatures.

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Designing a new breed of nuclear reactors, cont.

Nuclear primer

A traditional nuclear reactor works by juxtaposing nuclear fuel rods in close proximity, immersed in water. Nuclear fission – where atoms split and release energy in the form of heat - raises the temperature of the water. That water converts to steam and drives turbines that generate electricity.

One way of achieving higher temperatures without pressurizing, which ultimately represents a safety concern, is to using alternative cooling media, like gas, molten salts and liquid metals that can transfer heat. That's part of the advanced nuclear technology.

"For the next generation of nuclear reactors, since we need to increase temperature to increase plant efficiency, water is not suitable anymore," Couet said.

Surviving the elements

The environmental conditions in a reactor are harsh. It exposes materials to high temperatures, irradiation damage and corrosive media.

"When you build a structural material for a nuclear power plant, this material is in contact with the coolant. That is where it is transferring the heat or contain the coolant media. It can be quite corrosive," he said.

Take molten salts for instance. Leave your car next to the ocean for a year, and it's going to corrode. That's because the ocean air is saltier and salt is going to stick to your car. Salt is very corrosive. It's basically the same thing in an advanced nuclear reactor if you use molten salt, since molten salts can be much more corrosive than regular water.

"Corrosion is a big problem for advanced nuclear reactors in terms of materials, because, the liquid that is going to be used is more corrosive in general," Couet said

The expectation for advanced nuclear plants is in finding materials that can sustain the conditions of these next generation reactors. Engineers use nickel alloy, steel and other high-temperature alloys, for instance, in the aerospace industry as well as other high temperature applications. Researchers test new alloys for ones that can withstand this environment with less corrosion, and Couet is at the forefront of that effort.

The materials used in the reactor must withstand temperature corrosion and irradiation for decades. Those three conditions are going to degrade the material property over time. determination of TPC concentration.

Designing a new breed of nuclear reactors, cont.

Replicating conditions

It’s very challenging to reproduce those conditions in the lab,” Couet said. “You need the irradiation, the temperature, maybe molten salts or liquid metals. So it's a very challenging, and that's one thing my group is trying to reproduce.”

He tries to replicate the conditions of a nuclear reactor and test different alloys for degradation. To do this, he uses large furnaces that can raise the temperature of a liter sample to hundreds of degrees Celsius for thousands of hours.

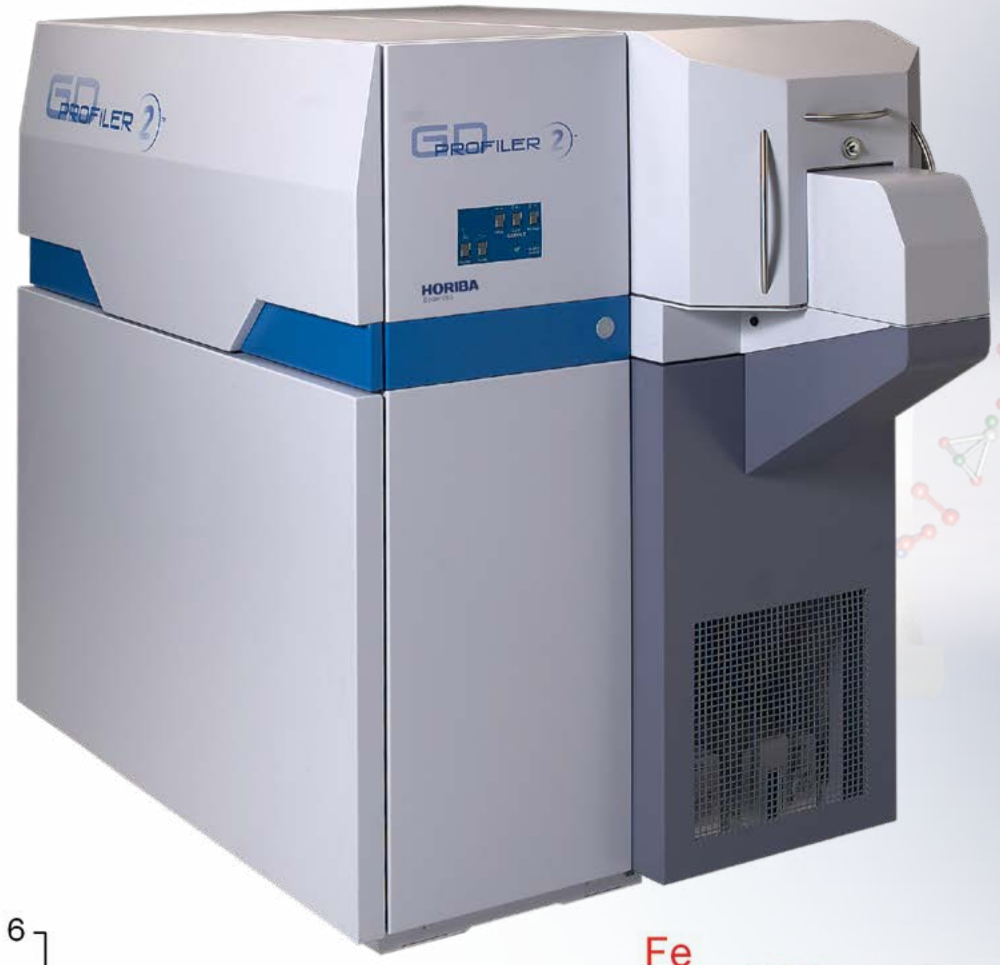
“We try to understand materials degradation and maybe model this degradation in the future,” he said. “If we can simulate or model degradation, then we can predict, for longer exposure times, how the materials are going to behave.”

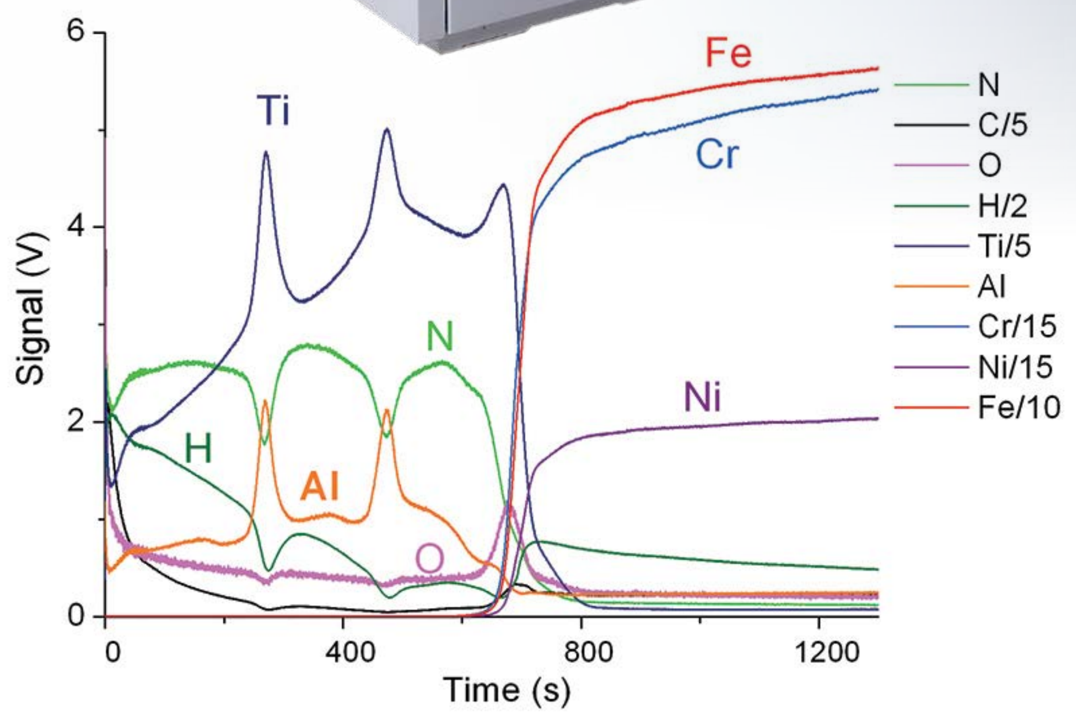
His team exposes each alloy and cooling medium samples to 1,000 hours or more under these conditions and then characterizes the samples. To test the samples, Couet uses multiple materials characterization techniques, including electron microscopes.

He also uses Glow Discharge Optical Emission Spectrometry (GD-OES) using a HORIBA GD-Profiler 2™. With it, he bores into the post-corroded sample, which may be a new alloy, and is able to measure the element concentrations in the alloy at nanometer depths. That helps him determine what effects the harsh, artificially created environment had on his sample.

“We want to know how deep the environment has penetrated into a sample or how deep the materials chemistry has degraded,” he said. “We are still using materials that have been used for 60 years, 70 years in nuclear power plants. They work well, but we still don't really understand the fundamentals of their corrosion. So even though the material works perfectly well and it's been used for decades, it's still interesting to try to understand the fundamental science, because then you can apply that to other advanced systems you do not have as much experience with.”They work well, but we still don't really understand the fundamentals of their corrosion. So even though the material works perfectly well and it's been used for decades, it's still interesting to try to understand the fundamental science, because then you can apply that to other advanced systems you do not have as much experience with.”

HORIBA GD-Profiler 2





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Designing a new breed of nuclear reactors, cont.

Changes in the funding environment leads to the next step

How far away are we from a new age of nuclear technology?

“Not very far, to be honest,” Couet said.

Countries across the world have built several test reactors with these new technologies. That includes the U.S., France, Russia, Japan and China. In the U.S., the private sector has been investing in these efforts over the past decade. That money is providing a funding stream, alongside money from the Department of Energy, for this new set of technology.

“There are a lot of investors, actually startups and new companies that have emerged,” Couet said. “Sometimes it's about developing a newer reactor, sometimes it's about a service to a nuclear sector. I think it is the first time since the birth of civil nuclear energy that there is some kind of a synergistic effect between public and private funding in the history of nuclear energy.”

A shifting landscape

Couet said that back in the days when students came to the university to do graduate level research, they wanted to work in a national lab or large utility. Now more and more students want to work in a startup, face new challenges and take risks.

It might be another 10 years before this new technology begins generating electricity, according to Couet. Moreover, it could be 2040 before it contributes to the electric grid.

Yet despite the need for better materials for reactors, and the demand for renewable energy and clean energy sources, Couet is still a pure scientist at heart.

“Sometimes you do the science for the sake of science,” he said. “Even though it can be a bit controversial, doing science for the sake of better understanding our universe and environment is also something that is beautiful.”

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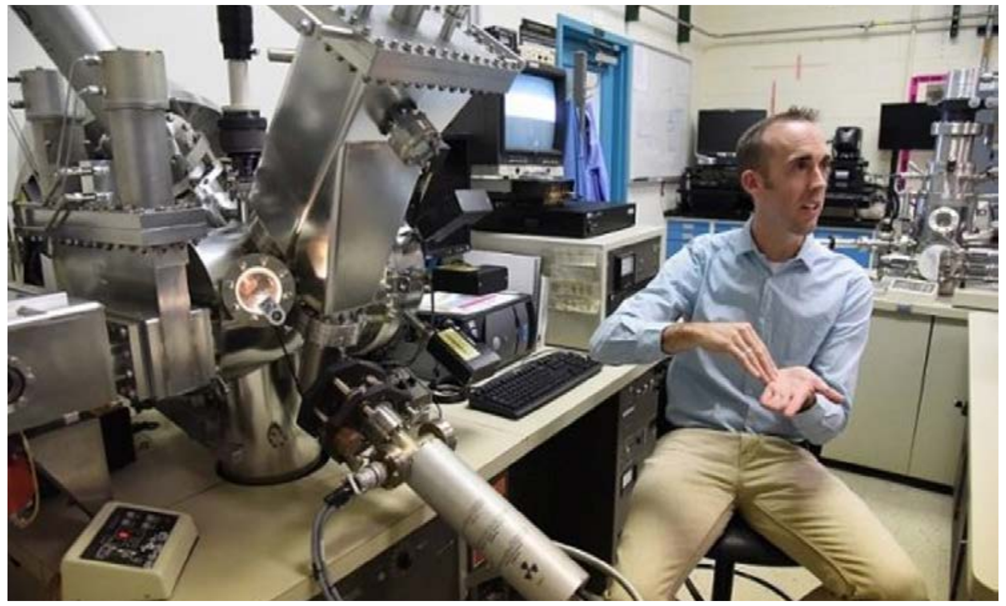
*Nuclear fusion is viewed as the holy grail of clean, renewable energy.
The science that goes into fusion is complex and getting there is a long road…*

How fusion breakthroughs will lead to clean renewable energy



Chase Taylor, Ph.D.

Nuclear fusion is viewed by many as the holy grail of clean, renewable energy.

Although studied since the 1920s, scientists have yet to overcome technological issues and the economics of this process that promises to deliver energy in the future.

Yet, in 2025, scientists plan to fire up the first fusion reactor slated to produce more energy than it takes to run it. That will prove the concept of an economically viable alternative contributor to the electric grid.

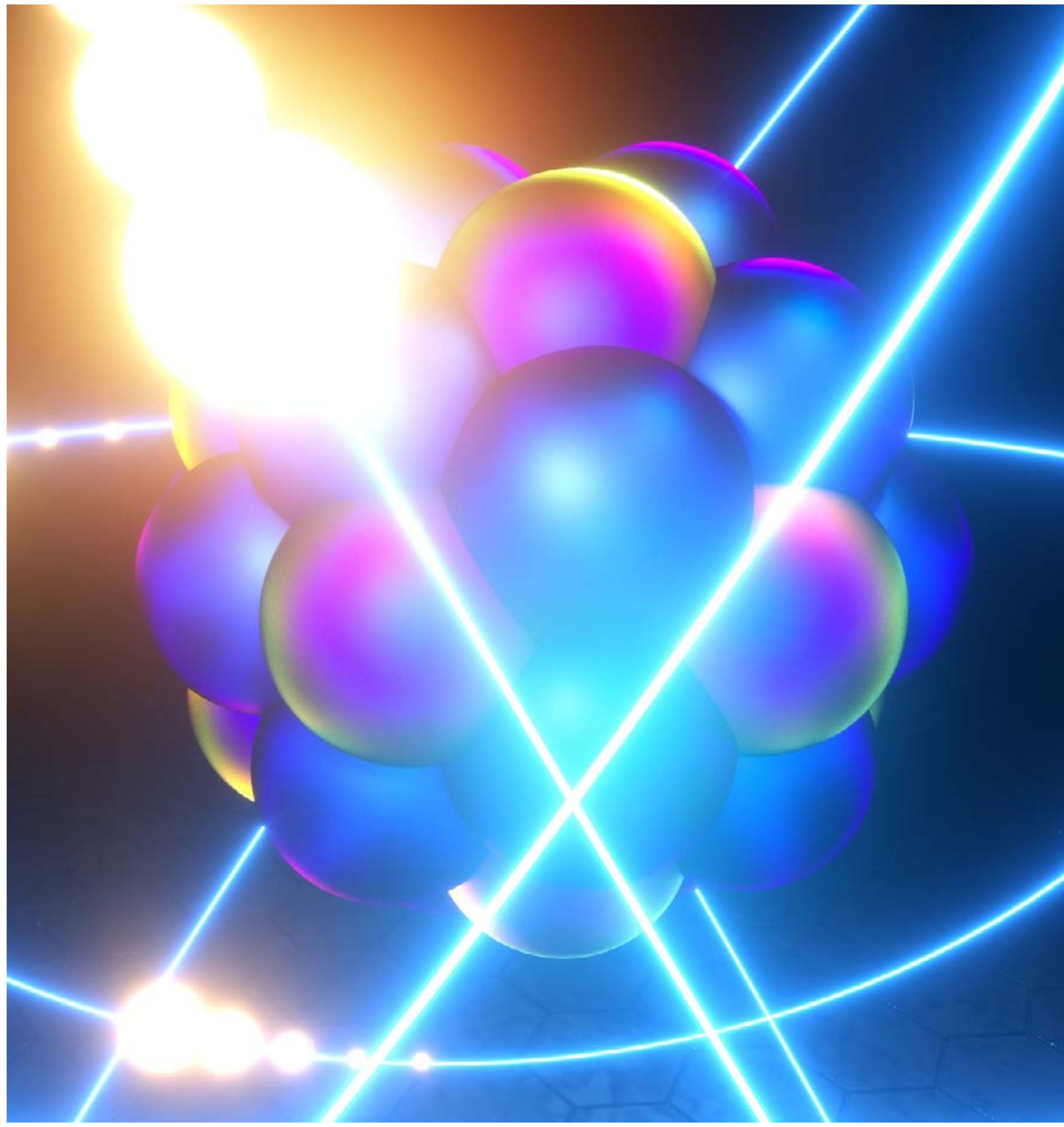
Getting there is a long road. Nuclear engineer Chase Taylor is a senior staff scientist with Idaho National Laboratory, a nuclear research facility. He is hard at work solving some of the problems that will make a commercially viable fusion reactor possible.

Converting energy

Power plants generate electricity by converting mechanical power, like the rotation of a turbine into electrical power. The plants rely on either fossil fuels, nuclear fission or renewable sources like hydro to turn the turbines.

Fission powers today's electricity-producing nuclear reactors. Nuclear fission releases heat energy by splitting atoms. The energy produced by the reaction heats water, which produces steam to turn turbines and ultimately produce electricity.

Fission has many advantages. It provides very reliable, low emission energy, is long-lasting, and has the lowest annual mortality rate of any energy resource. It is well understood, and advanced fission reactor systems can further amplify its benefits in smaller packages with even stronger safety performance than current designs.



Fission reactor systems can be dangerous when poorly designed or managed, as events in Chernobyl and Fukushima have shown. Large-scale plants have high initial capital investment and its waste products require very long-term storage if recycling efforts are not used.

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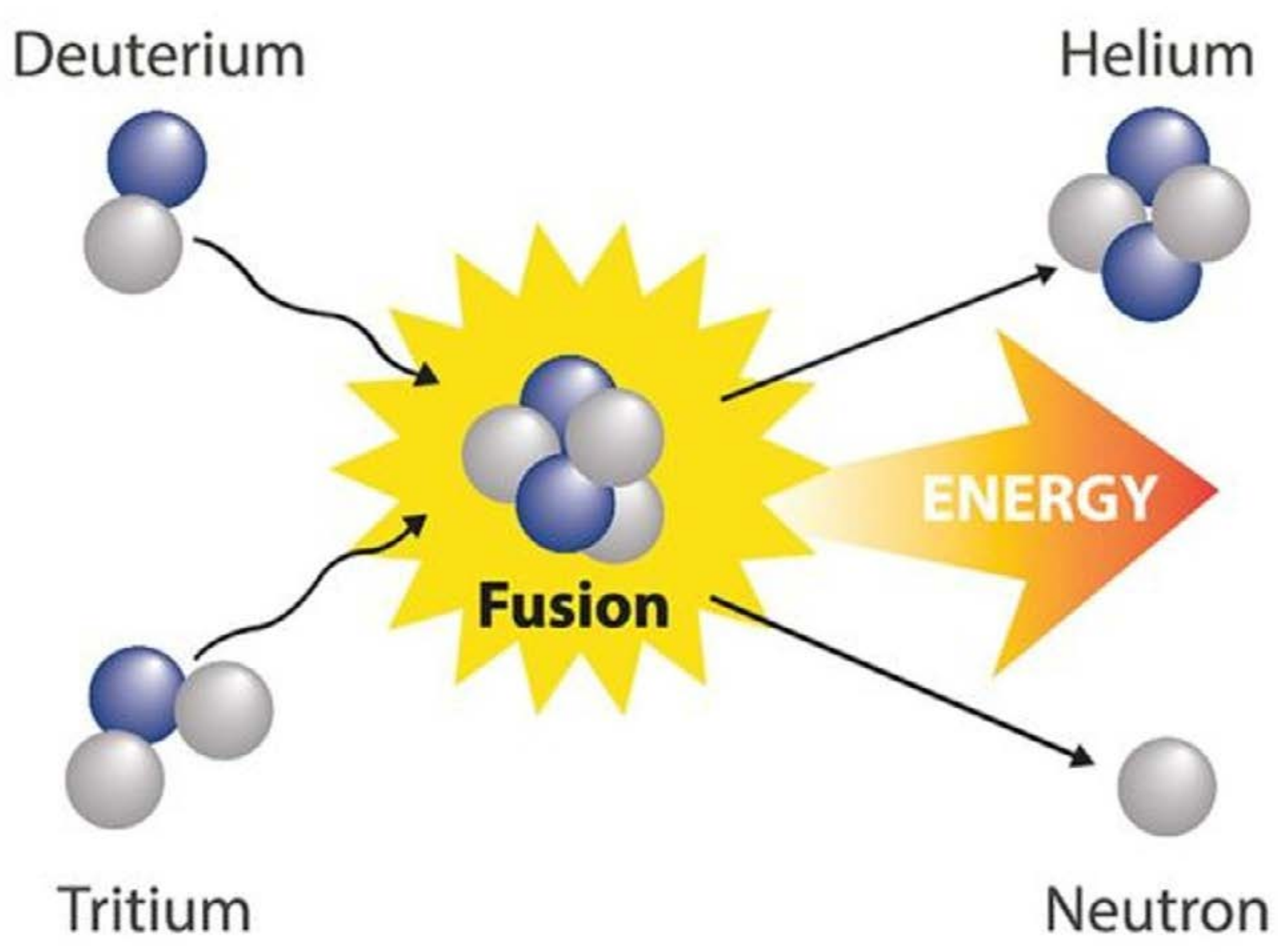
Stories

How fusion breakthroughs will lead to clean renewable energy, cont.



The Fusion Process

Fusion, a separate process, is a nuclear reaction where atomic nuclei of low mass fuse to form a heavier nucleus with an accompanying release of energy. It's what powers the sun.

A fusion reactor uses hydrogen isotopes, deuterium and tritium, as fuel. Isotopes are variations of a chemical element with different numbers of neutrons in the nucleus.

The supply of fusion fuel is virtually unlimited. Scientists can produce tritium during the fusion reaction while in contact with lithium, and deuterium can be found in seawater. Although seawater has a small fraction of the element, fusion fuel fills our oceans, giving us a virtually limitless fuel supply. Another benefit to fusion is that a fusion reactor cannot have a runaway catastrophic nuclear event, like a meltdown.

"It's not possible. Even in the most remote possibility," Taylor said. "It's not that it's engineered to be safe, it's just the physics of fusion. Simply, if anything goes wrong, the fusion reaction will stop. That's actually what makes fusion so challenging."

But fusion has some drawbacks. Controlling a fusion reaction has proved difficult. The hydrogen bomb is based on an uncontrolled fusion reaction. If the same amount of energy could be released gradually in a controlled manner, fusion could live up to its promise.

Fusion also leads to radiation damage on plant systems and some radioactive waste, and will also initially have high operating costs.

The problem

Fusion uses these isotopes in a plasma, a kind of flowing gas that responds to electric fields, like the gas in fluorescent lights. A fusion reactor heats that gas to extremely high temperatures and compresses it with magnets.

But some of the material leaks out and slams into the tungsten armor tiles that line the walls inside of the donut-shaped reactor. A fusion reactor produces neutrons, which can penetrate deep into the walls and create a pathway for the deuterium and tritium to follow. That's an inefficiency in the process and a possible safety issue.

"We are interested in looking at how much of that deuterium and tritium, that should be used as fuel is instead getting stuck in the tungsten tiles, and how deeply it is trapped in the tile," Taylor said.

How fusion breakthroughs will lead to clean renewable energy, cont.

Regulatory barriers

From a regulatory point of view, operators are limited to a certain amount of deuterium and tritium in a facility. But those isotopes are intended to be used as the energy source. If some of it is stuck in the walls of the reactor vessel, the deuterium and tritium aren't available to create heat and eventually electricity. Yet it still counts against the operator's regulated limit. That's an efficiency issue that must be overcome.

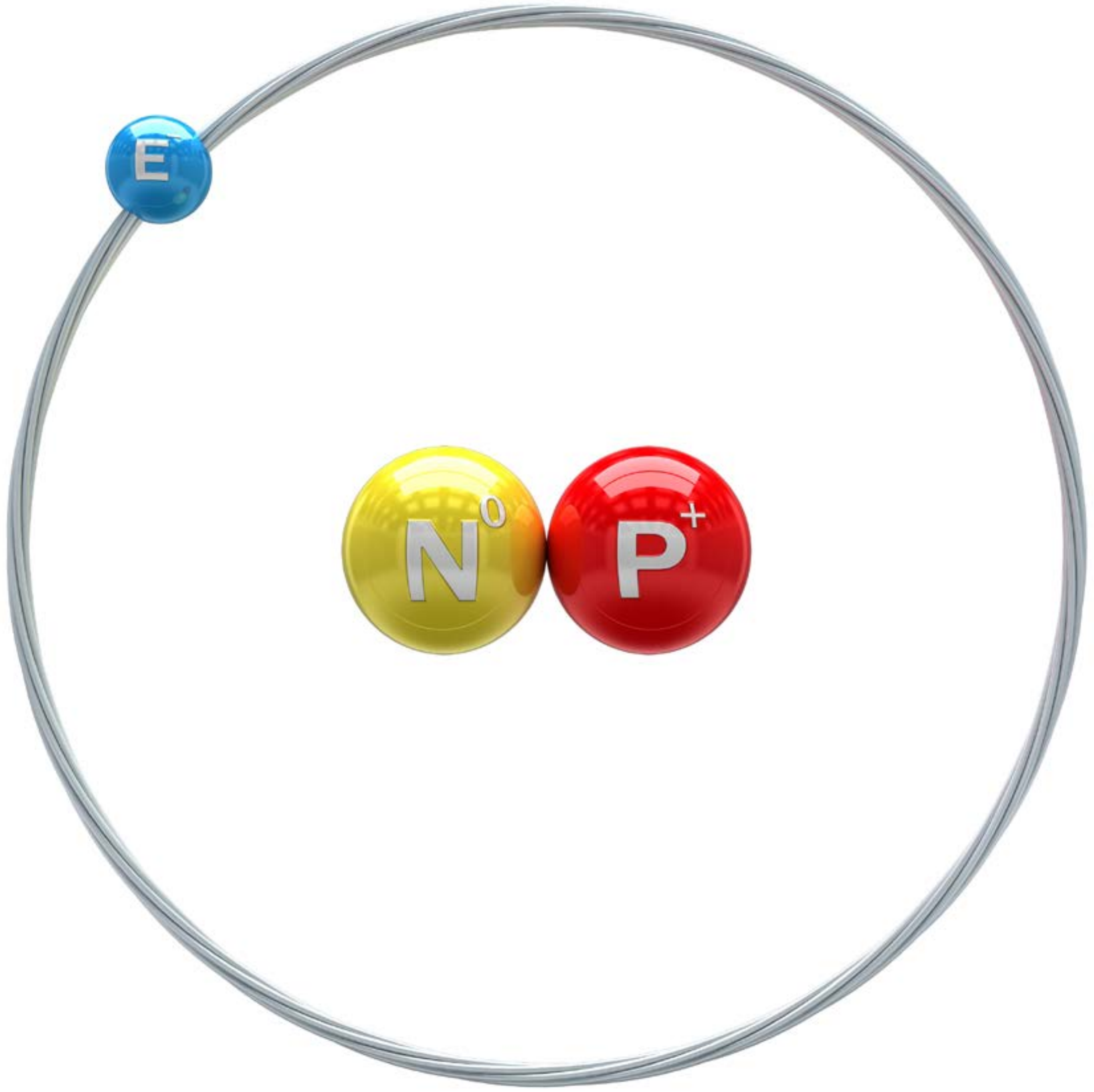
"By studying how much deuterium and tritium penetrate the walls and how deeply, we can better understand this process," he said.

Measuring the loss

Two standard techniques exist for measuring the total quantity of tritium that penetrates into the walls. One is by heating up the tile sample and boiling out all of the trapped deuterium and tritium. Scientists make the measurement with a quadrupole mass spectrometer. But that doesn't tell you anything about how deep the isotopes were trapped in the sample.

The second and somewhat less available technique is nuclear reaction analysis. Unfortunately, that is limited to just a few microns in depth, between three and eight microns in the tungsten. It can't see anything deeper than that.

"Some publications estimate that technique (nuclear reaction analysis) misses up to 90 percent of the total deuterium or tritium



that is stuck in the sample," Taylor said. "So it's difficult to say in an accident-based scenario, based on that technique, how much deuterium and tritium is actually stuck."

Taylor sought out a technique that would be sensitive and to allow him to look much deeper into the sample.

GD-OES

That's where Glow Discharge Optical Emission Spectrometry (GD-OES) comes into play. It measures the elements present in the sample, at what concentration levels, and of course, how deeply the materials have penetrated into the tungsten tiles.

GD-OES burrows into the sample, characterizes its composition and measures the depth of penetration of the radioactive substances.

"Very few techniques are even sensitive to deuterium and tritium," Taylor said. "But optical emission spectroscopy is one of those techniques that is sensitive to it. And this technique also lets us quickly look up to a hundred microns in the sample, which far surpasses the capabilities that are currently considered standard in the fusion materials community."

It also helps determine if the deuterium and tritium are going out the backside, permeating through the tiles or just stuck in the middle.

Taylor uses a HORIBA GD-Profiler 2 GD-OES instrument for his analysis.

The GD-Profiler 2 provides fast, simultaneous analysis of all elements of interest including nitrogen, oxygen, hydrogen and chlorine. It is an ideal tool for thin and thick films characterization and process studies.

"It's for managing the fusion fuel cycle essentially," he said.

How fusion breakthroughs will lead to clean renewable energy, cont.

Measuring the loss of isotopes

Permeation or penetration will occur. Knowing to what extent and how deep it is can allow scientists to engineer the systems to account for the loss of isotopes.

"If the deuterium or tritium is going to permeate through the first layer, then we need to control that at the second layer behind that," he said.

Taylor operates a linear plasma device that simulates the most energetic portion of a fusion reactor. He uses that linear plasma device to implant deuterium and tritium into tungsten tiles.

"We know how much deuterium and tritium hit the tile," he said. "Then we remove (the tile) and take it over to the glow discharge system, measure how much actually got stuck in it, and at what depth it got stuck."

He uses that information to feed into models so scientists can extrapolate into other conditions. It helps build a database showing what levels the materials permeated into the walls under various conditions. Those conditions include fluxes, or how many particles hit the tiles per second; temperature, which can range between 200 to 1,200 degrees Celsius; and the helium concentration, a byproduct of the fusion reaction.

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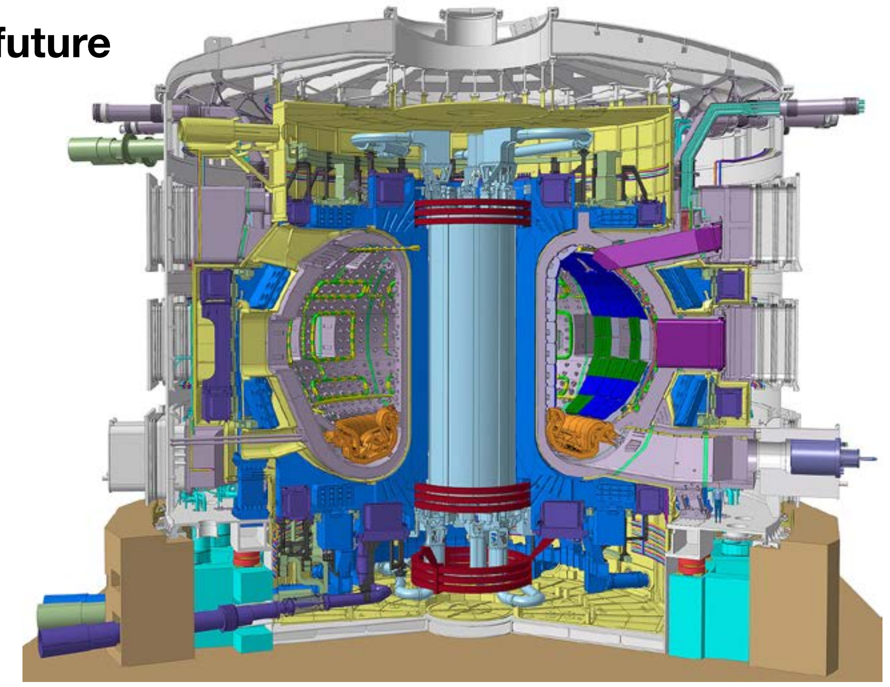
The future



An artist's rendering of a fusion reactor by the ITER, an international nuclear fusion research and engineering megaproject. It will reach temperatures hotter than the sun.

An enormous international collaboration called ITER – meaning "the way, or journey" in Latin – is building a fusion reactor in the south of France. Scientists designed the reactor to prove the feasibility of fusion as a large-scale and carbon-free source of energy.

ITER will be the first fusion device to produce net energy – when the total power produced exceeds the power required to heat the plasma. It's expected to be the first fusion device to test the integrated technologies, materials and physics regimes necessary for the commercial production of fusion-based electricity.

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The collaborators launched the joint experiment in 1985. ITER members include China, the European Union, India, Japan, Korea, Russia and the United States.

"If that's successful, then commercial entities would begin to develop their own (fusion reactors)," Taylor said. "We're probably couple of decades away from that, with development and regulatory issues."

Path to fusion

As an undergrad, Taylor was doing an internship related to fission.

"I attended a lunch seminar about fusion and really, my jaw hit the floor. I hadn't really heard about fusion up until that point."

The enormous complexity of the science that goes into fusion appealed to him.

"I was planning on going to grad school, and the next summer I got an internship in fusion. That was pretty cool."

Taylor earned his master's and doctorate degrees in nuclear engineering, and his research focuses on fusion materials.Now his future will help determine the health of the planet.

Contamination on a product can halt a production, generating huge daily losses, until it the source gets identified. Quick investigation is key…

Materials characterization crucial in Silicon Valley systems

Fuhe Li, Ph.D.

A semiconductor manufacturer in the Silicon Valley faced a sizable setback. Something was contaminating its product, and the company halted production. That cost the manufacturer millions of dollars a day.

The company called in Fuhe Li, the Director of Advanced Materials for Air Liquide Balazs. Li's division of the multinational, French-based company supplies material characterization services to support the semiconductor and electronics industries.

If the contamination were on the surface of the product, the cause would be in the manufacturing process. If it were in the interior layers of the product, it would point to faulty materials as the cause.

Li and his team used GD-OES, which stands for Glow Discharge Optical Emission Spectrometry. It analyses the layers of a material on the nanometer level and can reach depths of microns with a nano meter resolution. GD-OES contributes to the development of new materials with coatings at the nano-scale and upward.

Scientists also use GD-OES in many industries. That includes monitoring photovoltaic devices manufacturing, understanding the origin of corrosion on painted car bodies, assessing the composition of precious metals, controlling hard disks or LED manufacturing, improving Li batteries, detecting microchip defects and other applications.

The GD-OES analysis of the industrial contamination, using a HORIBA GD Profiler 2 revealed an impurity in the interior of the product. That meant the manufacturer had a materials supplier issue. The manufacturer's engineers were able to identify the source of the contaminant and production resumed shortly after.

"We're not just doing analytical work to produce results. We help them to solve their process related issues and provide solutions," Li said. "The Profiler can provide most of the information we need and vertical location of the elements containing the contaminant."

This was a typical situation facing the team at Air Liquide Balazs, which also works with other high tech customers like disc drive makers, the nanotech, the military and aerospace industries.

Li, who was recruited 22 years ago out of UC Berkeley where he conducted his postdoctoral studies, has a warm, hearty laugh. It belies the seriousness of his work. He problem-solves mission critical processes for technology-based companies.

Marjorie Balazs founded Li's division in 1975. She was a pioneer in Silicon Valley at about the time when the Intel and Fairchild semiconductor businesses began ramping up their semiconductor manufacturing. Balazs saw the need for analytical material characterization.

The company has thousands of clients with the majority in the U. S. and Canada. Others are located in Asia and Europe.

NEXT »

Materials characterization crucial in Silicon Valley systems, cont.

GD-OES

Finding contaminants in the interior of the product faces challenges. Scientists use pulsed RF GD-OES to investigate materials from the surface down to more than 150 microns with a depth resolution that can be as good as 1 nm. Once the contaminant is identified, Air Liquide - Balazs scientists and manufacturers' engineers will team up to locate the contamination source in the processes.

The production processes for these high-tech companies are secretive, involving a lot of intellectual properties. The clients do not share it with Balazs. In fact, non-disclosure agreements with the companies prevent Li from revealing the names of the company's clients or the material contaminants found in the supplier's materials.

The technology is a moving target.

"This industry is too dynamic, too technical, and the things that we talk about today, tomorrow, they will change the process again," Li said. "Every day there's some new."

Technological challenges and customer needs, Li said, are what drives this innovation.

"It's difficult to keep pace with a customer. New problems are popping up all the time. It's because of the competition. The customers are driving all those changes."

The importance of elemental composition

A substance's elemental composition affects many of its properties.

"Material used in aerospace or that people use to build bridges, those materials can crack, peel, fatigue or rust," he said. "It is in a very corrosive environment. So the elemental composition of the material is crucial in order to prolong the lifetime of the material."

Li's group uses the GD Profiler to look at the composition of the material and evaluate its content uniformity – to see whether the elements are distributed uniformly in the material. In Li's words, he looks at the concentration variation as a function of depth. A GD-OES profile provides surfacing and uniformity information, and bulk composition.

It's a way to make sure the supplier is sending the same product as ordered. Engineers perform incoming material characterization to make sure that they are getting the material they need.

"This is critical for several industries," Li said. "Aerospace, they must prevent the airplane from going down because of material fatigue over time. In semiconductors, it's to make sure parts won't fail."

Balazs services the consumer electronics industry, including digital multimedia communications. Every part the manufacturers use must pass rigid industrial specifications.

HORIBA GD-Profiler 2

Materials characterization crucial in Silicon Valley systems, cont.

Other techniques

GD-OES isn't the only method used to inspect materials. Many older, more expensive and slower techniques have been more widely accepted in industry because they have been around longer.

"There are no techniques can do it all," Li said. "It really depends on what problem they have. We are the experts that decide what technique to use."

Li is a proponent of glow discharge technology.

"The information that we provide to customers is very valuable," he said. "Some of the information we provide to our customers with a GD-OES is something that our customers have never had in the past."

For example, Li's group used GD-OES on insulating materials, and were able to profile it from its surface down to 50 to 60 micron in depth. No other technique, especially those traditional electron beam and ion bean technologies could accomplish that, Li said.

"The beam has charge," he said. "When you shoot the charged beam onto insulating materials, you will create a charging effect that usually prevents us from getting any reliable information from the material. That charging effect is very bad and very severe. Sometimes it doesn't generate any signal."

GD-OES doesn't have that effect. Li uses an RF alternating current, sputtering material layer by layer. He wrote a couple of papers on that, trying to promote GD-OES as a viable technology.

"We can change the industry techniques that are common practice by using a HORIBA non-traditional approach to improve industrial common practice."

Li said the stress and challenges brought by constant crisis is worthwhile.

"At the end of the day when I drive home, I just feel they're rewarding. You're helping customers, you're making direct impact on a society, and we're making a difference."

Air Liquide Balazs headquarters in Silicon Valley

Applications - Information Notes

Information Notes:

- GD-Profiler 2
- Steel, Aluminum, Packaging, Drill Bits, Corrosion and Electroplating
- Renewable Energy
- GD-OES, SEM and XPS

Applications - Application Notes

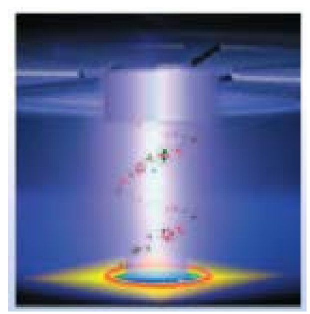
- Films with metal and metal oxide particles
- Electrodes of Li-ion batteries
- Measurement of hydrogen and deuterium
- Magnetron sputtering deposition
- Organic and organic/inorganic ltilayered materials
- Mobile phone screen cover
- Electroplated coating
- MoS_2/Pb nanocomposite coatings
- Perovskite solar cells

Since its invention in the late 60's-80's, HORIBA Scientific GD-OES went through numerous technical improvement to make it relevant to advanced materials characterization…

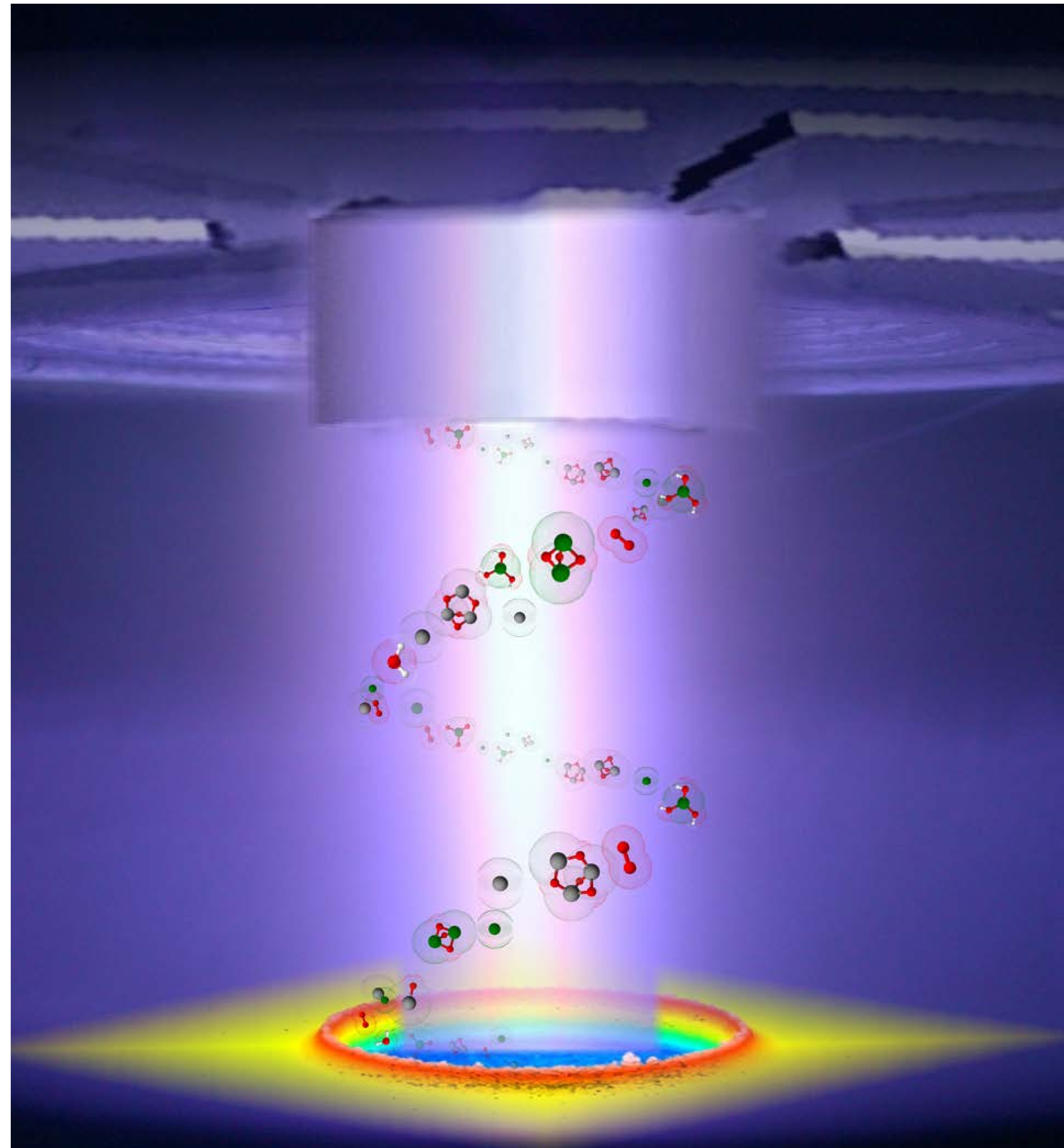
If the scientific examination of the glow discharge plasma happened during the latter half of the 19th century, the Glow Discharge Optical Emission Spectrometer in its closest form (compared to modern instruments), was invented in Europe by researchers from the metallurgical industry, mainly from Germany and France, in the late 60's – 80's and was first used for bulk analysis. Soon people realized that the GD source could perform depth profiling of thin and thick films. However, both the economic situation of metallurgy in Europe at that time, and the lack of scientific papers published – or published in French or German only – contributed to keeping GD-OES as a "confidential" technique.

Since then, and despite the first depth profiles published being of GaAs thin films in 1970, the GD-OES technique developed in the metals industry and is now widely used for elemental bulk analysis and depth profiling for electrically conductive metallic coatings characterization. Over the past fifteen years however, multiple new instrumental developments have enlarged the field of applications of GD-OES to include the characterization of coatings and thin films for advanced materials, making it an essential tool for quality control and process optimization/monitoring.

Glow Discharge is now able to characterize many different materials, electrically conductive and not, covering a wide range of applications from photovoltaic (CIGS, Perovskites…) to packaging, from organic electronics to energy storage (lithium batteries, fuel cells…) and is the characterization companion tool to a variety of deposition techniques of thin and thick films (plasmas, electroplating, anodization, etc.). GD-OES also became a complementary technique to XPS and SEM.



Discover all unique features of HORIBA Scientific GD-Profiler 2 to undertake new challenges in materials characterization



Applications - Information Notes

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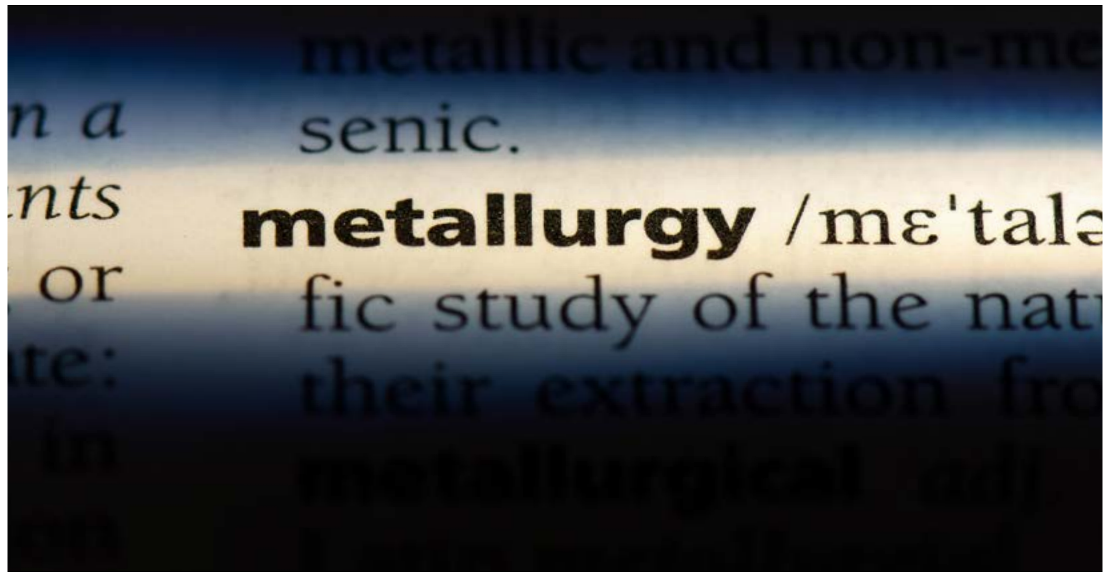
GD-OES is now widely used for the characterization in the fields of metallurgy, renewable energies and is also used as a complementary tool along with XPS and SEM…

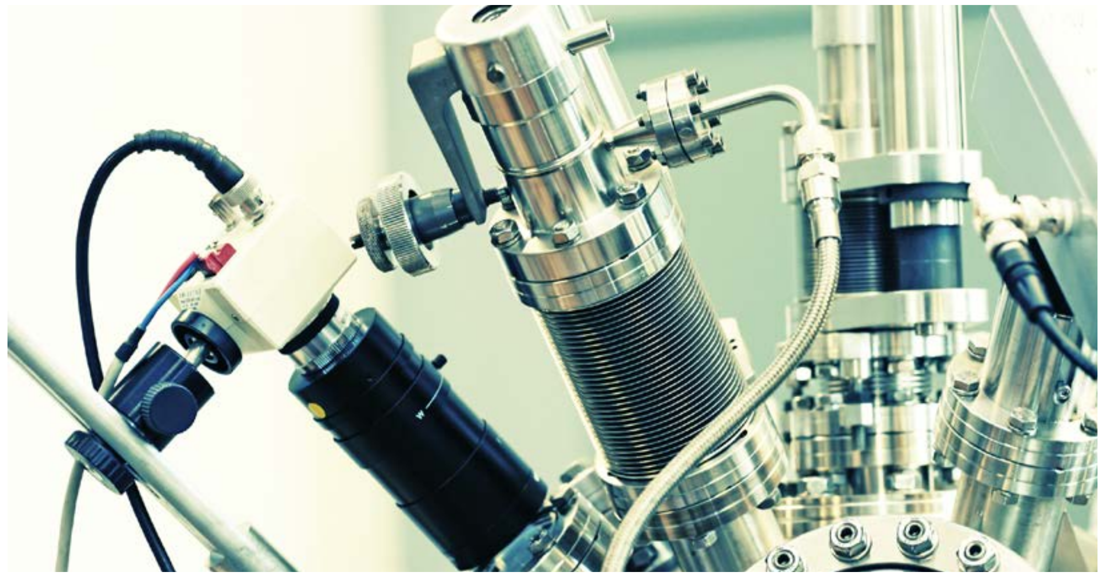


Recent applications of metals and alloys involve organic and inorganic coatings, thickness measurement and odd shape materials. Their characterization is more complex than in the past and brings new challenges to the analyst. Discover how HORIBA Scientific GD-OES can help you to undertake the new challenges for the characterization of your materials.

Read more about Bulk Analysis of Metals to Depth Profiling of Advanced Materials:
Focus on: Steel, Aluminum, Packaging, Drill Bits,Corrosion and Electroplating

GD-OES can help for the characterization of any of the materials involved in renewable energies. Elemental depth profiling, thickness measurement, diffusion and interfaces studies is possible for all elements including H, Li, Na, C, O and more. Discover how HORIBA Scientific GD-OES can address all challenges in the characterization of these materials.

Read more about Bulk Analysis of Metals to Depth Profiling of Advanced Materials:
Focus on Renewable Energy



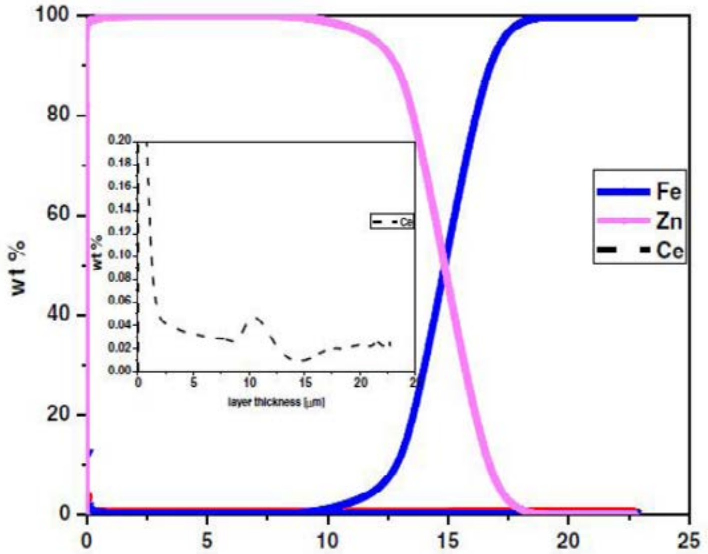
Discover GD-OES complementarity to SEM and XPS. In addition to providing additional information, GD-OES can also help preparing samples for SEM or help XPS reaching deep interfaces in minutes. Check applications involving SEM and XPS along with GD-OES.

Read more about Bulk Analysis of Metals to Depth Profiling of Advanced Materials:
Focus on GD-OES, SEM and XPS

Applications - Application Notes

Analysis of films containing metal and metal oxide nanoparticles

Metal and Metal oxides nanoparticles are introduced in films to enhance or add properties – corrosion resistance, photonics effects, antibacterial activity etc. Metal oxide nanoparticles are widely used for the fabrication of composites due to their availability. Nanoparticles like ZnO, $Al2O_3$, ZrO_2, TiO_2, CeO_2, etc., incorporated in various metals and alloys have been used to generate composite layers with superior properties. Similarly, polymer films with embedded metal nanoparticles can be fabricated for instances by melting a powder mixture (Co alloy in polyethylene) or by drying in a powder resin mixture (Ag particles in epoxy resin). HORIBA Scientific is partner of a French research project that, in the same chamber, couples a spray of nanoparticles onto PVD growing layers.Pulsed RF GDOES is used in many researches on these new materials to study the depth profile distribution of the particles and the thickness of the composite layer.

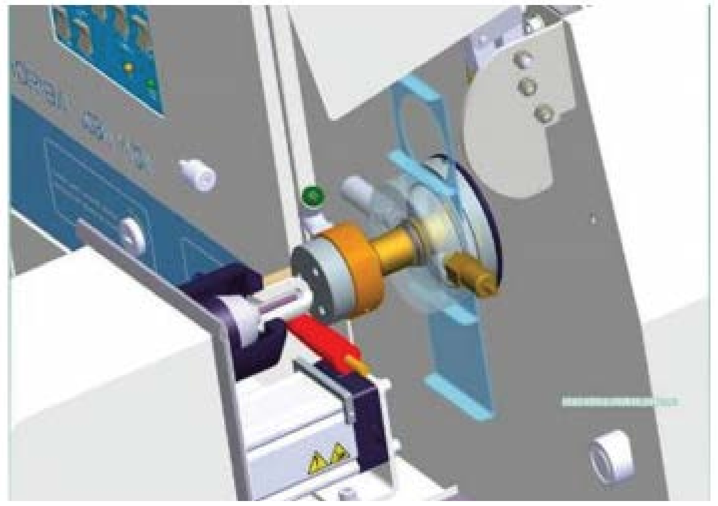


Pulsed RF GDOES depth profile (insert: the distribution in the layer)

Learn more

Features and Benefits of Pulsed RF GD OES for the Characterization of Electrodes of Li-ion Batteries.

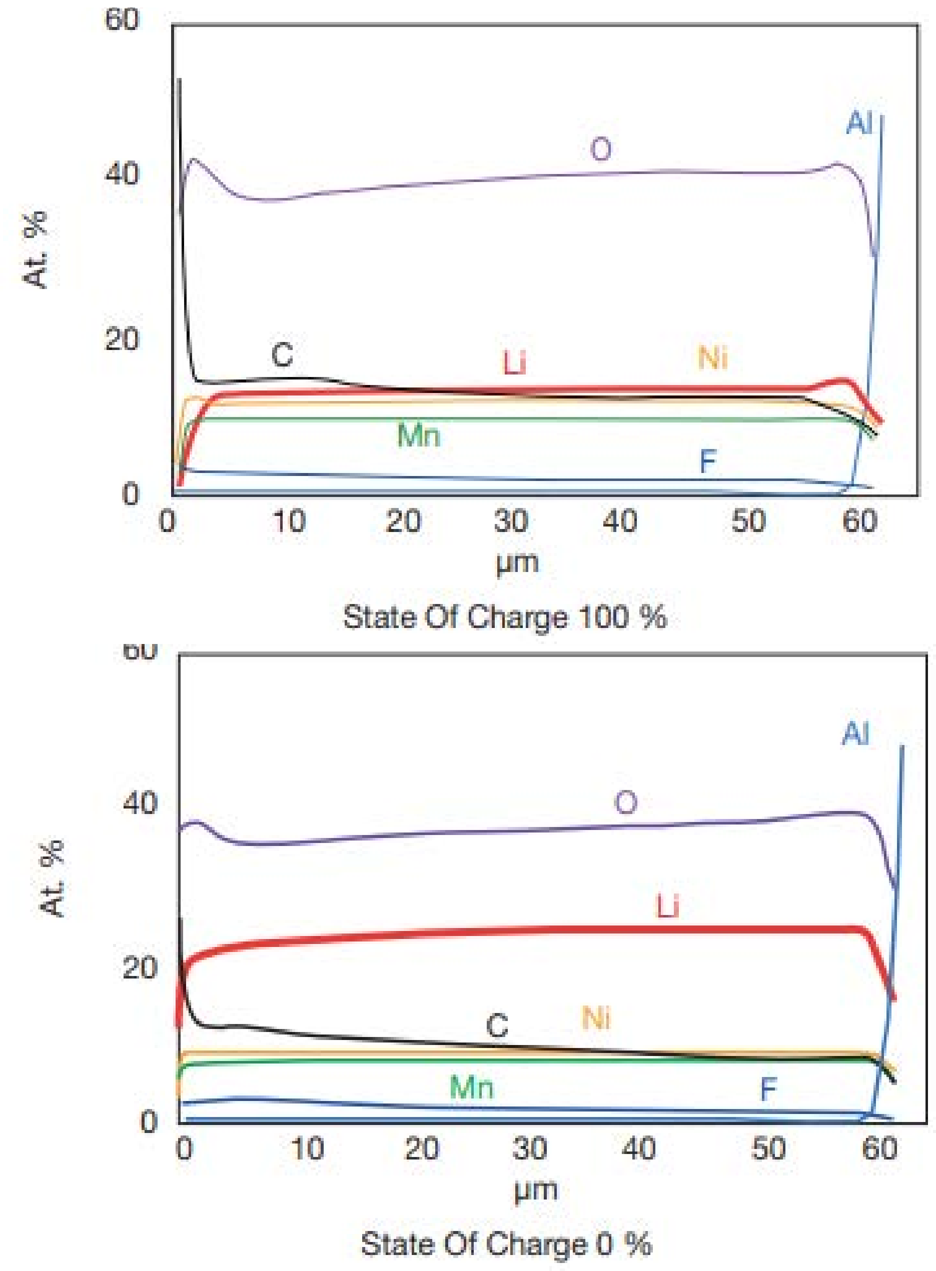
A Li-ion battery is a rechargeable battery in which lithium ions move between the anode and the cathode creating an electricity flow. The chemical reactions do not simply take place at the surface of the electrodes but rather affect depths of several tens of micrometers. Pulsed RF Glow Discharge Optical Emission Spectrometry provides Ultra Fast Elemental Depth Profiling of thin and thick films and has been successfully applied for the characterization of both positive and negative electrodes of Li ion batteries.



Schematic view of the LI bell mounted on the GD Profiler 2 Instrument

HORIBA Scientific has more than 15 references world wide in this field. Most results obtained are protected by Non Disclosure Agreement and cannot be communicated. We therefore invite people interested in the technique to contact us so that we could also describe more in depth, the unique proprietary features of our instrument and set up some performance tests.

Typical Results



Pulsed RF GD OES Depth Profile Analysis of the positive electrode of a battery fully charged and discharged

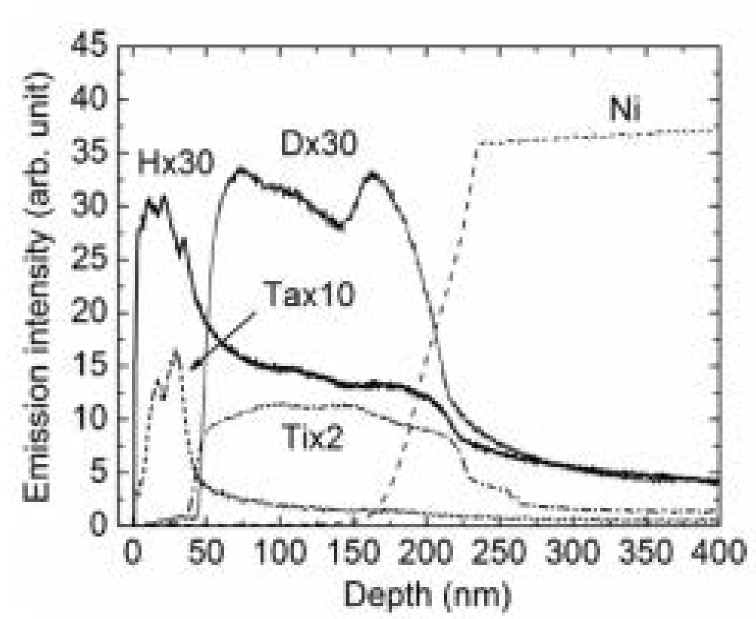
Learn more

Applications - Application Notes

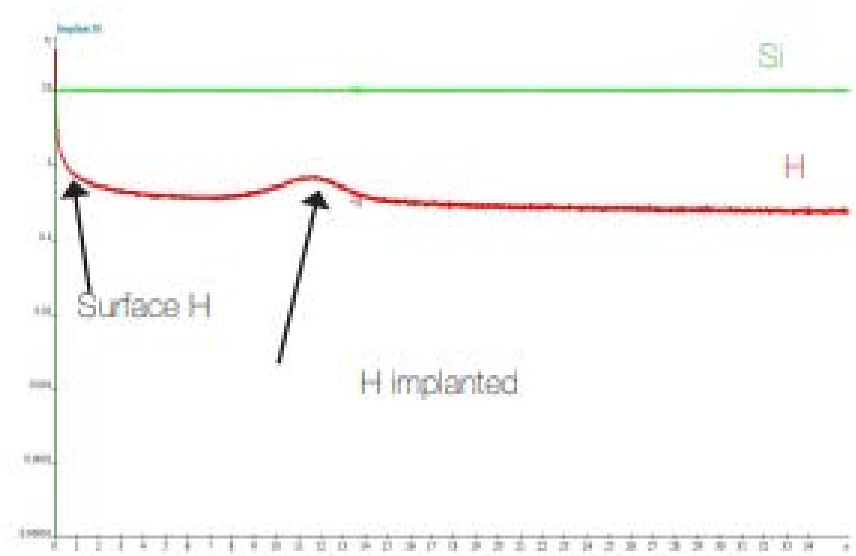
H&D Measurement of Hydrogen (and Deuterium)

RF GD OES is well known for ultra fast elemental depth profile of thin and thick films. All elements can be measured including Hydrogen (H) which is important in many application fields - for corrosion studies, for PV, in metallurgy, for the development of hydrogen storage materials and for all polymeric coatings studies to name a few. Isotopic separation usually requires MS spectrometry. However for Hydrogen, it is possible to separate it from its isotope Deuterium (D) in an optical spectrometer with adequate resolution: the 2 emission lines being at 121,534 nm (for D) and 121,567 nm (for H) – so separated by 30 picometers!

Deuterium profile is of great interest in fusion studies for plasma facing components, it is also of interest in corrosion studies (D being substituted to H in the corrosion medium.



Depth profiles of H, D, Ta, Ti and Ni in the Tz(H)Ti(D)/Ni layered structure



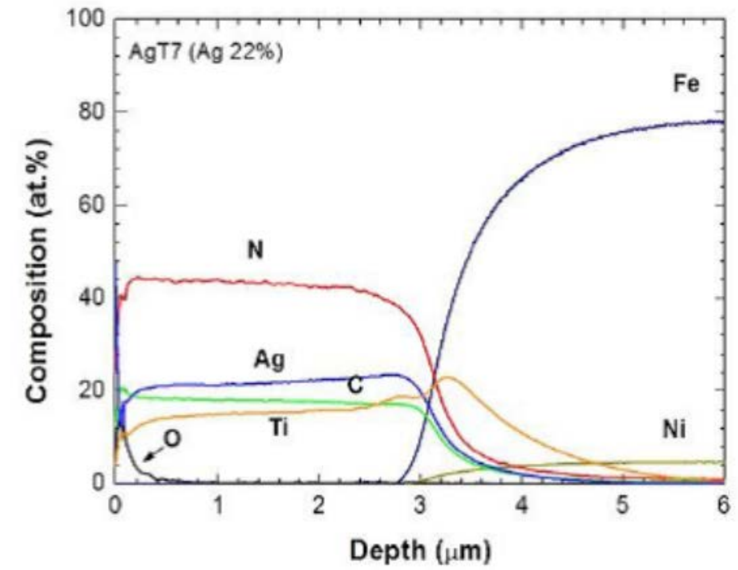
Learn more

GDOES, the Analytical Companion Tool for Magnetron Sputtering Deposition

Pulsed RF GDOES is a companion analytical tool for magnetron sputtering deposition. Magnetron sputtering is a type of Plasma Vapour Deposition. The vacuum chamber of the PVD coating machine is filled with an inert gas, such as argon. By applying a high voltage (RF, HIPIMS etc), a glow discharge is created, resulting in acceleration of ions to the target surface and a plasma coating. The argon-ions will eject sputtering materials from the target surface (sputtering), resulting in a sputtered coating laye on the products in front of the target. Often an additional gas such as nitrogen or acetylene is used, which will react with the ejected material (reactive sputtering). A wide range of sputtered coatings is achievable with this PVD coating technique.

Magnetron sputtering technology is very advantageous for decorative coatings (e.g. Ti, C Zr and Carbon Nitrides), because of its smooth nature. The same advantage makes magnetron sputtering widely used for tribological coating in automotive markets (e.g. CrN, Cr2N and various combinations with Diamond Like Carbon (DLC) coating.

Magnetron sputtering is somewhat different from general sputtering technology. The difference is that magnetron sputtering technology uses magnetic fields to keep the plasma in front of the target, intensifying the bombardment of ions. A highly dense plasma is the result of this PVDcoating technology.



GD depth profile of a coating deposited by magnetron sputtering

Learn more

HORIBA Scientific

Applications - Application Notes

Information Notes:

- GD-Profiler 2
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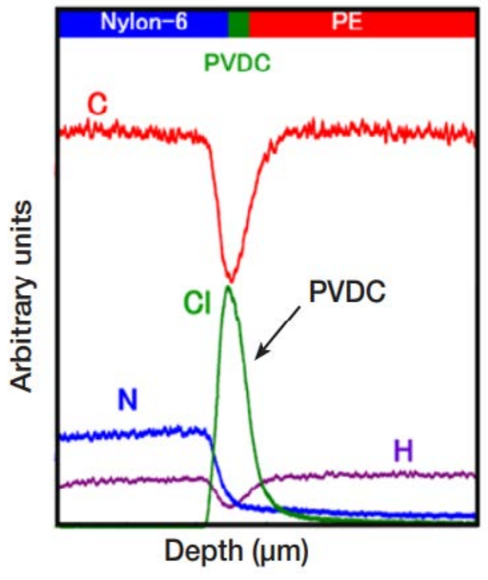
Depth Profile Analysis of Organic and Organic/Inorganic Multilayered Materials by Pulsed RF GDOES

GDOES relies on the fast sputtering of a representative area of the material of interest by a dense plasma and the real time optical analysis of the emission light produced by the sputtered species excited by the same plasma.

With the development of pulsed RF source and the invention of the “UFS”, the technique now contributes to the development and characterization of polymeric materials. Additives can be seen and localized, the structure of the material can be revealed quickly and therefore the elaboration process can be controlled and optimized.



TRC Presentation of the 2014 Japanese GD Day

Learn more

This figure illustrates a GDOES depth profile on a multilayered polymer. The layers are distinguished by representative elements. No molecular information (as given for instances by Raman Spectroscopy) is obtained with GDOES but the observation of representative elements and/or changes in the intensity levels for the same elements (that relate to changes in sputter rate from material to material) anyhow clearly reveals in many cases the structure of the multilayered compound.

What’s Protecting Your Mobile Screen? A Depth Profile of Polymer Protection Covers Using Raman and UFS-GDOES

The GD Profiler 2 can be a key instrument during the optimization and follow up of manufacturing processes. This is a fast technique which allows the easy comparison of different materials, the detection of defects and the presence of contaminants. Moreover, combined with the UFS system, it proves to be a flexible technique for organic and hybrid materials, providing numerous new application domains.

For the detection of thin hard coatings it is a prerequisite to have a full confocal Raman microscope with a spatial resolution at the diffraction limit otherwise it is impossible to spatially resolve and detect such thin layers.

Finally, microRaman and UFS-RF-GD-OES are useful complementary techniques for depth profiling of complex multilayered polymer materials. The results given by these techniques prove to be in excellent agreement.

Learn more

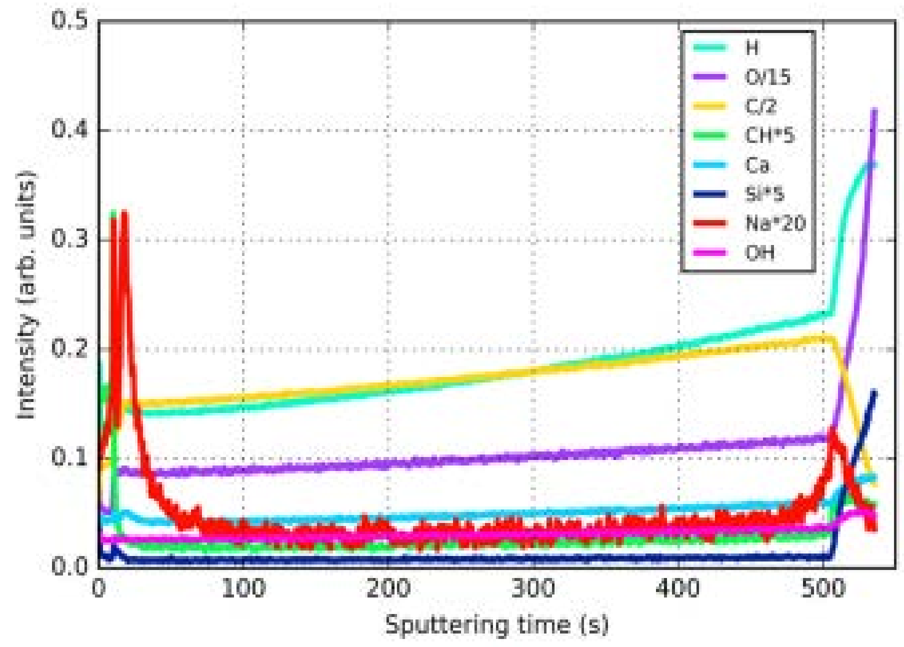
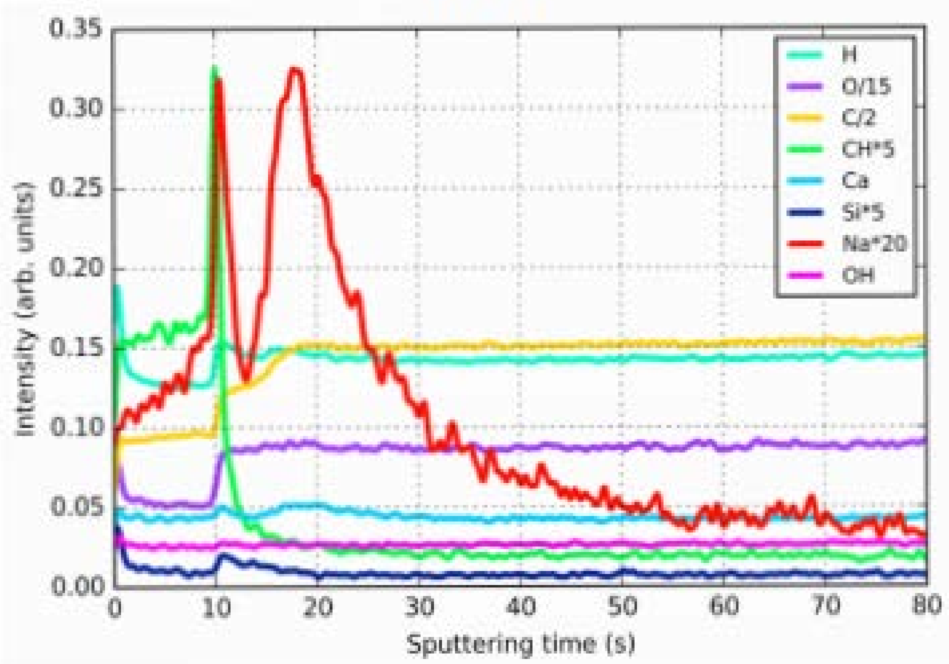


Figure 6a

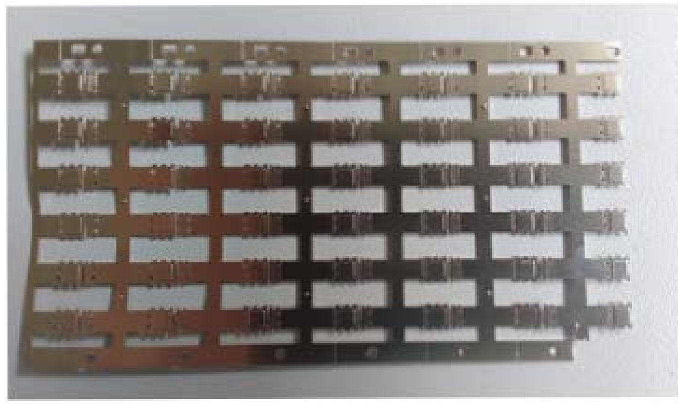


UFS-RF-GDOES depth profile of the PMSPC; and zoom of the first 80s of analysis

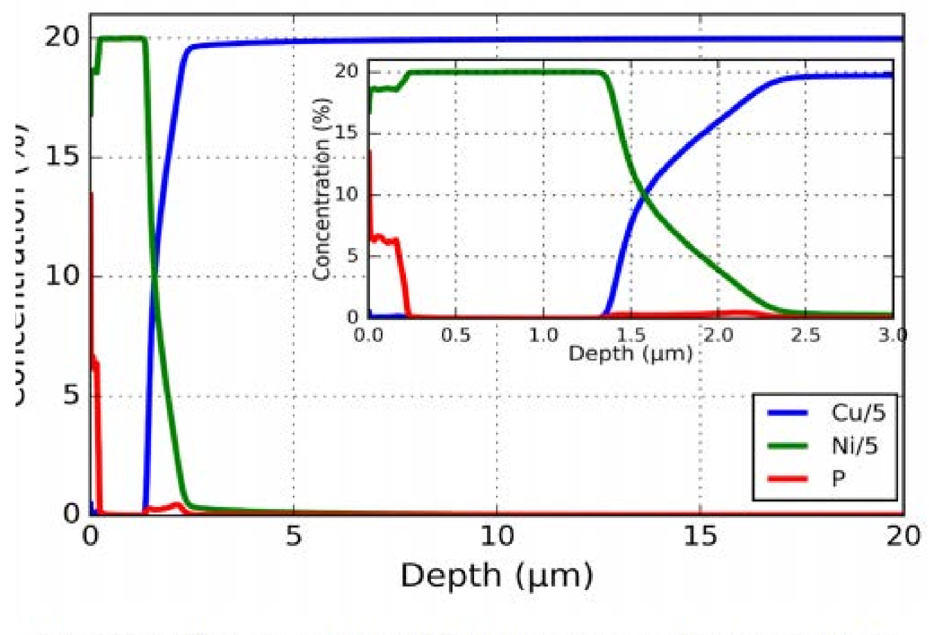
Applications - Application Notes

How to analyse your electroplated coating?

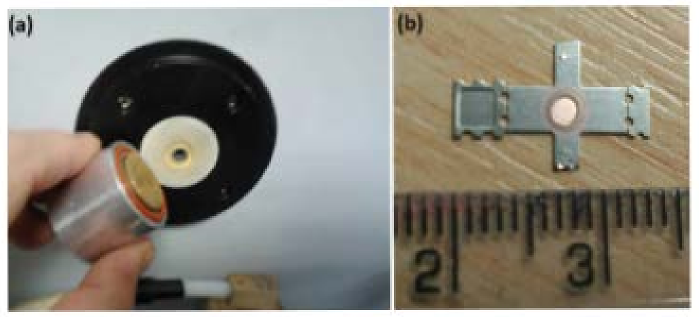
Even if alchemy does not exist, electroplating can be considered the next best thing. The idea is to coat relatively common metals (e.g. copper, steel, etc.) with a more precious one (e.g. gold, silver, etc) using electricity! Actually electroplating has many different uses, besides making cheap metals look expensive. Indeed, it can be used to make anti-corrosion coatings, to produce a variety of alloys like brass or bronze on steel, etc...



Example of an electroplated sample



Quantified elemental depth profile for the sample shown. By establishing a set of calibration curves, it has been possible to convert the relative GDOES analysis presented in a quantative result.



The small sample holder and a small analyzed sample with a 2 mm GD crater in the center.

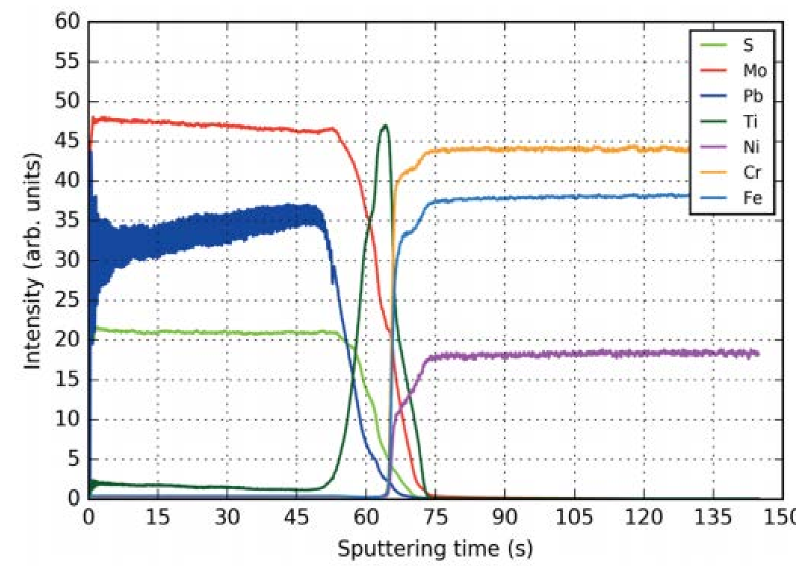
Learn more

Thin layers of MoS2/Pb nanocomposite coatings for solid lubricants

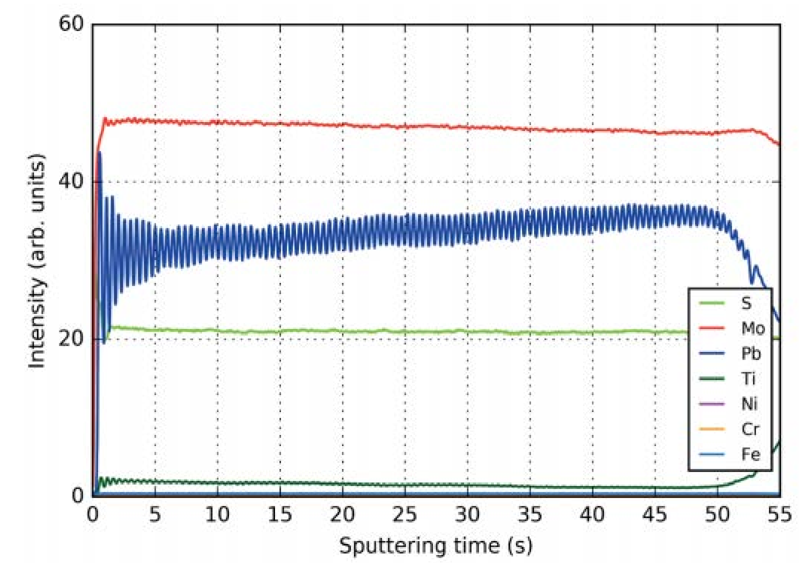
Pulsed RF Glow Discharge Optical Emission Spectrometry offers ultra-fast elemental depth profiling capability for the investigation of thin and thick films. Thanks to the use of a pulsed RF source, coupled with a high resolution optical spectrometer, the GD Profiler 2 provides an excellent depth resolution, allowing the fast evaluation of the coating quality.

In this application note, we focus on a MoS2/Pb composite multilayered sample, used as a solid lubricant. The analysis of such a sample shows the excellent performance of this instrument for the study of nm-thick complex coatings.

Learn more



Full depth profile of MoS_2/Pb coating obtained with the GD Profiler 2



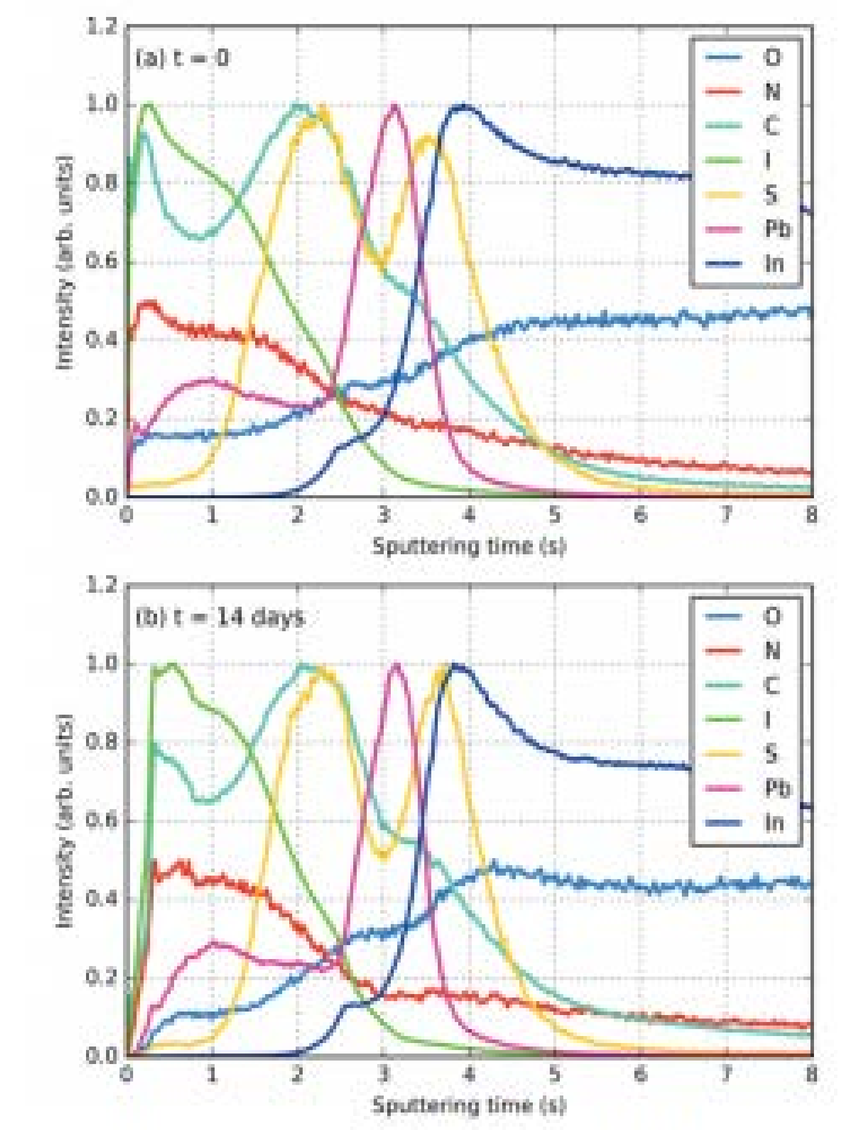
Zoom on the stacks coatings to see the depth resolution

Applications - Application Notes

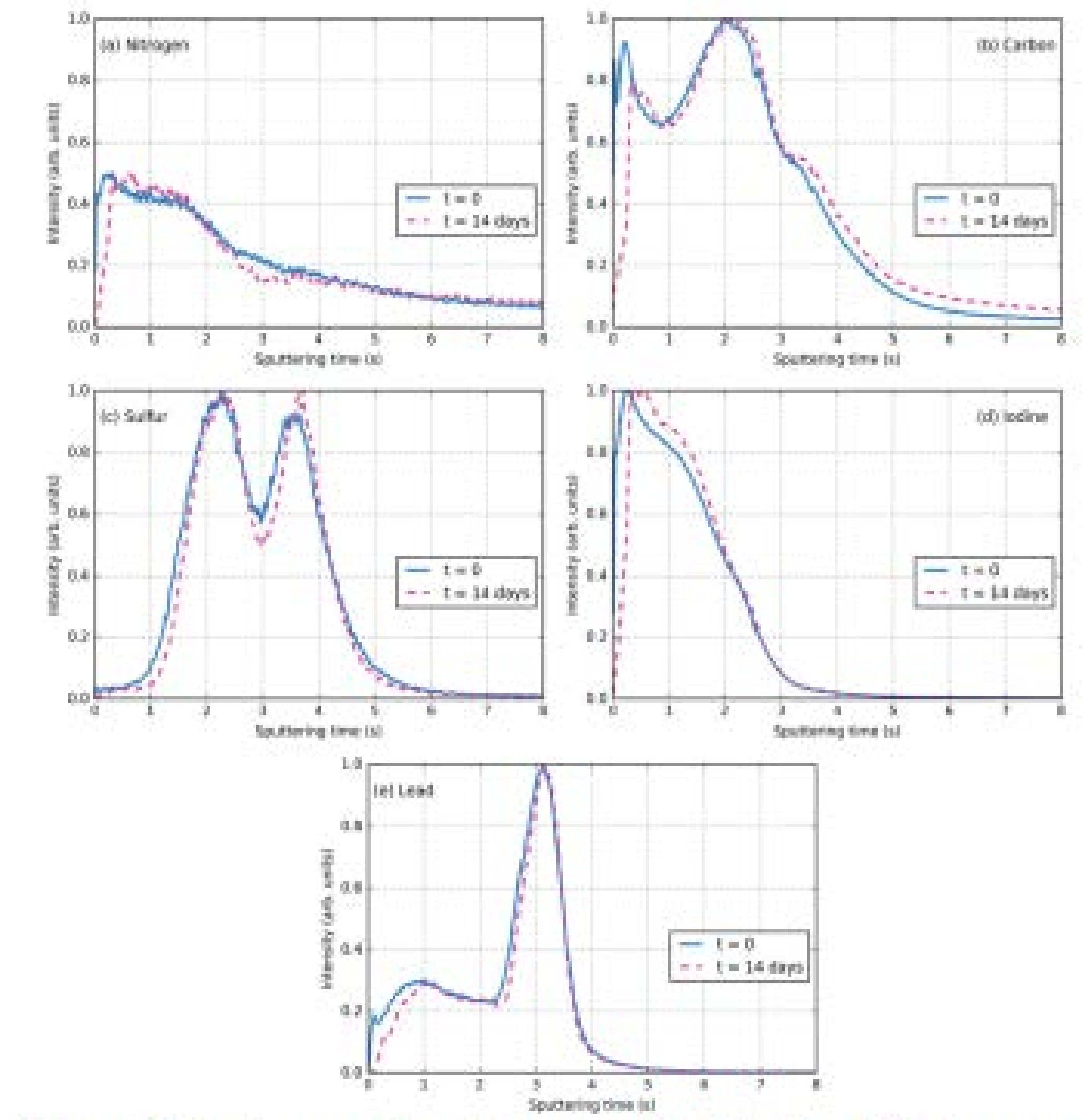
Studying perovskite solar cells with HORIBA Scientific equipment

With their ~20% efficiency, hybrid perovskite solar cells are the new promising candidate for next generation photovoltaics. Thanks to the wide HORIBA Scientific portfolio, different techniques can be used to gain in depth knowledge on the optoelectronic properties and mechanisms of this class of materials. In this application note we decided to use spectroscopic ellipsometry, steady-state and time-resolved fluorescence and Glow Discharge Optical Emission Spectroscopy to investigate the properties of $CH_3NH_3PbI_3$ thin films deposited on a spin-coated PEDOT:PSS. The impact of the exposure to air was addressed.

Learn more

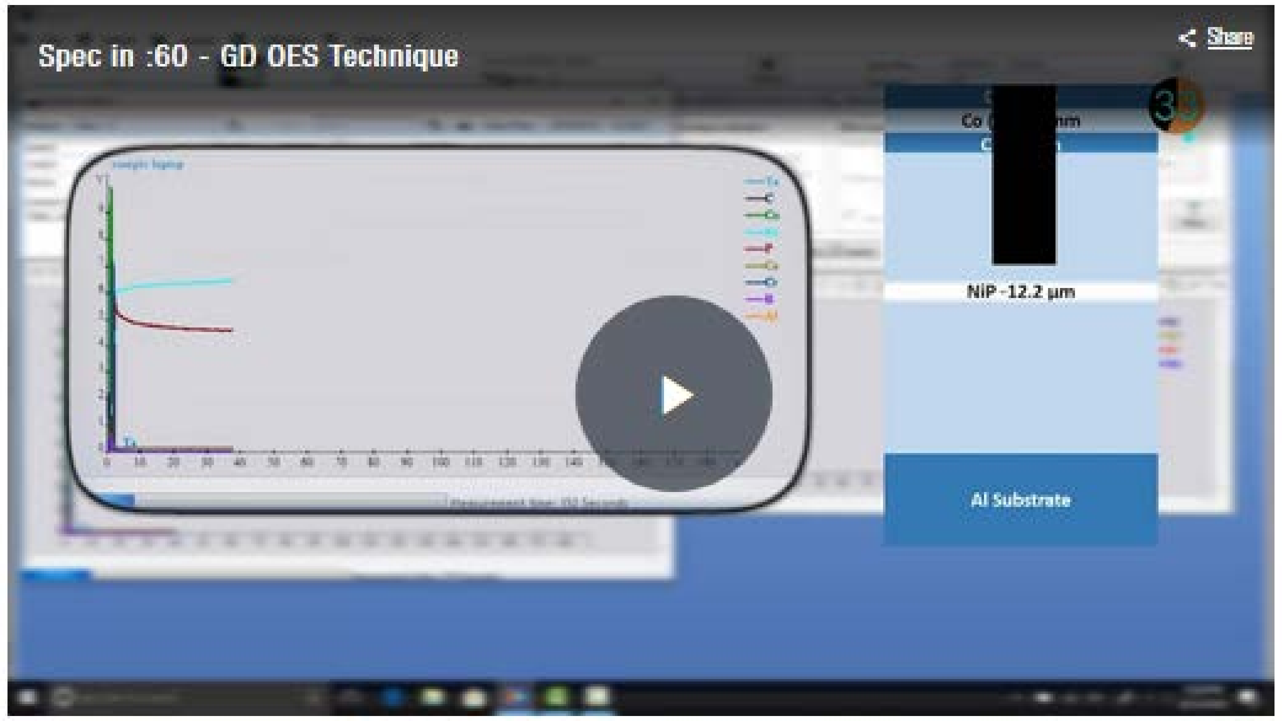


Qualittive elemental depth profile of a (a) *t*+0 and (b) *t*+14 days



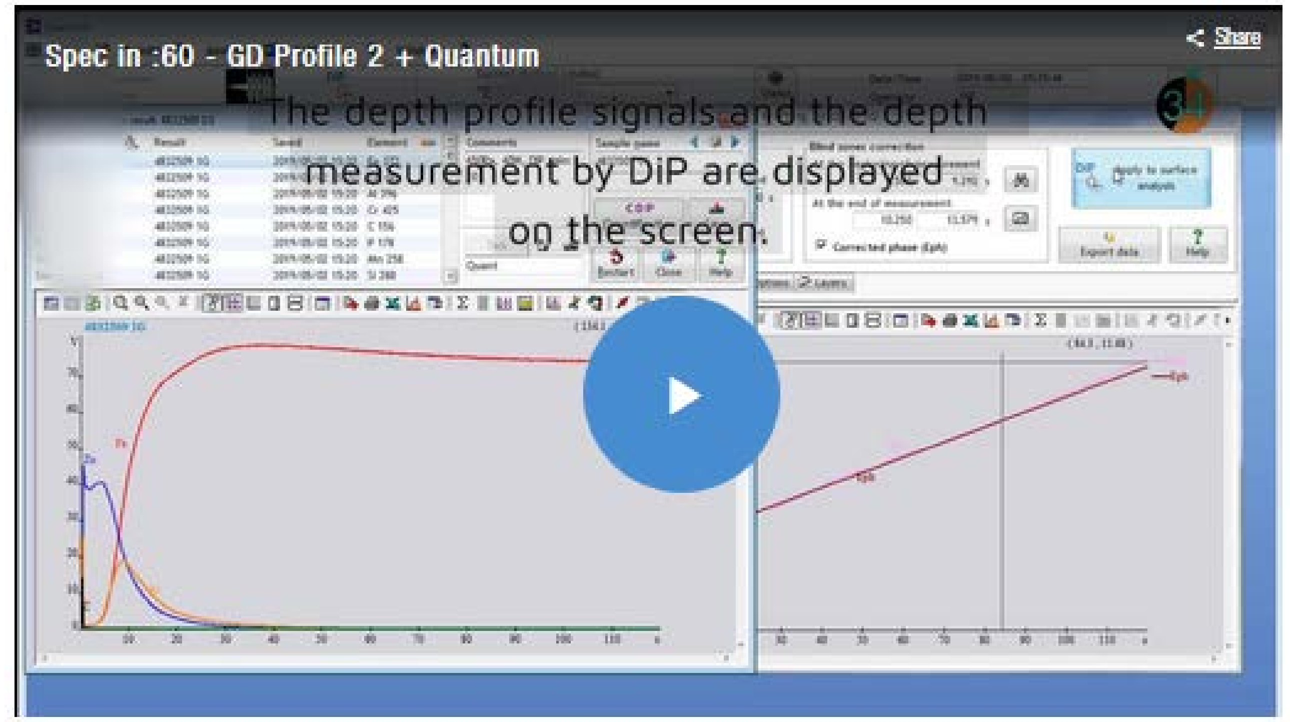
Direct comparison of the eletmental distribution atfirst exposure to air (continuous blue curve) and after 14 days of exposure to air (hatched pink curve) for (a) nitrogen, (b) carbon, (c) sulfur, (d) iodine and (e) lead.

Videos



[Spectroscopy in :60 - GD OES Technique](#)

[GD Profiler 2 performs elemental depth profiling really quickly](#)



[Spectroscopy in :60 - GD Profiler 2™ + Quantum™](#)

[Acquisitions, real-time measurement, and DiP conversion, wow!](#)

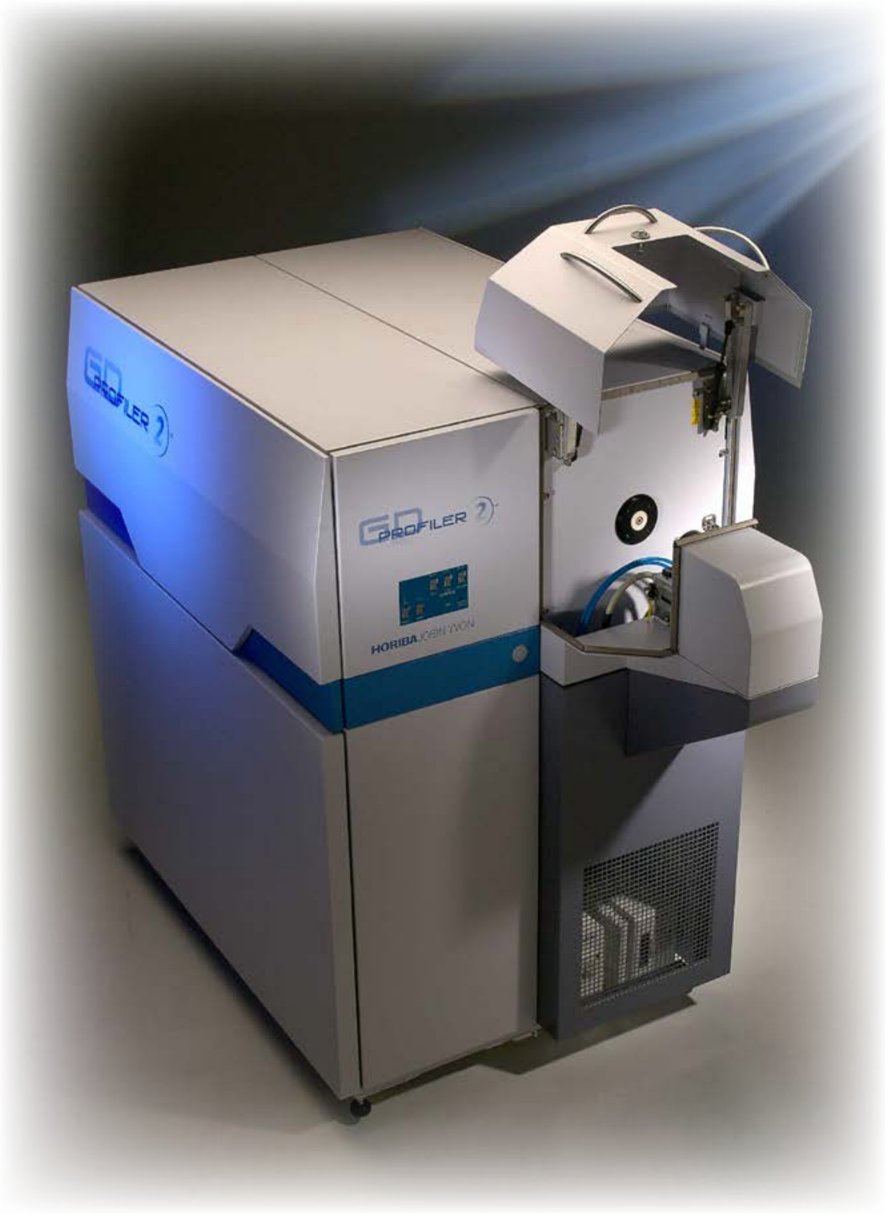
[Spectroscopy in :60 - GD Profiler 2 + accessories](#)

[Multiple accessories can be used on the GD-Profiler 2 to handle a large variety of samples](#)

Contact

Interested to discuss about GD-OES and how it can help on your application?

- **US/Canada/Mexico:** matthieu.chausseau@horiba.com
- **Japan:** akira.fujimoto@horiba.com
- **China:** yanhong.wu@horiba.com
- **Rest of the world:** patrick.chapon@horiba.com

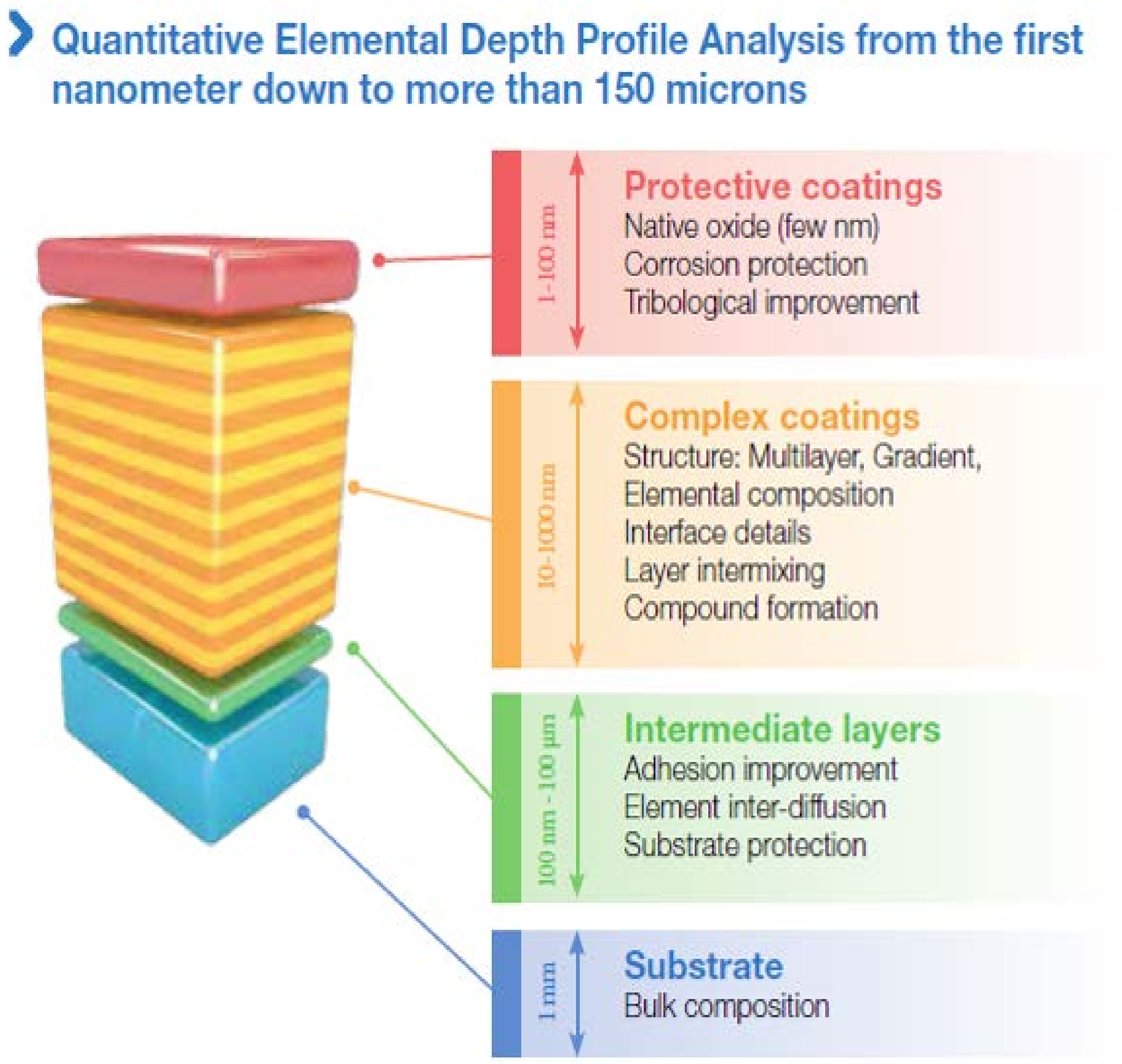


To learn more about GD-OES visit:

horiba.com/scientific

and

https://www.linkedin.com/groups/4175530/



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