HORIBA JOBIN YVON

Ultra Fast Elemental Depth Profiling

RF Glow Discharge Optical Emission Spectrometers

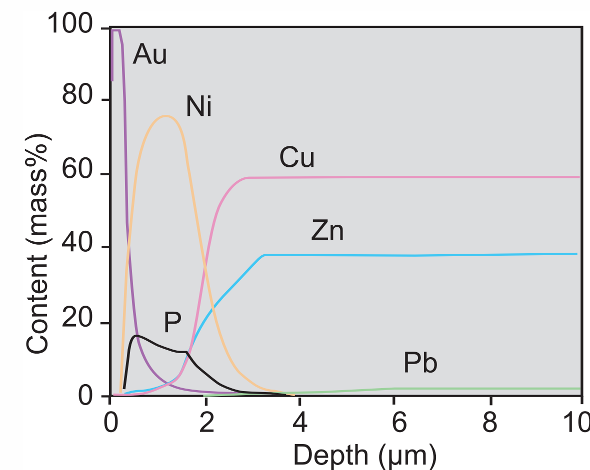
Look at the Surface and Beyond...

GD PROFILER 2™ and GD PROFILER HR™

GD PROFILER SERIES

GD-Profiler Series RF Glow Discharge Optical Emission Spectrometers

For many applications, it is essential to know the chemical composition of a material, both at the surface, at the interfaces and in the bulk. Whether the application is coated steels, e.g. for car bodies, or semiconductor wafers, e.g. for integrated circuit manufacture, the surface composition determines many important parameters such as appearance, corrosion resistance, adhesion, conductivity, etc, while the bulk composition is important for the stability and long life of the product. Only one technique, RF Glow Discharge Optical Emission Spectrometry (RF-GD-OES) can provide both the surface and bulk composition, quickly, inexpensively, and with high sensitivity to all elements (including the gaseous elements), for almost all solid materials, including metals, metal alloy coatings, semiconductors, polymer coatings, glass, ceramics, etc.



RF GD Depth Profile revealing a gold layer on NiP on brass and the GD spot on the sample after analysis. Total measurement time: 2 minutes. (Courtesy of EMPA Switzerland)

Elemental analysis from the first nanometer down to more than 150 microns

The GD-Profiler Series of RF-GD-OES offers two models, each with a variety of options to suit an extensive range of applications. The GD-Profiler 2 is a very compact instrument with high flexibility, while the GD-Profiler HR offers unmatched performance with its ultra high resolution optics.

The GD-Profilers use a Class E RF generator optimized for stability and crater shape allowing for real surface analysis. This is due to the RF source's constant 13.56 MHz frequency and the rapid response of the matching box. Since the source can be pulsed at a millisecond rate, very fragile samples may be analyzed.

View of the GD-Profiler 2

HORIBA Jobin Yvon (HJY) original, ion-etched holographic gratings assure the highest light throughput for maximum light efficiency and sensitivity.

High-performance, patented High Dynamic Detectors (HDD™) provide speed and sensitivity in detection without compromise – a dynamic range of 10^{10} with microsecond speed.

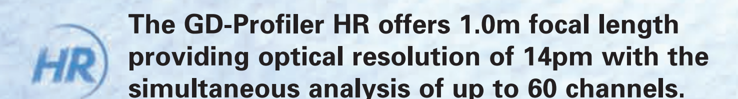
Easily accessible sample compartments allow plenty of room for sample loading, even with large samples. Accessories for small samples or non-flat samples are available. The patented CenterLite Laser Alignment System provides for precise sample positioning.

The GD-Profiler 2's simultaneous 0.5m polychromator allows the determination of up to 47 elements between 110 and 800nm. This allows the determination of H, O, C, N, and Cl and it also gives an optical resolution between 18 and 25 pm for all elements.

The monochromator option, available only from HJY, provides the perfect tool to increase instrument flexibility by allowing any element to be added to the analytical program(`n+1´).

The monochromator also allows full spectrum acquisition in as little as two minutes. Thus a complete sample fingerprint is available which can be examined with HJY's unique IMAGE software.

View of the GD-Profiler HR with ultra high resolution combined optics



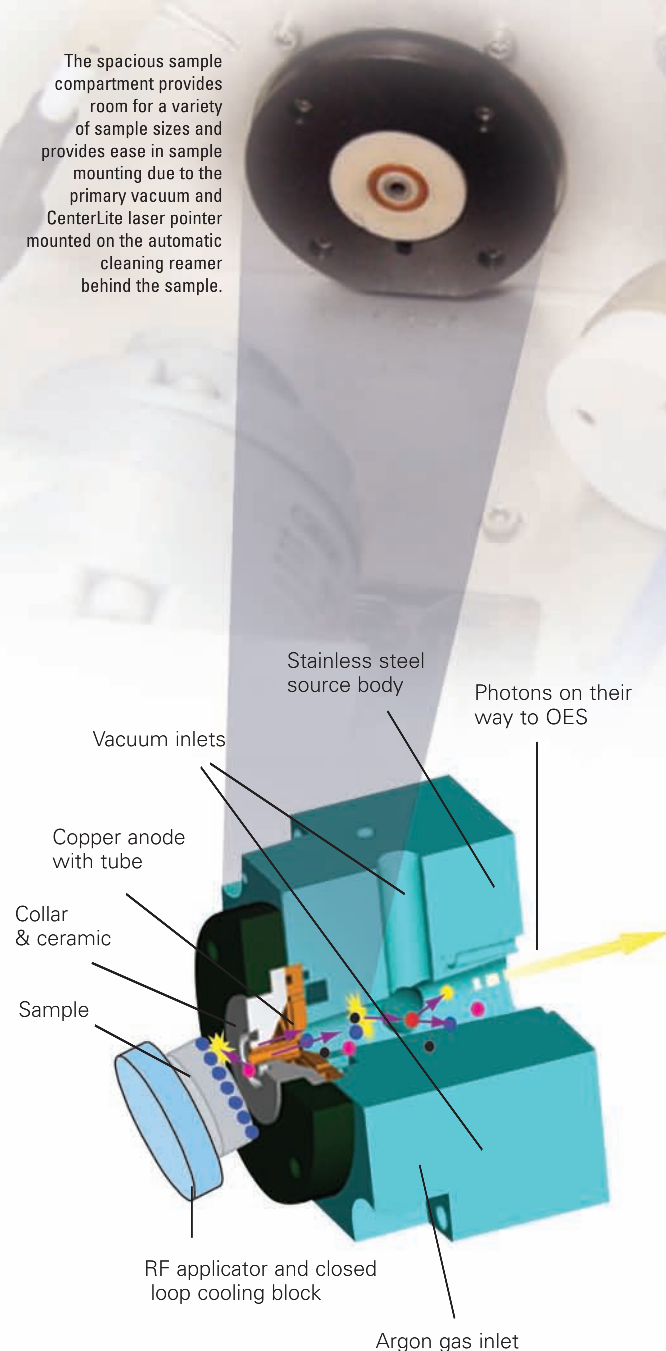
The GD-Profiler HR offers 1.0m focal length providing optical resolution of 14pm with the simultaneous analysis of up to 60 channels.

The optional 1.0m monochromator of the GD-Profiler HR offers the highest optical resolution available of 9pm in the UV.

Bulk, Surface and Depth Profiling, Conductive and non Conductive

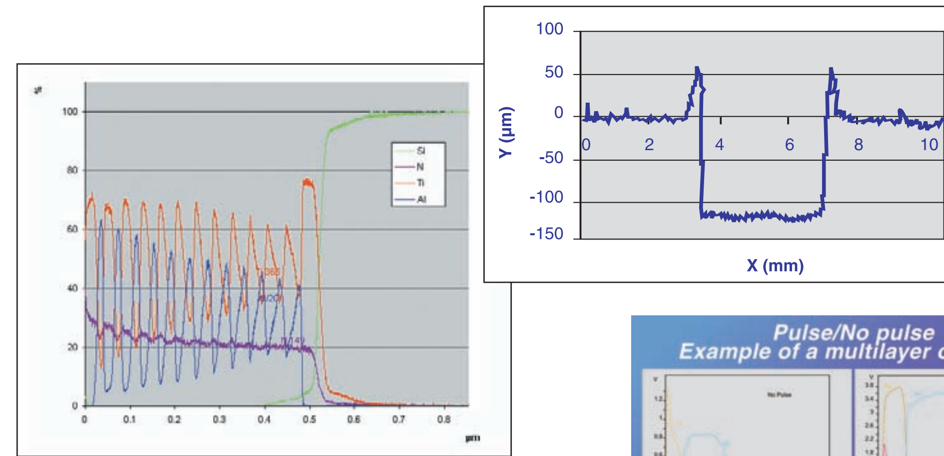
GD-Profiler Series RF-GD-OES system combines a glow discharge powered by a radio frequency (RF) source with an optical emission spectrometer. The essence of a glow discharge instrument is the source which supplies power to create the GD plasma. The HJY design allies the advantages of the Grimm lamp design featuring a dual pumping system with the RF Marcus lamp where RF is applied to the sample and not to a cathode block.

The technique is now internationally recognized (ISO 16962 on the analysis of Zn and Al coatings).



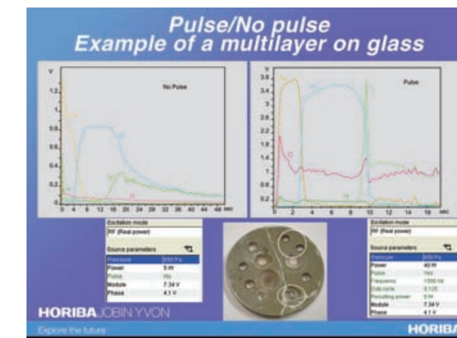
The spacious sample compartment provides room for a variety of sample sizes and provides ease in sample mounting due to the primary vacuum and CenterLite laser pointer mounted on the automatic cleaning reamer behind the sample.

- Separation of the sputtering and light emission area minimizes matrix effects, providing linear calibration curves and allows for the mixing of various alloys in one analytical program.
- Cooling of the sample with a recirculating water system allows temperatures close to zero degrees Celsius, making it possible for the analysis of thin films, such as polymers, which may melt at higher temperatures.
- Analysis of all elements including C, H, O, N, Cl, F.
- Low argon consumption of less than 0.3 L/minute minimizes the cost of analysis.
- The pulsable RF design allows the rapid analysis of both conductive and non-conductive layers or materials.
- Although ideal samples for GD are flat and large enough to cover the source O-ring, the need for only a primary vacuum allows the analysis of small or irregularly shaped samples through the use of special sample holders. Even coated wires have been successfully characterized.
- By attaching a DC Arc Source to the optical system, the trace analysis of powders is possible. The Time Resolved Measurement of the GD Profiler is ideally suitable for DC arc type analysis.

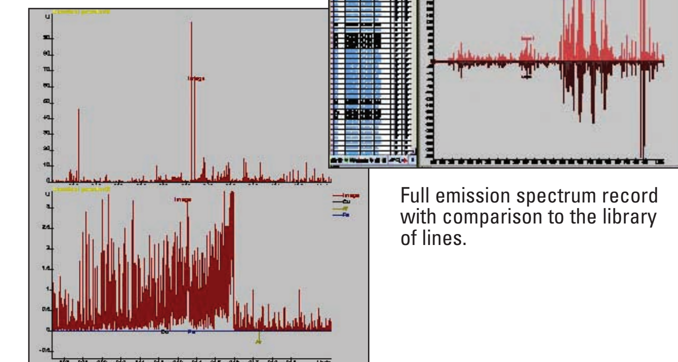


Typical 2-D crater obtained on Zn coated steel. The double pumping system of the HJY RF lamp minimizes the redeposition on the edges of the crater. This allows analysis down to 150 microns with excellent flatness, which is critical for depth resolution.

Hard coatings are of major commercial interest and are commonly prepared by chemical vapor deposition (CVD) or physical vapor deposition (PVD). RF-GD-OES has much to offer to this industry by assisting in the development of new products and in quality assurance and production problem solving. The GD-Profiler series of instruments offers the perfect solution for these types of samples, which are multi-layered, complex coatings. The above depth profile shows the analysis of 25 layers of AlN/TiN (20nm each) deposited on Si.

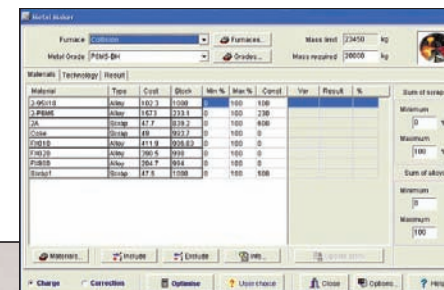


Benefit of the pulse source and synchronized acquisition. Application to thin layers on a fragile glass.

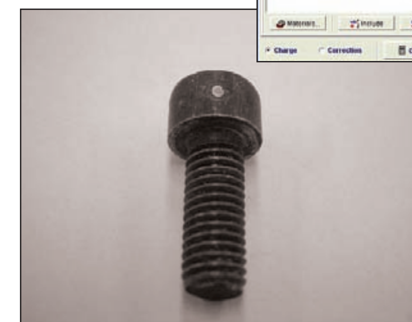


Full emission spectrum record with comparison to the library of lines.

Full spectrum of a sample featuring molecular bands that could affect the reading of elements at certain wavelengths.



Built in Metal Maker provides ultra fast charge calculation and correction, an essential tool in the metal industry.



Crater on a screw head using the universal sample holder attachment

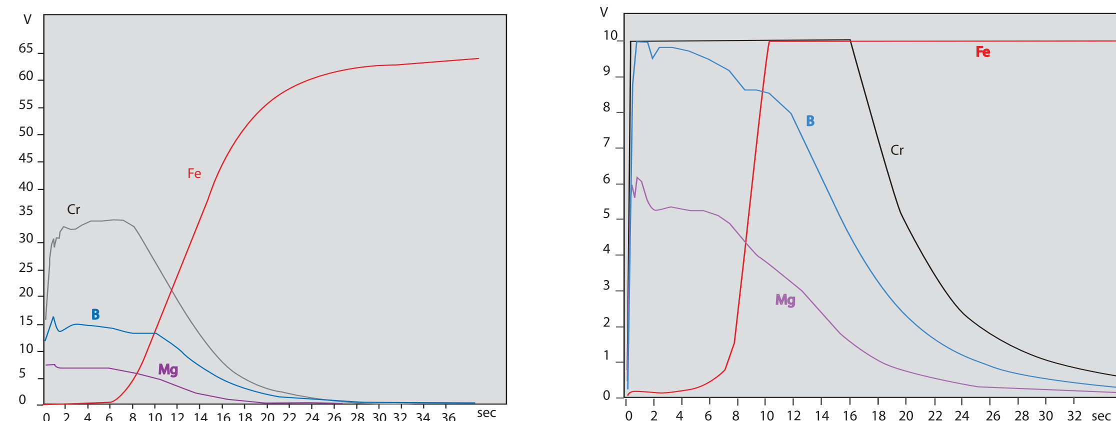
GD-PROFILER *Points*

- RF-GD provides the ability to perform the same types of bulk, elemental analysis as traditional OE systems (arc/spark) with a typical precision of 0.4% for the main elements and an analysis time of about 90 seconds
- HJY provides calibrations for both bulk and compositional depth profile (CDP) for alloys of Fe, Al, Cu, Ti, Ni, Co, Zn, Sn and Pb using Certified Reference Materials. Custom calibrations are also available
- The pulse mode's soft sputtering allows analysis of fragile materials (layers on glass, etc). Acquisition is synchronized with the pulses for optimum detection.
- The GD-Profiler Series offers a world of applications opportunities for thin films, PVD/CVD coatings, coated steels, oxidation/corrosion/passivation, surface treatments, semiconductors, metal alloys, ceramics, powders, and glasses
- HJY sample holders allow for the analysis of all sample shapes including tubes, rods, rough surfaces, etc.
- ISO Technical Committee TC 201 recognizes RF Glow Discharge as a valid technique for depth profile analysis

Speed and Sensitivity without Compromise for any Application

Depth profile analysis is a continuous process of sputtering through a sample at a rate of typically 3 microns per minute. At such a speed, two parameters are critical. First and foremost is time. Valuable information is lost with any time spent waiting or integrating. Second is the ability to respond accurately and immediately to the rapidly changing concentrations as you work through different layers and arrive at various interfaces. This means the ability to measure elements at both trace and major concentrations instantaneously. The same element, which may be at a trace level in one layer, could become a major component in another layer. This type of analysis demands a detection system that has high sensitivity for low intensity levels without using long integration times. It also requires an optical design and electronic acquisition system that can acquire a large quantity of information very rapidly.

HJY developed and patented the High Dynamic Detection (HDD) system. This unique design offers speed and sensitivity in detection without compromise. Because it utilizes a PMT, it offers unsurpassed detection capability for trace elements using short integration times, allowing the instrument to acquire more critical information at the surface and in the first seconds of sputtering or at the interfaces. The HDD design also extends the linear dynamic range of acquisition to 10^{10}, thus allowing the instrument to respond to signals at the interface varying from low counts to millions in the millisecond timeframe without saturation or voltage preadjustment. This response cannot be achieved with fixed high voltage systems or solid state detectors. With the IMAGE feature, the GD-Profiler instruments can also perform full spectrum fingerprinting of your sample or layer using the automatic HDD detection system and ultra fast acquisition electronics.



Complex coating on steel with HDD and without HDD. Without HDD the dynamic range is limited to 10V leading to saturation of the Cr signal in the coating (and partly of the B one) and of the Fe signal in the substrate. HDD mode is extensively used for calibration, bulk and surface analysis.

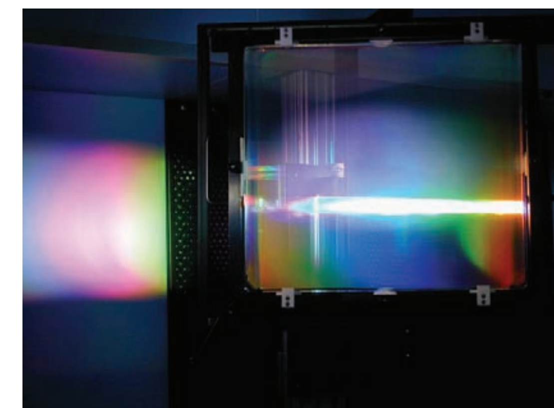
The main polychromator of the GD-Profiler instruments provide coverage from 110 to 620 nm. The optic offers a direct view of the plasma without the use of fiber optics, maximizing the efficiency in the UV. Ion-etched holographic gratings and few reflective surfaces mean maximum light throughput and efficiency for excellent detection limits. A nitrogen purge is used for low UV analysis. The enhanced resolution of the GD-Profiler HR provides the performance required for more difficult sample types and applications.

The flat field option extends the wavelength range to 800 nm to include Li, K, F and Cs. HJY was the first to design and patent the flat field in 1978. This unique design utilizes the primary optical beam directed to a second 1200 groove/mm grating.

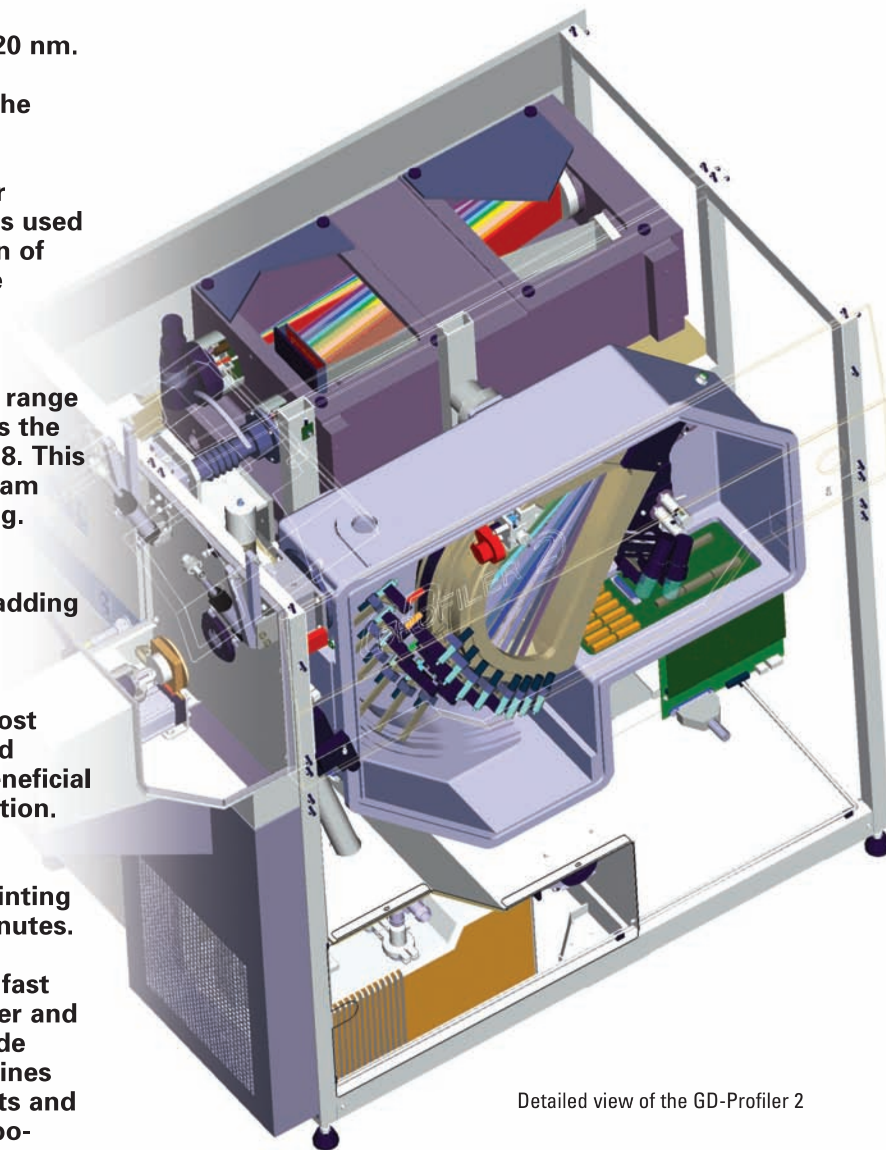
HJY offers the unique ability to extend the flexibility of your GD-Profiler instrument by adding a monochromator. The simultaneous "n+1" analysis during depth profiling is critical in research, where being able to determine an element not on the polychromator can be most useful. The mono option also offers improved resolution compared to the poly, which is beneficial for trace element or molecular band observation.

The Image software is provided with the monochromator. It allows spectrum fingerprinting of a sample or a thick layer in less than 2 minutes.

In addition to the poly, flat field and mono a fast CCD system could be added to the GD-Profiler and used in complement of the flat field to provide simultaneous measurements of some extra lines (essentially in the IR region for alcali elements and Ar lines). However by nature such CCD components have limited dynamics and are not fast enough for ultra thin layers.



The HJY gratings provide highest light throughput



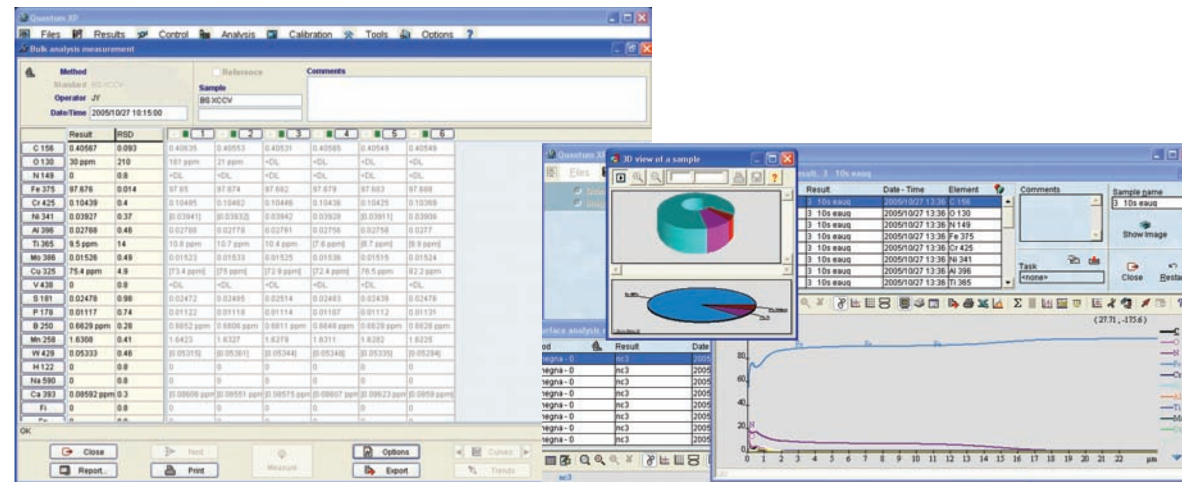
Detailed view of the GD-Profiler 2

GD-PROFILER Points

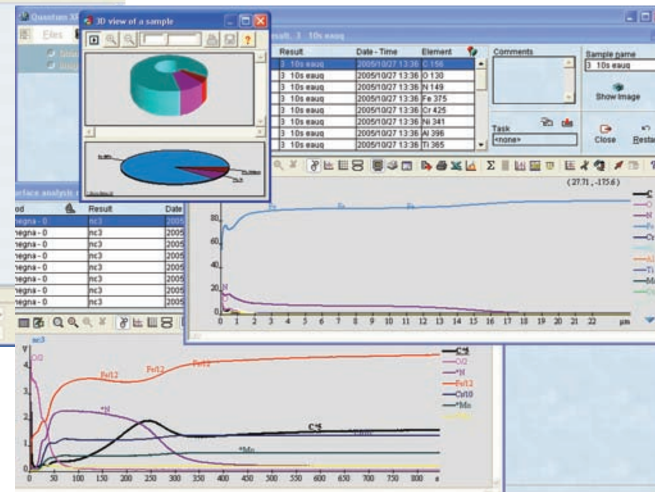
- HJY offers various optical combinations for maximum spectral coverage and flexibility. The monochromator option is a feature unique to HJY and not found on any other commercially available GD
- All GD-Profiler instruments offer patented HDD detection resulting in automatic high voltage adjustment
- HDD is perfect for small, unknown samples where only one spot can be made, as it provides a tailored acquisition for accurate analysis
- IMAGE provides a great tool for semi-quantitative analysis, method development and sample archiving

Quantum XP, Information and Technology in a Software

Quantum XP is a comprehensive multitasking management tool for the GD-Profiler Series allowing bulk and surface analysis in one package. It is loaded with features like **IQ** , Intelligent Quantification, that takes the mystery out of the math behind producing accurate Compositional Depth Profiles, **TimePlus** that allows one to increase the measurement time during a depth profile acquisition and the powerful **Quantum Tabler** that organizes your valuable data and presents them in easily customizable formats.

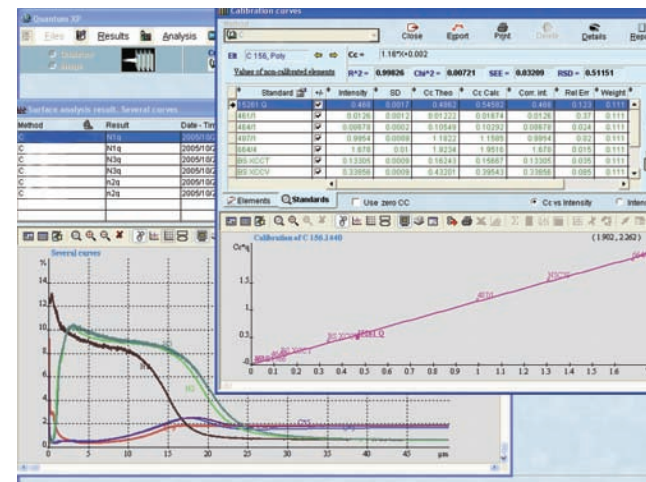


Bulk analysis window



Surface analysis. Multiple windows showing qualitative and quantitative analysis of a treated steel with 3D display.

- Time Plus function to increase analysis time during measurement
- Estimation of trends
- IQ Intelligent Quantification for Compositional Quantitative Depth Profiles
- Integrated libraries of reference materials, optical wavelengths and sputtering rates
- Multilingual software
- Flexible definition of tasks for repetitive work
- 3D display
- Powerful and flexible data handling
- Multitasking allows one to check calibration curves in parallel with the study of a single or multi surface result or to prepare reports while measuring a sample.



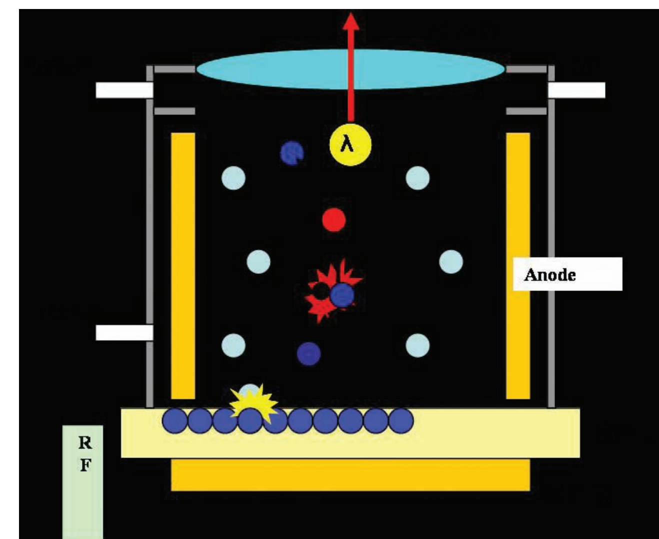
Comparison of Carbon and Nitrogen depth profiles on different treated steels and calibration curve of Carbon in CDP mode.

Two mechanisms yield a powerful technique.

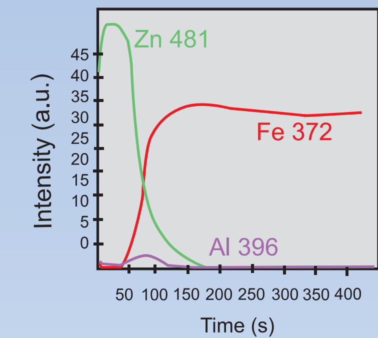
1) In RF GD-OES the material is efficiently sputtered by Ar ions. This first process is material dependent (i.e. the sputtering efficiency depends on the structure and the composition of the material).

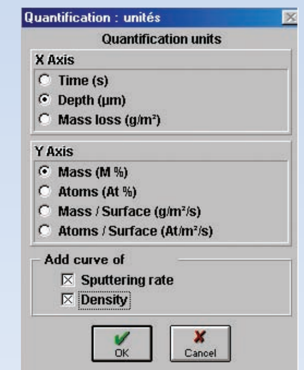
2) The ejected atoms navigate in the plasma which is located above the sample surface but away from the surface. They can be considered as free atoms. The light they produce is therefore only dependent on their number and of the plasma characteristics but not anymore on the sample itself so samples from different materials can be calibrated together, provided we know their sputtering efficiency. This light can then be measured and quantified by the optical spectrometer.

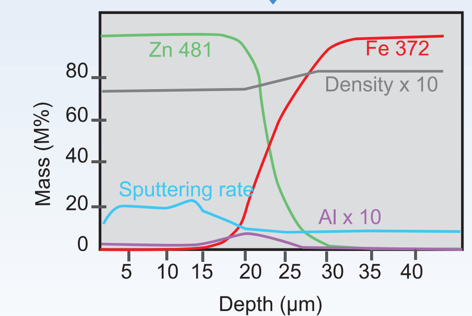
Combining the two processes it is therefore possible to derive the composition and the sputtering efficiency of a multilayer system simply from the recorded light intensities after proper calibration of the instrument.



An inside look at the principles of Glow Discharge and what is happening in the anode anode allows us to understand why RF-GD-OES is a truly quantitative technique. A double mechanism is in process.





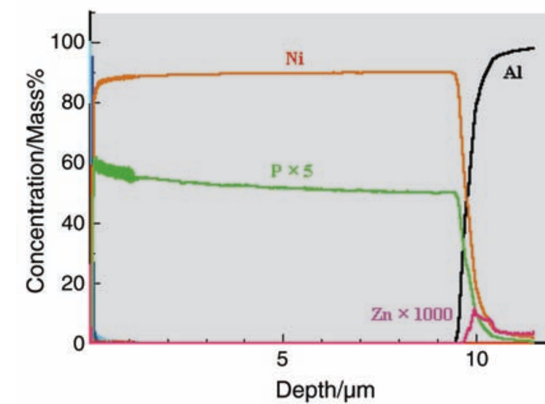


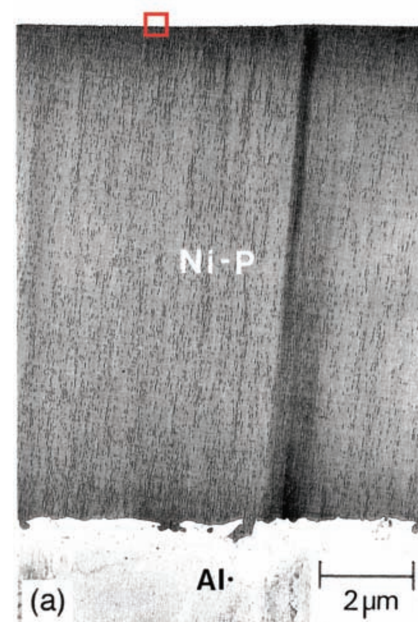
The Intelligent Quantification (IQ) takes full benefit of the knowledge of RF Glow Discharge to provide Compositional Quantitative Depth Profiles from the qualitative information recorded

GD PROFILER SERIES

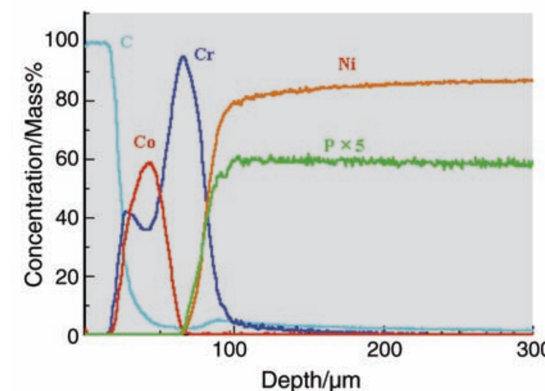
RF Glow Discharge Optical Emission Spectrometry for Surface and Thin Film Analysis

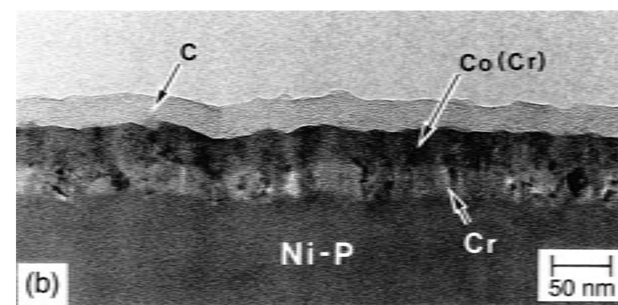
The capability of RF-GD-OES as a tool in surface science is now recognized. Though the technique does not provide lateral resolution, its speed and its ease of use make it the technique of choice for many investigations. Recent examples on hard disks, PZT layers, multilayers on glasses and thin oxides on metals have been published widely. For all these applications, the low energy of the incident particles in RF GD (50eV) assures minimal surface damage. The speed of RF-GD-OES allows depth profiles of both thin and thick films. The typical sputtering rate in RF-GD-OES is 10-150nm per second representing 30-500 atomic layers per second, but since the counting rates of the patented High Dynamic Detectors (HDD) are very fast the instrument is routinely capable of making 1-10 measurements per element per atomic layer.



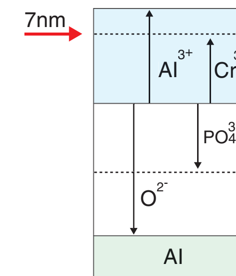


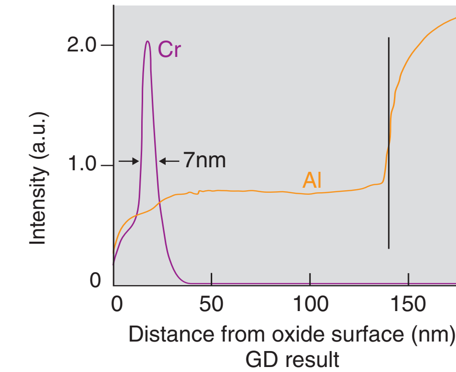
The analysis of commercial hard disks is a challenge for most surface techniques, due to the large variations in thickness throughout the layers of the disks. The fullscale depth profile above shows the ability of GD-OES to sputter through the 12 m amorphous Ni-P layer to the Al-Mg substrate in less than 200 seconds (sampling time 0.1 seconds). The transmission electron micrograph images of an ultramicrotomed section of the hard disk confirm the presence of these layers on the disk substrate. At the right, shown in (a), a general view at low magnification.

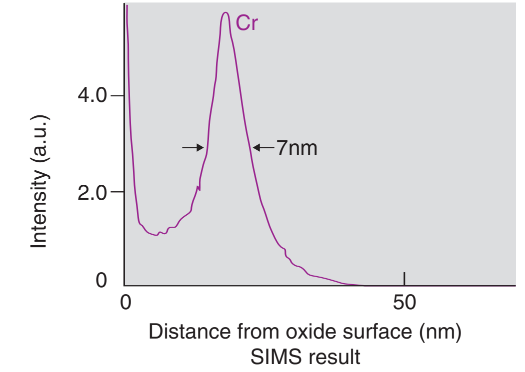




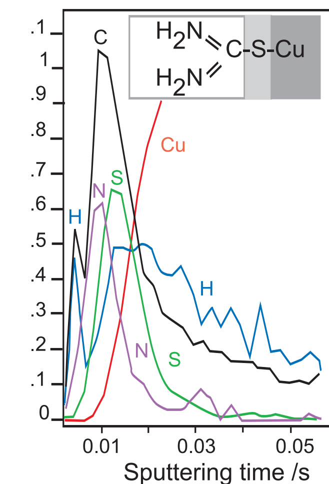
A zoom of the near-surface region above (left) reveals the lubricant layer, diamond-like carbon protective layer, and the cobalt-chromium magnetic alloy and chromium layers, each approximately 20-30nm thick, which were successively deposited over the disks. Sampling time of the near-surface region was 0.01 seconds. The image in (b) is a high magnification view of the area in the red box at the top of (a) above.







Anodic oxidation of aluminum films in appropriate electrolytes leads to the formation of thin (<1um) barrier anodic oxide films. The top left schematic shows such a film after anodizing in a sodium chromium solution. A comparison of the GD-OES analysis and that of secondary ion mass spectrometry (SIMS) was made for this sample. The distribution of chromium in the film compares well between the two techniques, however, the time of analysis was quite different. In GD-OES the time to reach the metal/oxide interface was 9 seconds, as compared to 60 minutes to reach this interface with SIMS.



Recent outstanding results have been published showing the capability of RF-GD-OES to depth resolve some molecules deposited on Cu. This is evidence of the resolution of the technique.

JY RECEIVED THE PRESTIGIOUS
NASA EXCELLENCE AWARD

Over 180 years of JY optical experience provides the platform for NASA projects. This same quality is at the heart of every GD-PROFILER Series instrument.

GD-PROFILER *Points*

- Analysis times are typically 100 times faster than classical surface techniques that operate in UHV environment.
- Sensitivity of RF-GD is in the range of 10^{17} atoms/cm^2
- Non conductors can be analyzed with RF GD without surface charging effects
- All elements can be measured in one shot (including H, O, Cl)
- Depth Resolution is in the nm range
- GD is complimentary to other techniques that provide imaging, lateral resolution or molecular information

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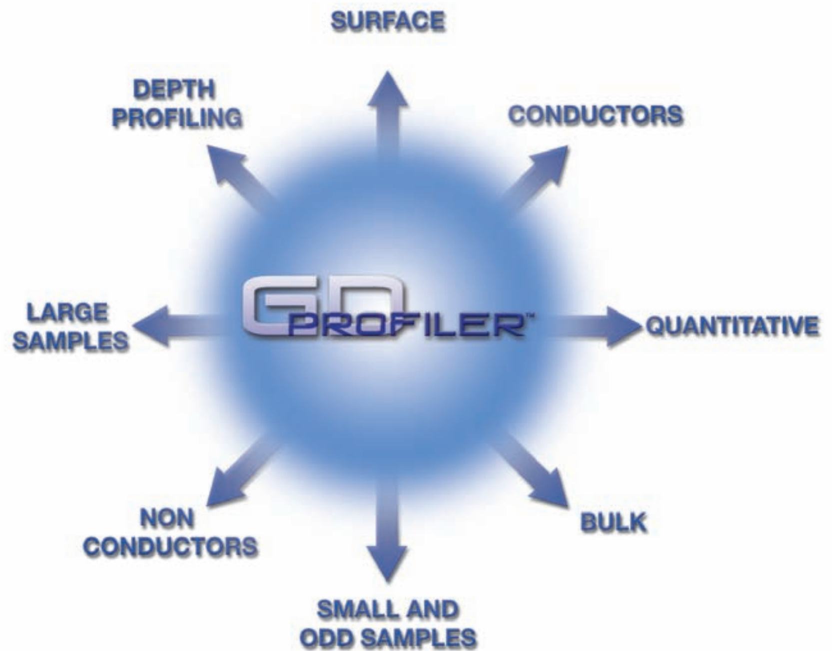
Discover A Whole New World of Support

We know Glow Discharge and its multiple application possibilities and we know that you need a dedicated solution. Our staff of analytical specialists, advice from world-recognized experts in GD, provision of reference materials, a comprehensive data base of publications and links to other users around the world provide the tools necessary to achieve results.

- Training programs that balance theory with practical hands-on experience
- World wide Applications assistance
- ALLIANCE on line catalog of supplies, accessories and upgrades
- Comprehensive partnership agreements for service maintenance
- User Group meetings and World Link Network provide continuous communication



Specifications and instrument appearance subject to change without notice.

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