Innovations in Optics for Emission Spectroscopy

Performance Meets Flexibility

Optical Emission Spectroscopy (OES) is generally regarded as the oldest instrumental technique for elemental analysis, tracing its origins to the mid 19th century. By the middle of the last century, developments in instrument design, optics, detector technology and electronics had created an analytical tool that had become one of the most widely used in materials testing. This is still the case today. OES instruments from SPECTRO Analytical Instruments have earned an enviable reputation for performance and reliability and thousands are to be found in laboratories and metallurgical industries throughout the world.

The heart of any OES instrument is the optics and detection system. Traditionally, the photomultiplier has been the detector of choice, but developments in semiconductor technology have yielded solid-state detectors such as CCD's that can approach the performance of the photomultiplier. Both types of detector have their advantages in terms of analytical performance, but compromises inevitably have to be made when choosing which to use in an instrument, and most commercially available instruments use either photomultiplier or CCD detection. The SPECTROLAB M10 has a revolutionary hybrid optical design that incorporates both types of detector in the same instrument in optimized configurations, allowing the analyst to choose the detector best suited for the individual application – truly the best of both worlds!

HISTORY OF OPTICAL EMISSION SPECTROSCOPY

Optical Emission Spectroscopy can be traced back to the work of pioneers such as Angström, Bunsen and Kirchoff in the second half of the 19th century, but perhaps the earliest reference is to Charles Wheatstone, who reported in 1835 that "when metals were volatilized in the electric spark, their light, examined through a prism, revealed certain rays which were characteristic of them". In 1853, Angström reported that an electric spark yields two superimposed spectra, one from the metal of the electrode and the other from the gas through which it passes. These phenomena were explored systematically by Bunsen and Kirchhoff, who developed a prototype spectroscope and used it to identify the emission spectra of sodium, lithium, and potassium. Later Bunsen went on to use spectroscopy to discover two new elements, cesium and rubidium. Spectrum analysis was applied in other areas of science, notably astronomy, and ultimately led to the development of atomic theory and to quantum mechanics.

The early experiments used gas flames and electric sparks to energize atoms and cause them to emit their characteristic spectra. Arcs and sparks are still in use today, but are now much more powerful, controlled and efficient. Other sources such as the Inductively Coupled Plasma (ICP-OES) and Glow Discharge (GD-OES) are also used. Early studies were in the visible region of the spectrum, but this has extended into the ultraviolet

– indeed many of the most useful emission lines are found there. The simple prism-based spectroscopes and photographic plates that were used to record the spectra have been replaced by high-performance optical systems and detectors, and these are discussed in this paper, illustrated by the advanced technology employed in the SPECTROLAB OES Spectrometer from SPECTRO Analytical Instruments.

INSTRUMENT BASICS

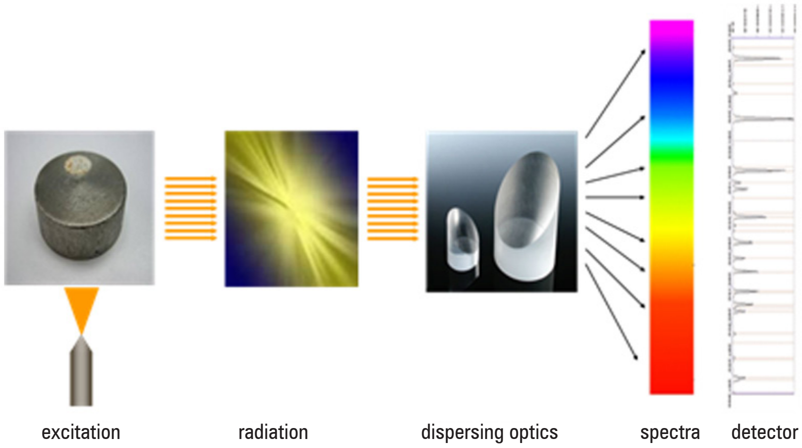


Fig 1: Schematic diagram of Emission Spectrometer

Fig 1 illustrates the essential components of an Emission Spectrometer, which are:

- An excitation device to provide the energy to excite the atoms in the test sample.
- Optics to isolate the specific wavelengths for the elements to be measured
- A detector system to measure the intensity of the light emissions
- Electronics to acquire the detector signals and to control the functions of the spectrometer
- A computer with software for calculation and display of emission spectra and concentration values

It is well known that the emission spectra generated can be very complex. Over 4000 different emission wavelengths (referred to by spectroscopists as spectral lines) have been identified for iron alone. Multi-element spectra can be very complex indeed, and any ionized atoms may give rise to emission spectra of their own, to further complicate the issue. Fig 2 shows the spectrum of just two elements, Fe and Co, covering a range of only 30nm in the ultra-violet region of the spectrum.

Many of the most useful analytical lines are found in the ultra-violet region of the spectrum, below 200nm, where oxygen in the atmosphere absorbs the radiation strongly.

A spectrometer to adequately cover the complete spectrum will therefore have the following characteristics:

- The ability to separate and identify closely adjacent spectral lines. This is essential for meaningful analysis and to avoid interelement interferences and is defined by the spectral resolution of the instrument. It should also be able to measure the required number of elements simultaneously, not only to save time but to eliminate any time-dependent variables and improve precision.
- A wide optical range covering the ultra-violet and visible regions of the spectrum. This requires an optical path free of atmospheric gases that might absorb the element lines of interest. It also means that any optical windows have to exhibit high transparency and any mirrors or other reflecting components need to have high reflectivity across the working range.
- To preserve the validity of calibrations and ensure accurate and precise results, the optical system must be very stable mechanically and not show any instability due to environmental changes such as temperature.

The optical system in SPECTROLAB exhibits all these characteristics.

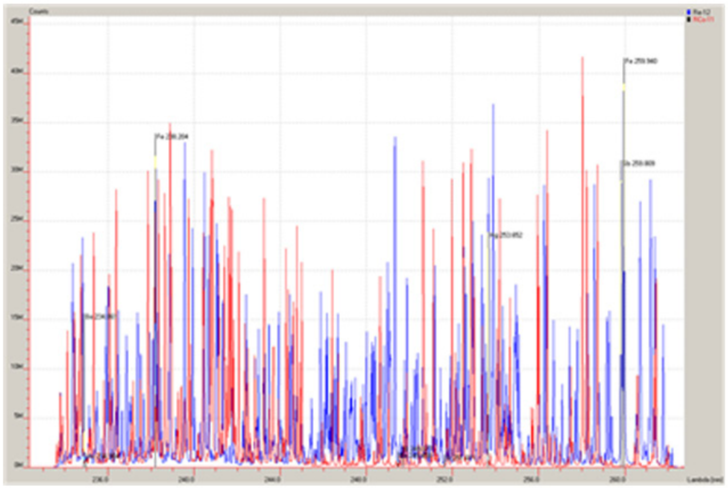


Fig 2: Partial Emission Spectrum of Fe and Co

Diffraction Gratings

The optical module, or polychromator, is the heart of any optical emission spectrometer, and the heart of the polychromator is the component used to disperse the spectral radiation and so isolate the spectral lines of interest. In the earliest days of emission spectrometry, prisms or even filters were employed, but today diffraction gratings are universally used.

The diffraction grating is a device with multiple angled parallel grooves across its surface. These grooves are very narrow - typically 1800 to 3600 per millimeter for a UV-visible grating.

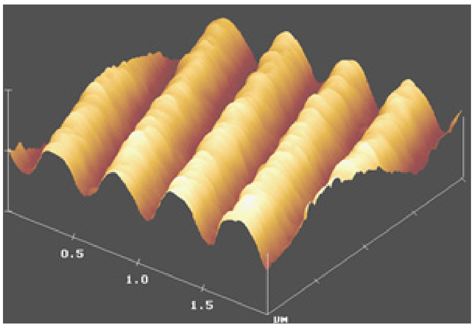


Fig 3: Grating Surface (highly magnified)

Light passing through or reflected from such a surface (almost all gratings used in spectroscopy are reflection gratings) will undergo diffraction, where essentially each groove acts as an individual source of light, with a very small path difference between them. Under these conditions, there will be interference between light from adjacent grooves, and reinforcement (maxima) and cancellation (minima) will occur at certain angles to the grating depending on the wavelength. For an incident light beam normal to the grating surface, the wavelength maxima for each wavelength λ will occur at an angle θ according to the grating equation:

$$n \lambda = d \sin\theta$$

Where d is the grating constant given by the groove spacing and n is an integer. This is for illumination normal to the surface; when the grating is illuminated at an incident angle θi, the equation becomes

$$n \lambda = d (\sin\theta + \sin\theta i)$$

As every wavelength leaves the grating at a different angle the spectrum is now dispersed. For a given geometry it is straightforward to calculate at which angle a particular wavelength (i.e. emission line) will occur. If a detector is then placed at that angular position, it should be possible to measure the intensity of the selected line. However in a typical spectrum, with many thousands of lines, it is necessary to isolate the line of interest. Traditionally this was done using a narrow slit, but modern CCD detector technology can satisfy that objective without the need for slits, as will be discussed later.

Looking again at the grating equation, n is an integer that is called the "order" of the grating, and the available light is distributed between the various orders. n can be negative, 0 or positive. At n=0 (the "zero order", all wavelengths are reflected at the same angle, like a normal mirror. It is possible to concentrate light into a particular order by controlling the depth or shape of the grooves on the grating during manufacture. This is termed "blazing" the grating.

Gratings used in the spectrometers produced by SPECTRO Analytical Instruments have between 1800 and 3600 lines per millimeter depending on the application. The working wavelength range is from 120 to 800 nm and they are normally optimized for maximum efficiency in the far (vacuum) ultra-violet region in the first or second order.

Originally, gratings were manufactured by laboriously machining grooves on to a substrate, usually glass, with a very precise and very expensive "ruling engine". The ruled grating was then coated with a reflective layer such as aluminum. Ruling individual gratings is a very slow and expensive process, so to save cost it was expedient to produce a number of cheaper "replica" gratings by casting copies in a suitable medium from the ruled "master". As in any copying process however, the quality of the replicas was not as good as the master, so for the best performance master gratings are always preferred. In recent times, the development of non-mechanical, laser-based holographic and photo-etching manufacturing processes have allowed master gratings to be produced more economically. All gratings used in SPECTRO OES instruments are masters, and many thousands have been manufactured in-house since 1988.

In a practical spectrometer it is necessary to focus the spectral image sharply at the exit slit in order to isolate the emission line of interest. In some designs, focusing mirrors are used to achieve this, but using optical manufacturing processes it is relatively straightforward to produce concave gratings that not only disperse the spectrum but also focus the light. This not only simplifies the optical design, but by eliminating a reflection also improves the energy transmission of the system.

When such a grating is illuminated from a slit as shown in Fig 4, the spectra produced are focused around the circumference of a circle, the diameter of which is the same as the focal length of the grating. The entrance slit and grating are also positioned on the circumference of the circle, which is termed the Rowland Circle of the grating.

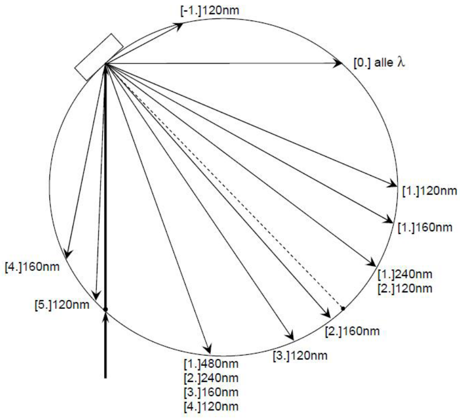


Fig 4: Rowland Circle of a concave grating

Fig 4 shows how the various orders are distributed around the circle. Detectors can then be placed around the circle to measure the light intensities. As can be seen, it is theoretically possible for lines with exact multiple wavelengths to coincide in the different orders: in practice this is not a major problem as corrections can be applied when it does occur. The full Rowland Circle is usually not used, and detectors are only set up for first and second order spectra. In such a configuration, the angular separation (or resolution) of adjacent lines depends on the grating and other factors such as slit widths, but is also a function of the size of the circle: the larger the circle, the better the resolution. In the SPECTROLAB spectrometer the Rowland Circle has a diameter of 750mm. The SPECTROLAB can achieve 9 picometer (0.009nm) resolution.

The Paschen-Runge mounting uses only a segment of the Rowland Circle. This is a very compact arrangement, and its simplicity and ruggedness makes it especially suitable for portable and mobile spectrometers.

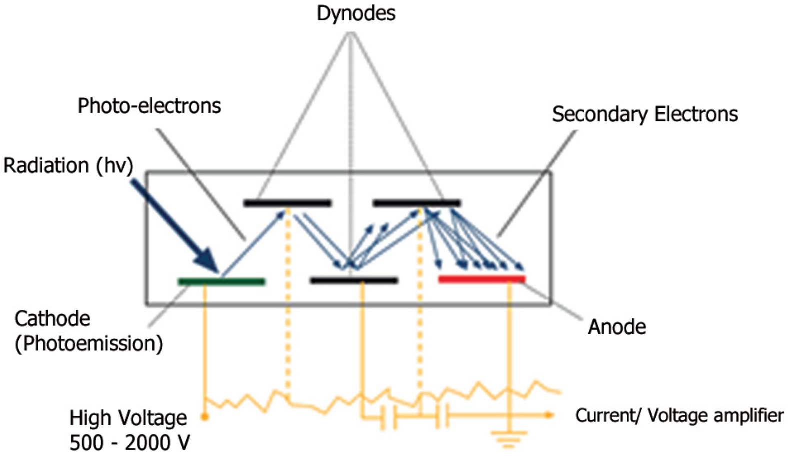


Fig 6: Schematic diagram of photomultiplier

Fig 5: Paschen-Runge mounting

In Fig 5, photomultiplier detectors are arranged around the Rowland Circle, one for each wavelength to be measured, and each with its own exit slit to select and isolate the spectral line. CCD detectors can also be used, indeed in the SPECTROLAB both are used, but before explaining how this is done these two types of detector will be described.

Detectors for Optical Emission Spectrometry

For many years, the preferred detector for OES was the photomultiplier tube (PMT). This is a vacuum tube device that detects and amplifies electromagnetic radiation to generate an electronic signal.

When light enters the photomultiplier, it first falls upon the photocathode, a photosensitive surface that emits electrons when light photons fall upon it. This signal is too small to be used immediately, and must be amplified. Photoelectrons emitted from the photocathode are accelerated by an electrical voltage towards the first of a series of dynodes, which has the property of emitting a number of secondary electrons when hit by each primary electron from the cathode. This happens at each dynode, thus achieving a very high cascade amplification of the original signal. The electrons emitted by the last dynode appear as a current signal at the anode and are passed to the instrument electronics. Typically, the accelerating voltage between each dynode is of the order of 100V, so high voltages, between 500 and 2000V, are required in total across the device.

PMT's are available in many different sizes and designs. They can be side-window or end-window. In either case the window material must be transparent to the radiation of interest, and fused silica is a common choice. Two popular tubes used in OES are the Hamamatsu IP 28, a side-window tube with a diameter of 28mm, and the R 300, an end-window tube 13mm in diameter.

The photomultiplier has several advantages as a detector. Although other types of detector are available, its speed of response makes it superior for tasks such as the study of individual sparks, and as will be discussed later this has a number of useful routine applications. In the SPECTROLAB the PMT signal is processed with micro-integrators that enable readings in microsecond ranges, so that a single spark can be divided into 100 or more steps giving detailed information about the spark for each spectral line. It is then possible to define integration windows with optimized dynamic range and the best signal to noise ratios for trace analysis (Time Resolved Spectroscopy -TRS). In contrast to conventional spectrometers it is now also possible to determine the light output for each individual spark instead of integrating the total output over a fixed measurement time. (Single Spark Evaluation - SSE). This allows elimination of "defective" sparks due to sample imperfection like inclusions or porosity, resulting in significantly improved analytical precision and accuracy. Conversely it is also possible to detect and quantify such inclusions! Both TRS and SSE can be independently defined for any of the photomultiplier tubes as

Fig 7: Typical side-window photomultiplier tube (Hamamatsu IP28)

required, multiple times if necessary. This flexibility enables optimal analytical results to be achieved with the shortest measurement time.

There are, however, some disadvantages. Each photomultiplier has to have its own exit slit, which must be very precisely aligned to select the required line. This is a highly skilled task. This usually means that the emission lines are chosen and installed in advance to suit the application, and changing them is an expensive process. This means that unexpected elements in the sample will not be measured and changes in the sample – perhaps a deliberate change in alloy composition – will require a reconfiguration of the instrument. The physical size of these detectors means that it is difficult or impossible to set up for very closely adjacent lines, which limits the choice available for analysis. It also limits the total number of detectors that can be accommodated in the spectrometer. Different types of PMT may need different mounting systems.

In recent years advances in semiconductor technology have led to the development of a new type of detector, the Charge Coupled Device or CCD.

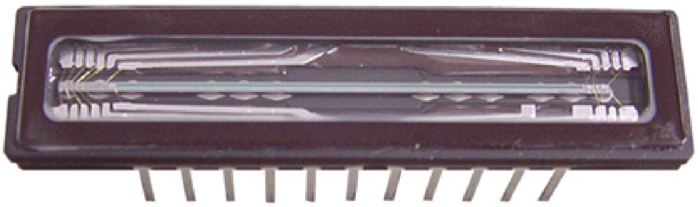


Fig 8: CCD detector

In these devices the light is converted not to a voltage or current, but to an electric charge, which accumulates in the device until it is read by external electronics. The more light that arrives at the detector, the greater the charge. A CCD module such as that in Fig 8 is actually a linear array of tiny detector elements, or pixels, each of which is a separate CCD detector. In the case of the detectors used in the SPECTROLAB, each array contains 3648 pixels, each of which is 8 μm wide and 200 μm high. Each pixel can thus act as its own exit slit, so such an array placed on the Rowland Circle will cover a segment of the total spectrum. In the SPECTROLAB optics, 18 CCD arrays are used to cover the wavelength range from 120 to 320 nm. This is equivalent to over 65,600 separate slits, each with its own detector. Some CCD detectors require on-chip cooling to well below ambient temperature to reduce their inherent noise characteristics, but this is not necessary with the SPECTRO detectors, which are maintained near ambient temperature. This has significant advantages: start-up times are greatly reduced and any accumulated humidity or vapor does not condense and freeze on to the detectors.

One potential problem with CCD detectors is that the chip housing is much wider than the sensor area itself. Placing the detectors straightforward to the Rowland Circle would cause significant vacancies in detecting spectral information due to the mechanical obstruction. This is solved by SPECTRO's patented arrangement of CCD sensors illustrated in Fig. 9.

The CCD detector arrays, each with its associated electronics, are mounted alternating above or below the plane of the Rowland Circle. The light from the grating is deflected either upwards or downwards by cylindrical mirrors. The mirrors are placed very close to each other. Moreover, during deflection the mirrors concentrate the light vertical to the Rowland Circle plane onto the CCD pixels. The arrangement in this way allows collecting virtually all spectral information along the Rowland Circle with high signal intensities. This use of CCDs, combined with the SPECTROLAB's 9 picometer resolution, allows complete freedom of line selection. As well as giving results on individual elements, the data can be displayed after the measurement as a complete emission spectrum, offering many possibilities for R&D and the analysis of unknown materials or new alloys. For routine measurements, based on selected lines, only the "Regions of Interest" (RoI) are transmitted, keeping measurement times to a minimum.

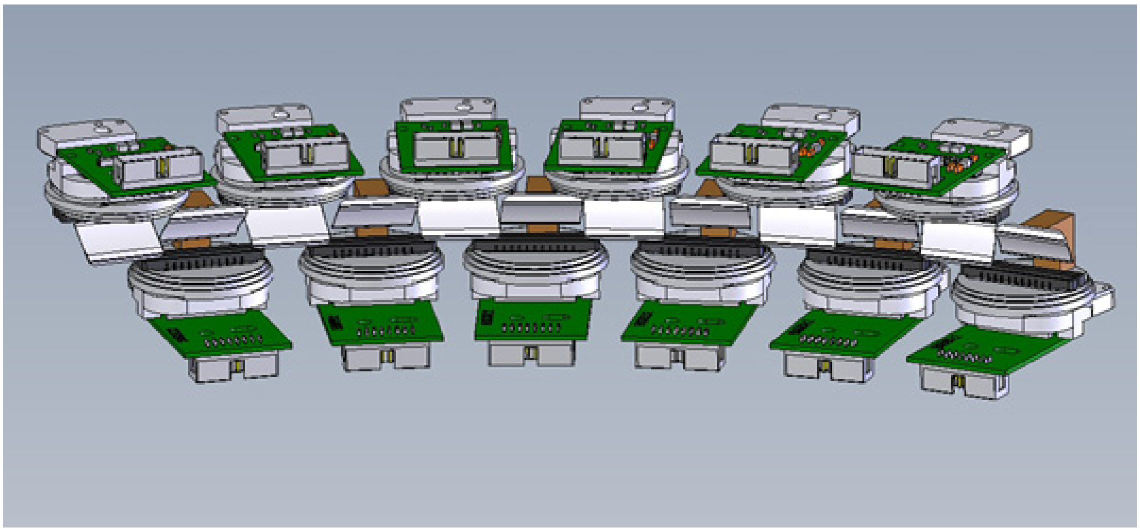


Fig 9: CCD Detector Assembly

Clearly both detector types have their advantages, and to choose one or the other compromises the performance that can be expected and limits the application scope of the instrument: the innovation of the SPECTROLAB design is that it allows both types of detector to be used, across the wavelength range and simultaneously.

THE SPECTROLAB HYBRID OPTICS

A schematic of the SPECTROLAB appears in Fig 10

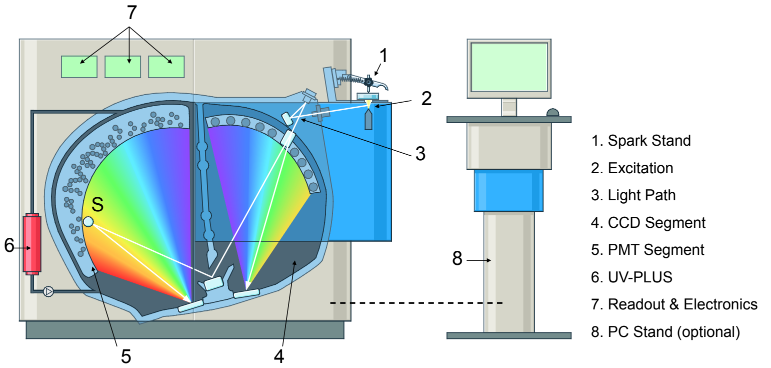


Fig 10: Schematic diagram of SPECTROLAB

The optics of the SPECTROLAB are in two sections, a CCD segment (4) and a PMT segment (5) The optical path can be followed at (3): light from the spark (2) enters the CCD segment via an entrance slit and falls directly onto a 3600lines/mm concave grating. The dispersed spectrum from this grating is focused on to the CCD arrays located around its Rowland Circle. Simultaneously, light passes through a separate aperture, through the CCD segment, refocused into the PMT segment by a concave mirror, passing an entrance slit at (S) onto the PMT grating. The dispersed spectrum from this grating is focused on to the exit slits infront of the photomultipliers around its Rowland Circle. Up to 108 PMT positions are available. The optical parameters are summarized in Table 1.

An optional additional CCD module, fiber-optic coupled to the spark stand, covers wavelengths from 233 nm - 780 nm, so that the instrument can then cover the entire spectrum continuously from 120nm to 780nm.
The readout system (parallel 12Bit analog/digital converters operating at a frequency of 1MHz for each channel) allows the data from both detector systems to be collected and processed simultaneously. This gives unrivalled flexibility: a complete sample spectrum can be obtained from the CCD module at the same time that the PMT module is performing the tasks at which it excels, such as ultra-trace analysis or the study of individual sparks.
Many of the best analytical lines are found in the ultra-violet and "vacuum" ultra-violet (UV and VUV) regions of the spectrum, below 200nm. At these wavelengths, oxygen absorbs UV light so air must be removed from the optical path. Most instruments use either a vacuum system or purging with relatively large volumes of argon to achieve this, but these methods are expensive and can cause contamination of the optical system with either vacuum pump oil or inert gas impurities. SPECTROLAB uses a unique system called UV-PLUS, in which the hermetically sealed optical chamber is filled just once with argon, which is circulated through the system by a membrane pump. Any traces of impurity are removed by filter cartridges built into the system (see (6), Fig 10). Operating costs are reduced and maintenance intervals extended.
Mechanical stability of the optical system is vital, particularly to maintain the alignment and calibration of the detector systems. The optical module of SPECTROLAB is itself an example of state-of-the-art aluminum engineering, shown diagrammatically in Fig 11.

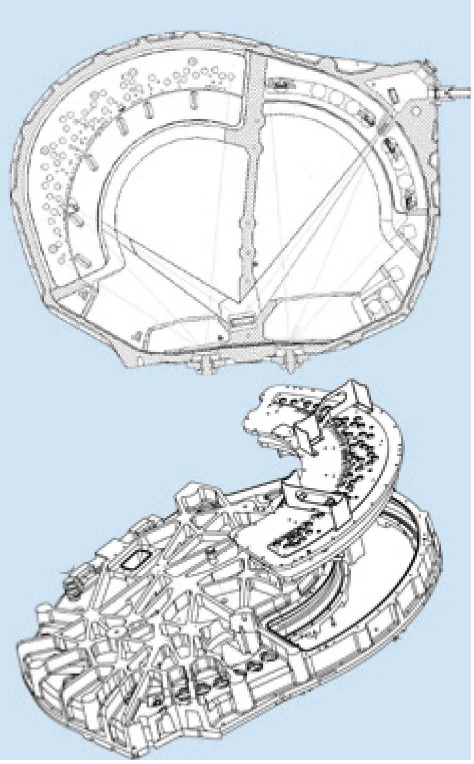


Fig 11: SPECTROLAB optical module

This is an extremely rigid and stable unit: in tests at its operating weight and at 200 mbar excess gas pressure, the maximum distortion measured at any point on the structure was less than 30μm. In normal operation, the optics are held at constant temperature and pressure to further improve stability.

Table 1: SPECTROLAB Optical Parameters

	CCD Module	PMT module
Grating	3600 lines/mm	2924 lines/mm
Spectral resolution	8.5 pm	12pm (1st order) 7pm (2nd order)
Pixel resolution*	3 pm	
Rowland Circle	750mm	750mm
Wavelength Range	120nm-320nm	120nm-500nm

** Pixel Resolution is the range of wavelengths that actually falls across each pixel. This is not the same as Spectral Resolution, which describes the smallest difference in wavelengths that can be discriminated by the instrument.*

OPTICAL PERFORMANCE = ANALYTICAL CONVENIENCE

The SPECTROLAB Hybrid Optics offers flexibility combined with performance. The analyst has an unrivalled choice when deciding on the conditions used to measure each sample.

The CCD optic provides the ability to examine the complete spectrum (after the measurement if necessary) to identify unexpected elements or to evaluate new alloys. Fig.12 shows the detail and resolution obtainable, showing a section of a spectrum.

The PMT module offers complementary facilities: for example for the instrument can be configured with a selection of PMT's optimized for the analysis, e.g. precious metals, aluminum alloys, etc. The speed of response of this detector also gives it the capability to examine individual sparks in detail.

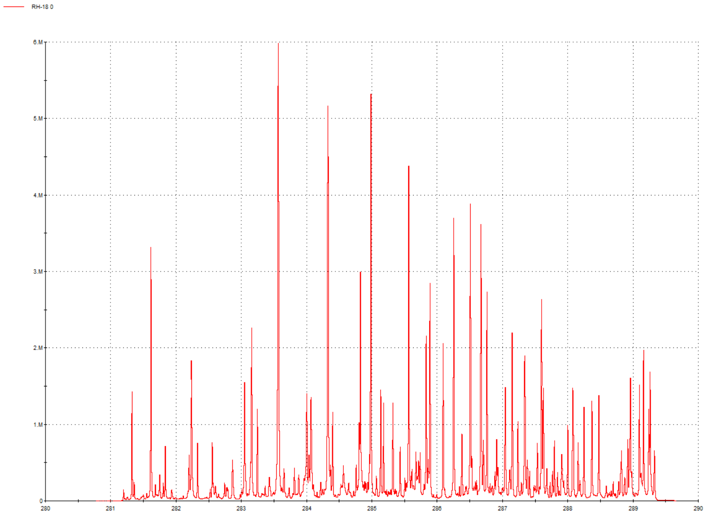


Fig 12: partial CCD spectrum

Each PMT is linked to a micro-integrator that scans the signal in 5 μsec steps, so each spark can be divided into up to 100 segments, allowing an intensity distribution analysis during the discharge for all the elements set up in the PMT module. Much useful analytical data can be obtained in this way, for example in the detection of inclusions in metallurgy.

Aluminum is widely used in the steel industry as a deoxidizing agent. In the steel it can be found as "soluble aluminum" e.g. as free metal, or as "insoluble aluminum" e.g. as aluminum oxide. In controlling the steelmaking process, it is important to know the relative proportions of the two forms. It would be possible to do this by classical methods, for example by dissolving the soluble part in acid, but this is far too slow for process control. It is possible, however, to perform this analysis by OES, by evaluating the statistics of the intensities of individual sparks (Single Spark Evaluation, SSE).

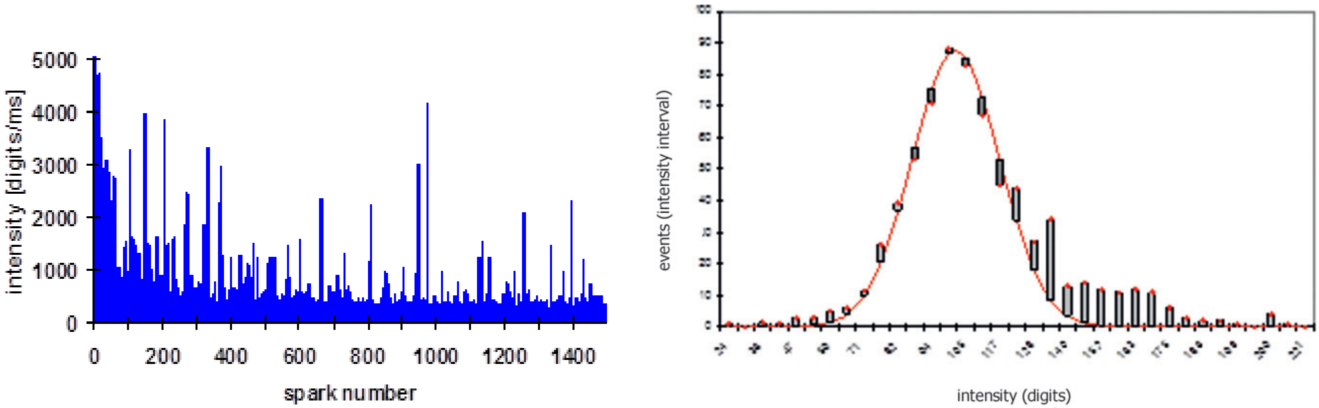
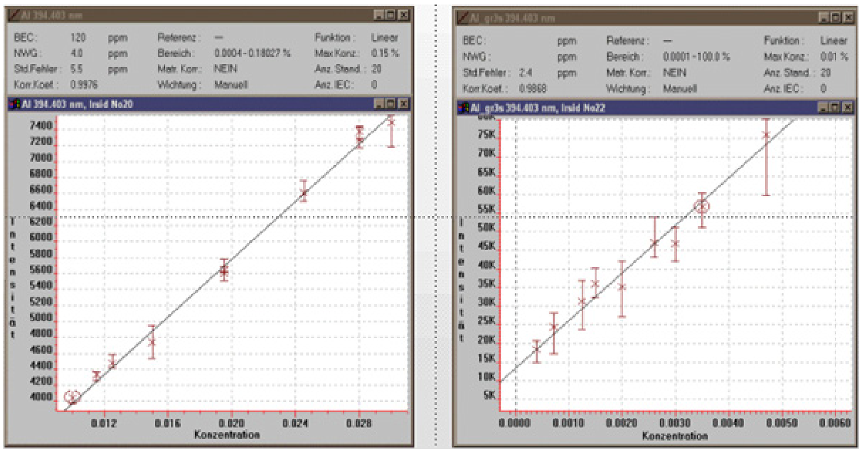


Fig 13: Determination of Soluble/Insoluble Al in Steel

As can be seen from the intensity graph, a certain number of sparks show significantly higher Al signals than the norm. The probability is that these are due to the spark hitting aluminum oxide, where the concentration of Al will be much higher than in the matrix (steel). Plotting the frequency of these events shows that most lie on a Gaussian distribution, whereas the higher intensities (to the right of the Gaussian peak) are due to the oxide. The values on the Gaussian distribution can be used to derive a calibration for "normal" analysis of Al, whereas from the others a calibration can be derived for "insoluble" Al:



Al 394.403 nm

BEC:	120	ppm	Referenz:	—		Funktion:	Linear
NWG:	4.0	ppm	Bereich:	0.0004 - 0.18027 %		Max Konz.:	0.15 %
Std.Fehler:	5.5	ppm	Matr. Korr.:	NEIN		Anz. Stand.:	20
Korr.Koeff.:	0.9976		Wichtung:	Manuell		Anz. IEC:	0

Al 394.403 nm, Insid No20

Al_gr3s 394.403 nm

BEC:		ppm	Referenz:	—		Funktion:	Linear
NWG:		ppm	Bereich:	0.0001 - 100.0 %		Max Konz.:	0.01 %
Std.Fehler:	2.4	ppm	Matr. Korr.:	NEIN		Anz. Stand.:	20
Korr.Koeff.:	0.9868		Wichtung:	Manuell		Anz. IEC:	0

Al_gr3s 394.403 nm, Insid No22

Fig 14: Calibrations for "Soluble" Al (left) and "Insoluble" Al (right)

Another SSE technique can be used for the identification and quantification of inclusions in metallurgical samples, by correlating the spark intensities according to the elements expected in the inclusions. Examples where this has been successfully performed with SPECTROLAB are Ti nitrides in steel, TiB in aluminum base, and MnS in steel. A typical example (TiN) in steel is shown in fig.15. In this example, the spark intensities of Ti at 337.3 nm and N at 149.3 are plotted. It can be seen that there is a correlation between certain sparks (shown in blue) that simultaneously show signals for Ti and N, i.e. they are signals due to the inclusions. From this data, an inclusion count can be derived.

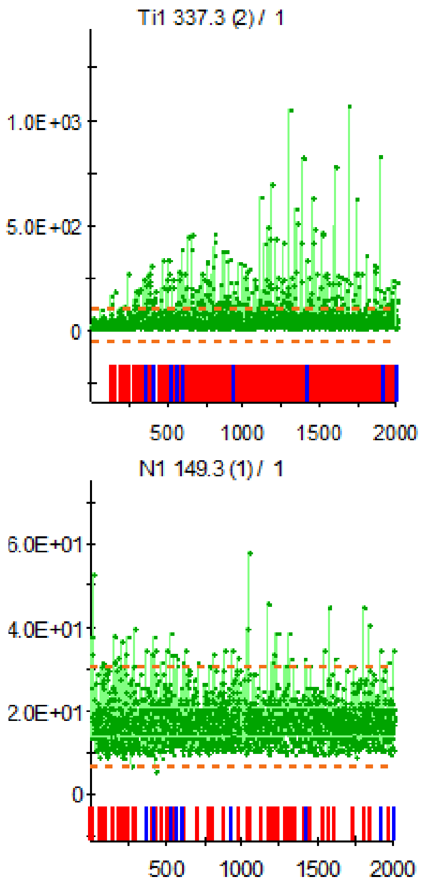


Fig 15: TiN in low alloy steel

Inclusion count results obtained by this technique correlate well with microscopically methods of inclusion counting, such as DIN 50602.

Yet another approach to spark evaluation is Time Resolved Spectroscopy, or TRS. The very fast response of the photomultiplier allows the emission due to each spark to be analyzed with respect to time. It is found that during the early part of the spark, background emissions and ion emissions may be significant, enhancing the measured signal.

It can be seen that when the electrical current in the spark is at its maximum, the background signals and ion signals that could interfere with the measurement are at their highest and are large compared with the elemental signal. The elemental signal, however, persists after the spark has finished, and so can be measured during a "TRS window" when the interference is at a minimum.

This can significantly improve the precision, as is shown in the comparison between data obtained with TRS and that obtained from the complete spark

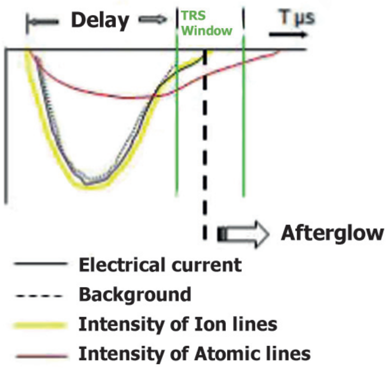


Fig 16: Time resolved OES peak

Full spark (left) and combined TRS and SSE (right)

In this example, using TRS has significantly reduced the variability in the measured intensities. The precision has been further improved by using SSE to eliminate the "outliers" from the result. The first approximately 250 (blue) sparks have been excluded from the result to remove variations that can occur when the spark first strikes the sample surface, before the excitation process stabilizes.

Of course, the ability to analyze individual sparks in detail in this way is only helpful if the sparks themselves are repeatable and consistent. For the first time in OES, the SPECTROLAB has full digital control over the spark. Power control, pulse definition and off-line control are all digital, using the latest 32MHz microcontroller to give much improved control resolution - a sampling rate of 400 times in a 200 µs discharge and a sampling resolution of 125 mW.

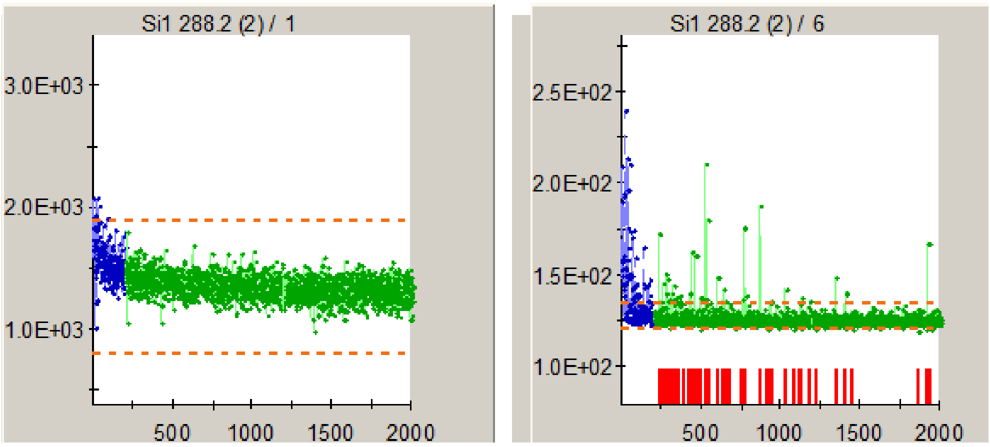


Fig 17: Si in High Alloy Steel.

CONCLUSIONS

The unique hybrid optical system employed in SPECTROLAB gives unrivalled flexibility and analytical power. For the first time, the different but complementary benefits of PMT and CCD detection are available to the analyst in one OES-instrument – the best of both worlds. This revolutionary optical system is combined with advances in other key areas like spark control and signal processing to create the first of a new generation of optical emission spectrometers.

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