

## A NEW INSTRUMENT IN ANALYTICAL SPECTROSCOPY – LASER ELEMENTAL ANALYZER (LEA)

SOLAR TII Ltd (Minsk, Belarus) reports here the principally novel development of an atomic-emission analyzer – the LEA-S500. The LEA's key feature is the originally-designed laser which is used as an excitation source. The first Analyzer model was designed for the analysis of both conducting (metals and alloys) and non-conducting (glass, ceramics, films, etc.) materials.



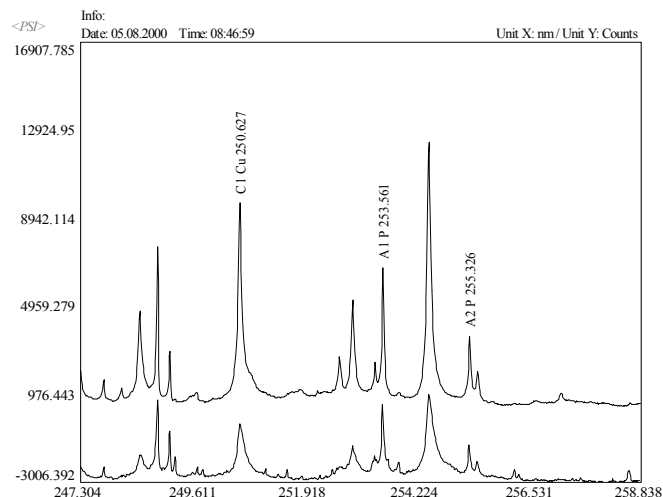
**Fig.1. External view of LEA-S500 Laser Analyzer.**

Due to the extensive work carried out in the research of the LEA's application range, we have recently developed the analysis techniques that can be used to analyze the elemental and bulk chemical compositions in different materials using the LEA. A variety of the relevant tasks have stimulated the development of the Analyzer's original configuration and advanced Software.

### Analyzer brief description.

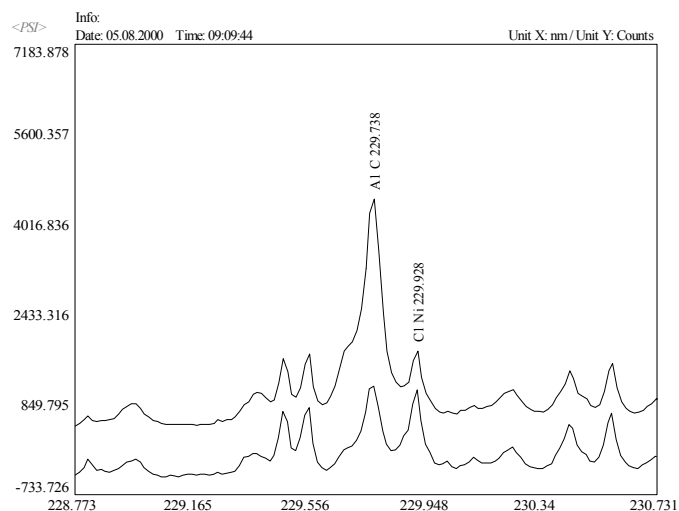
Fig.1 gives the Analyzer's external view.

The specifically developed Q-switched Nd:YAG laser is used as a spectrum excitation source. The laser generates an average power of 1 W at 1064 nm and provides a peculiar temporal kinetics for pulse repetition. Treatment of the material surface with pulses creates the specific conditions for the plasma cloud formation. Under these conditions the near-surface layer plasma density and erosivity reduce giving rise to an increase in the intensity of spectral lines with high excitation energies, the background level decreasing too and the spectral lines becoming narrower (Fig.2).



**Fig.2. Spectra of one and the same sample subjected to treatment with a conventional (bottom curve) and original (top curve) laser.**

The sample compartment, where spectrum excitation occurs, is equipped with a special pump to permit both air exhaust (down to  $10^{-1}$  Pa) and creation of the required atmosphere (argon, for instance) in the breakdown region. Air exhaust allows for the increase in the detection sensitivity of some volatile and difficult to excite elements, as, for instance, in carbon, whose concentration is very important for the steel analysis (Fig.3).



**Fig.3. Spectrum section of a steel sample containing 0.71% C (CIII 229.689) with air exhaust (top curve) and with no air exhaust (bottom curve) .**

The spectrograph and the beam focusing system in the Analyzer, also contribute to the Analyzer's high sensitivity. The number of optical components in the Analyzer is minimal. The spectrograph using a vertically-symmetric configuration with a F-number of 5.7, is an aberration-free astigmatism-compensated system with a high throughput to allow the most efficient utilization of a light signal.

The 1800 grooves/mm grating in use ensures a dispersion of 1 nm/mm. The spectrograph permits a fast scan between 190 and 850 nm.

With other gratings, the Analyzer can provide a dispersion of up to 0.5 nm/mm and cover a spectrum range of up to 1200 nm.

Spectra are detectable with a high-performance CCD-camera, specifically developed for short-time pulse processes.

Implementation of the above technique allowed us to produce an instrument with a variety of analytical functions. All performance characteristics of the Analyzer are computer-controlled via the dedicated Software.

## Software.

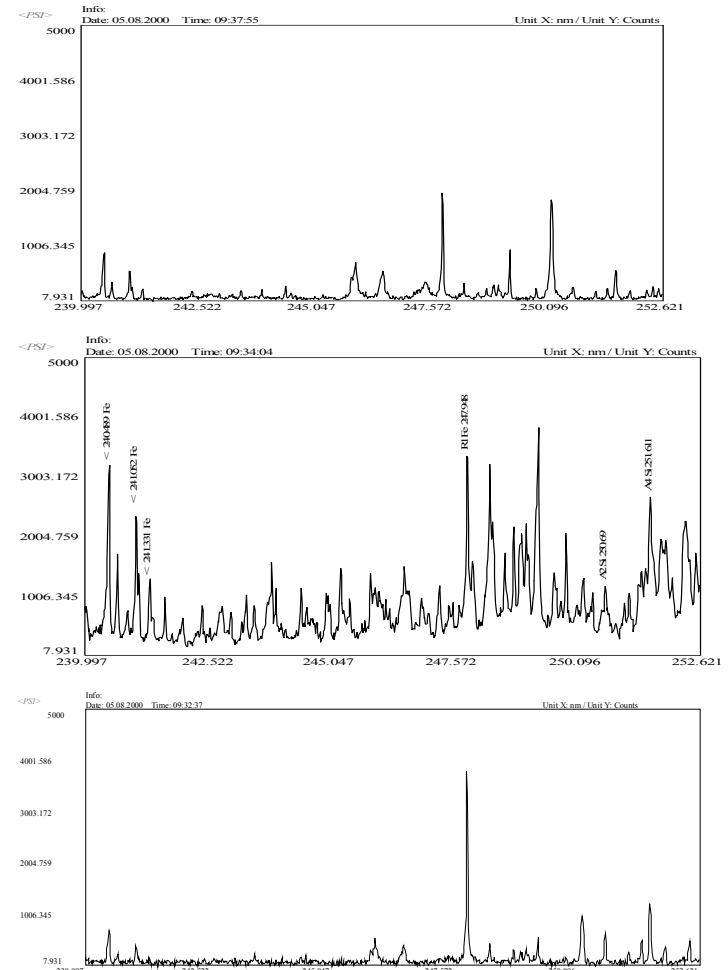
The Software permits an analyst, a technologist or an operator to obtain any sort of information. Today the progress in the Software development is ensured by utilization of internal algorithms and control functions combined with calculations. This approach to the Software will allow a variety of functions to be realized without compromising the simplicity of Interface, when in each particular case the user obtains the information he needs. This implies that with the development of some new analysis technique, the user's optimum interface and the instrument parameters will be simultaneously configured. As a result, one and the same Software program can be well employed by an expert-criminalist, a technologist at the chemical enterprise or by an operator at the metallurgical works.

The Analyzer Software provides:

- Availability of displaying the sample surface on the monitor during an analysis
- Analyzer's automatic control
- Wavelength automatic calibration
- System stability control
- Global re-calibration
- Database for material types
- Automatic identification of material type and elemental base
- Database for line wavelengths
- Indication of deviation from the specified material type
- Control of unexpected impurities
- Control of quality and reliability of analysis results
- Graphical representation of analytical signals
- Control of line profile
- Printout and mathematical treatment of analysis results

## Microstructural and global analysis within a single measurement cycle - the natural progress in analytical spectroscopy.

The laser capability of irradiating small areas (spots) on the material surface is used to expand the range of the Analyzer's functions as applied to metals and alloys.



**Fig.4. Spectra for different surface spots of aluminium alloy sample. The middle spectrum is the intermetallide (Fe) spectrum.**

The normally employed atomic-emission analyzers analyze the total chemical composition of a sample. Other methods are to be applied, however, to analyze the metal structure, the elemental distribution uniformity or gradient. By use of a laser and some computer-aided facilities to precisely position the analyzed spot, the measurements of the sample chemical composition and the analysis of the sample structure can be combined in a single run (Fig.4).

Using a short-focus system to focus the laser beam, an erosion spot can be minimized down to 30  $\mu\text{m}$ . Furthermore, making use of a built in camera to visualize the surface, one can directly identify the spots for the required sampling (Surface Mapping) from the computer screen. This allows you to simultaneously analyze a microstructure, impurities, the origin of point corrosion as well as to obtain the data on the distribution uniformity of the test element. With the use of the mathematical functions (integration or separation of the results,

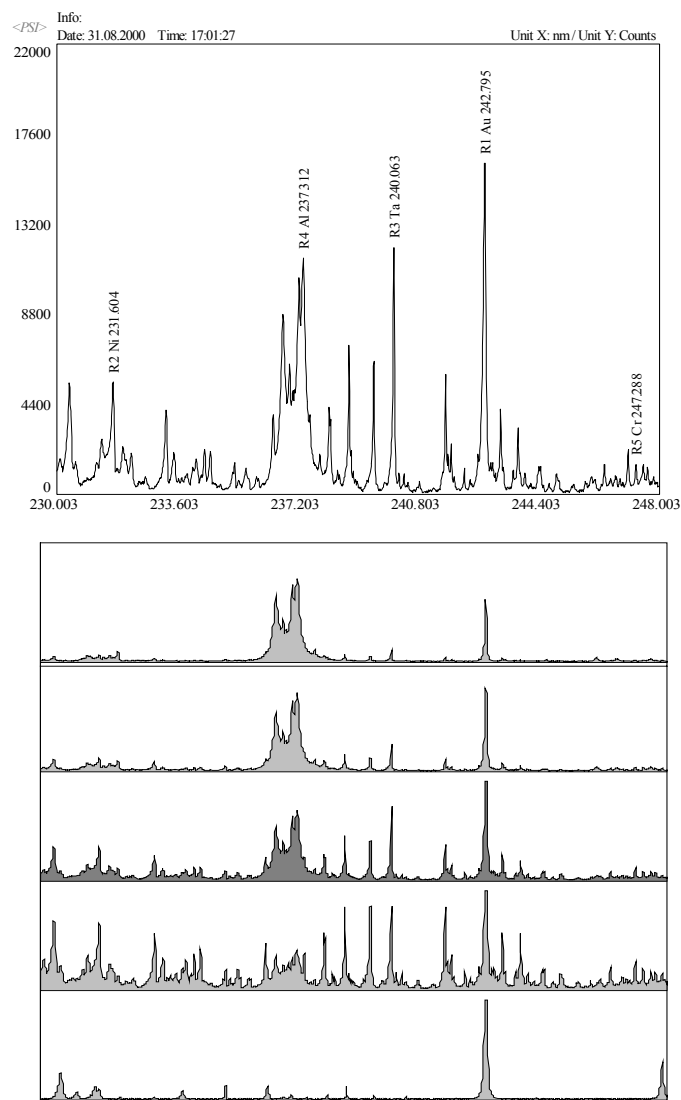
consideration of the base material, subtraction of buffer material spectra, etc.), the comprehensive information about materials can be obtained. Moreover, with suitable techniques in use, one can simultaneously analyze such waste materials as swarf, metallic dust or others.

### Sectioning. Coatings for ceramics, paper, glass, metals.

One more important application of the Analyzer is the analysis of simple and multi-layer coatings, i.e. the layer-to-layer analysis (sectioning). Such different materials as metals with electroplates, glasses with mirror multi-layer coatings, dyes on paper, glaze on ceramics were analyzed.

Both quantitative and qualitative analyses can be carried out with use of the calibration-free analysis technique. This technique also permits the thicknesses of coatings and layers to be determined together with their compositions.

Figure 5 presents an example of the spectra for a multi-layer coating on the  $Al_2O_3$  substrate.

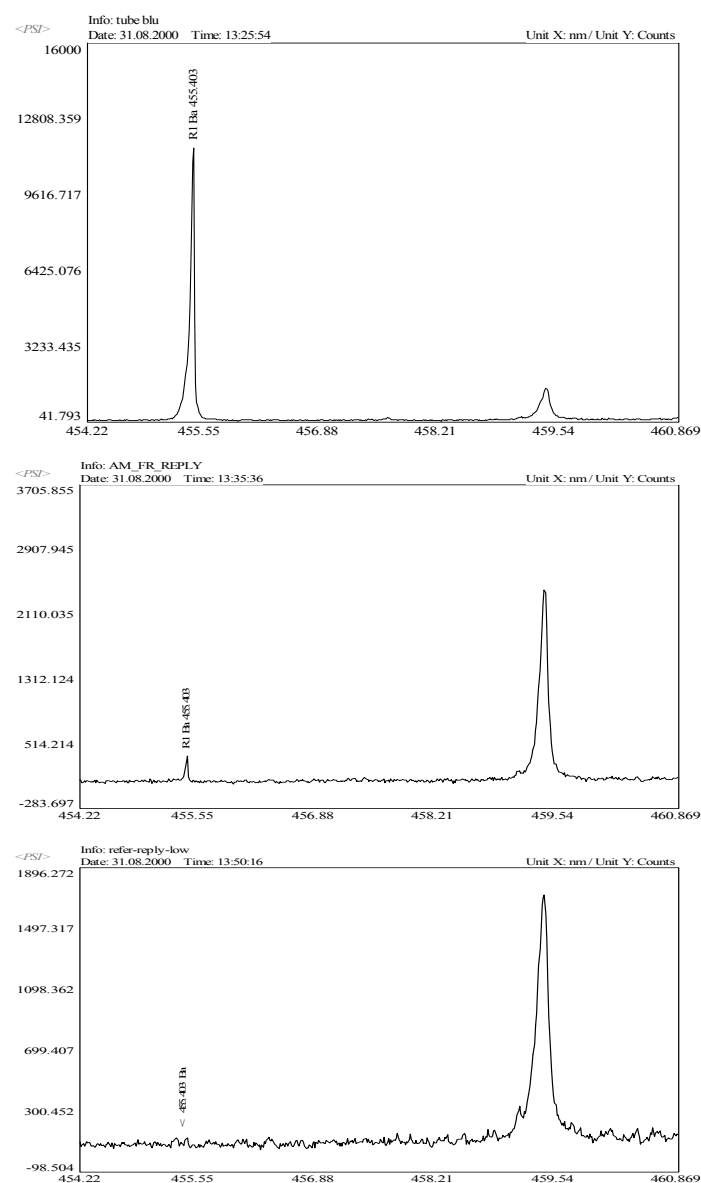


**Fig.5. Five spectra for a multi-layer coating (Au, Ta, Ni, Cr on  $Al_2O_3$ ) illustrating different concentrations of the elements in different coating layers.**

### Nonconducting materials: trace concentrations of catalysts in a plastic.

One of the main advantages of the LEA is its ability to analyze nonconducting materials directly, eliminating the complicated sample preparation step. Consider the case when the catalyst trace concentration is to be determined in a plastic.

A fragment - a transparent polyethylene tube, obtained with the aid of a barium-based catalyst - was used in the analysis. It was necessary to determine the barium concentration in the finished product. For the better accuracy of the results, the tube was made frosted. With this type of material the breakdown occurred at pulses whose intensity was considerably lower than that for the transparent material. The same sample preparation procedure was employed for the analysis of the glass surface.



**Fig.6. 455.403 nm Ba line. Ba concentration: (1) ~ 50 ppm, (2) 5 ppm, (3) < 1 ppm. Different Y-axis scales.**

From the analysis carried out it was concluded that the catalyst impurities (traces) are uniformly distributed over the entire bulk of the material. About ten pulses with a pulse

energy of 30 mJ were sufficient to carry out the analysis. The barium detection limit in such objects was nearly 1 ppm. Figure 6 shows the spectrum sections for several samples with different Ba concentrations.

Besides, a small concentration of Mg was also detected in the near-surface layer of the sample. This implies that it is always advisable to control the so called unexpected impurities during an analysis. This function is also Software-supported.

## Contact:

### **BELARUS**

#### ***SOLAR TII, LTD***

15/2, Akademicheskaya str.,  
Minsk 220072,  
Republic of Belarus  
Phone: +375 (17) 284 02 00  
Fax: +375 (17) 284 06 38  
E-mail: [laser@it.org.by](mailto:laser@it.org.by)  
Web: [www.solartii.com](http://www.solartii.com)

### **JAPAN**

#### ***Tokyo Instruments, Inc.***

6-18-14, Nishikasai,  
Edogawa-ku,  
Tokyo 134-0088, Japan  
Phone: +81 3(3686) 4711  
Fax: +81 3(3686) 0831  
E-mail: [sales@tokyoinst.co.jp](mailto:sales@tokyoinst.co.jp)  
Web: [www.tokyoinst.co.jp](http://www.tokyoinst.co.jp)

### **GERMANY**

#### ***PROSCAN elektronische Systeme GmbH***

Lechstrasse 40,  
D-86937 Scheuring, Germany  
Phone: +49 (8195) 999-511  
Fax: +49 (8195) 999-512  
E-mail: [solar@proscan.de](mailto:solar@proscan.de)  
Web: [www.proscan.de](http://www.proscan.de)