



Low Nitrogen determination in Steel

Alain Salaville and Jocelyne Marciano, Application Laboratory, HORIBA Jobin Yvon S.A.S., Longjumeau, FRANCE

Seiji Sakakura and Satoshi Yoshida, HORIBA Application Laboratory, Kyoto, JAPAN

Written by Kinumi Hayashi, HORIBA R&D Department, Kyoto JAPAN

Keywords: EMGA 920, Steel, Nitrogen, Elemental Analyzer

1 Introduction

Manufacturers have always understood the importance of controlling impurities in the steel production process as the level of impurities affects the mechanical properties of steels. Among these impurities is nitrogen, which can have a positive or negative effect on a steel's properties depending on its final use.

At each step in the process, the nitrogen level may need to be checked: in raw materials, in the different steps of production as well as in the final forming industry. As the EMGA-920 has a wide measurement range with a high precision all over the range, it's the ideal tool for an accurate and quick determination of this element at all the production steps.

The following example especially illustrates the ability of EMGA-920 to analyze a low level of nitrogen in a steel standard with an excellent repeatability.

2 Instrumentation

2.1 Principle (see figure 2)

The sample is loaded into a graphite crucible which has been placed on the lower electrode and then elevated to make contact with the upper electrode of the impulse furnace. A high current passes through the crucible to create a high temperature (up to 3000°C).

The gases extracted during the fusion are directly analyzed after the dust filter.

The Oxygen concentration is measured by CO and CO₂ non-dispersive infrared analyzers (NDIR) in order to achieve very good accuracy over the full measurement range.

The Nitrogen in the sample is extracted as nitrogen gas (N₂) and its concentration is determined by a thermal conductivity detector (TCD).



Figure 1: Model EMGA-920 for analysis of O/N concentration in solids



Gas flow diagram



Figure 2: Gas flow diagram of the EMGA-820/EMGA-920

2.2 Super High Performance

Wide measurement range

Thanks to dual NDIR detectors measuring CO and CO₂ for Oxygen determination and optimized TCD design for Nitrogen determination, the EMGA series provide the widest measuring range: for a 1g sample Oxygen can be measured up to 5% and Nitrogen up to 3%.

Precision

Likewise these optimizations lead to the World's best Oxygen/Nitrogen precision with a SD ≤ 0.02ppm or a RSD ≤ 0.5%, whichever is larger, measured with reference gas.

Standard method

Finally the EMGA-820/920 fulfills requirements of the standard methods for analysis of steel, titanium, tantalum, ceramics, etc. The main ones are listed here:

- ISO 10720:1997, ISO17053:2003
- JIS G1228:1997
- ASTM E1019, E1569, E1409

2-3. Unique features of EMGA-series

Programmable temperature curves (figure 3)

Different functions allow the users to easily optimize the fusion temperature according to the sample.

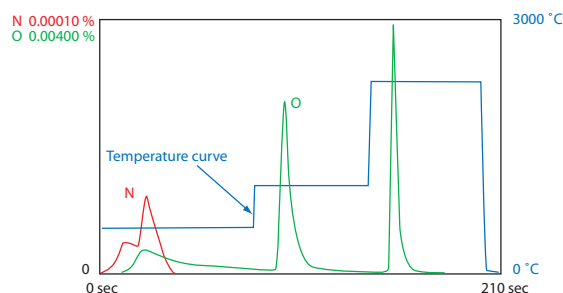


Figure 3: Oxide differentiation in iron powder

Dual Sample/Flux Introduction mechanism (figure 4)

This system allows the decontamination of the crucible and the flux. The following diagrams illustrate the 3 steps and the graphs illustrate the associated parameters evolution.

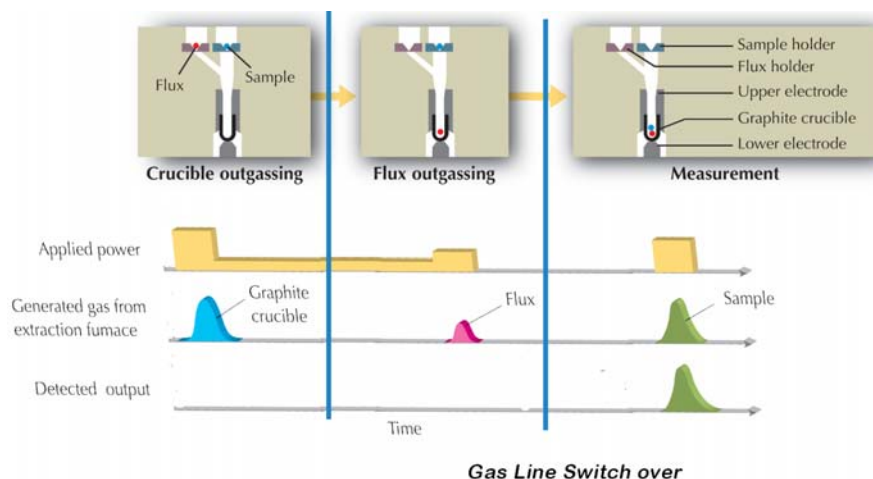


Figure 4: Dual sample/flux introduction mechanism



User friendly software with maintenance counter & navigator (figure 5)

In the maintenance window, you can reach pictures and videos illustrating maintenance operations by a simple click.

Maintenance counters inform operators when to replace consumables to assure consistent and accurate results, which is especially useful in a multi-user facility.

EMGA-920 model: Fully integrated accessories with simplified operations (figure 6 and 7)

With integrated automations, operations are much simpler and faster. Just enter sample name, weight and put the sample in the EMGA. Analysis starts immediately and all operations are done fully automatically till next sample. It is total "hands clean" use, so the operator doesn't need to touch crucibles anymore and doesn't have contact with carbon dust. Furthermore, as automatic operations are done while the next sample is being prepared, the gain in terms of time is about 40 s. compared with conventional systems.

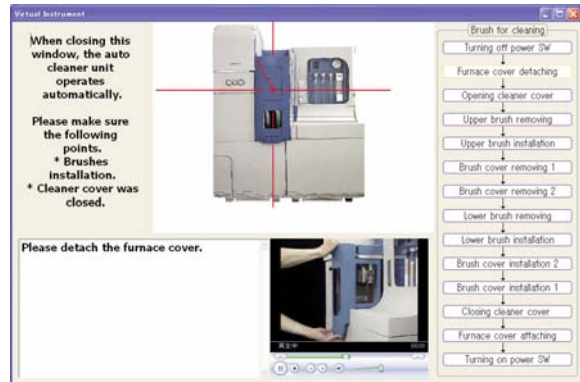


Figure 5: Software with maintenance counter

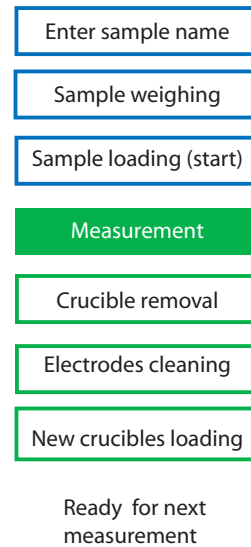


Figure 6: Sequence of measurement. (Steps in green are automatic)

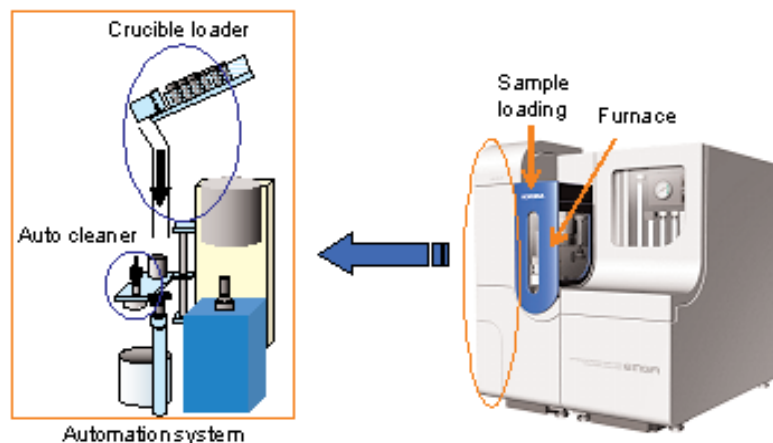


Figure 7: Details of the automation of the EMGA-920



3 Sample Information

Sample: JSS367-8 (N: 34ppm)
 Sample type: Swarf type steels
 Sample weight: Approx.,1.0g
 Calibration sample: JSS366-8 (N: 7.5ppm),
 JSS-368-8 (N: 144ppm)
 Flux: No flux
 Crucibles: Graphite crucibles (ECC. 3200043542)

4 Sample preparation

No sample preparation

5 Measurement & Results

Table1. Analysis data of samples

Sample name	Sample weight (g)	Nitrogen (ppm)
JSS 367-8	1.0042	31.93
	1.0090	31.83
	1.0045	32.02
	1.0048	31.74
	1.0020	31.33
Average		31.77
SD		0.27
RSD(%)		0.84

6 Conclusion

The EMGA-920 is the ideal instrument for controlling Nitrogen in steel industries: wide range of analysis, simple operations and software leading to multi-users, fully automated instrument that provides a quick result.

The above example especially shows the performance of the EMGA-920 in the determination of low nitrogen level in steel.

Moreover this model also gives excellent results for oxygen determinations, which is a very important element for the ductility of steel and consequently for forming.

info-sci.fr@horiba.com

www.horiba.com/scientific

HORIBA
Scientific

Explore the future

Automotive Test Systems | Process & Environmental | Medical | Semiconductor | Scientific

HORIBA

USA: HORIBA Jobin Yvon Inc., 3880 Park Avenue, Edison, NJ 08820-3012, Toll-Free: +1-866-jobinyvon
 Tel: +1-732-494-8660, Fax: +1-732-549-5125, E-mail: info-sci.us@horiba.com, www.horiba.com/scientific
 France: HORIBA Jobin Yvon S.A.S., 16-18, rue du Canal, 91165 Longjumeau cedex
 Tel: +33 (0) 1 64 54 13 00, Fax: +33 (0) 1 69 09 07 21, www.horiba.com/scientific
 Japan: Horiba Ltd., 2 Miyahigashi, Kisshoin, Minami-ku, Kyoto 601-8510
 Tel: +81 (0) 3 38618231, Fax: +81 (0) 3 38618259, www.horiba.com
 Germany: +49 (0) 89 46 23 17-0 Italy: +39 0 2 57603050 U.K.: +44 (0) 20 8204 8142
 China Beijing: +86 (0) 10 8567 9966 China Shanghai: +86 (0) 21 3222 1818 Spain: +34 (0) 91 724 16 57