Oxygen and Nitrogen determination in Boron Nitride

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

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1 Introduction

Boron Nitride (BN) has become a popular material as it can be used in many fields as varied as semiconductors, photovoltaics, cutting tools, lubrication or even nano tubes depending of its crystallographic form. The success of this manufactured ceramic is due to its excellent heat conductivity, chemical stability, electrical insulation, inertness and, finally, machinability.

Impurity levels, especially oxygen, can have a negative influence on the mechanical and thermic properties of the ceramic as B_2O_3 is formed at sintering temperature. High purity BN is also needed in semiconductor applications. On the other hand, in the synthesis of c-BN (cubic) from h-BN (hexagonal) a small amount of oxygen is positive as it decreases the conversion temperature and pressure.
This application note will show the relevance of the EMGA-920 to measure high llevels of nitrogen while simultaneously determining low levels of Oxygen with good precision.

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 CO_2 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 (N_2) and its concentration is determined by a thermal conductivity detector (TCD).

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

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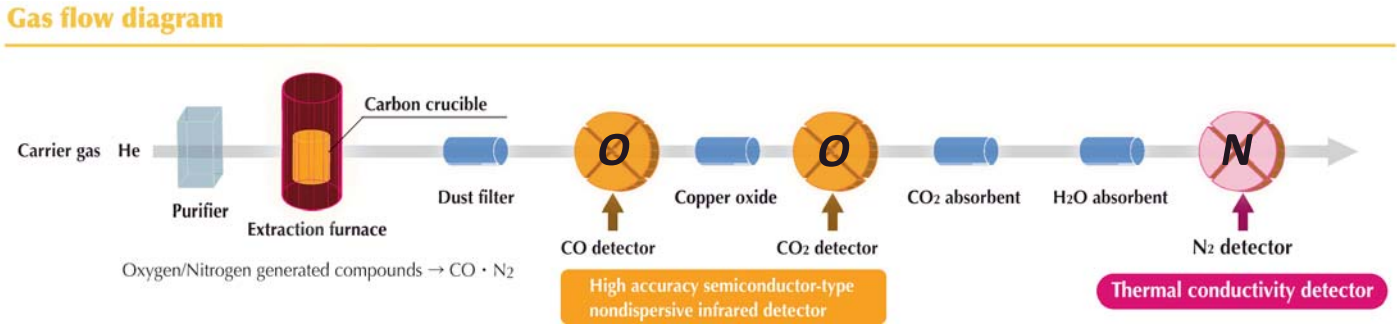

Figure 2: Gas flow diagram of the EMGA-820/EMGA-920

2.2 Super High Performance

Wide measurement range

Thanks to dual NDIR detectors measuring CO and CO_2 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.

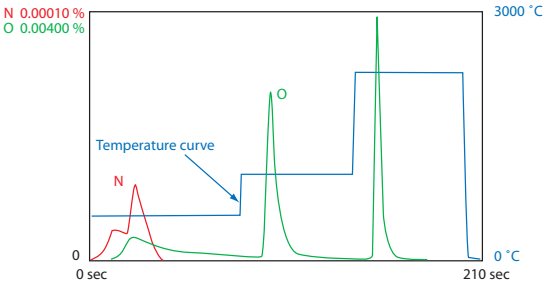

Figure 3: Oxide differentiation in iron powder using the programmable temperature curves feature

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.

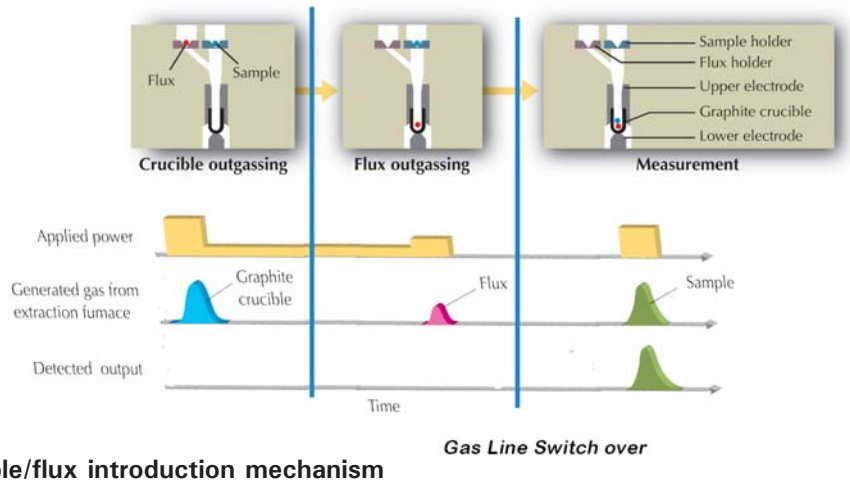

Figure 4: Dual sample/flux introduction mechanism

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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 preparations are done while the next sample is being prepared, the gain in terms of time is about 40 s. compared with conventional systems.

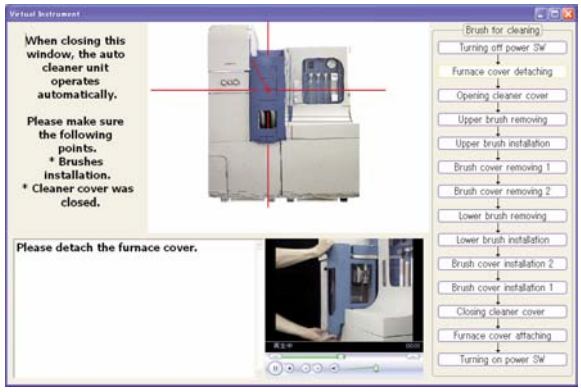

Figure 5: Software with maintenance counter

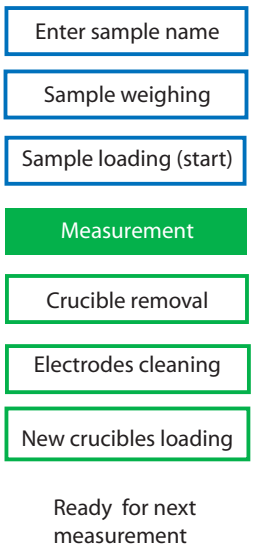

Figure 6: Sequence of measurement. (Steps in green are automatic)

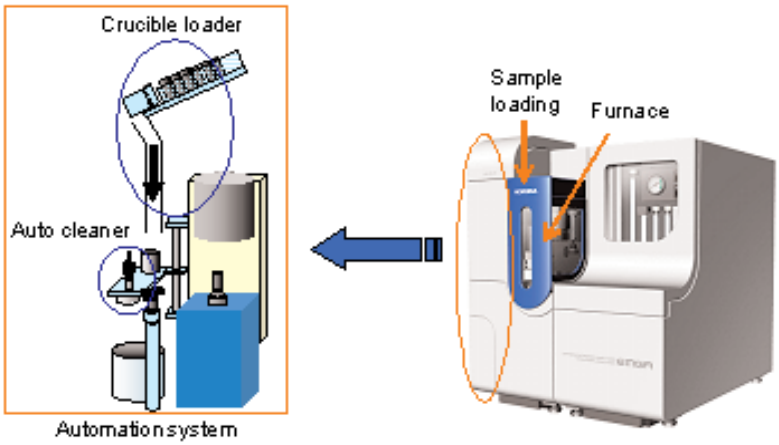

Figure 7: Details of the automation of the EMGA-920

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3 Sample Information

Sample: Boron nitride (BN)
Sample type: Powder
Sample weight: Approx., 0.02 g
Calibration sample: JCRM R003, 40 mg (Oxygen: 1.27mass %/ Nitrogen: 39.00mass %)
Flux: For sample and calibration sample: Sn pellet, 0.5 g, Ni capsule
Crucibles: Graphite crucibles (ECC. 3200043542)

4 Sample preparation

Put the calibration samples and samples into Ni capsules, press and fold them into small pieces. Calibrate the instrument per the Instruction Manual.

5 Measurement & Results

Put Sn pellet into the hopper for flux and approx. 0.02 g sample enclosed by Ni capsule into the hopper for sample, and then measure them.

Table1. Analysis data of samples

Sample name	Sample weight (g)	Oxygen (mass%)	Nitrogen (mass%)
BN	0.0248	0.651	54.50
	0.0250	0.655	53.83
	0.0252	0.662	53.84
	0.0251	0.657	54.15
	0.0249	0.656	53.87
Average		0.656	54.04
SD		0.004	0.29
RSD(%)		0.60	0.54

6 Conclusion

The EMGA-920 has been developed to be able to analyze low as well as high levels of nitrogen and oxygen maintaining very good accuracy over the full analytical range. It's particularly interesting when analyzing materials with one of the elements is a major constituent of the matrix and the other is an impurity. Boron Nitride is a good example of this.
The above example especially shows the performance of the EMGA-920 in determining oxygen and nitrogen with excellent precision even with a high level nitrogen.

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/scientfic
Japan: Horiba Ltd., 2 Miyanohigashi, Kisshoin, Minami-ku, Kyoto 601-8510
Tel: +81 (0) 3 38618231, Fax: +81 (0) 3 38618259, www.horiba.com/scientific
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 Shangai: +86 (0) 21 3222 1818 Spain: +34 (0) 91 724 16 57