APPLICATION NOTE

Oxygen and Nitrogen determination in Aluminium 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

Keywords: EMGA 920, Aluminium nitride, Oxygen, Nitrogen, Elemental Analyzer

1 Introduction

Aluminium nitride is principally used for its high thermal conductivity combined with a high electrical resistivity. So it's an interesting heat dissipator in electric and electronic applications especially as its thermal expansion coefficient is close to the silicon. Moreover, AlN is resistant to oxidation and abrasion.
Aluminium nitride is synthesized by reduction of alumina under N2 atmosphere. In its production, circuit board sintering processes are used. To make the material denser the always remaining surface oxygen of the AlN grains is used in the presence of a catalyst (rare earth or alkaline-earth oxides).
So, some remaining oxide is necessary for a good sintering. But, as oxygen content degrades the final product properties, the oxygen level in AlN powder should be adjusted as to be mostly eliminated at the end of sintering. That means that variation in oxygen % should stay within a range of 0.2% mass.
This application note will show the relevance of the EMGA-920 to measure high level of Nitrogen while maintaining the needed precision for Oxygen determination in AlN.

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

HORIBA Scientific

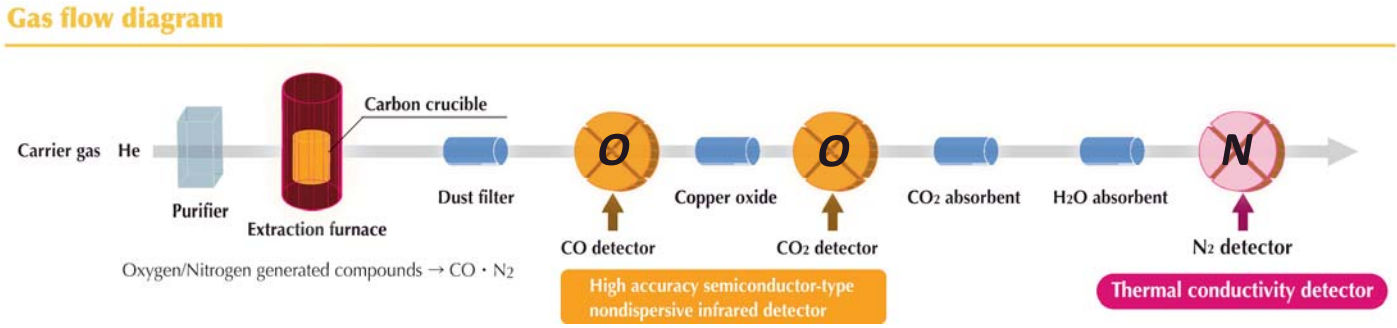

Figure 2: Gas flow diagram of the EMGA-820/EMGA-920

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.

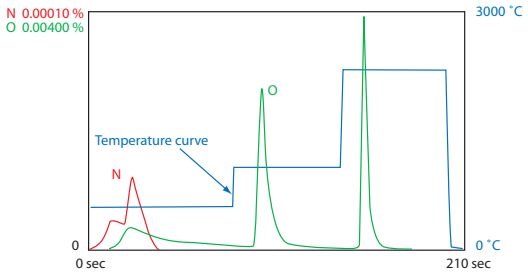

Figure 3: Oxide differentiation in iron powder

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.

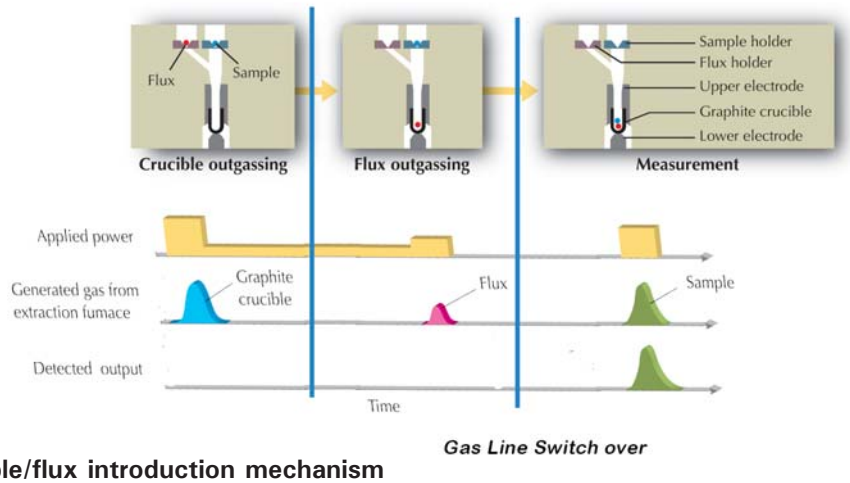

Figure 4: Dual sample/flux introduction mechanism

HORIBA Scientific

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.

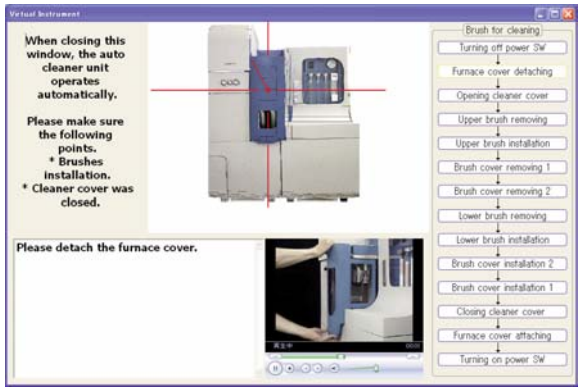

Figure 5: Software with maintenance counter

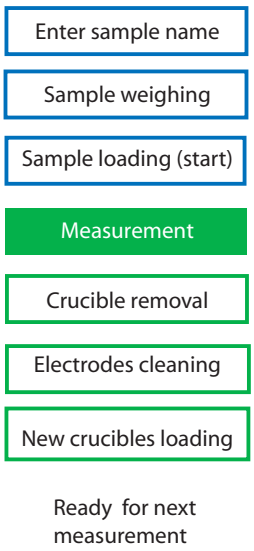

Figure 6: Sequence of measurement. (Steps in green are automatic)

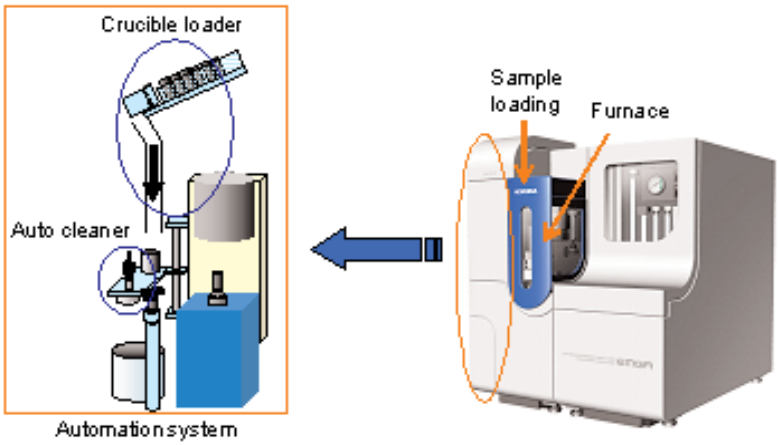

Figure 7: Details of the automation of the EMGA-920

HORIBA Scientific

3 Sample Information

Sample: AlN
Sample type: Powder
Sample weight: Approx. 0.03 g
Calibration sample: 0.04 g JCRM R003 (Silicon Nitride powder) O: 1.27 mass%, N: 39.00 mass%
Capsule: Ni capsule
Flux: Sn pellet, 0.5 g, and Ni pellet, 0.5 g, for sample, Sn pellet, 0.5 g, for calibration sample
Crucibles: Graphite crucibles (ECC No. 3200043542)

4 Sample preparation

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

5 Measurement & Results

Put Sn pellet and Ni pellet into the hopper for flux and 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%)
AlN	0.03005	1.30	31.45
	0.03004	1.29	31.47
	0.03013	1.30	31.50
	0.03000	1.30	31.58
	0.02983	1.30	31.53
Average		1.30	31.51
SD		0.004	0.051
RSD(%)		0.3	0.2

6 Conclusion

For production of sintered AlN products the impurity level of AlN powder needs to be strictly controlled. The oxygen level especially should stay within a narrow range to obtain at the same time good sintering and low oxygen content in the final product. This application note shows the ability of the EMGA-920 to mainytain very good precision for oxygen determinations even when a high level of nitrogen is analyzed.

info-sci.fr@horiba.com

www.horiba.com

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@jobinyvon.com, www.jobinyvon.com
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.jobinyvon.fr
Japan: Horiba Ltd., 2 Miyanohigashi, Kisshoin, Minami-ku, Kyoto 601-8510
Tel: +81 (0) 3 38618231, Fax: +81 (0) 3 38618259, www.jobinyvon.jp
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