Application Note

Instrument: ONH836

Determination of Oxygen, Nitrogen, and Hydrogen in Iron, Steel, Nickel-base, and Cobalt-base Alloys: Optimizing Cycle Time and Analytical Performance

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Introduction

The determination of the amount of oxygen, nitrogen, and hydrogen in iron, steel, nickel-, and cobalt-base alloys represents some of the most important quality metrics for these materials. Oxygen is used to create steel from pig iron by removing excess carbon. Oxygen content must be controlled to limit the amount of carbon monoxide that can be formed during solidification which may cause excessive porosity. Nitrogen is considered both an impurity as well as an important alloying agent. It can be present as a nitride or interstitially in its gaseous form. Increased nitrogen content is known to increase yield and tensile strength, thus decreasing ductility and formability. Excessive levels may evolve during solidification thus increasing porosity. High hydrogen content is the primary cause of embrittlement, blistering and flaking due to its high mobility through the lattice and provides no potential alloying benefits. The ONH836 utilizes a high-power electrode furnace to quickly and efficiently release the target gases from within the sample, which allows for a very rapid simultaneous determination of oxygen, nitrogen, and hydrogen.

Sampling and sample preparation is an important issue because traditional methods used to obtain samples for oxygen and nitrogen determination are different from those recommended for hydrogen, especially when sampling molten metal. The main difference in steel sampling procedures for oxygen/nitrogen and hydrogen is due to the mobility of hydrogen. Special precautions must be used when sampling for hydrogen. From molten steel and iron, a sample must be quickly quenched in cold water and chilled in a refrigerant such as liquefied nitrogen or a mixture of acetone and solid carbon dioxide in order to reduce losses of hydrogen from diffusion. Losses of oxygen and nitrogen from diffusion are not a problem. A sample that is taken for hydrogen and chilled in a refrigerant can also be used for oxygen and nitrogen determination. However, a sample that is typically taken for oxygen and/or nitrogen determination is not suitable for hydrogen determination due to hydrogen loss (diffusion). Surface contamination must be removed by filing or light grinding, using care not to overheat the sample.

Subsequently the prepared sample is washed in a suitable solvent such as acetone and dried with warm air. The prepared sample must be analyzed immediately after preparation. ASTM E 1806 and ISO 14284 are sampling/sample preparation documents specific for steel and iron and are an excellent source of information.

Accessories

776-247 Graphite Crucibles; 611-351-182 Lower Electrode Tip for 776-247 Crucibles without automation; 611-351-181 Lower Electrode Tip for 776-247 Crucibles with automation.

NOTE: The 611-351-181 Lower Electrode Tip is only required if the instrument is equipped with automation.

Reference Materials

LCRM®, LRM®, NIST, or other suitable reference materials.

Method Selection

Two methods are described in this application note; either method can be used to analyze iron, steel, nickel-, and cobalt-base alloys. The Precision Method is recommended for general use and will provide the best precision and accuracy throughout the typical O, N, and H concentrations found in this group of metals; approximate cycle time is 3.5 minutes. The Fast Track method can be used where speed of analysis is a critical component; for example, when molten metal is being sampled and results are required in the shortest possible time. This method will produce suitable results for most samples; approximate cycle time is 2.25 minutes. As noted above, sampling and sample preparation are key elements to accurate O, N, and H determination as well. It is up to the user to determine which method best meets their needs.

Method Parameters

General Parameters	**Precision Method**			**Fast Track Method**		
Sample Introduction	Automated Sample Drop			Automated Sample Drop		
Analysis Delay	25 s			0 s		
Auto Analyze on Mass Entry	No			No		
Outgas Before Mass Entry	No			No		
Wait for User to Load Sample	Yes			Yes		
Vacuum On Time	18 s			7 s		
Element Parameters	*Oxygen*	*Nitrogen*	*Hydrogen*	*Oxygen*	*Nitrogen*	*Hydrogen*
Integration Delay	5 s	15 s	10 s	5 s	15 s	10 s
Starting Baseline	2 s	2 s	2 s	2 s	2 s	2 s
Use Comparator	No	No	No	No	No	No
Integration Time	30 s	60 s	60 s	25 s	35 s	35s
Use Endline	Yes	Yes	Yes	Yes	Yes	Yes
Ending Baseline	2 s	2 s	2 s	2 s	2 s	2s
Range Select	Auto	—	—	Auto	—	—
Range Lower Limit	800	—	—	800	—	—
Range Upper Limit	900	—	—	900	—	—
Furnace Parameters						
Furnace Control Mode	Power			Power		
Outgas Furnace Settings						
Cycles	2			1		
Power Mode	Constant			Constant		
Power	5500* W			5500* W		
Time	20 s			10 s		
Cool Time	5 s			5 s		
Surface Oxide Removal						
Remove Surface Oxides	No			No		
Analyze Furnace Settings						
Step 1 Power Mode	Constant			Constant		
Power	4800* W			4800* W		
Approximate Cycle Time	3.5 Minutes			2.25 Minutes		

**May vary, depending on line voltage. Level can be adjusted to facilitate recovery and/or reduce crucible burn-through.*

Automation Parameters (if equipped)

General Parameters	
Auto Cleaner State	Enabled
Auto Cleaner Mode	During Analysis
Clean Time	8 s

Procedure

1. Prepare the instrument as outlined in the operator's instruction manual.
2. Determine the instrument blank.
   a. Log in a minimum of 3 Blank replicates.
   b. Press the Analyze button on the instrument screen. After a short delay, the loading head slide-block will open.
   c. Press the Analyze button on the instrument screen again, the loading head slide-block will close and the lower electrode will open.
   d. Clean the upper and lower electrode manually, or, if applicable, remove the crucible and press the analyze button to clean with the automatic cleaner.
   e. Firmly place a graphite crucible on the lower electrode tip.
   f. Press the Analyze button on the instrument screen, the lower electrode will close and the analysis sequence will start and end automatically.
   g. Repeat steps 2b through 2f a minimum of three times.
   h. Set the blank following the procedure outlined in the operator's instruction manual.
3. Instrument calibration/drift correction.
   a. Log in a minimum of 3 Standard replicates.
   b. Weigh a ~1.000 gram of a calibration/drift standard, enter the mass and standard identification into appropriate replicate fields.
   c. Press the Analyze button on the instrument screen. After a short delay, the loading head slide-block will open.
   d. Place the calibration/drift standard into the open port at the top of the loading head.
   e. Press the Analyze button on the instrument screen again, the loading head slide-block will close and the lower electrode will open.

f. Clean the upper and lower electrode manually, or, if applicable, remove the crucible and press the analyze button to clean with the automatic cleaner.
g. Firmly place a graphite crucible on the lower electrode tip.
h. Press the Analyze button on the instrument screen, the lower electrode will close and the analysis sequence will start and end automatically.
i. Repeat steps 3b through 3h a minimum of three times for each calibration/drift standard used.
j. Calibrate/drift following the procedure outlined in the operator's instruction manual.

4. Analyze Samples.
   a. Log in Sample with the appropriate number of reps.
   b. Weigh ~1.000 gram of sample, enter mass and sample identification into appropriate rep fields.
   c. Repeat steps 3c through 3h for sample analysis.

Typical Results

		Precision Method				**Fast Track Method**		
Sample	*Mass g*	*O%*	*N%*	*H ppm*		*O%*	*N%*	*H ppm*
LECO	1.0	0.0031	0.0372	5.3		0.0033	0.0362	5.5
502-416		0.0030	0.0362	5.5		0.0029	0.0369	5.7
0.0028% O		0.0028	0.0366	5.3		0.0028	0.0371	5.3
0.0365% N		0.0029	0.0369	5.3		0.0028	0.0370	5.8
5.4 ppm H		0.0027	0.0364	5.2		0.0027	0.0371	5.3
		0.0029	0.0361	5.6		0.0025	0.0359	4.9
		0.0028	0.0364	5.4		0.0026	0.0351	4.9
		0.0029	0.0366	5.5		0.0029	0.0364	5.6
		0.0028	0.0364	5.3		0.0030	0.0365	5.6
		0.0029	0.0361	5.4		0.0030	0.0367	5.6
	***X* =**	***0.0029***	***0.0365***	***5.4***	***X* =**	***0.0028***	***0.0365***	***5.4***
	***s* =**	***0.0001***	***0.0003***	***0.1***	***s* =**	***0.0003***	***0.0006***	***0.3***
LECO	1.0	0.0362	0.0024	1.1		0.0370	0.0021	0.7
501-646		0.0363	0.0024	0.9		0.0370	0.0024	0.6
0.0363% O		0.0361	0.0024	1.2		0.0360	0.0021	1.0
0.0022% N		0.0364	0.0024	0.8		0.0362	0.0019	0.9
		0.0364	0.0023	1.1		0.0362	0.0021	1.0
		0.0363	0.0024	1.3		0.0362	0.0024	0.6
		0.0361	0.0023	0.9		0.0364	0.0023	1.1
		0.0362	0.0022	0.7		0.0356	0.0018	0.6
		0.0360	0.0023	0.7		0.0365	0.0023	1.1
		0.0369	0.0023	0.9		0.0358	0.0020	0.7
	***X* =**	***0.0363***	***0.0023***	***1.0***	***X* =**	***0.0363***	***0.0021***	***0.8***
	***s* =**	***0.0002***	***0.0001***	***0.2***	***s* =**	***0.0005***	***0.0002***	***0.2***

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