Oxygen and Hydrogen Determination in Titanium Hydride

LECO Corporation; Saint Joseph, Michigan USA

Instrument: ONH836 Series

Introduction

Titanium hydride is used for multiple applications, in place of titanium powder, as the production process is less costly and produces products with comparable mechanical properties. The fact that it is composed of theoretically 4.04% hydrogen by weight increases its usefulness for even more applications. Some of its uses include a raw material for the sintering of complex titanium parts, a raw material for titanium carbide and nitride production, a hydrogen source for metal foaming, an additive in powder metallurgy, a getter for impurities, and a roadmap for hydrogen storage research such as fuel cells and battery applications.

Due to the nature of its applications, the amount of hydrogen and oxygen present provides the user with a measure of future performance, making oxygen and hydrogen determination a principle quality control metric. The ONH836 series of inert gas fusion analyzers provides the hardware and performance necessary to measure the amount of both oxygen and hydrogen in titanium hydride simultaneously during a single analysis. This application note outlines the method parameters for a low-temperature determination of hydrogen prior to a high-temperature determination of oxygen. Nitrogen determination is not applicable to titanium hydride by inert gas fusion.

Sample Preparation

Samples should be uniform powder or chip. Samples should be weighed into an open nickel capsule.

NOTE: Use the same part number and lot number of capsules that will be used for the analysis of samples. Leave capsule open during the weighing and introduction of the sample into the nickel capsule. The entire operation must be accomplished using clean tweezers only. For best results, a five-place balance is recommended.

Accessories

782-720 Graphite Crucibles; 502-822 Nickel Capsules; 782-721 Lower Electrode Tip for 782-720 crucibles without automation; 618-376 Lower Electrode Tip for 782-720 crucibles with automation

NOTE: The 618-376 Lower Electrode Tip is only required if the instrument is equipped with automation.

Calibration Samples

Reagent Grade TiH_2, 501-147 Copper Pin, 501-148 Copper Pin, and 501-149 Copper Pin

Analysis Procedure

1. Prepare the instrument as outlined in the operator's instruction manual.
2. Determine the instrument blank.
   a. Login a minimum of three Blank replicates.
   b. Press the Analyze button on the instrument screen. After a short delay, the loading head slide-block will open.
   c. Place an empty 502-822 Nickel Capsule into the loading head or appropriate autoloader position if applicable.
   d. Press the Analyze button on the instrument screen again, the loading head slide-block will close and the lower electrode will open.
   e. Clean the upper and lower electrode either manually or, if applicable, remove the crucible and press the Analyze button again to clean with the automatic cleaner.
   f. Firmly place the graphite crucible on the lower electrode tip.
   g. Press the Analyze button on the instrument screen. The lower electrode will close and the analysis sequence will start and end automatically.
   h. Repeat steps 2b through 2g a minimum of three times.
   i. Set the blank following the procedure outlined in the operator's instruction manual.
3. Instrument calibration/drift correction.
   a. Login a minimum of three Standard replicates.
   b. For hydrogen calibration, weigh ~0.10 g of Reagent Grade TiH_2 calibration/drift sample into a 502-822 Nickel Capsule; enter the mass and sample identification into appropriate replicate fields. For oxygen calibration, weigh ~1.0 g of a suitable oxygen calibration/drift sample into a 502-822 Nickel Capsule; enter the mass and sample identification into the appropriate replicate fields.
   c. Place an empty 502-822 Nickel Capsule into the loading head or appropriate autoloader position if applicable.
   d. Press the Analyze button on the instrument screen. After a short delay, the loading head slide-block will open.
   e. Place the open nickel capsule containing the calibration/drift sample into the open port at the top of the loading head.
   f. Press the Analyze button on the instrument screen again, the loading head slide-block will close and the lower electrode will open.

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Analysis Procedure (continued)

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

4. Analyze Samples.
   a. Login the appropriate number of Sample replicates.
   b. Weigh ~0.10 g of TiH_2 sample into a 502-822 Nickel Capsule; enter mass and identification into appropriate replicate fields.
   c. Repeat steps 3c through 3h for sample analysis.

Method Parameters**

General Parameters		
Sample Introduction	Automated Sample Drop	
Analysis Delay	30 s	
Auto Analyze on Mass Entry	No	
Outgas Before Mass Entry	No	
Wait for User to Load Sample	Yes	
Vacuum On Time	18 s	
Element Parameters	**Oxygen**	**Hydrogen**
Integration Delay	5 s	10 s
Starting Baseline	2 s	2 s
Use Comparator	No	No
Integration Time	210 s	210 s
Use Endline	Yes	Yes
Ending Baseline	2 s	2 s
Range Select	High	—
Furnace Parameters		
Furnace Control Mode	Power	
Outgas Furnace Settings		
Cycles	3	
Power Mode	Constant	
Power	5300* W	
Time	20 s	
Cool Time	5 s	
Analyze Furnace Settings		
Step 1 Power Mode	Constant	
Power	1200* W	
Time	100 s	
Step 2 Power Mode	Ramped	
Low Power	1200* W	
High Power	4800* W	
Time	20 s	
Step 3 Power Mode	Constant	
Power	4800* W	
Time	90 s	

**May vary, depending on line voltage. Level can be adjusted to facilitate recovery and/or reduce crucible burn-through.*
***The method parameters listed in the table above are optimized for the use of helium as a carrier gas. The use of argon as a carrier gas will require lengthened integration times, as well as reduced outgas and analysis power levels. Please contact the LECO Technical Services Laboratory for additional details.*

Automation Parameters (if equipped)

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

Typical Results

Sample	*Mass (g)*	*% Oxygen*	*% Hydrogen*
Reagent Grade TiH_2	0.10053	0.53	4.03
4.02% Hydrogen	0.10047	0.54	4.02
	0.10078	0.54	4.01
	0.10101	0.56	4.01
	0.10041	0.54	4.03
	X=	***0.54***	***4.02***
	s=	***0.01***	***0.01***
LECO 501-149	1.0029	0.0539	0.00008
0.0541% Oxygen	1.0058	0.0542	0.00011
Copper Pin	1.0064	0.0541	0.00013
	1.0012	0.0540	0.00009
	1.0034	0.0543	0.00013
	X=	***0.0541***	***0.00011***
	s=	***0.0002***	***0.00002***
TiH_2 Sample	0.10151	0.67	3.99
	0.10064	0.67	3.98
	0.10171	0.68	3.98
	0.10090	0.66	4.00
	0.10125	0.68	3.99
	X=	***0.67***	***3.99***
	s=	***0.01***	***0.01***

Hydrogen Detector calibrated using Reagent Grade Titanium Hydride linear forced through origin.
Oxygen Detector calibrated using LECO 501-149 Copper Pin linear forced through origin.

Inorganic Application Note

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