Oxygen and Hydrogen Determination in Titanium Hydride

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Instrument: TCH600

Introduction

Titanium hydride is used in place of titanium powder as the production process is less costly and produces products with comparable mechanical properties. Being composed of 4.04% hydrogen by weight increases its usefulness for additional applications. Some of its uses include as 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 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 purity, making oxygen and hydrogen determination a principle quality control metric. The TCH600 series of inert gas fusion analyzers provides the hardware and performance necessary to measure the amount of both oxygen and hydrogen in titanium hydride 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.

Sampling and Sample Preparation

If powder or chip samples are to be analyzed, they cannot be placed directly in the loading head. Instead, samples must be weighed into a nickel capsule, analyzed in the Semi-Auto Analysis mode. Note: Use the same part number and lot number of capsules that will be used for the analysis of samples. Leave the capsule open during the weighing and analysis. The capsules should always be handled with clean tweezers.

Accessories

782-720 Graphite Crucibles, 502-822 Nickel Capsule, 501-073 Graphite Powder

Calibration Samples

For hydrogen calibration use Reagent Grade TiH_2. For Oxygen Calibration use LECO 502-201 or other suitable reference materials.

Method Parameters

Analysis Parameters

Outgas Cycles	3
Analysis Delay	25 seconds
Analysis Delay Comparator	1.000
Analysis Type	Auto Analysis
Pre-Analyze Purge Time	60 seconds
Auto Analyze on Mass Entry	Disabled
Pre-Analyze Crucible Outgas	Disabled

Note: Disable low oxygen must be checked.

Element Parameters

	Oxygen	*Hydrogen*
Minimum Analysis Time	120 seconds	200 seconds
Significant Digits	5	5
Conversion Factor	1.000000	1.000000
Integration Delay	5 seconds	10 seconds
Comparator Level	1.000000%	1.000000%
Stop if below (%)	0.000000	0.000000

Furnace Parameters

Furnace Control Mode	*Power*
Purge Time	15 seconds
Outgas Time	20 seconds
Outgas Cool Time	5 seconds
Outgas Low Power	6000 Watts*
Outgas High Power	6000 Watts*
Outgas Ramp Rate	0 watts/sec
Analyze Low Power	1200 Watts*
Analyze High Power	5200 Watts*
Analyze Ramp Rate	0 watts/sec
Sample Prep Time	0 seconds
Sample Prep Power	0 watts
Furnace On Time	30 seconds

Temperature Sustain

Step	*Start Power*	*End Power*	*Time*
1	1200 watts	1200 watts	45 seconds
2	1200 watts	5200 watts	20 seconds
3	5200 watts	5200 watts	135 seconds

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

Procedure

1. Determine Blank.
   a. Enter 1.0000 g mass into Sample Login (F3) using Blank as sample name.
   b. Press Loader Switch on front of furnace, after a short delay the loading head slide block will open.
   c. Place an open 502-822 Nickel Capsule into open port at top of loading head.
   d. Press Loader Switch again, the loading head slide block will close, and the furnace lower electrode will open. Clean the upper and lower electrode manually.

Inorganic Application Note

e. Add ~0.05 g 501-073 Graphite Powder to a 782-720 Graphite Crucible.
f. Firmly place the crucible on the lower electrode tip.
g. Press the Loader Switch. The lower electrode will close and the analysis sequence will start and end automatically
h. Repeat steps 1a through 1g a minimum of three times.
i. Set the blank following the procedure outlined in the operator's instruction manual.

2. Calibrate/Drift Correct.
   a. Weigh calibration sample (~0.08 g for TiH_2) into a 502-822 Nickel Capsule and enter the mass and sample identification into Sample Login (F3).
   b. Press the Loader Switch on the front of furnace. After a short delay, the loading head slide block will open.
   c. Place the open nickel capsule containing the calibration sample into the open port at top of loading head.
   d. Press the Loader Switch. The lower electrode will open. Clean the upper and lower electrode manually.
   e. Add ~0.05 g 501-073 Graphite Powder to a 782-720 Graphite Crucible.
   f. Firmly place the crucible on lower electrode tip.
   g. Press the Loader Switch. The lower electrode will close and the analysis sequence will start and end automatically.
   h. Repeat steps 2a through 2g a minimum of three times for each calibration/drift sample used.
   i. Calibrate or Drift Correct the instrument following the procedure outlined in the operator's instruction manual.
3. Analyze Samples.
   a. Weigh ~0.080 g TiH_2 sample into a 502-822 Nickel Capsule and enter mass and sample identification into Sample Login (F3).
   b. Proceed as directed in steps 2b through 2g.

Typical Results

Sample	*Mass (g)*	*Oxygen (%)*	*Hydrogen (%)*
Reagent Grade TiH_2	0.0823	0.520	4.01
4.02% Hydrogen	0.0808	0.520	4.03
	0.0784	0.519	4.02
	0.0837	0.520	4.02
	0.0867	0.525	4.03
	$\bar{\chi}$=	***0.521***	***4.02***
	***s*=**	***0.002***	***0.01***

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