Hydrogen in Reactive and Refractory Metals

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Instrument: H836*

**ONH836/OH836 also applicable*

Summary

Titanium is a metal that can be combined with elements such as aluminum, vanadium, molybdenum and tin to produce high-strength, low-density, and corrosion-resistant alloys. Titanium alloys are used by the military, medical devices, sporting goods, and aerospace industries because of these properties, and due to the strict demands of these industries, effort needs to be taken to assure that the material meets the highest quality standards.

Oxygen and nitrogen are alloying elements in titanium, and are also classified as alpha stabilizing elements as they promote alpha phase alloys. Interstitial oxygen and nitrogen levels can be used to regulate the tensile strength of the material, but due to its high solubility can cause unwanted surface embrittlement. This phenomenon can be leveraged, however, under controlled processing to create surface films that increase surface hardness and wear properties.

One of the more critical chemical specifications of titanium alloys is the hydrogen content. Too high a hydrogen content can cause hydrides to precipitate, which can lead to embrittlement and subsequent cracking when the alloy is stressed. Hydrogen pickup typically occurs during downstream processing steps such as heat treating, pickling, and cleaning.

LECO Corporation offers several configurations of fusion determination for this analysis. The following application note outlines the process from sample generation to data.

Sample Preparation

Sampling and sample preparation of refractory metals such as titanium and zirconium is somewhat different from that of steel. Unlike steel samples, hydrogen is not as mobile in this group of materials; therefore, storage in liquid nitrogen or dry ice is not required. However, it is important to keep the sample cool when cutting or sectioning. ASTM E 1447 "Determination of Hydrogen in Titanium and Titanium Alloys by the Inert Gas Fusion Thermal Conductivity/Infrared Detection Method" permits surface preparation by abrading (if necessary to remove contamination).

Accessories

782-720 Graphite Crucibles; 782-721 Lower Electrode Tip for 782-720 Crucibles without automation; 618-376 Lower Electrode Tip for 782-720 Crucibles with automation; 761-739 Tin Flux Pellets; 501-059 or 502-040 Tin Capsule (for powder or granular samples)

Calibration

LECO 502-024, 502-135, 762-741; NIST or other suitable reference materials.

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 reps.
   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 either manually, or if applicable, remove the crucible and press the analyze button to clean with an automatic cleaner.
   e. Place two 761-739 Tin Flux Pellets into a 782-720 Graphite Crucible.
   f. Firmly place the 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 reps.
   b. Weigh approximately 0.15 to 0.35 grams of a calibration/drift standard, enter the mass and standard identification into appropriate rep fields.

   Note: LECO Reference Materials do not require preparation. See preparation statement on the reference material certificate.

   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. Remove the crucible and clean the upper and lower electrode either manually, or if applicable, press the analyze button to clean with an automatic cleaner.

Inorganic Application Note

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	*Hydrogen*
Integration Delay	5 s
Starting Baseline	2 s
Use Comparator	No
Integration Time	35 s
Use Endline	Yes
Ending Baseline	2 s
Furnace Control Mode	Current
Outgas Furnace Settings	
Cycles	3
Current Mode	Constant
Current	850* A
Time	20 s
Cool Time	5 s
Surface Oxide Removal	
Remove Surface Oxide	No
Analyze Furnace Settings	
Step 1 Current Mode	Constant
Power	765* A
Approximate Cycle Time	3.5 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 (continued)

g. Place two 761-739 Tin Flux Pellets into a 782-720 Graphite Crucible.
h. Firmly place the 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 standard used.
k. Calibrate/drift following the procedure outlined in the operator's instruction manual.

4. Analyze Samples.
   a. Login Sample with the appropriate number of reps.
   b. Weigh approximately 0.15 to 0.35 grams of appropriately prepared sample, enter mass and sample identification into appropriate rep fields.
   d. Repeat steps 3c through 3i for sample analysis.

Typical Results

Sample	*Mass*	*H ppm*
NIST 352c	0.25	49.0
49.0 ppm H		49.6
±0.9 ppm H		48.4
		49.1
	***X* =**	***49.0***
	***s* =**	***0.49***
Unknown	0.25	7.5
Unalloyed		6.3
Titanium		6.5
		7.5
	***X* =**	***7.0***
	***s* =**	***0.64***

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