Carbon in Refractory and Reactive Metals

LECO Corporation; Saint Joseph, Michigan USA

Instrument: C844

Introduction

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 of quality standards. Carbon, like oxygen and aluminum, is an alloying element in titanium, and is classified as an alpha stabilizing element as it promotes alpha-phase alloys. Alpha-phase titanium alloys are less ductile and have less strength than the beta-phase alloys, but have higher corrosion resistance and deformability. Carbon is considered to be an impurity, commonly introduced during the manufacturing process, of commercially pure (CP) titanium grades.

Sample Preparation

Sample should be clean and free of contamination. Contamination on the sample can cause significant errors in the analytical data; therefore, care must be taken to ensure a clean representative sample is analyzed.

Reference Method ASTM E1941

Accessories

528-018 or 528-018HP Crucible (preheated*); 502-492 Copper; 502-231 Iron Chip; 773-579 Metal Scoop; 761-929 Tongs.

**Ceramic crucibles are baked in a muffle or tube furnace (LECO TF-10) at a minimum of 1250°C for a minimum of 15 minutes, or at 1000°C for 40 minutes. The crucibles are removed from the furnace, allowed to cool for 1 to 2 minutes, and then transferred to a desiccator for storage. If the crucibles are not used within four hours, they should be re-baked.*

Calibration

501-995 LECO Titanium Reference Material; 502-402 LECO Steel Reference Material; NIST Reference materials may be used as well. Due to limited availability of refractory/reactive reference materials, steel reference materials can be used to calibrate.

Method Parameters

Analysis Parameters	
Purge Time	15 s
Analysis Delay	20 s
Sample Cool Time	20 s
Furnace Mode	Constant
Furnace Power	100%

Element Parameters	Carbon
Integration Delay	0 s
Starting Baseline	2 s
Use Comparator	No
Integration Time	50 s
Use Endline	Yes
Ending Baseline	2 s
Range Select	Auto
Range Lower Limit	800
Range Upper Limit	950

Procedure

1. Prepare the instrument and crucibles as outlined in the operator's instruction manual.
2. Blank Analysis.
   a. Login a minimum of three Blank reps.
   b. Add ~1.5 g of 502-092 Copper and ~1 g of 502-231 Iron Chip accelerator to a preheated 528-018 or 528-018HP Crucible.
   c. Place the crucible on the furnace pedestal (or appropriate autoloader position if applicable), and initiate analysis.
   d. Repeat steps 2b through 2c a minimum of three times.
   e. Set the blank by following the procedure outlined in the operator's instruction manual.
3. Calibrate/Drift Correct
   a. Login a minimum of three Standard reps.
   b. Weigh ~0.5 g of 501-995 LECO Titanium Reference Material or other suitable calibration/drift material into the crucible and enter the mass and standard identification of the reference material.
   c. Add ~1.5 g of Copper and ~1 g of Iron Chip accelerator on top of the reference material.
   d. Place the crucible on the furnace pedestal (or appropriate autoloader position if applicable), and initiate analysis.
   e. Repeat steps 3b through 3d a minimum of three times for each calibration/drift reference material intended for calibration/drift.
   f. Calibrate/drift correct by following the procedure outlined in the operator's instruction manual.

4. Sample Analysis
   a. Login a Sample with appropriate number of reps.
   b. Weigh ~0.5 g of sample into the crucible and enter the mass and sample identification of the sample.
   c. Add ~1.5 g of Copper and ~1 g of Iron Chip accelerator on top of the sample.
   d. Place the crucible on the furnace pedestal (or appropriate autoloader position if applicable), and initiate analysis.

Typical Results

Sample	*Mass (g)*	*% Carbon*
NIST 173a	0.5005	0.0245
Titanium Alloy	0.5052	0.0252
(6Al-4V)	0.4999	0.0248
0.025% C	0.5026	0.0248
	0.5028	0.0257
	0.5034	0.0243
	0.4995	0.0247
	0.4995	0.0254
	0.4977	0.0251
	0.5041	0.0255
	$\bar{\chi}$=	***0.0250***
	s=	***0.0005***

Sample	*Mass (g)*	*% Carbon*
Ta Powder	0.5007	0.0020
	0.5015	0.0020
	0.5012	0.0023
	0.5015	0.0019
	0.5004	0.0019
	0.5021	0.0019
	0.5010	0.0019
	0.5014	0.0020
	0.4999	0.0020
	0.5022	0.0021
	$\bar{\chi}$	***0.0020***
	s=	***0.0001***

Sample	*Mass (g)*	*% Carbon*
Zircaloy Chip	0.4995	0.0159
	0.5016	0.0157
	0.5010	0.0163
	0.5023	0.0157
	0.5005	0.0159
	0.5023	0.0157
	0.4977	0.0157
	0.4997	0.0158
	0.4984	0.0161
	0.5000	0.0159
	$\bar{\chi}$	***0.0159***
	s=	***0.0002***

Sample	*Mass (g)*	*% Carbon*
Ti Wire	0.4674	0.0652
	0.4922	0.0652
	0.4851	0.0660
	0.5381	0.0658
	0.4686	0.0659
	0.5190	0.0652
	0.4519	0.0653
	0.4746	0.0653
	0.5101	0.0651
	0.4754	0.0652
	$\bar{\chi}$	***0.0654***
	s=	***0.0003***

Based on a single standard calibration using NIST SRM 173a

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