Oxygen in Aluminum and Aluminum Alloys

LECO Corporation; Saint Joseph, Michigan USA

Instrument: TC600-Series

Introduction

The determination of oxygen in aluminum is an extremely important quality control measure for both wire and integrated circuit (IC) manufacturers, but for different reasons. Oxygen in aluminum is always in the form of aluminum oxide since it is insoluble in aluminum and subsequently acts as a dielectric particle (nonconductor of electricity). The conductivity of the wire is relative to the oxygen level. Target performance is relative to the oxygen level in an aluminum sputtering target. Since aluminum oxide is a source of contamination during the sputtering process, the use of a low oxygen target will improve device yield.

Sample Preparation

This method is written for solid samples; Aluminum powder, chip or granular samples can be analyzed following the procedures used for reactive/refractory metals (Titanium). Reference LECO Application Note 203-821-185 for more information).

A clean representative sample is required in order to obtain optimum results. Surface contamination must be removed by machining on a lathe or abraded with a clean file, followed by rinsing in acetone and drying with warm air. The prepared sample must be analyzed immediately after preparation.

Accessories

776-247 Graphite Crucible, 611-351-182 Electrode Tip, 501-073 Graphite Powder, 761-739 Tin Pellet, 501-263 Copper Chip.

Calibration Standard

LECO 501-643, 501-644, or other suitable reference materials.

Method Parameters

Analysis Parameters

Outgas Cycles	3
Analysis Delay	20 seconds
Analysis Delay Comparator	1
Analysis Type	Semi-Auto Analysis
Auto Analyze on Mass Entry	Disabled
Preliminary Crucible Outgas	Disabled

Element Parameters	**Oxygen**
Minimum Analysis Time	35 seconds
Significant Digits	5
Conversion Factor	1.00
Integration Delay	5 seconds
Comparator Level	1.00
Stop if below (%)	0.00000

Furnace Parameters

Furnace Control Mode	Power
Purge Time	15 seconds
Outgas Time	15 seconds
Outgas Cool Time	5 seconds
Outgas Low Power	6000 watts*
Outgas High Power	6000 watts*
Outgas Ramp Rate	0 watts/second
Analyze Low Power	5000 watts*
Analyze High Power	5000 watts *
Analyze Ramp Rate	0 watts/second
Sample Prep Time	0 watts/second
Sample Prep Power	0 watts/second
Temperature Sustain	None

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

Procedure

1. Prepare instrument for operation as outlined in the operator's instruction manual.
2. Determine Blank.
   a. Enter 1.0000 g mass into Sample Login (F3) using Blank as the sample name.
   b. Press Loader Switch on front of furnace, after a short delay the loading head slide block will open.
   c. Press Loader Switch again, the loading head slide block will close and the lower electrode will open.
   d. Add one 761-739 Tin Pellet, ~0.5 g 501-263 Copper Chip, and ~0.05 g 501-073 Graphite Powder into a 776-247 Graphite Crucible.
   e. Place crucible on electrode pedestal.
   f. Press Loader Switch; the lower electrode will close and the analysis sequence will start and end automatically.
   g. Repeat steps 2a through 2f a minimum of three times.
   h. Set the blank following the procedure outlined in the operator's instruction manual.

Inorganic Application Note

3. Calibrate/Drift Correct.
   a. Weigh ~1.0 g of a steel calibration sample; enter mass and sample identification into Sample Login (F3).
   Note: Oxygen in aluminum reference materials are very rare; typically steel reference/ calibration samples are used for this application. If oxygen in aluminum reference/calibration sample is used, 0.1 to 0.3 g sample mass is recommended.
   b. Press Loader Switch on front of furnace, the loading head slide block will open.
   c. Place sample into open port at top of loading head.
   d. Press Loader Switch again, the loading head slide block will close and the lower electrode will open.
   e. Add one 761-739 Tin Pellet, ~0.5 g 501-263 Copper Chip, and ~0.05 g 501-073 Graphite Powder into a 776-247 Graphite Crucible.
   f. Place crucible on the electrode pedestal.
   g. Press Loader Switch; the lower electrode will close and the analysis sequence will start and end automatically.
   h. Repeat steps 3a through 3g 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.
4. Analyze Samples.
   a. Weigh a freshly prepared aluminum sample of ~0.1-0.3 g; enter mass and sample identification into Sample Login (F3).
   b. Proceed as directed in steps 3b through 3g.

Typical Results

Sample	Mass g	O ppm
Aluminum Rod	0.2218	8
	0.2188	10
	0.1923	8
	0.1989	9
	0.1764	10
	***X* =**	***9***
	***s* =**	***1.0***
Aluminum Disc	0.2287	20
	0.2390	19
	0.2369	15
	0.1803	17
	0.2285	18
	***X* =**	***18***
	***s* =**	***1.9***

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