Oxygen Determination in Aluminum

LECO Corporation; Saint Joseph, Michigan USA

Instrument: ONH836 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

Typical samples are solid metal samples. Samples should be abraded prior to analysis to remove surface oxides. Samples should be handled with clean tweezers only. Note: Due to the formation of surface oxides, the use of an autoloader is not recommended. Samples should be analyzed immediately after preparation. This method is for solid samples; Aluminum powder, chip or granular samples can be analyzed following the method used for reactive/refractory metals (Titanium) chips and powder procedure. Reference Application Note 203-821-427 for more information.

Accessories

776-247 Graphite Crucibles; 761-739 Tin Flux Pellets; 501-263 Copper Chip; 611-351-182 Lower Electrode Tip for 776-247 Crucibles

Calibration Samples

There are several suitable steel or copper reference materials available from LECO. Likewise, NIST, JK, JSS, and BCS are certified bodies that have a variety of certified reference materials (SRM/CRM) available as well. 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.

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. 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 again to clean with the automatic cleaner.
   e. Add one 761-739 Tin Flux pellet and approximately 0.5 g of 501-263 Copper Chip to a 776-247 crucible.
   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. Weigh ~1.0 g of the calibration/drift sample; enter the mass and sample identification into appropriate replicate fields.
   c. Press the Analyze button on the instrument screen. After a short delay, the loading head slide-block will open.
   d. Place the sample 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. 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.
   g. Add one 761-739 Tin Flux pellet and approximately 0.5 g of 501-263 Copper Chip to a 776-247 crucible.
   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/drift following the procedure outlined in the operator's instruction manual.
4. Analyze Samples.
   a. Login the appropriate number of Sample replicates.
   b. Weigh ~0.25 g of Aluminum sample; enter mass and identification into appropriate replicate fields.
   c. Repeat steps 3c through 3i for sample analysis.

Inorganic Application Note

Delivering the Right Results

Method Parameters

General Parameters	
Sample Introduction	Automated Sample Drop
Analysis Delay	25 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**
Integration Delay	5 s
Starting Baseline	2 s
Use Comparator	No
Integration Time	30 s
Use Endline	Yes
Ending Baseline	2 s
Range Select	Auto
Range Lower Limit	800
Range Upper Limit	950
Furnace Parameters	
Furnace Control Mode	Power
Outgas Furnace Settings	
Cycles	2
Power Mode	Constant
Power	5800* W
Time	20 s
Cool Time	5 s
Analyze Furnace Settings	
Step 1 Power Mode	Constant
Power	5000* W

**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

Typical Results

Sample	*Mass (g)*	*% Oxygen*
Aluminum Rod	0.2896	0.00018
	0.2780	0.00015
	0.3010	0.00017
	0.3030	0.00016
	0.2764	0.00012
	X=	***0.00016***
	s=	***0.00003***
AlSi Alloy	0.2185	0.00060
	0.2855	0.00062
	0.2721	0.00055
	0.2477	0.00062
	0.2521	0.00051
	X=	***0.00058***
	s=	***0.00005***

Note: Results based on linear force through origin calibration using LECO 501-644 Steel Pin, verified with 501-653 Copper Pin.

Inorganic Application Note

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