Oxygen in Copper and Copper Alloys

LECO Corporation; Saint Joseph, Michigan USA

Instrument: TC600

RO600/TCH600/ROH600 also applicable

Introduction

Copper is the most important material in the electrical conductor wire industry due to its high benefit-cost ratio. Copper provides admirable surface quality, mechanical properties, and high conductivity at a cost much less than silver or gold. Aluminum is the only metal that is routinely substituted for copper, but only in applications where weight is a major factor. One of the most important factors when grading copper is the level of residual impurities, primarily the oxygen level. Oxygen levels in electrical conductor wires typically range from 5-10 ppm (OF – oxygen free, OFE – oxygen-free electronic grades) to around 650 ppm (ETP – electrolytic tough pitch grade). Oxygen levels of up to 200 ppm can actually improve conductivity as it acts as a scavenger element that forms metal oxides with metal contaminants. Too much oxygen has been associated with an increase in hydrogen embrittlement problems.

Oxygen determination by the inert gas fusion infrared detection method is the most widely used and reliable method for broad range and high precision oxygen determination in copper and copper alloys. 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

This method is written for solid samples; a clean representative sample is required in order to obtain optimum results. Surface contamination must be removed by etching the sample according to the procedure outlined in ASTM E2575 section 10. The following is the procedure for etching.

a. Cut sample to an appropriate size (0.5-2.0 g).
b. Etch sample with concentrated HCl at 20°C for 3 minutes.
c. Etch sample in a mixture of equal parts concentrated HNO_3, CH_3COOH and concentrated H_3PO_4 at 70°C for 1 minute.
d. Rinse sample with three (3) successive distilled water rinses.
e. Rinse sample with three (3) successive methanol rinses.
f. Dry in a stream of warm air.

Alternately, surface contamination can be removed by abrading the sample using a clean, flat mill file, followed by rinsing in methanol and drying with warm air. The prepared sample must be analyzed immediately after preparation.

Accessories

776-247 Graphite Crucibles; 501-073 Graphite Powder; 611-351-182 Lower Electrode Tip for 776-247 Crucibles without automation; 611-351-181 Lower Electrode Tip for 776-247 Crucibles with automation.

Note: The 611-351-181 Lower Electrode Tip is only required if the instrument is equipped with automation.

Calibration Samples

LECO 501-147, 501-148, 501-149, 501-990 One-Gram Nickel Plated Copper Pins with O content determined; NIST, BCR or other suitable reference materials.

Note: Most LECO copper calibration materials are plated with nickel for stability and oxidation resistance. Sample preparation is not required and the samples are suitable for use straight from the bottle. Refer to the certificate of analysis for details.

Method Parameters

Analysis Parameters	
Outgas Cycles	3
Analysis Delay	20 seconds
Analysis Delay Comparator	1.000
Analysis Type	Semi-Auto Analysis
Auto Analyze on Mass Entry	Disabled
Pre-Analyze Crucible Outgas	Disabled

Element Parameters	*Oxygen*
Minimum Analysis Time	40 seconds
Significant Digits	5
Conversion Factor	1.000000
Integration Delay	5 seconds
Comparator Level	1.000000%
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	—
Analyze Low Power	5000 watts*
Analyze High Power	5000 watts *
Analyze Ramp Rate	—
Sample Prep Time	—
Sample Prep Power	—
Temperature Sustain	None
Furnace on Time	30 seconds
Temperature Sustain	None
Peak Find Parameters	Oxygen
Look for Shoulders	No
Peak Threshold	0.000000

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

Inorganic Application Note

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. Clean upper and lower electrodes either manually or with an equipped automatic cleaner.
   d. Place ~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.
3. Calibrate/Drift Correct.
   a. Weigh ~1.0 g of a calibration sample; enter mass and sample identification into Sample Login (F3).
   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. Clean upper and lower electrodes either manually or with an equipped automatic cleaner.
   e. Place ~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 Copper sample of ~0.5-2.0 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%*	*Sample*	*Mass (g)*	*O%*
CRM	1.0 g	0.0582	CRM	1.0 g	0.0584
CMO Cu600/I		0.0578	CMO Cu600/I		0.0580
Copper Rod		0.0586	Copper Rod		0.0582
0.0581% O		0.0584	0.0581% O		0.0583
±0.0012%		0.0575	±0.0012%		0.0578
Etched		0.0584	Abraded		0.0583
		0.0575			0.0581
		0.0581			0.0582
		0.0584			0.0574
		0.0577			0.0580
	***X*=**	***0.0581***		***X*=**	***0.0581***
	***S*=**	***0.0004***		***S*=**	***0.0003***
CRM	1.0 g	0.0314	CRM	1.0 g	0.0315
NIST 885		0.0316	NIST 885		0.0313
Refined		0.0317	Refined		0.0313
Copper		0.0318	Copper		0.0315
0.031% O		0.0315	0.031% O		0.0312
±0.002%		0.0316	±0.002%		0.0316
Etched		0.0314	Abraded		0.0315
		0.0319			0.0314
		0.0317			0.0315
		0.0313			0.0317
	***X*=**	***0.0316***		***X*=**	***0.0315***
	***S*=**	***0.0002***		***S*=**	***0.0001***
OFHC	1.0 g	0.00012	OFHC	1.0 g	0.00016
Copper Wire		0.00006	Copper Wire		0.00021
Uncertified		0.00008	Uncertified		0.00016
Etched		0.00007	Abraded		0.00020
		0.00007			0.00014
		0.00005			0.00014
		0.00012			0.00012
		0.00016			0.00012
		0.00008			0.00014
		0.00011			0.00012
	***X*=**	***0.00009***		***X*=**	***0.00015***
	***S*=**	***0.00003***		***S*=**	***0.00003***
LECO	1.0 g	0.0235			
501-147		0.0236			
Nickel Plated		0.0237			
Copper		0.0238			
0.0239% O		0.0239			
±0.0003%		0.0238			
As Received		0.0235			
		0.0236			
		0.0238			
		0.0235			
	***X*=**	***0.0237***			
	***S*=**	***0.0001***			

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