Application Note

Instrument: H836EN

Determination of Hydrogen in Aluminum

LECO Corporation; Saint Joseph, Michigan USA

Introduction[1,2]

Aluminum has several qualities that make it the preferred material for applications in the construction, aerospace, electronics, container, and automotive industries. These qualities include low density, high strength, high conductivity, a desirable appearance, and superior corrosion resistance. Unfortunately, Aluminum also has some disadvantages, including a relatively high cost and unfavorable Hydrogen solubility characteristics.

Hydrogen gas has a high solubility in molten Aluminum and is readily introduced during processing. Once solidification begins, the solubility decreases approximately twenty-fold, and Hydrogen is forced out of the Aluminum. Although most of the Hydrogen is diffused, a small percentage may get trapped, creating Hydrogen-filled voids. This remaining Hydrogen gas is the root cause of numerous failure mechanisms in Aluminum products, such as voids in casting and blisters in sheets. As a result, the determination of total, surface, and bulk Hydrogen content in Aluminum is a critical quality control procedure for ensuring material integrity and performance.

Instrument Model and Configuration

The LECO H836EN is a Hydrogen determinator that utilizes an electrode furnace, Argon carrier gas, and thermal conductivity detection to meet the needs of the Aluminum industry. The H836EN software takes advantage of the existing ability to perform stepped furnace analysis and incorporates it in such a way that separation of surface and bulk Hydrogen can be performed and corresponding results reported.

A pre-weighed sample is placed in a graphite crucible, which is heated in an impulse furnace to release analyte gases into a flowing stream of Argon carrier gas. Evolved Oxygen reacts with the graphite crucible to form CO and CO_2. Separate reagents remove moisture from the analyte gases present as H_2O and convert CO to CO_2, which is then scrubbed from the analyte gases by an additional reagent. A molecular sieve column is used to separate the analyte gases, H_2 and N_2. Then a thermal conductivity (TC) cell is used for the detection of Hydrogen (H_2).

Method Reference

ASTM E2792: Standard Test Method for Determination of Hydrogen in Aluminum and Aluminum Alloys by Inert Gas Fusion

Sample Preparation

Proper sample preparation is critical for the determination of Hydrogen in Aluminum. Samples should be sectioned and machined on a lathe to a uniform sample dimension. This facilitates surface/bulk Hydrogen determination. Sampling of molten metal is typically done using a well-fed, chill wedge-bar Copper book mold (referred to as a Ransley mold[3]) to minimize formation of voids. The sample is sectioned using a water-cooled cut-off saw. Final sample preparation is completed using a lathe. The lathe chuck and tool must be clean and free of lubricants.* It is important that the entire original surface is removed. The maximum/ideal sample dimension is 10.5 x 24 mm. The sample may be rinsed in acetone and dried with warm air prior to analysis to ensure the removal of any remaining surface contamination.

**A lathe speed between 600-700 rpm is recommended for optimal performance.*

The prepared sample should be analyzed as quickly as possible to minimize surface contamination/Hydrogen pickup. Handle prepared sample with clean tweezers only.

Click here to watch an Aluminum Sample Preparation video.

Note: LECO Reference Materials typically do not require preparation prior to analysis. Refer to preparation statement on the reference material certificate for details. Please reference the appropriate Safety Data Sheets (SDS) for safe handling of all reference materials and samples.

Accessories

764-330 Graphite Crucibles, 611-351-183 Electrode Tip, 761-739 Tin Flux Pellets**, 766-053 Crucible Tweezers, 760-138 Sample Tweezers.

***The Tin flux pellets are used for steel calibration analysis only and are not required for Aluminum analysis.*

Reference Materials

LCRM®, LRM®, NIST, or other suitable reference materials.

Note: Due to the limited availability of Aluminum reference materials, a steel reference material may be used for calibration.

Method Summary

When steel reference materials are used to calibrate the instrument for Aluminum analysis, the furnace, outgas, and analyze furnace parameters used for instrument calibration will differ from those used for sample analysis.

Instrument Steel Calibration Parameters

Analysis of steel reference materials is performed utilizing a furnace current setting of 900 A for crucible outgassing and 850 A for reference material analysis. A two-step furnace method is utilized.

Aluminum Sample Analysis Parameters

Analysis of Aluminum samples is performed utilizing a furnace power setting of 4500 W for crucible outgassing. Sample analysis is performed utilizing a stepped furnace analysis, allowing for separation of surface and bulk Hydrogen sources. A five-step furnace method is utilized.

Each of the five steps serves a specific purpose in ensuring accurate analysis of Aluminum samples, as detailed below:

Step 1: Liberates surface Hydrogen
Step 2: Allows the surface Hydrogen to separate from the bulk Hydrogen for peak integration
Step 3: Ensures complete liberation of surface Hydrogen prior to bulk analysis
Step 4: Liberates bulk Hydrogen
Step 5: Applies a reduced power to prevent Aluminum from boiling into the upper electrode.

Method Parameters†

General Parameters

Sample Introduction	Automated Sample Drop
Analysis Delay	70 s
Auto Analyze On Mass Entry	No
Outgas Before Mass Entry	No
Outgas At End Of Analysis	No
Wait For User To Load Sample	Yes
Nominal Mass	1.0000 g
Vacuum On Time	20 s

Element Parameters

Integration Delay	30 s
Starting Baseline	2 s
Use Comparator	Yes
Comparator Level	0.10 %
Integrate From Furnace Method	Yes
Minimum Integration Time	440 s
Maximum Integration Time	445 s
Use Endline	--

Furnace Method Parameters†

Steel Calibration Furnace Method Parameters

Apply Peak Find to This Furnace Method	No

Furnace Parameters

Furnace Control Mode	Current

Outgas Parameters

Cycles	3
Current Mode	Constant
Current	900†† A
Time	30 s
Cool Time	5 s

Surface Hydrogen

Analyze Surface Hydrogen	No

Analyze Furnace Settings

Step 1	Current Mode	Constant
	Current	850†† A
	Time	60 s
Step 2	Current Mode	Constant
	Current	0 A
	Time	30 s

Aluminum Furnace Method Parameters

Apply Peak Find to This Furnace Method	Yes

Furnace Parameters

Furnace Control Mode	Power

Outgas Parameters

Cycles	4
Power Mode	Constant
Power	4500†† W
Time	30 s
Cool Time	5 s

Surface Hydrogen

Analyze Surface Hydrogen	No

Analyze Furnace Settings

Step 1	Power Mode	Constant
	Power	950†† W
	Time	20 s
Step 2	Power Mode	Constant
	Power	0 W
	Time	60 s
Step 3	Power Mode	Constant
	Power	450†† W
	Time	70 s
Step 4	Power Mode	Constant
	Power	1400†† W
	Time	35 s
Step 5	Power Mode	Constant
	Power	0 W
	Time	150 s

Peak Find Settings

Strategy	User Specified
Peaks	2

Peak 1 - Surface Hydrogen

Name	Surface Hydrogen
Peak Start	Fixed Time
Start Time	5.0 s
Peak End	Fixed Time
End Time	140.0 s

Peak 2 - Bulk Hydrogen

Name	Bulk Hydrogen
Peak Start	Link to End of Previous Peak
Peak End	End of Sample Data

†Refer to 836 Series Operator's Instruction manual for parameter definitions.
††May vary based on the line voltage. Adjust to improve recovery or to reduce crucible burn-through.

Procedure

1. Prepare the instrument as outlined in the operator's instruction manual.
2. Determine the steel calibration blank‡.
   a. From the Analysis Screen, use the Login Bar to add three or more blank replicates and select the appropriate method. Next, select the appropriate furnace method.
   b. Press the Analyze button on the Analysis Screen. After a short delay, the loading head slide-block will open.
   c. Press the Analyze button on the Analysis 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 with an equipped automatic cleaner.
   e. Add one 761-739 Tin Flux Pellet into a 764-330 Graphite Crucible and firmly place the graphite crucible containing the Tin pellet on the lower electrode tip.
   f. Press the Analyze button on the Analysis Screen, and the lower electrode will close. The analysis sequence will start and end automatically.
   g. Perform steps 2b through 2f a minimum of three times.
   h. Set the blank for the steel reference material replicates following the procedure outlined in the operator's instruction manual.
3. Determine the Aluminum sample blank‡.
   a. From the Analysis Screen, use the Login Bar to add three or more blank replicates and select the appropriate method. Next, select the appropriate furnace method.
   b. Press the Analyze button on the Analysis Screen. After a short delay, the loading head slide-block will open.
   c. Press the Analyze button on the Analysis 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 with an equipped automatic cleaner.
   e. Firmly place a 764-330 Graphite Crucible on the lower electrode tip.
   f. Press the Analyze button on the Analysis Screen, and the lower electrode will close. The analysis sequence will start and end automatically.
   g. Perform steps 3b through 3f a minimum of three times.
   h. Set the blank for the Aluminum sample replicates following the procedure outlined in the operator's instruction manual.

   ‡The steel calibration blanks are used to set the blank for the steel reference materials, and the Aluminum sample blanks are used to set the blank for the Aluminum samples.
4. Instrument calibration or drift correction.
   a. From the Analysis Screen, use the Login Bar to add three or more calibration/drift replicates and select the appropriate method. Next, select the appropriate furnace method.
   b. Weigh a steel calibration/drift reference material and enter the mass and sample identification information into the appropriate replicate fields in the Analysis Screen.
   c. Press the Analyze button on the Analysis Screen. After a short delay, the loading head slide-block will open.
   d. Place the reference material sample into the open port at the top of the loading head.
   e. Press the Analyze button on the Analysis 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 with an equipped automatic cleaner.
   g. Add one 761-739 Tin Flux Pellet to a 764-330 Graphite Crucible and firmly place the crucible containing the Tin pellet on the lower electrode tip.

   Note: Tin flux pellets are used for steel calibration analysis only and are not required for Aluminum analysis.
   h. Press the Analyze button on the Analysis Screen, and the lower electrode will close. The analysis sequence will start and end automatically.
   i. Perform steps 4b through 4h a minimum of three times for each calibration/drift sample utilized.
   j. Calibrate/drift following the procedure outlined in the operator's instruction manual.
   k. Verify the calibration/drift correction by analyzing an appropriate mass of another/different suitable reference material, following steps 4b through 4h, and confirm that the results are within the acceptable tolerance range.
5. Analyze the samples.
   a. From the Analysis Screen, use the Login Bar to add the desired number of sample replicates and select the appropriate method. Next, select the appropriate furnace method.
   b. Weigh a prepared sample and enter the mass and sample identification information into the appropriate replicate fields in the Analysis Screen.
   c. Press the Analyze button on the Analysis 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 Analysis 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 with an equipped automatic cleaner.
   g. Firmly place a 764-330 Graphite Crucible on the lower electrode tip.
   h. Press the Analyze button on the Analysis Screen, and the lower electrode will close. The analysis sequence will start and end automatically.
   i. Perform steps 5b through 5h for each sample being analyzed.

Typical Results

Data was generated utilizing a linear, force through origin calibration using LECO 503-537 (Lot 0735) LCRM Steel Pin (6.9 ppm Hydrogen). The calibration was verified using LECO 502-916 (Lot 0825) LCRM Nickel Plated Steel Pin (1.3 ppm Hydrogen).

Sample	*Mass (g)*	*Total H (ppm)*	*Surface H (ppm)*	*Bulk H (ppm)*
Aluminum Rod	5.5037	0.134	0.034	0.099
ALRO Steel Corp.	5.6161	0.227	0.110	0.117
PN: 17002900	5.5140	0.172	0.055	0.117
	5.6268	0.096	0.011	0.084
	5.6543	0.141	0.051	0.090
	$\bar{x}$ =	***0.154***	***0.052***	***0.102***
	s =	***0.049***	***0.037***	***0.015***
Aluminum Rod	5.4782	0.154	0.056	0.097
Alcoa (Aluminum Company of America)	5.3474	0.154	0.076	0.079
Alcoa Alu-H2 (Lot A)	5.4409	0.130	0.072	0.057
0.068 ±0.018 mg Hydrogen/kg Al	5.5842	0.107	0.033	0.075
	5.2730	0.191	0.127	0.065
	$\bar{x}$ =	***0.147***	***0.073***	***0.074***
	s =	***0.031***	***0.035***	***0.016***

$\bar{x}$ = Sample Mean; s = Sample Standard Deviation

References

[1]D. E.J. Talbot, "The Effects of Hydrogen in Aluminum and Its Alloys", Maney, London, 2004.

[2]P. D. Hess and G. K. Turnbull, Proceedings of International Conference on the Effects of Hydrogen on Materials Properties and Selection and Structural Design, Champion, 1973, 277-287.

[3]C.E. Ransley and D.E.J. Talbot, "The Routine Determination of the Hydrogen Content of Aluminum and Aluminum Alloys by the Hot-Extraction Method". Journal of the Institute of Metals, Vol. 84, 1955-1956, 445.

LECO Corporation | 3000 Lakeview Avenue | St. Joseph, MI 49085 | Phone: 800-292-6141 | 269-985-5496

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