Hydrogen Determination in Aluminum and Aluminum Alloys

Instrument

RHEN602

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 a majority 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. Thus, an accurate hydrogen determinator is a necessary quality control device for all aluminum producers.

The LECO RHEN602 is a hydrogen determinator which utilizes an electrode furnace, an argon carrier gas, and thermal conductivity detection to meet the needs of the aluminum industry. The RHEN602 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.

Each of these steps serves a specific purpose for this analysis.
- Step 1 is used to liberate surface hydrogen
- Step 2 provides the surface hydrogen time to separate from the bulk hydrogen
- Step 3 is optional and used to ensure complete liberation of surface hydrogen prior to bulk analysis
- Step 4 is used to liberate bulk hydrogen
- Step 5 is a power reduction step used to prevent aluminum from boiling into the upper electrode

The RHEN602 software allows for separate programming of minimum analysis time and analysis delay for surface and bulk hydrogen. The acquisition screen plots two different windows (Surface and Bulk) based on these values. Next, the Surface and Bulk values can be summed and the Total Hydrogen value reported.

The following application note is specifically written for use with the LECO RHEN602.

RHEN602

Sampling and Sample Preparation

This application is written for solid aluminum samples. Samples are 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 wedge is separated from the bar using a water-cooled cut-off saw. Wrought products are sectioned using a water-cooled cut-off saw. Final sample preparation is completed using a lathe. Lathe chuck and tool must be clean and free of lubricants. Nominal lathe speed is 600 to 700 RPM, advance at ~0.04 mm/revolution. It is important that the entire original surface is removed. The maximum/ideal sample dimension is 10.5 x 24 mm. Wash sample in acetone and dry with warm air prior to analysis. Prepared sample should be analyzed as quickly as possible to minimize surface contamination/hydrogen pick-up. Handle prepared sample with clean tweezers.

Accessories

764-330 Graphite Crucibles, 611-351-183 Electrode Tip.

Calibration Samples

NIST or other suitable reference materials. Gas dosing may also be used to calibrate; refer to operator's instruction manual.

Method Parameters

Analysis Parameters

Outgas Cycles	4
Analysis Delay	120 seconds
Analysis Delay Comparator	1.000
Analysis Type	Semi-Auto Analysis
Pre-Analysis Crucible Outgas	Disabled

Element Parameters	Hydrogen Surface	Hydrogen Bulk	Standards*
Minimum Analysis Time	130 seconds	340 seconds	90 seconds
Significant Digits	7	7	7
Conversion Factor	1.000000	1.000000	1.000000
Integration Delay	35 seconds	155 seconds	32 seconds
Comparator Level	2.000000 %	2.000000%	2.000000%

When steel calibration samples are used for calibration.

Gas Dose Parameters*	Hydrogen
Minimum Gas Dose Time	70 seconds
Integration Delay	32 seconds
Comparator Level	1.000000%
Bypass Furnace	Yes (check box)
Gas Dose Cycles	1

Helium or Nitrogen may also be used as dose gases.

Furnace Parameters		Standards*
Furnace Control Mode	Power	Power
Purge Time	15 seconds	15 seconds
Outgas Time	30 seconds	15 seconds
Outgas Cool Time	5 seconds	5 seconds
Outgas Low Power	4500 watts	4500 watts
Outgas High Power	4500 watts	4500 watts
Outgas Ramp Rate	0 watts/seconds	0 watts/seconds
Analyze Low Power	0 watts	3700 watts
Analyze High Power	1400 watts	3700 watts
Analyze Ramp Rate	0 watts/seconds	0 watts/seconds
Sample Prep Time	0 seconds	0 seconds
Sample Prep Power	0 watts	0 watts
Furnace On Time	300 seconds	70 seconds

When steel or titanium calibration sample are used for calibration.

Temperature Sustain

Step	Start Power	End Power	Time
1	900 watts	900 watts	20 seconds
2	0 watts	0 watts	60 seconds
3	450 watts	450 watts	70 seconds
4	1400 watts	1400 watts	30 seconds
5	0 watts	0 watts	150 seconds

Temperature Sustain Standards: None

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.
   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. Place crucible on electrode pedestal.
   e. Press Loader Switch, the lower electrode will close and the analysis sequence will start and end automatically.
   f. Repeat steps 2a through 2e a minimum of five times.
   g. Set the blank following the procedure outlined in the operator's instruction manual.
3. Calibrate/Drift Correct.
   a. Weigh a calibration sample and enter mass into sample login.
   b. Press Loader Switch on front of furnace, the loading head slide block will open.
   c. Place prepared 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. Place crucible on the electrode pedestal.
   f. Press Loader Switch, the lower electrode will close and the analysis sequence will start and end automatically.
   g. Repeat steps 3a through 3f a minimum of five times for each calibration/drift sample used.
   h. Calibrate or Drift Correct the instrument following the procedure outlined in the operator's instruction manual.

   Note: Gas dosing may be used to calibrate/drift correct in lieu of analyzing solid calibration samples. Refer to the operator's instruction manual for details.
4. Analyze Samples.
   a. Weigh ~3.0 to 6.5 g prepared aluminum sample and enter mass into sample login.
   b. Proceed as directed in steps 3b through 3f.

*Typical Results—Solid Samples

Sample	Mass g	Surface H ppm	Bulk H ppm	Total H ppm
6061	5.8061	0.017	0.065	0.082
Aluminum Alloy	5.9035	0.026	0.057	0.083
	5.3544	0.022	0.058	0.080
	5.1521	0.026	0.061	0.087
	X =	*0.023*	*0.060*	*0.083*
	s =	*0.0043*	*0.0033*	*0.0029*
AlMg 5%	3.8328	0.050	0.150	0.200
Aluminum Alloy	3.9159	0.047	0.174	0.221
0.16 ppm H (bulk)	3.4060	0.059	0.160	0.219
Reference Standard**	3.9018	0.049	0.166	0.215
	X =	*0.051*	*0.163*	*0.214*
	s =	*0.0054*	*0.0101*	*0.0095*
AlMgSi 0.5%	6.3068	0.026	0.133	0.159
Aluminum Alloy	6.1883	0.037	0.128	0.165
0.14 ppm H (bulk)	6.0590	0.034	0.140	0.174
Reference Standard**	5.7480	0.020	0.138	0.158
	X =	*0.029*	*0.135*	*0.164*
	s =	*0.0080*	*0.0051*	*0.0073*

*Results based on calibration with LECO 501-529 Steel Pin.

**Al-reference materials from Senter for Industriforskning, Oslo, Norway.

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.