DEVELOPMENT OF A METHOD TO ANALYZE ALUMINUM ALLOYS POWDERS USING THE GAS ANALYZER LECO ONH836

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Introduction

LECO methods exist to analyze oxygen (ONH836_O_ALUMINUM_203-821-459) and hydrogen (RHEN602_H_ALUMINUM_203-821-300) in aluminum. The goal of this work is to determine if a method able to analyze oxygen, nitrogen, and hydrogen in aluminum at the same time is feasible. Past work at MetaFensch has shown that ONH836_O-H_TITANIUM_HYDRIDE_203-821-458 (Oxygen and Hydrogen Determination in Titanium Hydride) can be used to analyze oxygen and hydrogen in titanium powders and also adapted to include nitrogen with good repeatability. For this reason, this method was taken as a starting point for development in aluminum.

Description of the initial method

The materials used to prepare the sample are shown in the table below. The powder sample (around 0.1 g) is placed along with graphite powder within a Nickel capsule and then put in a graphite crucible for melting and gas analysis.

Materials	LECO reference
Graphite crucible	782-720
Nickel capsule	502-822
Graphite powder	501-073

Table 1: Reference of the additives used in the initial method.

Based on past experience with titanium powder, the addition of a tin pellet helps desorb nitrogen from the sample and also increases the volume in the crucible. The utility of this additional pellet for aluminum powder is a subject of investigation.

Furnace parameters and the heat cycle are listed below:

- 6000 W for drying 3 times (1000 w for 20 sec)
- 100 seconds at 1200 W
- An increase from 1200 W to 4800 W over 20 seconds
- A plateau at 4800 W for 20 seconds

Starting from the initial method, the objective is to adjust parameters specifically for aluminum. Different levels of each parameter can be explored: heating power, powder quantity and the type/number of additives. To set these parameters, the following experiments were carried out:

- 5 measurements at different power levels to target the optimal power to melt aluminum powder
- 16 measurements with different quantity of powder
- 5 measurements to evaluate the impact of the additives (Ni capsule, Sn pellet and Graphite powder)

In this study, a new alloy powder for Additive Manufacturing was chosen to adjust the method.

Tests and results

Before each series of tests, a blank analysis is performed: 3 measurements without aluminum powder and without recording the results in order to heat the machine up and increase stability.

Power

To investigate the power needed to melt aluminum powder, 5 different levels were tested: 3500 W, 4000 W, 4800 W, 5200 W and 6000 W. The other parameters were fixed: powder quantity (0.10 g) and additives (1 Sn pellet, 1 Ni capsule and 0.5 g of Graphite powder).

Titanium or tungsten powders require 4800 W to melt, a lower power is thus expected for aluminum, as the melting temperature is lower. The results of the test are shown in Figure 1.

Figure 1: Sample and crucible after the analysis of aluminum powder at each power.

To evaluate the optimal power, the sample after analysis is observed. The materials within the crucible (aluminum powder + additives) need to be perfectly melted, resulting in a spherical shape with no remaining free powder. A melting temperature that is too high leads to degradation of the crucible.

At 5200 W and 6000 W, cracks are observed on the crucible and the base has eroded or even become detached from the crucible. At 3500 W melting is not quite complete while the sample is deteriorated at 4800 W. Therefore, 4000 W seems to be optimal, with a good melt and an undamaged crucible.

Powder quantity

To obtain good reproducibility, an optimal sample quantity is needed: if there is not enough powder, dispersion within the results increases, whereas too much powder leads to incomplete melting and, therefore, erroneous results. To determine the quantity of powder necessary to produce representative results, 16 tests with increasing amounts of aluminum powder were performed: from 0.05 g to 0.20 g, in steps of 0.01 g. The precision of the scale is 0.001 g. The other parameters were fixed: power at 4000 W and additives (1 Sn pellet, 1 Ni capsule and 0.5 g of Graphite powder)

The oxygen content is used to compare the results and study the influence of sample quantity.

The oxygen level and the shape of the melted sample obtained for different quantities of aluminum powder are shown in Figure 2.

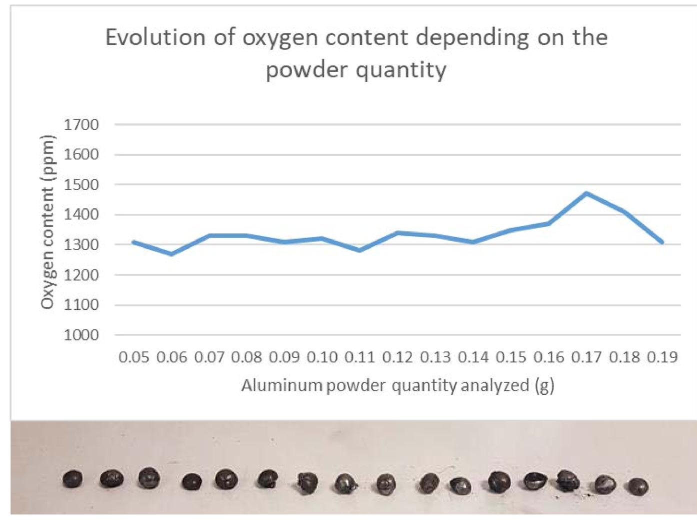

Figure 2: Graph representing the evolution of oxygen content based on powder quantity (top), and, from left to right, picture of the samples after analysis, from 0.05 to 0.20g of aluminum powder (bottom).

Apart from 2 measurements with higher mass, the oxygen content remains approximately 1300 ppm. Therefore, the quantity of powder has no major influence on the analysis. Considering this observation, it is decided to use a mass sample of 0.1 +/- 0.01 g for the analysis of aluminum powder (as is recommended in the LECO method: Oxygen and Hydrogen in Titanium hydrid).

Additives

The next step is to analyze the impact of additives. Measurements were carried out with varying the number of additives: tin pellet, graphite powder and nickel capsules. The other parameters were unchanged, powder quantity of 0.1 g and power of 4000 W

The average values obtained after analysis are presented in Table 2 below, and the picture of the samples to observe the melt is in Figure 3.

in ppm	Additives	Oxygen	Nitrogen	Hydrogen
Oxygen and Hydrogen Determination in Titanium Hydride (LECO)	1 Sn pellet 0.5 g of graphite powder 1 Ni capsule	1310 +/-26	16 +/-1	23 +/-7
Without Graphite powder	1 Sn pellet 1 Ni capsule	1330 +/-11	16 +/-1	19 +/-9
Without Tin pellet	0.5 g of graphite powder 1 Ni capsule	1140 +/-68	18 +/-1	14 +/-1
Ti method with 2 Tin pellets	2 Sn pellets 0.5 g of graphite powder 1 Ni capsule	1350 +/-16	17 +/-1	21 +/-2
Ti method with 2 Nickel capsules	1 Sn pellet 0.5 g of graphite powder 2 Ni capsules	648 +/-870	17 +/-1	30 +/-7

Table 2: Results of O, N, H contents with different additives (type and number).

Figure 3: Sample aspect after analysis with different additives, with 2 tin pellets for the 2 samples on the left, and 2 Nickel capsules for the 2 samples on the right.

Comparison using the Titanium method as a reference was carried out; the additives for this method are 1 Ni capsule, 1 Sn pellet and 0.5 g of graphite powder. Removing the graphite powder makes no difference in the O, N, H results for aluminum. On the contrary, removing the tin pellet disturbs the measure by decreasing oxygen content and increasing result spread. Tin pellets can thus be considered to play a role in measurement accuracy. Increasing the number of pellets from 1 to 2 does not have any significant influence, therefore, 1 pellet is sufficient.

Nickel capsules cannot be eliminated as they are used to contain the aluminum powder before melting. Nevertheless, a method with an addition of Ni capsule is not relevant, as it deteriorates the measurement of oxygen (600 ppm vs. 1300 ppm expected) and leads to melting problems, as it is shown in Figure 3.

Comparison with certified materials and laboratory

Finally, to verify that the method is correct, 2 certified reference materials, where dissolved gas levels are already known, were tested. Using the aluminum method developed, the following results were obtained (Table 3).

Certified reference materials	in ppm	Oxygen	Nitrogen	Hydrogen
502-876 (N°0300)	Target (certified)	3070 +/- 50	60 +/- 10	17.3 +/- 3.2
	Mean value (3 measurements)	3050 +/- 35	91 +/- 2	19.3 +/- 2.7
502-867 (N°0739-1)	Target (certified)	1380 +/- 50	120 +/- 10	(25.4)
	Mean value (3 measurements)	1340 +/- 20	99 +/- 4	23.8 +/- 5.1

Table 3: Analysis of CRMs using the method developed in this study

Comparing obtained measurements with CRM standards, it can be seen that average oxygen and hydrogen contents are within standard deviation. However, this is not the case for nitrogen. Measurements can be used qualitatively to determine trends but further work is required in order to improve accuracy. To consolidate the relevance of the developed method, measured oxygen content on 2 aluminum alloy powders has been compared to a certified laboratory (Table 4).

in ppm		Oxygen content (3 measurements)			Mean	Standard deviation
Certified lab	Al Powder 1	1200	1400	1400	1300	89
MetaFensch	Al Powder 1	1130	1100	1070	1070	64
Certified lab	Al Powder 2	470	490	310	430	76
MetaFensch	Al Powder 2	270	300	330	300	25

Table 4: Comparison of Oxygen contents for 2 Al powders, measured by MetaFensch with the new method and a certified laboratory.

The comparison of these results is quite good within the current calibration interval (range from 1000 to 3000 ppm). To further improve the method and measure lower oxygen content in aluminum powder (< 1000 ppm, as for sample Al powder 2 in Table 4), a lower oxygen content reference material is required.

Conclusion

The titanium method previously developed corresponds to a high temperature crucible, a tin pellet, approximately 0.5 g of Graphite and a capsule of Nickel, for a titanium powder sample of 0.1 g (+/- 0.015 g) with an optimal furnace parameters and heat cycle. A parametric study was carried out to adjust this method for aluminum.

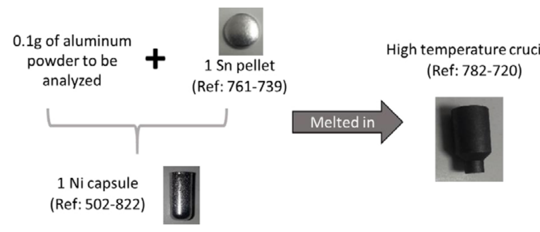

Figure 4: Illustration of the method developed to analyze aluminum powder using LECO836.

The results show that the use of graphite, which lowers the melting point of titanium in the original method, is not critical for aluminum. Experiments also show that the amount of tin required is 1 pellet and a standard nickel capsule, in order to ensure a good melting of the sample. Figure 4 summarizes these findings.

About the authors

MetaFensch and IRT-M2P have a joint research laboratory located in the northeastern France, working with industrial partners on materials and metallurgical R&D projects. In this laboratory, the characterization of oxygen, nitrogen, and hydrogen contents in titanium alloys (bulk materials and powders), using the existing "Oxygen and Hydrogen Determination in Titanium hybrid" method from LECO, has been studied. With the arrival of a new VIGA atomizer (Vacuum Induction Gaz Atomization) dedicated to the study of light alloy powders, a need for aluminum powder characterization appeared. The development of a method dedicated to analyzing O, N, H contents in aluminum powders using LECO ONH836 has been carried out for this purpose.

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