IGA FRACTIONAL O/N/H ANALYSES OF POWDER FEEDSTOCKS FOR ADDITIVE MANUFACTURING

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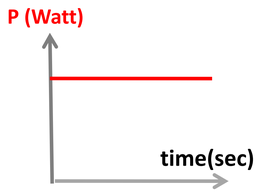
Introduction

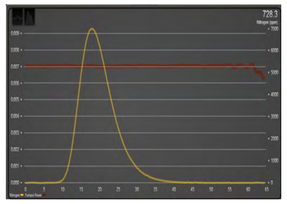
Additive manufacturing calls for powder feedstock of metals, alloys and ceramics, with particle size typically in 100s nm – 10s µm range. This leads to orders-of-magnitude increase in surface area from the bulk materials. Driven by the increase in surface free energy (i.e., thermodynamic favorable) and the decrease in diffusion length (i.e., kinetic favorable), the surface chemistry of powders becomes equally important to, if not greater than, the bulk chemistry. Of particular interest is to understand and control the O, N and H chemistries of powders, which are prone to change depending on the material type, the powder manufacturing technique, working atmosphere, packaging/handling, etc.

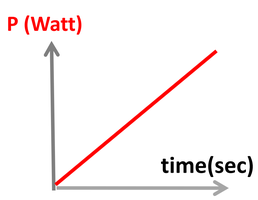
Interstitial Gas Analyses (IGA) is the standard method to determine bulk content of O, N and H in inorganic materials, such as metals, alloys and ceramics, via carbo-reductive inert gas fusion, or hot extraction-induced dehydrogenation, followed by infrared or thermal conductivity detection. Taking advantage of the temperature-programming capability of LECO® ONH836 instrument, herein we wish to demonstrate that by analyzing samples in different temperature modes (flash heating, ramping and stepped heating), it is possible to quantitatively speciate the O, N and H chemistries in powders, such as surface and interface oxygen, oxide precipitates, interstitial oxygen, surface -OH or physiosorbed moisture, from % levels to ppm levels.

IGA Fractional Analysis Method Development – N in High Purity Graphite

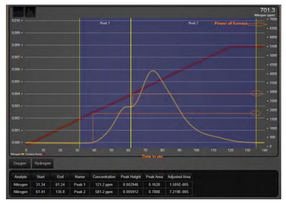


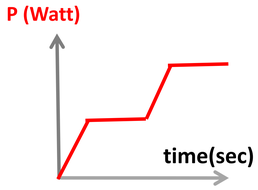
*In **flash mode** the total N is determinated*



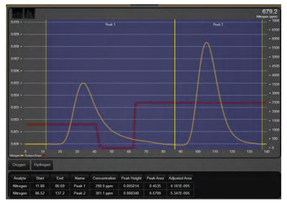


*In **ramp mode** two N chemistries are identified*



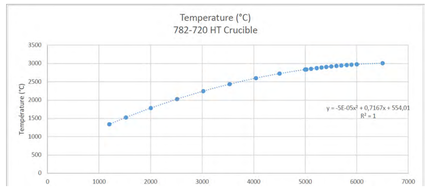


*In **steps mode** the N chemistries are resolved and quantified*

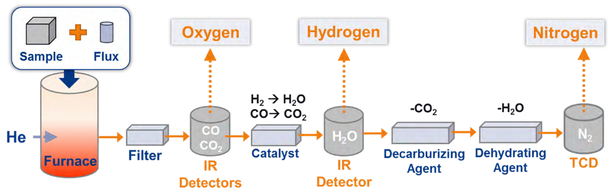


Instrument Operating Principle

LECO® ONH836
O/N from ppm to 50%wt
H from 0.1 ppm to 2%wt

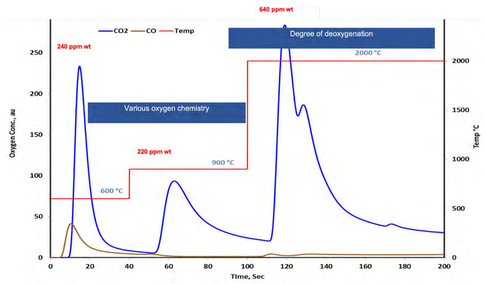


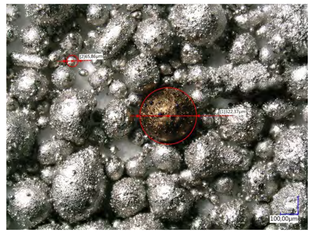
Abaqus Power (Watt) / Temperature (°C)



Case Study I: Fractional O Analysis of Ta6V Powder for Additive Manufacturing

Fractional O Analysis of Ta6V Powder





IGA fractional analysis gives:

- Information on various oxygen chemistries
- Surface oxygen/bulk oxygen ratio
- Quantitative and precise results

ELEMENT	H	O	N
AVERAGE, wt%	0.029	0.11	0.065
% RSD (n = 3-5)	4	10	12

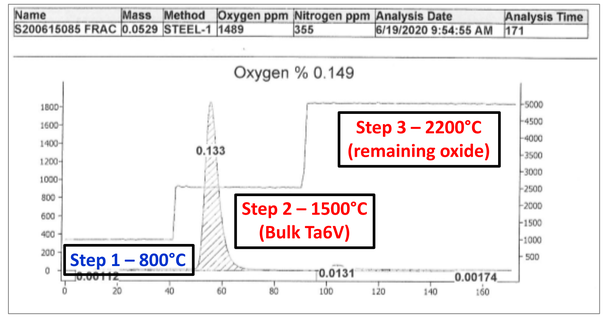
Comparative IGA Flash / Fractional

Operating Mode	REP. 1	REP. 2	REP. 3	AVG (ppmw)	RSD (%)
IGA Flash	3990	3850	3960	3930	1.9
IGA Fractional	1490	1430	1505	1475	2.6

The difference is attributed to moisture and M-OH bonds on the powder

Step 1 – 800°C

Step 1 is set to 800°C. This temperature step is high enough to drive off moisture and to dehydrate M-OH bonds, but low enough to prevent them from conversion to CO and CO_2 for IGA dosing.



Case Study II: Fractional O and H Analysis of Al Powder for Additive Manufacturing

	Lot A		Lot B		Lot C	
ppmwt	O	H	O	H	O	H
IGA Flash	1855	66	2826	93	2091	81
IGA rampe	1539	39	2701	82	1743	52
Flash - rampe	316	27	125	11	348	29
H/O (%)	8.5%		8.8%		8.3%	

Since most H/O bonds exist in H_2O and M-OH form with stoichiometric ratios of 12.5% and 6.3%, it is estimated these lots contain ~35% H_2O and ~65% M-OH. Further characterization and verification can be done by determining moisture on LECO RC 612, surface oxide thickness with Auger Electron Spectroscopy for, O/H bonding chemistries with XPS and FTIR.