SAND2017-10109D

Compositional Analysis of Additively Manufactured Metals

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Move Towards Additive Manufacturing

AM builds a part layer by layer

AM offers flexibility in design
(not restricted by manufacturability)

Reduction in design cycle
(ability to iterate through designs)

EOS® M290 Direct Metal Laser

Powder Bed Fusion - high thermal energy beam fuses layered metal powders

- Powder Bed Fusion
  - Electron Beam Melting
  - Selective Laser Melting
  - Direct Metal Laser Sintering

Needs in Compositional Analysis for AM

- Additive manufacturing presents new challenges in qualification.
  - Compositions of built parts are not necessarily the same as compositions of starting materials.
  - Powder, as starting material, has potential to introduce light elements (particularly C and O) at much higher levels than may be desired.
  - Powder recycling may alter powder chemistry.
  - Use of different materials in a single machine can lead to compositional carry-over from previous powders.
- Quantitative composition analysis with low detection limits (~0.01%).
- Sensitivity to light elements (C, S, O, N, H, and B) at ppm levels.
- Flexibility for different materials.
- Matrix independent
- Rapid, non-destructive, adaptable to different shapes/sizes/surfaces
- Low cost
- Spatial resolution and/or depth resolution
- Potential to be implemented as an in process monitor.

Compositions from CF8 (304L equivalent)

CF8 – stainless steel	GDS-OES (CCD)	GDS-OES (PMT)	arc-spark-OES (CCD)	micro-XRF	private lab (ICP)	ICP-OES/MS	EPMA / WDS microprobe	XPS-sputtered	nominal
Fe	71.95	71.85	71.4	69.35 ± 0.27	71.83	72.41	72.038 ± 0.581	67.9	63.8-74
Cr	18.12 ± 0.397	18.14 ± 0.095	18.1±0.0434	20.33 ± 0.28	18.097 ± 0.163	17.595 ± 0.091	19.39 ± 1.067	7.6	18-21
Ni	8.45 ± 0.099	8.15 ± 0.125	8.46±0.0344	7.4 ± 0.27	8.363 ± 0.225	7.991 ± 0.027	8.097 ± 0.873	1.7	8-11
Mn	0.89 ± 0.0075	0.88 ± 0.0066	0.89±0.0063	0.59 ± 0.05	1.083 ± 0.278	0.863 ± 0.027	0.818 ± 0.037		<1.5
Mo	0.053 ± 0.0018	0.051 ± 0.0003	0.0796±0.0015	0.05 ± 0.01		0.061 ± 0.004	0.048 ± 0.012		<0.5
P	0.030 ± 0.0015	0.028 ± 0.0010	0.0277±0.0004	0.47 ± 0.08	0.035 ± 0.003	0.049 ± 0.002	0.036 ± 0.01		<0.04
Si	0.37 ± 0.023	0.40 ± 0.0043	0.404±0.003	1.59 ± 0.19	0.540 ± 0.199	0.445 ± 0.004	0.381 ± 0.02		<2.0
Al	0.007 ± 0.0001	0.005 ± 0.0001	0.004±0.0003			0.004 ± 0.0			
Cu	0.19 ± 0.0075	0.18 ± 0.0030	0.184±0.003	0.16 ± 0.01		0.247 ± 0.005	0.18 ± 0.014		
Nb	< 0.02	0.008 ± 0.0011							
Ti	< 0.005	0.003 ± 0.0003	<0.0005						
V	0.070 ± 0.0019	0.066 ± 0.0011	0.0696±0.0008			0.088 ± 0.004			
Co			0.179±0.0019			0.239±0.005			
W			0.0075±0.0017						
Sn			0.005±0.0004						
Se			0.022±0.0033						
Ta			0.054±0.0019						
K						0.004±0.001			
Ca								0.2	
C	0.056 ± 0.0001	0.056 ± 0.0013	0.0501±0.0022		0.0497 ± 0.0059			15.7	<0.08
S	0.003 ± 0.0003	0.003 ± 0.0001		0.0 ± 0.05	0.003 ± 0.001		0.006 ± 0.004		<0.04
O								6.9	
N			0.065±0.0027						
H (ppm)									

Light Elements

Instrumental Gas Analysis (IGA)

CF8 – stainless steel	IGA	IGA-2nd lab	nominal
C	0.055±0.001	0.0602 ± 0.0088	<0.08
S	0.0029±0.000	0.0042 ± 0.0003	<0.04
O	0.020±0.0002	0.0254 ± 0.0014	
N	0.052±0.0002	0.0522 ± 0.0006	
H (ppm)	4.27±1.4		

	C	S	O	N
Kovar Fine	0.0093+/-0.0005	0.0028+/-0.0000	0.6470+/-0.0019	0.0019+/-0.0000
Kovar Medium	0.0054+/-0.0002	0.0022+/-0.0002	0.3910+/-0.0014	0.0025+/-0.0001
Kovar Course	0.0027+/-0.0004	0.0027+/-0.0003	0.1860+*-0.0011	0.0033+/-0.0003

KOVAR	weight %
Nickel	29
Iron	53
Cobalt	17
Carbon	<0.04
Manganese	<0.5
Silicon	0.2
Aluminum	<0.1
Chromium	<0.2
Magnesium	<0.1
Zirconium	<0.1
Titanium	<0.1
Copper	<0.2
Molybdenum	<0.2

Aluminum alloy

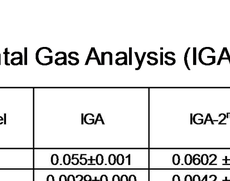
	additive AlSi10Mg
Al	90.2
Si	9.5 ± 0.14
Fe	0.099 ± 0.031
Ni	0.007 ± 0.0
Ti	0.009 ± 0.001
Mg	0.073 ± 0.001
V	*0.015 ± 0.001*
Ga	*0.004 ± 0.0*
C	0.004 ± 0.001
S	<0.003
O	0.108 ± 0.007
N	<0.003
g/cm³	2.577

AlSi10Mg	Nominal Composition	
	Min. %	Max. %
Aluminum	87.1	89.35
Silicon	9	11
Iron	0.55	0.55
Nickel	≤0.05	≤0.05
Titanium	≤0.15	≤0.15
Magnesium	0.2	0.45
Zinc	*≤0.1*	*≤0.1*
Lead	*≤0.05*	*≤0.05*
Tin	*≤0.05*	*≤0.05*
Copper	*0.05*	*0.05*
Manganese	*0.45*	*0.45*

literature density = 2.67 g/cm³

Titanium alloy

	Ti64	additive Ti64
Ti	92.5	91.9
Al	3.2 ± 0.05	3.68 ± 0.06
V	4.00 ± 0.03	3.91 ± 0.05
Fe	0.199 ± 0.016	0.07 ± 0.001
Hf		0.174 ± 0.001
Zr		0.058 ± 0.001
Ta		0.031 ± 0.0
Ni	0.011 ± 0.0	
C	0.012 ± 0.0047	0.0121 ± 0.0054
S	< 0.003	< 0.003
O	0.0906 ± 0.0064	0.1150 ± 0.0067
N	< 0.003	< 0.003
g/cm³	4.402	4.368



Ti 6Al-4V		
	Min. %	Max. %
Titanium	88	91
Aluminum	5.5	6.75
Vanadium	3.5	4.5
Iron		0.4
Hydrogen		0.015
Carbon		0.1
Nitrogen		0.05
Oxygen		0.02
Other, Total		0.4

Stainless Steel

	316L	additive 316L
Fe	67	68.3
Cr	16.39 ± 0.11	16.3 ± 0.11
Ni	11.87 ± 0.08	9.86 ± 0.07
Mo	2.90 ± 0.09	2.42 ± 0.05
Mn	1.06 ± 0.01	1.53 ± 0.01
Si	0.52 ± 0.01	0.31 ± 0.01
Co	0.08 ± 0.00	0.53 ± 0.00
W	0.04 ± 0.00	0.11 ± 0.00
Nb	0.02 ± 0.00	0.07 ± 0.00
P	0.04 ± 0.01	0.07 ± 0.00
Cu	0.01 ± 0.00	0.42 ± 0.00
Sb		0.02 ± 0.00
Sn		0.01 ± 0.00
C	0.0220 ± 0.0080	0.0200 ± 0.0080
S	0.0049 ± 0.0004	0.0069 ± 0.0009
O	0.0051 ± 0.0009	0.3090 ± 0.0008
N	0.0410 ± 0.0012	0.0154 ± 0.0031
g/cm³	7.948	7.905

316L:- specification		
	Min. %	Max. %
Iron	62.045	69.045
Nickel	10	14
Chromium	16	18
Molybdenum	2	3
Manganese	2	2
Carbon	0.03	0.03
Phosphorus	0.045	0.045
Sulfur	0.03	0.03
Silicon	0.75	1
Nitrogen	0.1	0.1

U.S. DEPARTMENT OF ENERGY

NNSA National Nuclear Security Administration

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