Microstructure & Mechanical Properties of Direct Metal Laser Sintered (DMLS) CoCrMo in the As-Printed and Heat Treated Condition

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as part of Summer Internship at INTECH, DMLS
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Work carried out at Materials Engineering Department, IISc, Bangalore

July 13th 2018

May 14th 2018 – July 14th 2018

INDIAN INSTITUTE OF SCIENCE
भारतीय विज्ञान संस्थान

ACKNOWLEDGEMENTS

INTECH DMLS PVT LTD
Sridhar Balaram
Naniah M A
Raghunandan M
Chidanand Hiremath
Rekha Rs
Production Team INTECH-DMLS

Indian Institute of Science (IISc)	
Prof Suryasarathi Bose	Project Faculty
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Pooja S G	SEM
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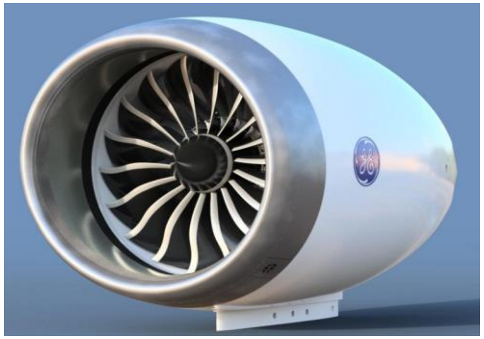
Shashidhara S	Tensile Tests
Sandeep, Ananth Prof. A S ShetraBalan	AIMIL (Particle Size Distribution) VIT (Surface Roughness)

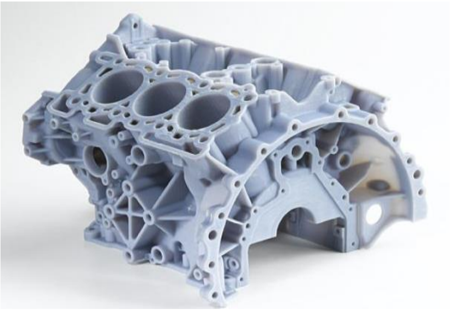
Background on Additive Manufacturing

Additive Manufacturing technology is seen as an emerging technology for repair as well as new make (near net shape) applications. The advantages of the process are in being able to result in near net shape, complex parts, with weight savings

Additive Manufacturing Applications

Aerospace | **Healthcare** | **Automotive Parts**





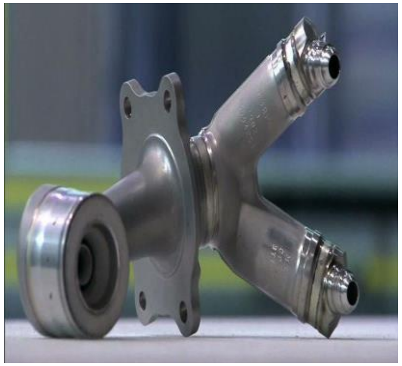
Background – CoCrMo

Cobalt-base alloys are generally described as non magnetic, wear, corrosion and heat-resistant (high strength even at elevated temperature). Many properties of the alloy originate from the crystallographic nature of cobalt, the solid-solution-strengthening effect of chromium and molybdenum, the formation of extremely hard carbides and the corrosion resistance imparted by chromium.

Due to its excellent resistance to degradation in the oral environment, the first medical use of cobalt-base alloys was in the cast of dental implants. Today, the use of Co alloys for surgical applications is mainly related to orthopaedic prostheses for the knee, shoulder and hip as well as to fracture fixation devices. Joint endo-prostheses are typical long-term implants and the applied implant material must therefore meet extremely high requirements with regard to biocompatibility with the surrounding body tissue material and corrosion resistance to body fluids.

Objective – Characterization of DMLS CoCrMo

- To optimize parameters for dense part in the As-Printed sample without any Hot Isostatic Pressing (HIPing).
- To characterize DMLS CoCrMo (Cobalt-Chromium-Molybdenum) and evaluate its suitability for orthopaedic implant materials.



Properties of CoCrMo

1) **High Strength**
2) **High Fatigue Resistance**
3) **Biocompatibility**
4) **High Resistance to Wear and Corrosion.**

The objective of this analysis is to characterize Direct Metal Laser Sintered CoCrMo in the as printed and heat treated condition, for Praxair (powder and bulk)

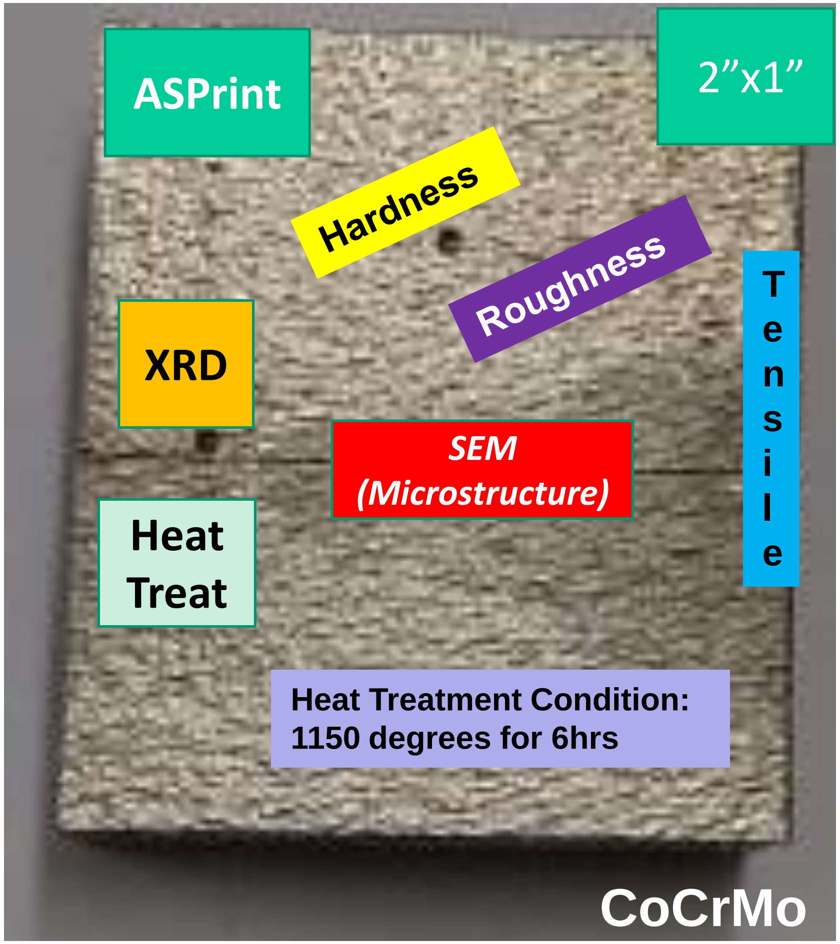
a. **Roughness**
b. **Hardness**
c. **Microstructure (porosity, particle size analysis, chemistry and phases)**
d. **X-ray diffraction (phase analysis)**
d. **Tensile strength (Room Temperature) - small scale testing**

in order to serve as a baseline for building various products

AM Fuel Nozzle used in General Electric's LEAP Jet Aircraft engine. One of the greatest inventions of additive manufacturing. (Source: General Electric Company, All rights reserved)

Characterization DOE

Particle Size Analyser	•Malvern 3000; Dry Method; Size Range (10nm to 3.5mm)
Surface Roughness	• Mahr MarSurf GD 120; Measuring Speed: 0.1mm/s • Traversing Length: 1.75mm
Microstructure	•Zeiss Axiocam HRc with Axiovision & ImageJ software
Microhardness	•Future Tech FM 800 Tester Load=300gf; Vickers Method
SEM	•FEI Quanta200 •ThermoFisher EDS
X-Ray Diffraction	•Xpert PRO PANAnalytica Cu K_{α} λ=1.54 Å ; Angle Range (20-100) deg, Step Size: 0.033, Step Time: 118.5 seconds
Tensile Strength	• Instron 5987, 2 KN, Strain Rate : 0.006mm/s ;
Heat treatment	Delta Power Tools High Temperature Tubular Furnace

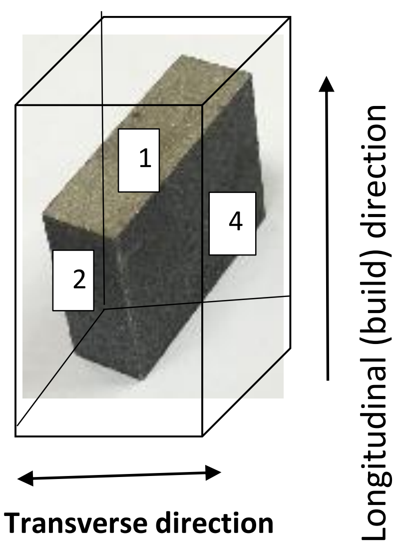


Etchant for CoCrMo As-Printed/HeatTreated Electrolytic etching with 5% HCl at 6V

Nominal Composition of CoCrMo (wt%) *from EOS Data Sheet*

Element	Co	Cr	Mo	Mn	Si	Fe	C	Ni
Percentage	60-65	26-30	5 to 7	<1	<1	<0.75	<0.16	<0.1

Process Parameters for DMLS sample	May not be accurate
Laser Power	290W
Beam Diameter	80μm
Layer Thickness	40μm
Hatch Distance	110μm
Scan Speed	950mm/s
Powder Size	35μm



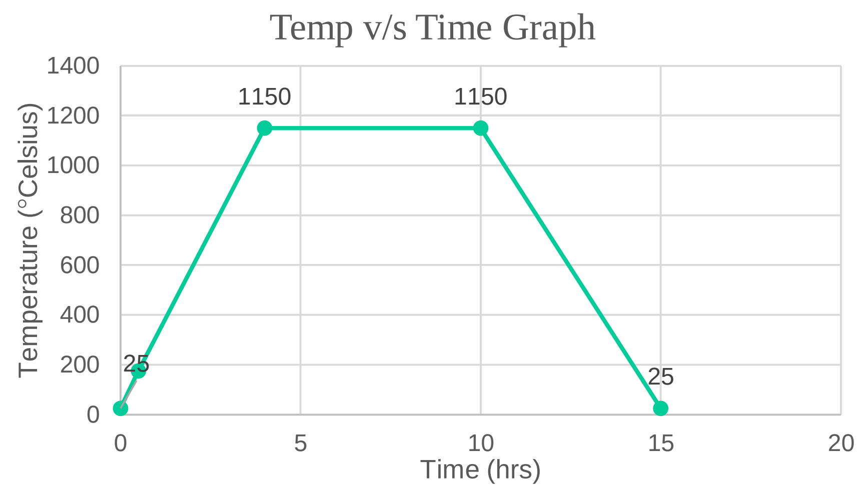
Legend: Cube

1 Top surface (where name/code is)

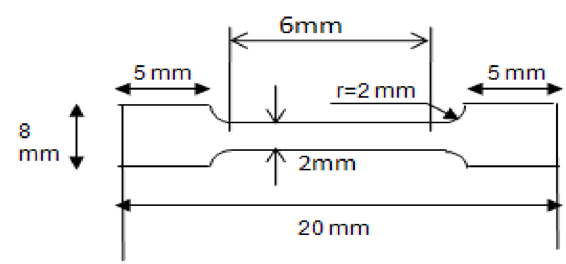
2 Transverse surface used

4- Side surface (Any side can be Longitudinal)

Heat Treatment Process

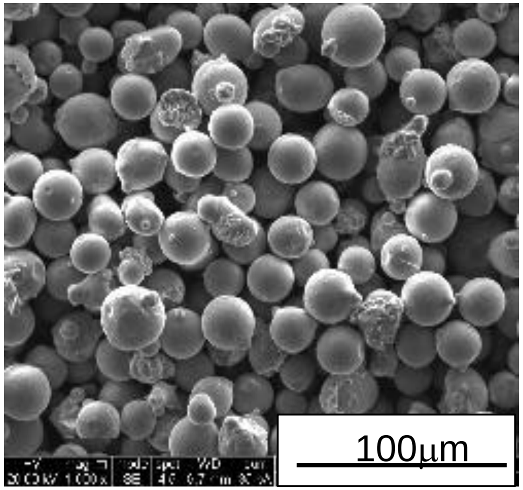


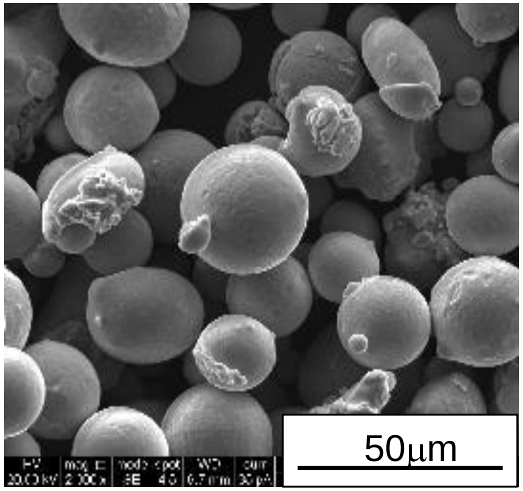
Tensile Sample



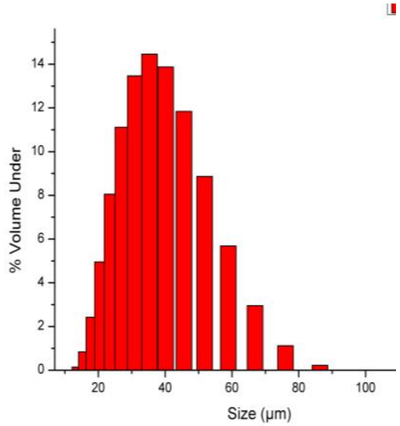
GaugeLength:6mm
Thickness: 0.5mm
StrainRate: 0.006 mm/s

Powder Analysis



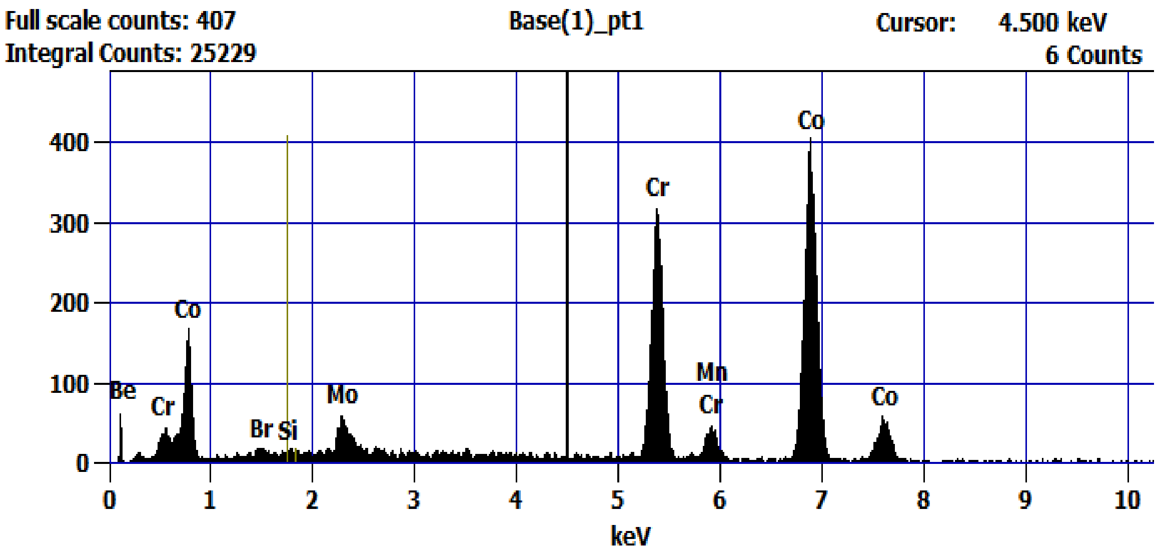


Particle Size Distribution



Particle Range	Avg.Size µm
Dv10	21.32
Dv50	32.96
Dv90	50.81

Energy Dispersive Spectroscopy (EDS)



Composition of CoCrMo (wt%)

Co	66.74
Cr	28.91
Mo	4.14
Mn	0
Si	0.21

Average Particle Size was found to be approx : 33 µm

Surface Roughness

Glass bead blasting conditions

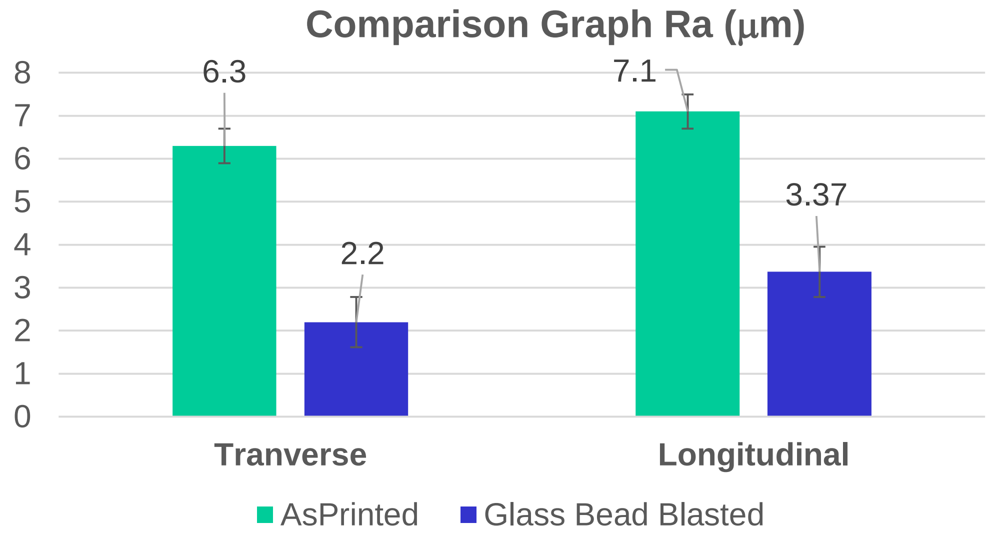
Pressure	7.5 bar
Time	5min
Distance bw gun & workpiece	Average 155mm
Angle bw gun and workpiece	45 deg

Glass bead Blaster

$$R_a = \frac{1}{n} \sum_{i=1}^{n} |y_i|$$

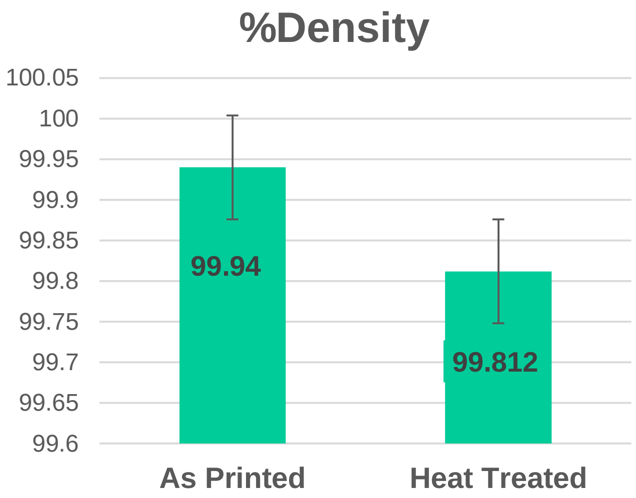
As-Printed

Grit Blasted

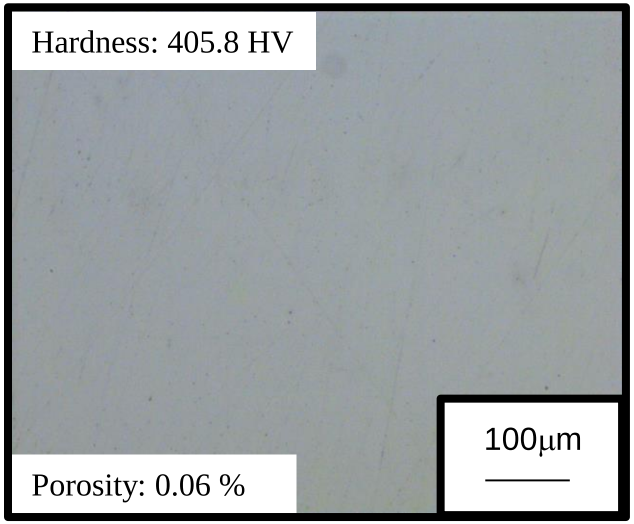


Roughness decreases by almost 60% upon glass bead blasting in both Transverse and Longitudinal directions

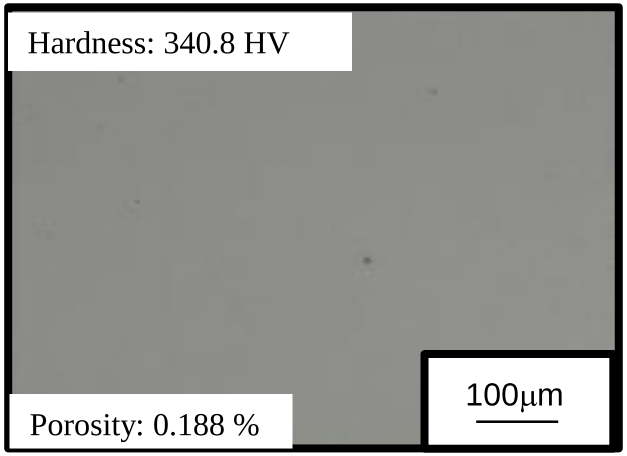
Porosity Optical Images



As-Printed



Heat-Treated

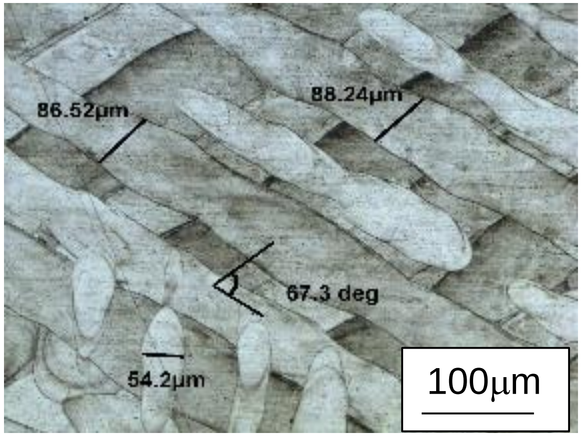


% Porosity:

The porosity of the As-Printed sample
As-Printed: 0.06 %
Heat Treated: 0.188%
Porosity increases upon Heat Treatment

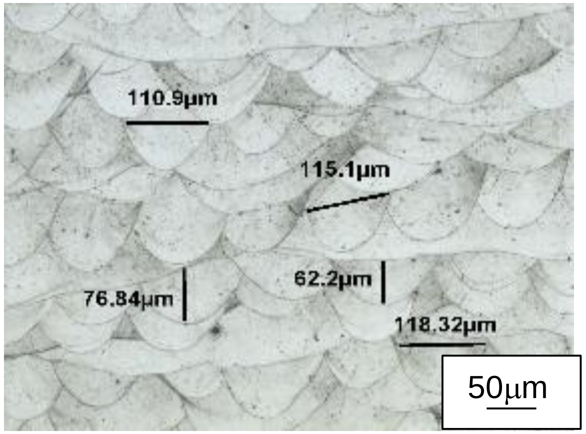
Optical Microstructure

Transverse



Average width : 76.32 microns
Angle between layers of 3D printed sample as measured was: 67.3 degrees

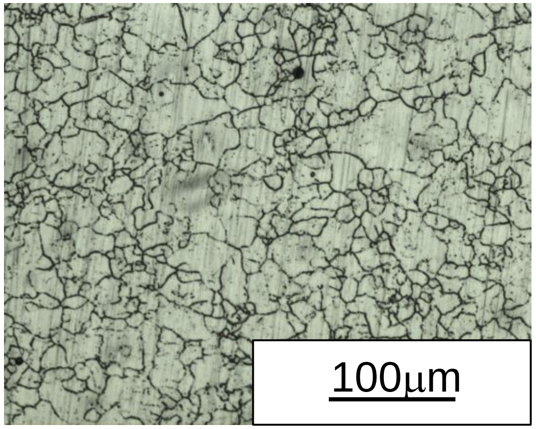
Longitudinal



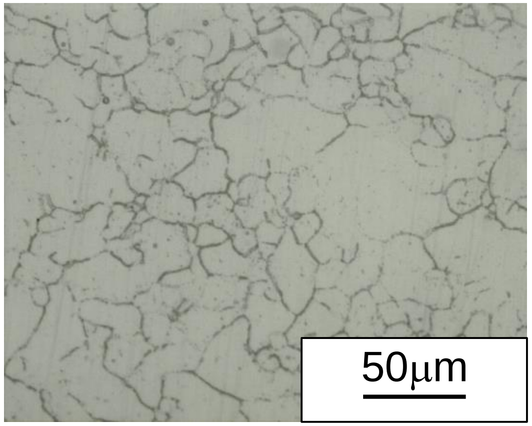
Average Diameter: 114.7 microns
Average Depth: 69.5 microns

As-Printed

Transverse



Longitudinal

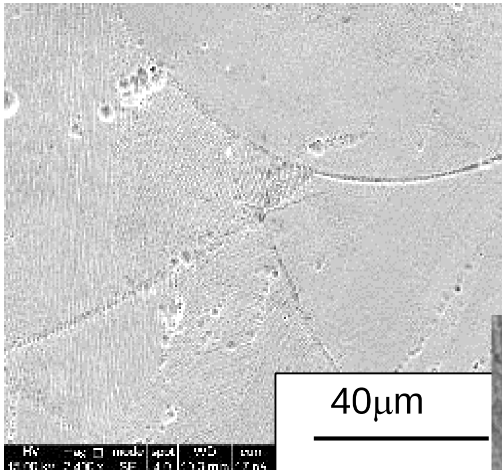


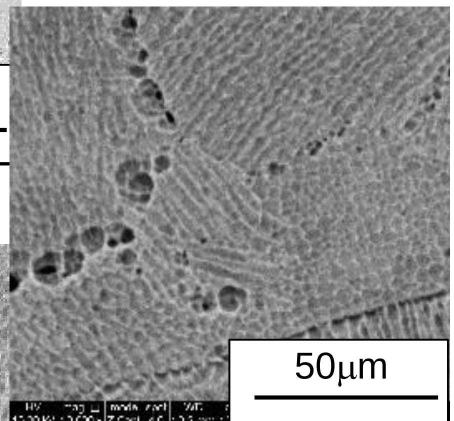
Heat Treated

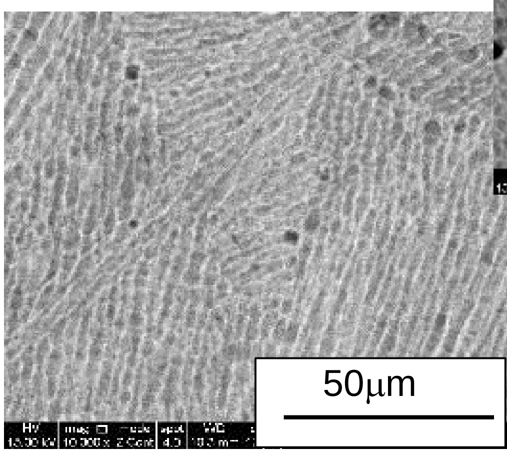
Average grain size (approx. diameter equals 40-45 µm)

Scanning Electron Microscope Images (Microstructure)

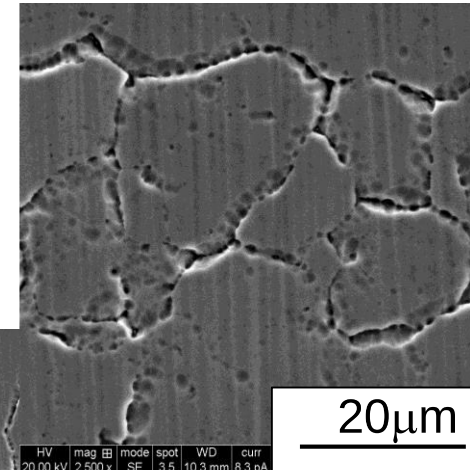
As-Printed

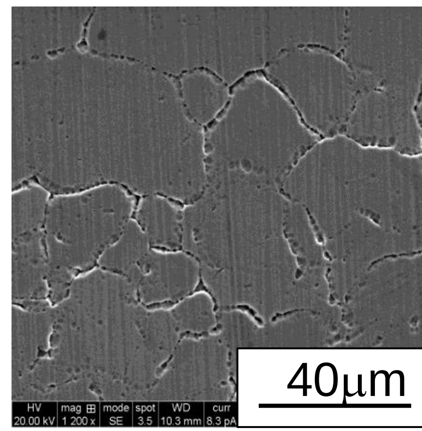


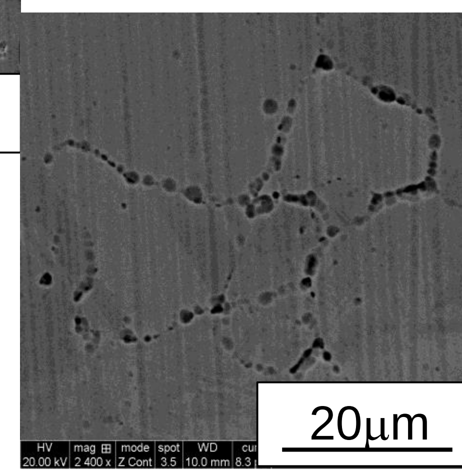




Heat Treated



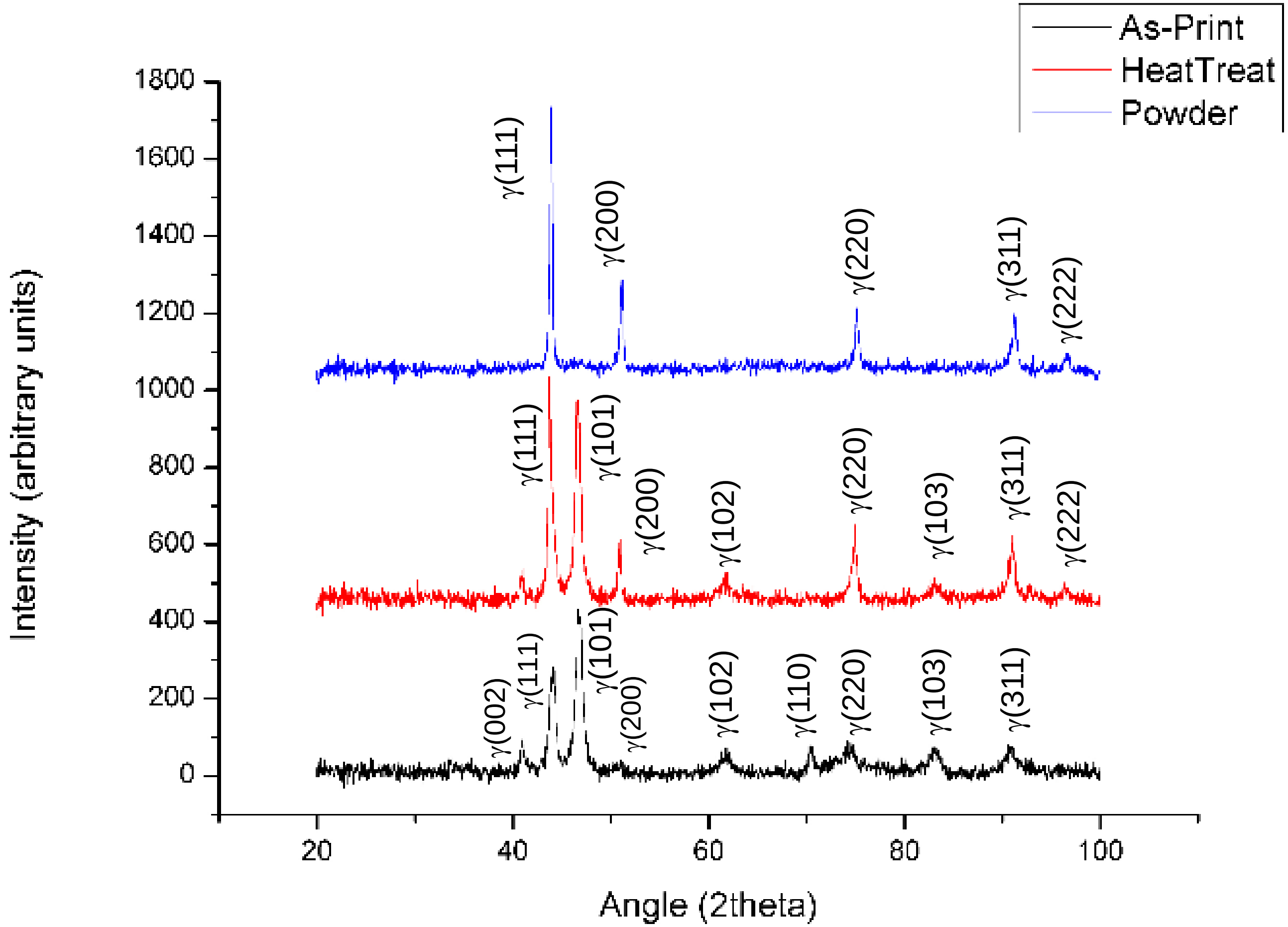




Dendritic structures and Melt Pool Boundaries are seen in As-Printed images. Upon Heat Treatment, the sample gets homogenized, hence grain size becomes independent of melt pool size.

X-Ray Diffraction

Parameter	Values
Starting angle (deg)	20
Finishing angle (deg)	100
Wavelength (Å) Cu K alpha	1.54
Voltage(KV)	40
Current (mA)	30
Step size(deg)	0.003



As-Printed has HCP and FCC phases

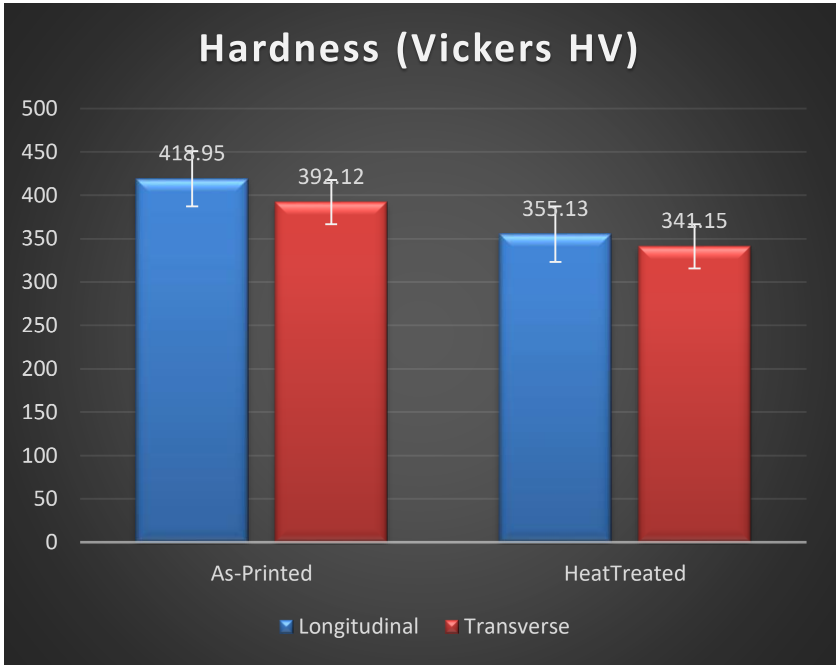
Heat Treated majorly has FCC phases

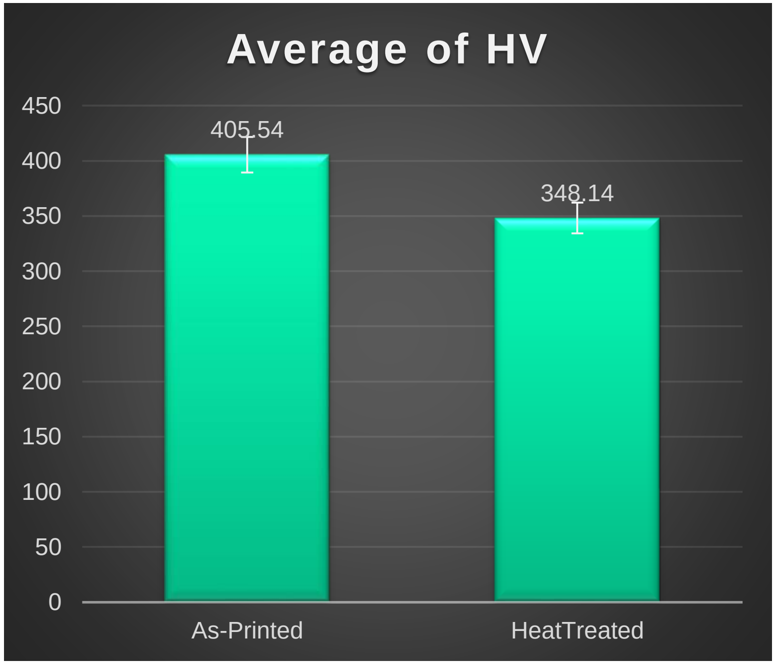
Referring to close matches in

1) ***ICDD 00-005-726 for ASP and HT***
2) ***ICDD 00-015-0806 for powder***

The peaks are matched for γ FCC and ε HCP phases of cobalt.

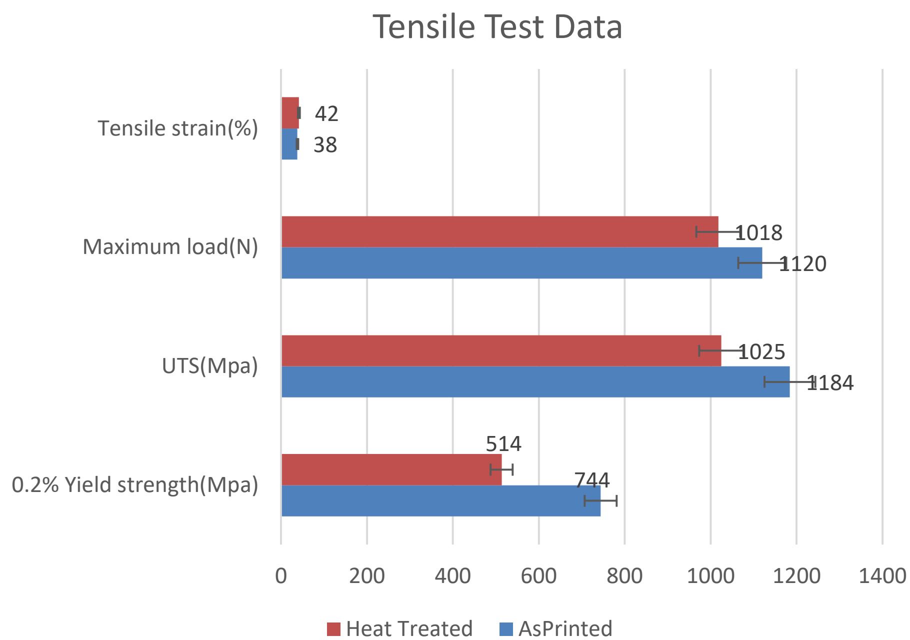
Hardness Measurements





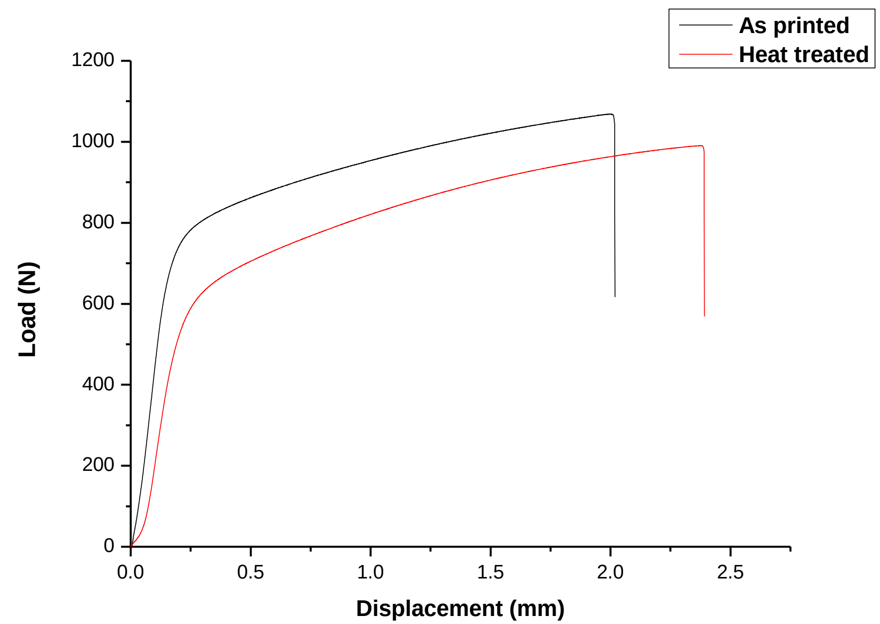
Hardness decreases upon Heat Treatment due to formation of equiaxed structure upon recrystallisation. The residual stress gets relieved upon heat crystallisation.

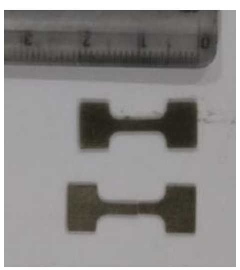
Tensile Testing- Room Temperature- Small scale test



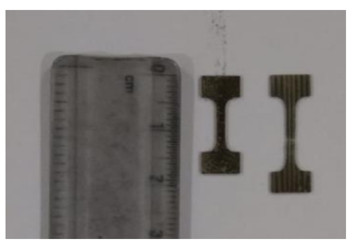
Load v/s Displacement

As Printed and Heat Treated CoCrMo plot





As Printed
Extension: 1.9mm



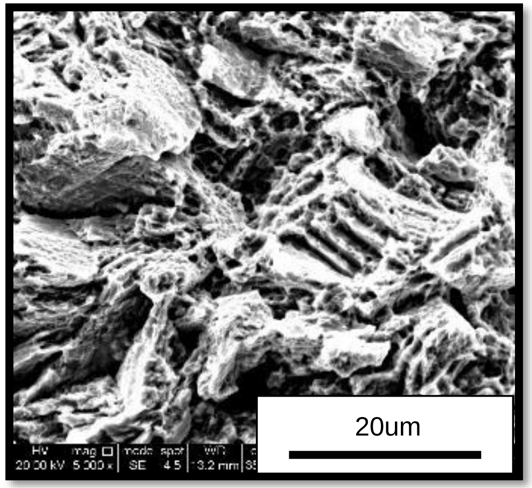
Heat Treated
Extension: 2.3mm

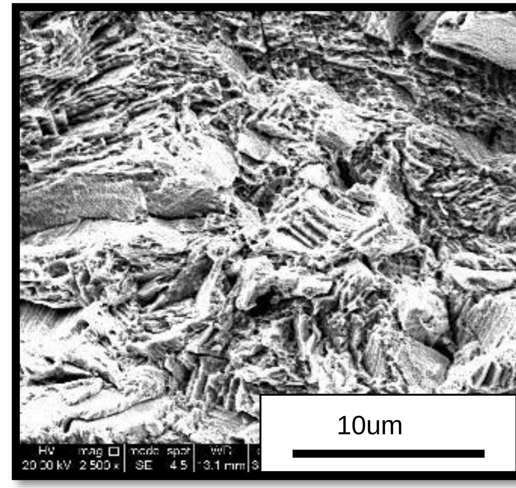
The As-Printed sample has higher 0.2% yield strength than Heat Treated sample (difference >200MPa)
The small scale tensile test results match with EOS regular size sample standards.

%Elongation: As-Printed= 38% and Heat Treated=42%

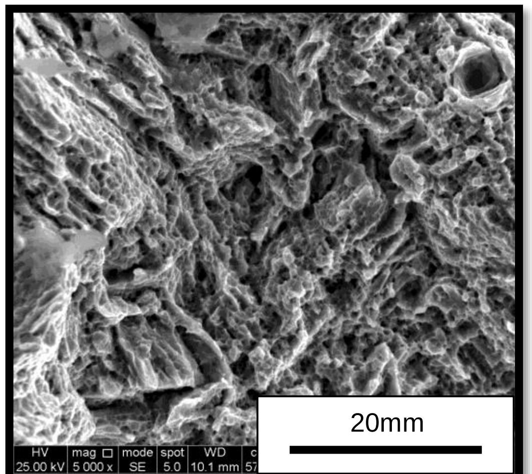
Fractographs

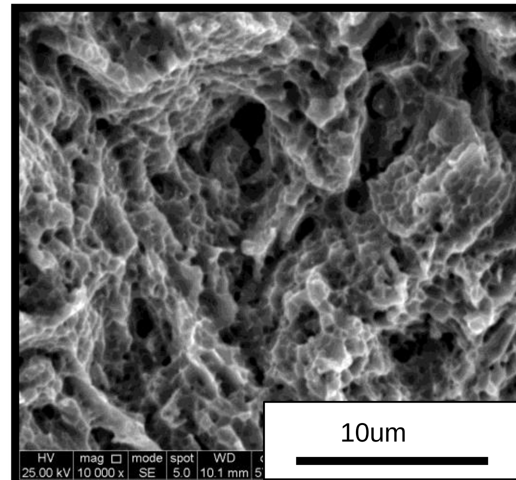
As-Printed





Heat Treated



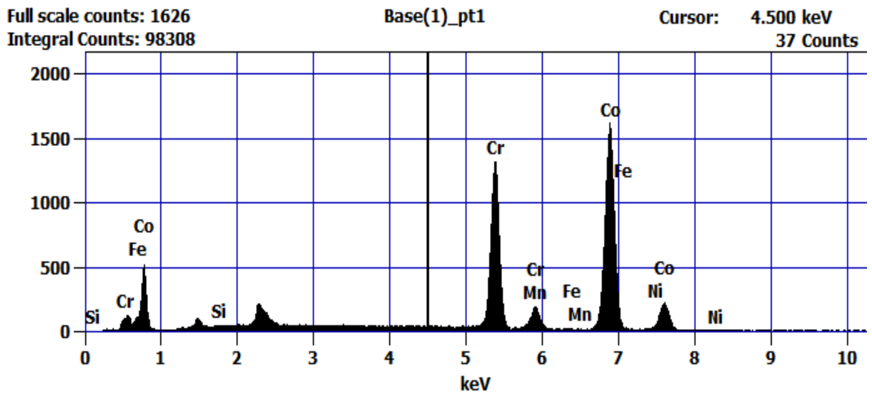


Some lamellar tearing can be seen in the as printed fractographs , corroborating with the presence of the hcp-beta phase in the as printed microstructure. In some sense it appears like a cleavage fracture in appearance characterized by a well defined crystallographic appearance in the facets. Upon heat treatment the occurrence of an equiaxed recrystallized microstructure renders the alloy with a ductile matrix as indicative of the heat treated fractographs, corresponding to a much higher ductility of 42%.

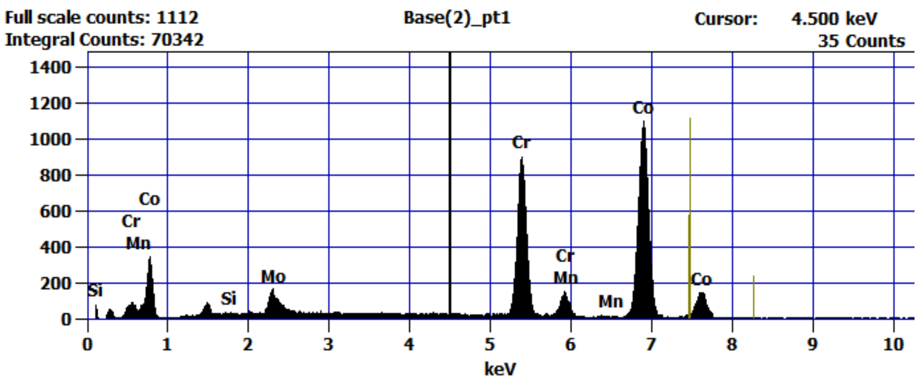
EDS Summary

Weight %	Sample Type					
Elements	Powder	AsPrinted		Heat Treated		Nominal Composition
		Transverse	*Longitudinal*	*Transverse*	*Longitudinal*	
Cobalt	**66.74**	**64.21**	**64.23**	**65.74**	**66.1**	62-66%
Chromium	**28.91**	**28.67**	**28.1**	**29.42**	**28.86**	27-30%
Molybdenum	**4.14**	**6.8**	**6.96**	**4.6**	**4.21**	5-7%
Silicon	**0.21**	**0**	**0.55**	**0.25**	**0.35**	<1%
Manganese	**0**	**0.15**	**0.16**	**0**	**0.49**	<1%
Iron		**0.17**				<0.75%
Carbon						<0.35%
Nickel						<0.5%

As-Printed



Heat Treated



Executive Summary

Attribute	Overall	As-Printed (μm)	Heat Treated (μm)
Powder Particle size	33μm		
Surface Roughness (As-Printed)	6.7	6.7	
Surface Roughness (Glass Blasted)	3		
Porosity		0.06%	0.19%
Weld Pool: Avg. width	76.3μm		
Angle bw layers	67.3 deg		
Weld Bead: Avg. Diameter	114.7μm		
Weld Bead: Avg. Depth	69.5μm		
XRD		ε HCP & γ FCC phase	γ FCC phase
Hardness		405 HV	348 HV
% Elongation		38%	42%

References:

1) http://www.georgevandervoort.com/metallography/
2) https://www.finishing.com/
3) www.metallographic.com/Etchants/Etchants.htm
4) https://www.cartech.com/
5) *'Microstructural Evolution and Mechanical Properties of Direct Metal Laser-Sintered (DMLS) CoCrMo After Heat Treatment '* Bawane et.al **Metallurgical and Materials Transactions A, 2018**; DOI 10.1007/s11661-018-4771-4
6) '*Study of Process Parameter and Powder Variability on the Properties and Recrystallization Behavior of Direct Metal Laser Sintered CoCrMo'* **Santhosh Kumar Rao Chandrasekara, Anigani Sudarshan Reddy, Dheepa Srinivasan, Durga Ananthanarayanan; Proceedings of the ASME 2017 Gas Turbine India Conference**