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ECOPARTS ADDITIVE METAL SOLUTIONS

EOS StainlessSteel 17-4PH



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EOS StainlessSteel 17–4PH is a stainless steel powder intended for manufacturing parts on EOS metal systems with EOS DMLS processes.

This document provides information and data for parts built using EOS StainlessSteel 17–4PH powder EOS art.-no. 9011–0041 on the following system sp e cif ic ations:

DMLS® System: EOS M 290

- \rightarrow Ceramic blade (2200-3013)
- → Grid nozzle (2200-5501)
- → IPCM M extra Sieving Module with 75µm mesh size (200000315) recommended

Manual sieve with 75µm mesh size (200000321) recommended; standard manual sieve with 80µm mesh possible

→ Argon atmosphere

Software: EOSYSTEM 2.5 or newer / EOSPRINT 1.5 or newer EOS Parameter Set: 17–4PH 40µm Stainless

 \rightarrow (Default Job: 17-4PH_040_StainlessM291_100)

Description

Precipitation hardening steels are widely used in engineering applications, which require corrosion resistance and strength. Parts built from EOS StainlessSteel 17-4PH can be machined, shot-peened and polished in as-built or heat treated states. Solution annealing together with ageing treatment are necessary in order to achieve proper hardness and mechanical properties (ASTM A564 – 13). Due to the layerwise building method, the parts have a certain anisotropy which can be eased by solution annealing.

Quality Assurance of EOS StainlessSteel 17–4PH powder material

The quality of the delivered EOS StainlessSteel 17-4PH powder lots is ensured by the Quality Assurance procedures which are part of EOS Quality Management System. The procedures include quality assurance of both the powder and process.

Quality assurance of the powder product includes: \rightarrow sampling (ASTM B215)

- → sieving (ASTM B214)
- \rightarrow particle size analysis (ASTM B822)
- → chemistry analyses (ASTM E2823/E1479/E1019)
- \rightarrow apparent density (ASTM B212/B329/B417)

Heat treatment

Vacuum H900 heat treatment procedure:

- → Solution annealing: Hold at 1040°C (1904°F) ±15°C (± 59°F) for 30 minutes, air cooling under 32°C (89°F).
- → Ageing: Hold at 480°C (896°F) for one hour, air cooling under 32°C (89°F).

Atmospheric HT procedure (preferred atmosphere: Argon):

- → Solution annealing: Hold at 1040°C (1904°F) ±15°C (± 59°F) for 30 minutes, air cooling under 32°C (89°F).
- → Ageing: Hold at 460°C (860°F) for one hour, air cooling under 32°C (89°F).

The quality of the process is assured with each delivered powder lots by building a quality assurance job with a qualified EOS M 290 system.

Process quality is assured by:

- → tensile tests (ISO6892, ASTM E8M)
- \rightarrow density measurement (ISO3369)
- \rightarrow hardness measurement (ISO 6508)
- \rightarrow chemistry analysis of the solid part
 - (ASTM 2823/E1479/E1019).

The results of the quality assurance tests are given in the lot specific Mill Test Certificates (MTC) according to EN-10204 type 3.1.

Technical Data	Μ
Powder properties	E
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The chemical composition of the	+
powder is in compliance with	+
standards "F899 – 12b Standard	
Specification for Wrought Stainless	
Steels for Surgical Instruments" and	
"A564M – 13 Standard Specification	
for Hot-Rolled and Cold-Finished	
Age-Hardening Stainless Steel Bars	
and Shapes".	

Material composition

Element	Min.	Max.
Cr Ni Cu Si Mn C P S Nb	15.0	17.50
Element Cr Ni Cu Si Mn C P S Nb + Ta	0	5.00
	3.00	5.00
	3.00	1.00
		1.00
		0.07
	0.15	0.040
		0.030
		0.45

Acc. to standard

Particle size

D50 [1] Particles	36-44 µm approx. 1.4-1.7 · 10-3 inch
>53µm [2]	Max 6.0 wt%
Particles >63µm [2]	Max 1.0 wt%
Powder density Apparent density [3]	Mean 3.83 g/cm3 Mean 13.84 lbs/in3
Tap density ^[4]	Mean 4.7 g/cm3 Mean 1.7 lbs/in3

 $^{\rm [1]}$ According to ASTM B822 [2] According to ASTM B214. According to ASTM B212, $^{\rm [3]}$ ASTM B329 & ASTM B417. [4] According to ASTM B527.

General process data

Layer thickness	40 μm 1.6 · 10–3 inch
Volume rate ^[5]	3.32 mm ³ /s (11.95 cm ³ /h) 0.73 in ³ /h

[5] The volume rate is a measure of build speed during laser exposure of the skin area. The total build speed depends on this volume rate and many other factors such as exposure parameters of contours, supports, up and downskin, recoating time, Home-In or LPM settings.

Physical and chemical properties of parts1

The chemical composition of parts is in compliance with standards "F899 – 12b Standard Specification for Wrought Stainless Steels for Surgical Instruments" and "A564M – 13 Standard Specification for Hot-Rolled and Cold-Finished Age-Hardening Stainless Steel Bars and Shapes". Composition complies the material composition in "powder properties" section. Part accuracy is adjustable by changing the "Beam Offset, X-, Y- and Z-Shrinkage"-parameters.

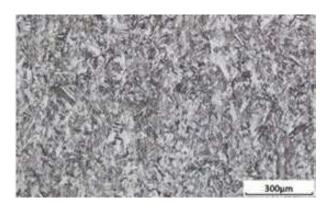
Part density [6]	Mean 7.79 g/cm3 Mean 28.14 lbs/in3
Part accuracy [7]	
Small parts	approx. \pm 50 µm approx. \pm 1.1 \cdot 10-3 inch
Min. wall thickness [8]	approx. 0.4 mm approx. 0.016 inch
Typical shrinkage after HT (for parts 50mm)	0.2%

Thermal expansion after atmospheric HT^[9]

25 – 100°C	10.4 10-6/K
25 – 200°C	11.0 10-6/K
25 – 300°C	11.4 10-6/K
25 – 400°C	11.8 10-6/K
25 – 500°C	12.0 10-6/K

Microstructure of heat treated parts

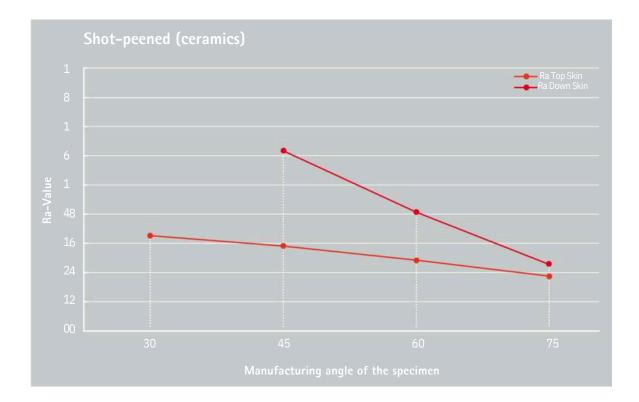
Average porosity [10]	0.030%
Average pore size [10]	7.2 µm
N (number of samples)	70



Atmospheric furnace (Atmospheric HT) was used to heat treat etched part. Etchant: Marble's reagent. 10 X magnification

Surface roughness after shot-peening (approx.) [11]

Horizontal	Ra 3.5 – 5.9 μm; Rz 17.3 – 27.7 μm Ra 0.12 – 0.20 · 10-3 inch; Rz 0.67 – 1.06 · 10-3 inch
Ver tic al	Ra 3.4 – 5.5 μm; Rz 15.9 – 28.5 μm Ra 0.12 – 0,20 · 10-3 inch; Rz 0.59 – 1.12 · 10-3 inch
Angled surfaces	Surface roughness measured in a function of manufacturing angle



⁽⁶⁾Weighing in air and water according to ISO 3369. [7] Based on users' experience of dimensional accuracy for typical geometries, e.g. ± 50 µm when parameters can be optimized for a certain class of parts or ± 70 µm when building a new kind of geometry for the first time. Part accuracy is subject to appropriate data preparation and postprocessing. [8] Mechanical stability is dependent on geometry (wall height etc.) and application. [9] According ASTM E228. [10] Porosities were measured from 15x15mm cross cuts using optical microscope according to internal procedure. Average porosity and pore size value depends on the job load. [11] Measurement according to ISO 4287. The numbers were measured at the horizontal (up-facing) and vertical surfaces of test cubes. Due to the layerwise building the roughness strongly depends on the orientation of the surface, for example sloping and curved surfaces exhibit a stair-step effect. Angles under 45° should be supported.

Statistical analysis of part properties1

Process and powder validation was performed using several powder lots and EOS M 290 systems. Number of samples used in process and powder validation are shown in tables below. All heat treated mechanical properties showed over 3 sigma performance against ASTM A564M H900 requirement. Vacuum H900 validation data includes two EOS M 290 systems and four powder lots. Atmospheric HT validation data includes one EOS M 290 system and two powder lots.

	As built	Vacuum H900	Atmospheric HT	ASTM A564 (H900)
Ultimate tensile strength, Rm		4 Sigma		
In horizontal direction (XY)	Mean 886.0 MPa	Mean 1335.8 MPa	Mean 1340.0 MPa	
	StDev. 70.4 MPa	StDev. 5.2 MPa	StDev. 5.9 MPa	min. 1310 MPa
N (number of samples)	72	144	36	
	Mean 924.2 MPa	Mean 1342.6 MPa	Mean 1345.5 MPa	min. 1310 MPa
In vertical direction (Z)	StDev. 65.9 MPa	StDev. 7.7 MPa	StDev. 2.8 MPa	
N (number of samples)	84	168	42	
Yield strength, Rp 0.2		6 Sigma		
In horizontal direction (XY)	Mean 860.6 MPa	Mean 1235.2 MPa	Mean 1235.5 MPa	
	StDev. 75.7 MPa	StDev. 9.8 MPa	StDev. 8.7 MPa	
N (number of complex)	72	144	36	
N (number of samples)	Mean 861.3 MPa	Mean 1250.7 MPa	Mean 1242.6 MPa	
In vertical direction (Z)	StDev. 44.7 MPa	StDev. 13.5 MPa	StDev. 10.1 MPa	min. 1170 MPa
	84	168	41	
N (number of samples)	04	100	41	
Elongation at break A		Almost 4 Sigma		
In horizontal direction (XY)	Mean 19.9%	Mean 14.0%	Mean 13.5%	min. 10%
	StDev. 1.2%	StDev. 0.8%	StDev. 0.9%	
N (number of samples)	72	144	36	
In vertical direction (Z)	Mean 20.1%	Mean 13.5%	Mean 12.6%	min. 10%
	StDev. 1.5%	StDev. 0.7%	StDev. 0.9%	
N (number of samples)	84		42	
Hardness HRC		Almost 4 Sigma		
IHardne s ^{g3]}	Mean 23.9 HRC	4 Sigma Mean 42.1 HRC	Mean 42.1 HRC	min. 40 HRC
	StDev. 3.6 HRC	StDev. 0.5 HRC	StDev. 0.5 HRC	11111. 40 T IIIC
N (number of complete)	20	40	10	
N (number of samples)	20	40	TO	

Mechanical properties at room temperature^[12]

^[12] Tensile testing according to ISO 6892 & ASTM E8M. ^[13] Rockwell Hardness, HRC, according to ISO 6508.

Additional information1

Modified Heat treatment

Modified heat treatment may improve properties further. Lower aging temperature 460°C (860°F) has proven to be more suitable for DMLS[®] manufactured 17-4PH. Tensile data with vacuum furnace – Solution anneal as [14] following by ageing in 460°C.

	Vacuum 460°C ^[14]	
Ultimate tensile strength, Rm		
Both directions	Mean 1358.1 MPa	
	StDev. 6.7 MPa	
N (number of samples)	39	
Yield strength, Rp0.2		
Both directions	Mean 1262.4 MPa	
	StDev. 12.9 MPa	
N (number of samples)	39	
Elongation at break, A		
In horizontal direction (XY)	Mean 13.8%	
	StDev. 0.6%	
N (number of samples)	39	
Hardness, HRC		
Hardne s s	Mean 42.8 HRC	
	StDev. 0.3 HRC	
N (number of samples)	5	

Cytotoxicity

Cytotoxicity tests were done according to ISO 10993-5. It included growth inhibition tests evaluated from two endpoints (XTT & BCA). Tests were done with as-manufactured cubes. EOS StainlessSteel 17-4PH cubes were extracted under agitation for 24±2h with DMEM 10% FBS. L929 cells were then incubated for 68 – 72h with the following concentrations of the test extract: 13.2%, 19.8%, 29.6%, 44.4%, 66.7% and 100%. Surface/volume ratio used was 3cm2/mL.

Cytotoxicity results

In this study under the given conditions no leachable substances were released in cytotoxic concentrations from the test item as confirmed by two different endpoints (XTT, BCA).

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