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DE LA RECHERCHE À L'INDUSTRIE

<u>Ceaden</u>

INFLUENCE OF PROCESS PARAMETERS ON THE FINAL 316L STAINLESS STEEL PROPERTIES MANUFACTURED BY SELECTIVE LASER MELTING (SLM)

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Ceaden INTRODUCTION: CONTEXT

Additive Manufacturing: growing technology in the industrial world for different sectors: aeronautics – automotive – medicine.

- Nuclear industry? Exploring by DOE (U.S.A) since 2012 for different materials: IN600, IN718, IN800, 316L SS, ODS steels.
- Benefits:
- \rightarrow Complex manufacturing of geometrical parts for nuclear applications.
- → Components manufacturing which are impossible to process by others traditional processes (cast, forge, rolling,...).
- → Processing steps reduction depending on parts complexity (i.e. GE-SAFRAN Leap-X fuel injector).
- \rightarrow Waste reduction: powder recyclability.
- \rightarrow Rapid prototyping.

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Question: Is additive manufacturing adapted for producing nuclear components? (GEN IV – Sodium Fast

Cooled Reactor).

- \rightarrow Material: 316L SS.
- \rightarrow Mechanical, corrosion and irradiation properties ?
- → <u>Understand relation between process and microstructure</u>.

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The influence of substrate temperature on the microstructure and mechanical properties of 316L SS samples







Ceaden 316L SS POWDER PROVIDED BY SLM SOLUTIONS

□ Chemical composition

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	Majority elements by ICP-OES (% wt) (uncertainly related of 3%)						Minority elements (%wt) By GDOES and LECO (uncertainly related of 5%)				
	Cr	Ni	Mn	Si	Мо	С	Ν	0	S	Р	
Standard RCC- MRx	16,5-18,50	10-13	2 max	1 max	2-2,5	300 max	1100 max	-	150 max	300 max	
316L SS powder	17,35	11,45	1,06	0,51	2,07	230	982	1876	130	305	

- ➤ Chemical composition in good agreement with RCC-MRx Code → French Nuclear Standard for Experimental Reactors
- Particles size



316L SS POWDER PROVIDED BY SLM SOLUTIONS

MAG: 500x HV: 15 kV WD: 10.8 mn

□ Morphology

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Argon atomized powder.

Typical heterogeneous shapes.



Rod-like particles





EXPERIMENTAL PROCESS: SELECTIVE LASER MELTING





- Selective laser melting
- Power max: 400 W
- Ytterbium doped laser fiber.
- Laser's spot size: 75 µm

✤ Fixed process parameters:

- → Power: 175W
- \rightarrow Layer thickness, e=30 µm
- \rightarrow Scan speed, v=700 mm/s
- \rightarrow Hatching distance, HD: 100 µm





 \rightarrow Scan Strategy:

incrementation of 67° between each layer



- ***** Variable process parameter:
- → Heating Platform

EXPERIMENTAL PROCESS: SELECTIVE LASER MELTING



Flow diagram

- Build enveloppe size: Ø=100 mm and height 100 mm.
- Aim: reduce thermal stresses and formation of cracks in the parts.



- Samples manufactured at different temperatures: → RT, 100°C, 200°C, 350°C, 500°C and 600°C.
- Cylindrical and Beam-like samples (X, Y, Z and 45° from XY plane).

Aim of In situ heat: →Decrease internal thermal stresses. →Grain sizes modification. →"In situ" heat treatment in order to reduce post process steps.





OUTLINE



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MICROSTRUCTURE

MECHANICAL PROPERTIES

Micro scale: Grains





- Columnar grains along B.D.: length around 4 times width.
- Grains grow across layers (30 μm).
- Grains are more elongated at 600°C on the BOTTOM (L=220 µm)
 - → Grains size influenced by the substrate temperature (Temperature higher at the bottom).
- No influence of temperature on grains width.

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MICROSTRUCTURE

Micro scale: Grains

MECHANICAL PROPERTIES



- No significant influence of the substrate temperature on the cells size:
- intra granular cells size between 0.6 and 1 µm.
- High cooling rate (10³-10⁶ K/s) - Casati and al. Journal of Meterials Science and Technology ,2016, 32: 738-744

Inside a same grain: (grain 2) Direction of growth of S/L interface can vary from one melt pool to another – Mertens and al., Materials Science, 2011, 783-786, 898-903

MICROSTRUCTURE

MECHANICAL PROPERTIES

Nano scale: Precipitates

- □ Analyse at the middle of the sample height (4.5 cm).
- □ No influence of the substrate temperature on:
 - > Structure: amorphous.
 - > Shape: spherical.
 - Size: diameter ranging 10 to 170 nm.
 - ➢ Composition: O, Si, Mn.

Trace of S, Mo and Al in some precipitates.

- ❑ Nano oxydes formation in situ during the build process → improve mechanical properties - K. Saeidi Ph. D. Thesis (2016).
- Precipitates size and density are highly influenced by the oxygen content in the build chamber Zhou et al. Mater. Sci. Eng., 2017, 167.
- □ Nano precipitates can improve swelling resistance under neutron irradiation *Sun et al., Sci. Rep., 2015, 5, 7801.*





MICROSTRUCTURE

MECHANICAL PROPERTIES

Micro Hardness



 \rightarrow No influence of the height on microhardness for RT, 200°C and 350°C.

- → At 600°C, microhardness increases linearly according to sample height.
 - → Grains more columnar (Hall Petch effect)
 - \rightarrow Internal stresses relieved on the **BOTTOM**
 - \rightarrow Thermal gradient.

- → Microhardness decreases as a function of temperature.
- → Same trend observed after heat treatment Herliansyah and al., Proceedings, 2015.

Zhong et al. J. Nucl. Mat. 470 (2016) 170.								
Micro-hardness measured on the side (top and bottom in building direction) and cross-section surface (HV1) of as-built sample, <i>cf</i> . discussion in the text.								
Bottom of side surface	Top of side surface	Cross-section	HIPed(Hv0.3) [26]					
239 ± 5 219 ± 5		228 ± 4	~220					

 \rightarrow Same values reported by *Zhong:*

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MICROSTRUCTURE

MECHANICAL PROPERTIES

Samples machined according the ISO 6892-1 standard. Test at room temperature.

Tensile test Samples manufactured along B.D. Influence of the substrate temperature

Forged 316LSS, RCC-MRx France nuclear Standard



> Strength and Yield strength decrease linearly as a function of temperature.

> Similar trend followed by the microhardness.

> Elongation increases linearly as a function of temperature.

Same trend observed after heat treatment:

Dislocation / Internal stresses relieved ?



Grains are more columnar along building direction when temperature increases.

Mechanical properties respect the minimum requirement of the nuclear standard.14

Ceaden conclusion/perspectives

☐ Microstructure:

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- * Columnar grains along the Z-direction with intra granular cells (around 0.6 to 1 μm).
- * Grains length influenced by substrate temperature especially at 600°C on the bottom of sample.

Mechanical properties:

- Same trend observed after heat treatment.
- * Mechanical properties respect the minimum requirement of the nuclear standard.

□ Perspectives:

- Measurement of the internal stresses.
- * Mechanical properties as a function of **build direction** at high substrate temperature.
- Post-processing heat treatments and hot isostatic pressing.

Thank you for your attention.