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Microstructure impact on high temperature corrosion behavior of AISI 316L stainless steel additively manufactured by Selective Laser Melting (AM-SLM)

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Abstract

Additive Manufacturing by Selective Laser Melting (AM-SLM) is a near-net shape method producing dense and geometrically complex materials from micrometric powders. This process involves complete melting and very high cooling rates who induce a **refinement of** microstructure, improving the mechanical properties of the material [1,2]. However, the impact of these new microstructures on real functioning properties, like for instance the high temperature durability, needs to be studied. In this purpose, AISI 316L is considered in this work. Samples elaborated by AM-SLM and by conventional metallurgy were oxidized under laboratory air at 900°C for periods up to 3000h. The results highlight better behaviour for AM samples, which present a very good corrosion resistance throughout the 3000 hours. The conventional samples show a good resistance only during the first 1000 hours. These differences were related to the different composition of the oxide layers growing on the surface of the samples during the high temperature ageing: **protective chromia for AM-SLM** and **non-protective iron oxides for conventional samples**. To explain these differences of reactivity, several hypotheses were considered. XRD, SEM-EBSD and TEM studies were performed on initial samples in order to evaluate their microstructure, surface mechanical state and crystallographic orientation. The most promising hypothesis seems to be related to the microstructure (probably at **nanoscale**) of the samples. For AM-SLM coupons, a typical cellular structure was identified by TEM, with a size between 0.2 and 1 µm, corresponding to a very dense network of dislocations, which accumulate forming cell walls. Dislocation might act during the high temperature exposure as short-circuit paths for Cr diffusion from the bulk to the surface allowing the growth of protective scales for long ageing times. Moreover, AM-SLM samples found to contain large quantity of nano-inclusion that might equally influence corrosion behaviour.

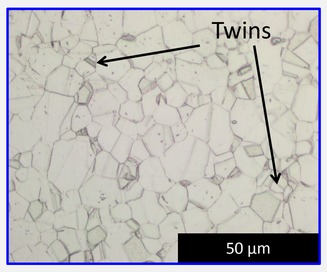
Materials and methods

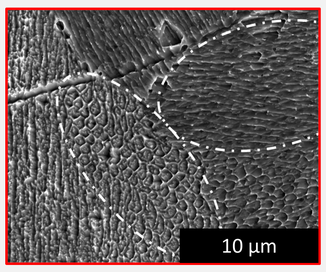
Raw materials → Conventional metallurgy: **Cold-rolling**; Additive Manufacturing : **SLM**

❖ *Chemical composition (% weight)*

	Cr	Ni	Mo	Mn	Si	C	P	S	Fe
Cold-rolled	**17,37**	**11,03**	2,16	1,64	**0,56**	0,02	0,01	0,002	Bal.
SLM	**18,00**	**12,00**	2,30	0,03	**0,47**	0,01	≤0,04	≤0,005	Bal.

❖ *Microstructures of cold-rolled and SLM samples*

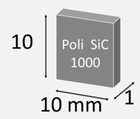




Experimental

❖ *Samples preparation*

- Size: 10 * 10 * 1 mm
- SiC polishing up to 1000 grit
- Ultrasonic cleaning in ethanol



Raw SLM samples: (a) Wall, (b) Rod

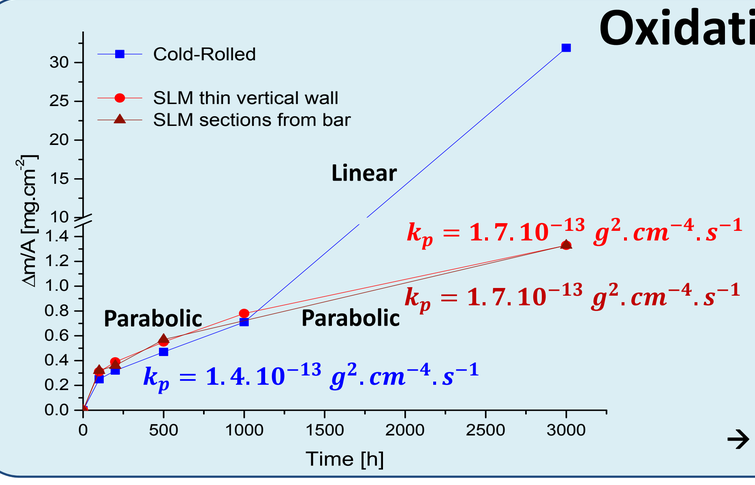
SLM sections

Carbolite muffle furnace

❖ *High temperature oxidation tests*

- Muffle furnace
- Temperature : **900°C**
- Atmosphere : Laboratory air
- Time: from 100 to 3000h

Oxidation kinetics



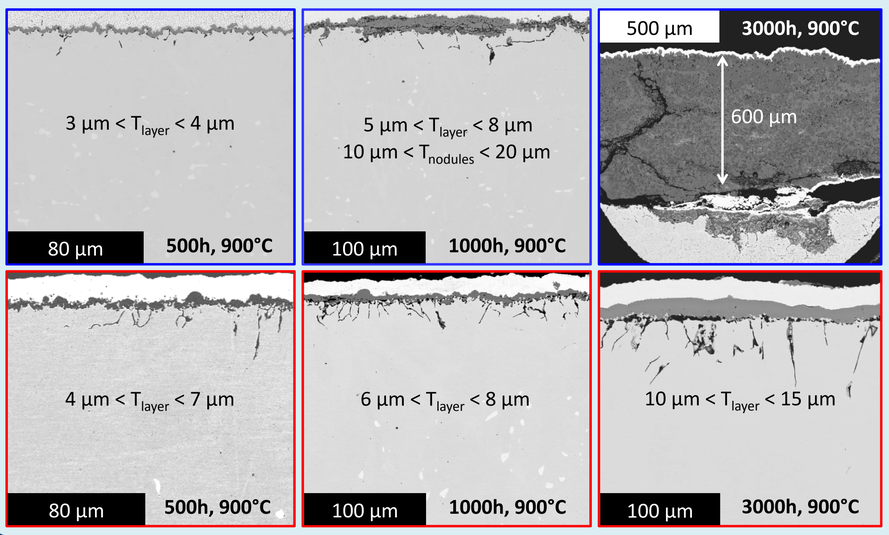
- **Protective** oxides : parabolic kinetics $\left(\frac{\Delta m}{A}\right)^2 = k_p t$
  $k_p \equiv parabolic\ constant\ rate$
- **Non protective** oxides : linear kinetics $\frac{\Delta m}{A} = k_l t$
  $k_l \equiv linear\ constant\ rate$

✓ k_p(SLM) ≈ k_p(cold-rolled) up to 1000h
✓ Breakaway oxidation only for cold-rolled samples after 1000h
✓ Parabolic behaviour throughout 3000 hours for SLM samples

→ **Better behaviour for SLM samples during 3000h of exposure in air at 900°C**

Oxides scales characterization

SEM cross-section observations



✓ **Oxides thickness** ↗ when exposure time ↗

Rolled samples:

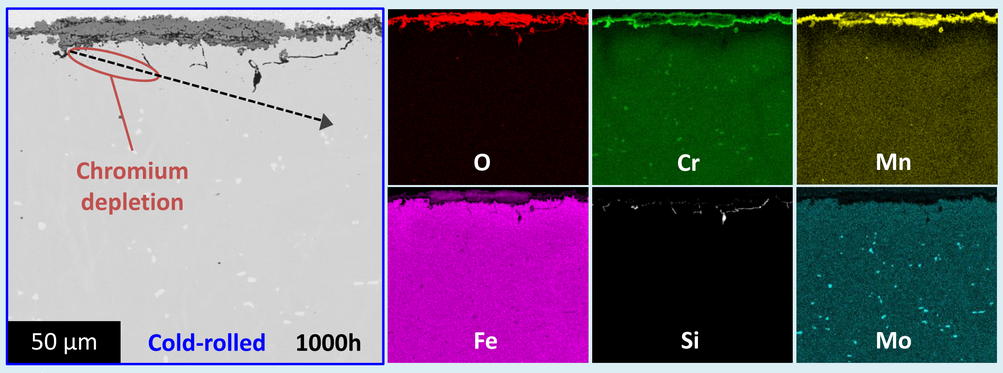
✓ Presence of **non-protective iron nodule oxides** $(Fe,Cr)_2O_3$ / Fe_3O_4 **after 1000h of exposure**
✓ **Very thick iron oxide layer** and **disastrous internal oxidation after** 3000h

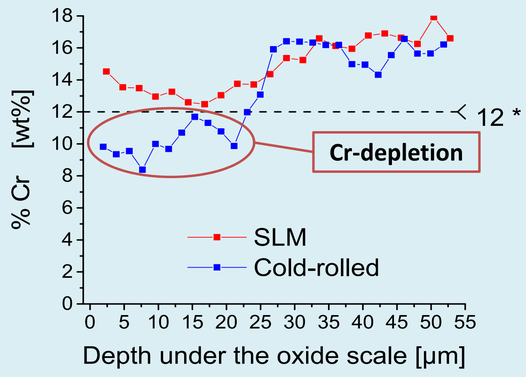
SLM samples:

✓ **thin oxide layers: ≈ 12 µm** after 3000h of exposure
✓ **Protective scale** composed of Cr_2O_3 for SLM coupons

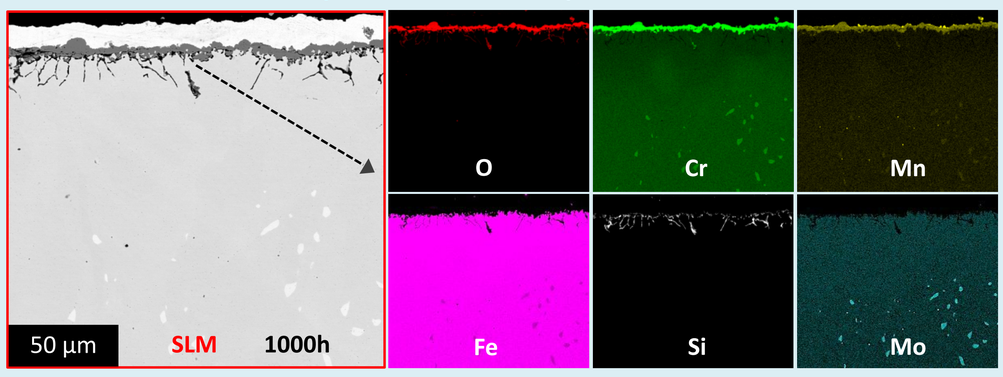
Metal substrate evolution during high temperature exposure (SEM)

Rolled samples: 1000h of exposure at 900°C





SLM samples: 1000h of exposure at 900°C



✓ **Cr-depletion below 12%wt** at the outer part of the metal (in the first 20 µm): **protective scale cannot form anymore**

✓ **Cr-content** does **not decrease below the 12%wt** for SLM coupons

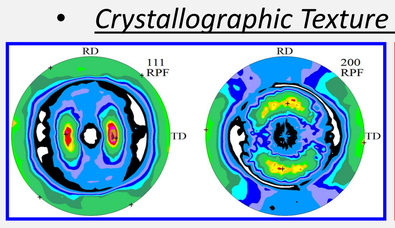
→ **Very good resistance** against long term high temperature oxidation **for SLM** can be related to a **sufficient supply** of **chromium from bulk to the surface** which allows the **formation** of a **protective** chromia layer Cr_2O_3.

Cr present at the outer part of the metallic substrate is **consumed by the growth of Cr_2O_3** during the high temperature oxidation. Its **recovery depends on the diffusion from bulk**.
This diffusion is known to **depend on the mechanical conditioning** of the surface and on the **presence of defects** such as **precipitates** and **dislocations** [3].

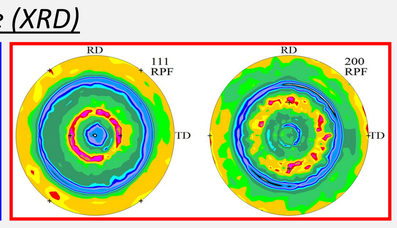
*12%mass is the lower limit of which chromia formation is favoured [4]

Characterisation of raw samples

- *Crystallographic Texture (XRD)*



"**Brass**" **texture** {110} <1-12>, planes {110} oriented // to the rolling surface



Fiber texture, planes {110} oriented // to the building platform.

→ **Similar crystallographic textures :** crystallographic planes {110} // to the surface exposition → **Texture does not explain the reactivity difference**

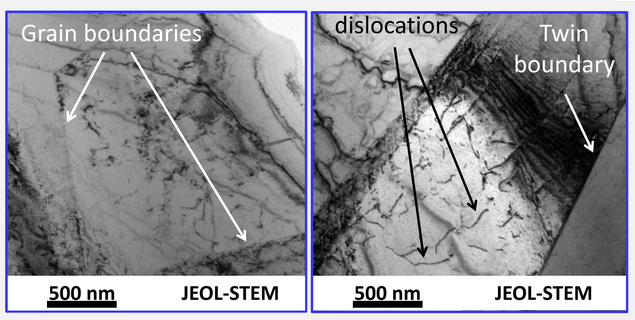
- *Mechanical surface state: Residual stresses (XRD)*

Sample	Cold-rolled		SLM wall		SLM from pillar	
	σ_{11}	σ_{22}	σ_{11}	σ_{22}	σ_{11}	σ_{22}
Stresses [MPa]	-494 ±57	-135 ±22	-90 ±27	-93 ±44	-275 ±30	-227 ±19

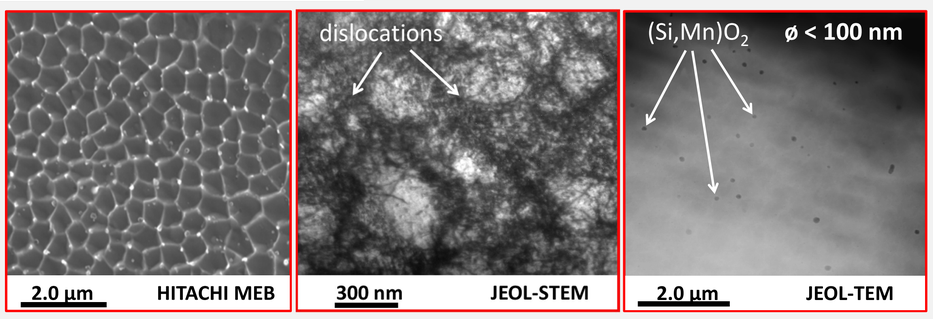
σ_{11} = Rolling direction

→ **The mechanical surface** condition of the samples **does not explain** the **differences** in behavior **between cold-rolling and SLM**

- ***Nano-scale characterization (TEM)***



✓ Microstructure with **polyhedral grains** accompanied by **twins** and some **isolated dislocations**



✓ **Cellular grain sub-structure** formed by the accumulation **of a large number of dislocation** accompanied by **nano-inclusions**

→ **Higher amount of dislocations for SLM than rolled samples**
→ **Larger number of dispersed nanoparticles only in the case of SLM samples**

Dislocations act as a **diffusion short-circuit for Cr** in the substrate [3,5] and thus **facilitate the diffusion of chromium** from bulk to the surface of the samples.
Nano-inclusions may equally **improve corrosion resistance** [6].

Conclusions

❑ **Promising results after oxidation under air of laboratory for SLM samples**

❑ **Differences** in term **of reactivity** and **corrosion products**

❖ **Parabolic behavior** for **SLM** throughout **3000 hours**
❖ **Parabolic behavior** for **cold-rolled** only up to **1000 hours**
Breakaway oxidation from 1000h for **conventional coupons**
Chromium depletion (< 12%wt) jut for **as-rolled** samples

The cellular network, formed by the accumulation of a **large number of dislocations**, as well as the presence of **ultra-fine nanoparticles** would **improve the high-temperature behavior** of stainless steel and explain the **best behavior of SLM specimens**.

Perspectives

➢ Quantify the **dislocation content** by XRD (modified Warren-Averbach method)
➢ Determined **Cr diffusion coefficient** from EDS-SEM profiles at the metal surface
➢ Confirm the better behaviour of SLM samples **for longer exposure times and different temperature / atmospheres**

References

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