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# Mitigating oxygen pick-up during laser powder bed fusion of Ti-6Al-4V by limiting heat accumulation

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## ABSTRACT

The dissolution of oxygen in Ti-6Al-4V during laser powder bed fusion (L-PBF) is a limitation for the final ductility of the produced components and a challenge for the end-users. In the present work, the effect of the residual oxygen in the process atmosphere of a laboratory scale L-PBF machine, as well as the role of heat accumulation, are studied. It was shown that oxygen content in the as-built Ti-6Al-4V is determined by the size of the scanned area and build time. The heat accumulation aspect was investigated by adjusting the inter-layer time (ILT), by increasing the recoating time or the number of produced parts. The results showed that oxygen pick-up could be limited by reducing residual oxygen level in the atmosphere or heat accumulation. A 400 ppm O<sub>2</sub> reduction measured at the top of a 70 mm column was achieved by increasing the ILT manually by 4.5 s, and a 1200 ppm O<sub>2</sub> reduction by increasing the scanned area by 7 times. By doing so, the hardness at full height was reduced by approximately 30 HV10. It is shown that design features characterised by high aspect ratio can absorb significant amount of oxygen resulting in increased brittleness.

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## 1. Introduction

Ti-6Al-4V is a well-established material in metal additive manufacturing (AM) and is one of the most used materials in laser powder bed fusion (L-PBF). However, this alloy is characterised by high affinity to oxygen [1] and the high solubility of oxygen in  $\alpha$ -phase (up to 33 at.% in  $\alpha$ -Ti [2]). Besides stabilizing the  $\alpha$  phase, oxygen is responsible for solid solution strengthening and detrimental embrittlement [3]. The L-PBF process typically operates under a residual oxygen level of about 1000 ppm O<sub>2</sub> [4], and a laser spot size of around 100  $\mu$ m, determining the melt pool size characterised by a short lifetime and high cooling rates (around 10<sup>6</sup> K/s [5]).

There are few studies considering inter-layer time (ILT), defined as the sum of the time when the laser is scanning the powder bed and the recoating time, as a critical parameter for L-PBF, while it was identified as important for direct energy deposition [6]. Recent study by Mohr et al. [7] indicates that short ILT during L-PBF of 316L stainless steel results in an increase of keyhole porosity. Also, Williams et al. [8] used *in situ* infrared thermography and

highlighted that a reduction of ILT leads to an increase of the build parts surface temperature.

The current work investigates the effect of increasing the ILT on the pick-up of oxygen, monitored externally, from the process atmosphere by the Ti-6Al-4V parts.

## 2. Methods

Ti-6Al-4V Grade 23 powder (Heraeus) with particle size distribution 15 to 45  $\mu$ m and oxygen content of 1051 ppm was employed. Laboratory L-PBF system TruPrint1000 equipped with a laser of 30  $\mu$ m spot size was used. Developed by manufacturer, standard laser parameters (*TruPrint\_1000\_Spot30\_Ti64\_ELI-A\_Surface\_v2.0*) were used to print columns of 22 mm in diameter and 70 mm in height, with a 20  $\mu$ m layer thickness. Argon 5.0 was employed as inert gas to generate the process atmosphere and the machine was operated at an oxygen level of 100 ppm. In parallel, the ADDvanceO2<sup>®</sup> precision system equipped with both, a lambda probe and an electrochemical cell, was analysing variations in oxygen concentration in the process atmosphere, by sampling process gas close to the samples.

Three build jobs were conducted: i) a single column placed at the centre position of the baseplate; ii) the same column with the recoating time increased manually by 4.5 s; iii) no recoating

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time was added manually but a total of 7 columns were built at once, also increasing the ILT.

Oxygen analysis in the built columns along their height was done using combustion gas analysis (LECO ONH836). The Vickers

hardness (HV10) was measured using a DuraScan20. X-ray diffraction (XRD) was conducted with a PANalytical X'Pert Pro diffractometer equipped with Cu source using the  $K\alpha$  radiation and operating at 45 kV and 40 mA in the  $30^\circ$  to  $85^\circ$  range.

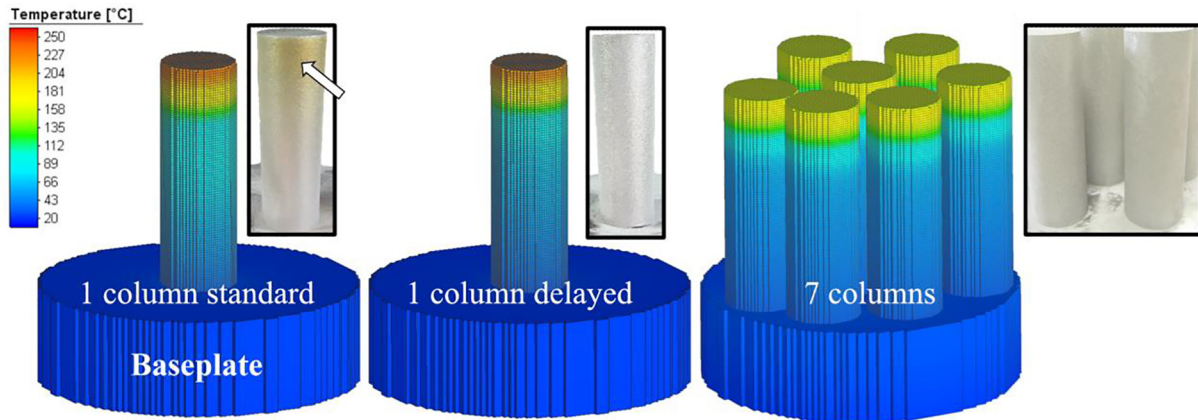


Fig. 1. FE analysis using Simufact Additive 4.1: temperature distribution within the columns for the last computed AM layer, with images of built columns.

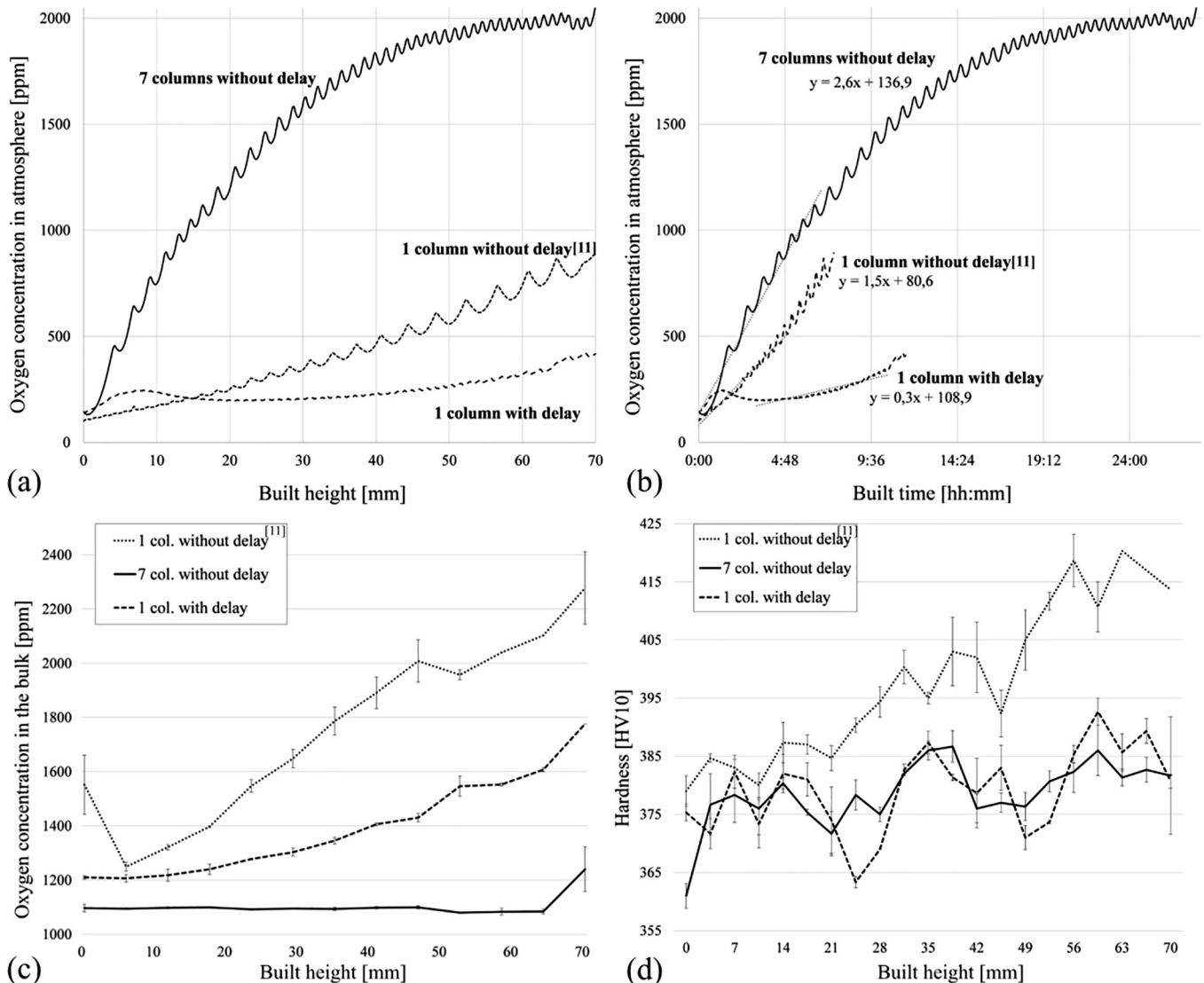
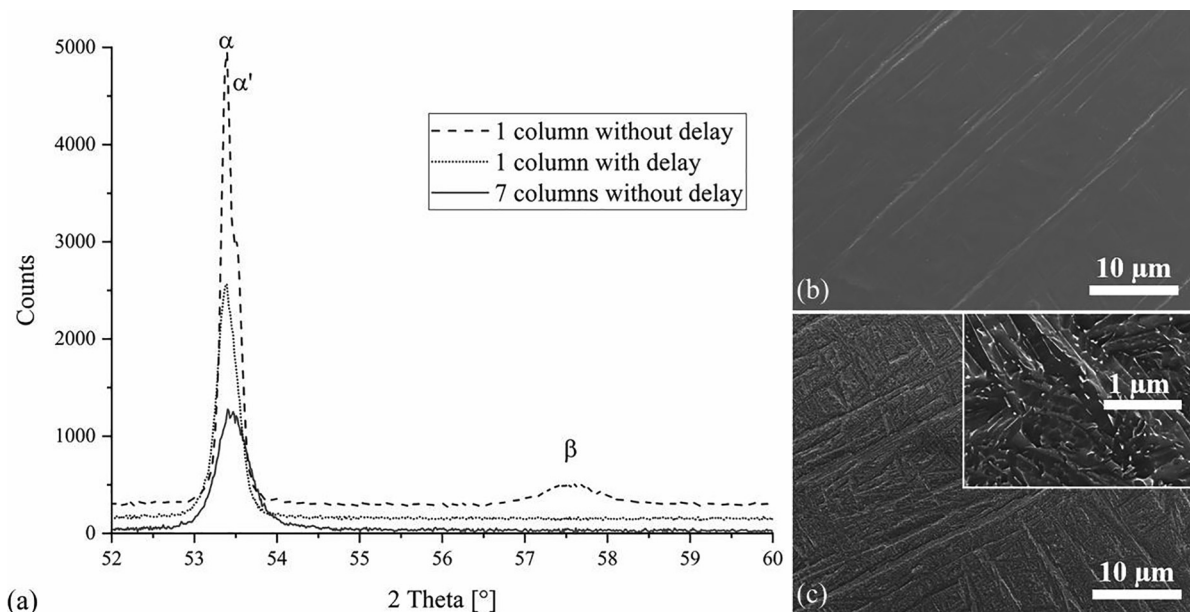


Fig. 2. Oxygen concentration during the build jobs measured with the electrochemical sensor: (a) along the columns height, (b) as a function of time. (c) Variations in oxygen content along the columns height, and (d) corresponding HV10 in their bulk.



**Fig. 3.** (a) XRD pattern of the polished cross-sections of the top of columns. (b) Microstructure of the cross-section of the top of the single column built with delay, (c) of the single column without delay.

Characterization was completed by high-resolution scanning electron microscopy (HR SEM) with a LEO Gemini 1550 on the polished and etched (using Kroll's reagent) cross-sections of the columns.

Finite Element (FE) analysis of the heat accumulation in the columns was performed using Simufact Additive 4.1 using the powder bed initial temperature, the gas and baseplate temperatures of 25 °C, the parts emissivity of 0.85, and the heat transfer coefficient of 12 W/m<sup>2</sup>K.

### 3. Results and discussions

Fig. 1 displays the temperature distribution for the last computed AM layer. It is evident that the increase of ILT by increasing the number of columns, permits to limit heat accumulation at the scale of the component. At full height the temperature is 80 °C less with an additional 21 s ILT for the 7 columns, compared to the one column standard layout (see Fig. 1, left). The lower retained temperatures allowed to inhibit the part's surface discoloration observed under standard conditions (see insert in Fig. 1). No discoloration was observed for the single column with manually increased ILT, but simulation approach used didn't allow to capture heat accumulation to the level of single deposited layer.

Fig. 2a,b displays the variations of the oxygen concentration in the atmosphere measured with the electrochemical sensor with respect to the height of columns and time. The increase of ILT is reflected in the overall longer build jobs. The deviation of the oxygen concentration from the setpoint of 100 ppm is attributed to the presence of hydrogen molecules probably coming from humidity. While the oxygen content measured with the electrochemical sensor increased during the build job, that of the lambda probe remained close to the setpoint. As discussed elsewhere [9], the accuracy of the lambda probes usually degrades in presence of H<sub>2</sub>, CO, NO and hydrocarbons due to their oxidation on the head of the hot ceramic sensor [10], leading to an underestimation of the oxygen level. The early higher oxygen level for the single column built with delay is attributed to a consequence of the machine purging. In addition, the longest built job with the 7 columns is characterized by the highest oxygen increase, and thus, the highest deviation between the two types of sensor. This suggests that

hydrogen variations in the process atmosphere are also connected to the size of scanned area, and hence possible humidity adsorbed on the powder particles.

Fig. 2c displays the variations in oxygen content in the columns bulk. It is clear that the increase in oxygen content within the bulk is connected to a coupled effect of the oxygen concentration within the process atmosphere and the heat accumulation which enhances the kinetics of oxidation (see Fig. 1 and Fig. 2). When the heat accumulation is minimal, so becomes the impact of the residual oxygen in the process atmosphere. This is highlighted by the composition of the 7 columns built together with about 1200 ppm O<sub>2</sub> at full height, which is approximately 1000 ppm less than the single column (and 150 ppm more than the powder). When both the residual oxygen level in the atmosphere and the heat accumulation are reduced, the oxygen pick-up in the bulk is also limited (by about 400 ppm O<sub>2</sub> at full height), as proves the comparison of the two single columns. At the same time, when the heat accumulation remains the same and the residual oxygen decreases from approx. 800 to 400 ppm, the oxygen pick-up is also reduced by approx. 400 ppm O<sub>2</sub> at full height [11]. Hence, controlling the residual oxygen appears to be a more robust alternative than increasing the ILT considering process productivity. The two cases for which the heat accumulation and pick-up of oxygen were limited, exhibit lower hardness than the standard column (Fig. 2d). The higher hardness is connected to the precipitation of the β phase for standard ILT; while the microstructures remained fully martensitic for the longer ILT cases (see XRD and HR SEM results, Fig. 3).

### 4. Conclusions

The present work highlights that longer ILT allows to limit heat accumulation, resulting in reduced oxygen pick-up, especially critical for high aspect ratio components. Longer ILT permitted to limit both the kinetics of oxygen pick-up as well as inhibit the precipitation of the β phase. These changes were reflected in the hardness of the produced part, reduced by approx. 30 HV10 at the columns full height for both longer ILTs, under the investigated conditions and used system. Results clearly indicate necessity of oxygen control

in case of sensitive material such as Ti-6Al-4V, requiring oxygen level down to 100 ppm (an order of magnitude lower than commercial standard) to limit oxygen pick-up, since increasing the ILT is counter-productive. Longer ILTs are of interest for critical design features such as thin/high aspect ratio sections or in case oxygen control during the processing is limited.

### CRediT authorship contribution statement

**C. Pauzon:** Data curation, Formal analysis, Investigation, Methodology, Writing - original draft. **K. Dietrich:** Formal analysis, Investigation. **P. Forêt:** Project administration, Resources, Supervision. **E. Hryha:** Formal analysis, Methodology, Supervision, Project administration, Writing - review & editing. **G. Witt:** Project administration.

### Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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### References

- [1] G. Lütjering, J.C. Williams, *Titanium : Engineering Materials and Processes*, Ed. SPRINGER, second edi (2007) 1–442. doi:10.1007/978-3-540-73036-1.
- [2] E. Hryha, R. Shvab, M. Bram, M. Bitzer, L. Nyborg, Surface chemical state of Ti powders and its alloys: Effect of storage conditions and alloy composition, *Appl. Surf. Sci.* (2015) 1–10, <https://doi.org/10.1016/j.apsusc.2016.01.046>.
- [3] Q. Yu, L. Qi, T. Tsuru, R. Traylor, D. Rugg, J.W. Morris, M. Asta, D.C. Chrzan, A.M. Minor, Origin of dramatic oxygen solute strengthening effect in titanium, *Science* (80-.). 347 (2015) 635–639. doi:10.1126/science.1260485.
- [4] C. Pauzon, E. Hryha, P. Forêt, L. Nyborg, Effect of argon and nitrogen atmospheres on the properties of stainless steel 316L parts produced by laser-powder bed fusion, *Mater. Des.* (2019), <https://doi.org/10.1016/j.matdes.2019.107873>.
- [5] I. Yadroitsev, I. Yadroitsava, Evaluation of residual stress in stainless steel 316L and Ti6Al4V samples produced by selective laser melting, *Virtual Phys. Prototyp.* 10 (2015) 67–76, <https://doi.org/10.1080/17452759.2015.1026045>.
- [6] E.R. Denlinger, J.C. Heigel, P. Michaleris, T.A. Palmer, Effect of inter-layer dwell time on distortion and residual stress in additive manufacturing of titanium and nickel alloys, *J. Mater. Process. Technol.* 215 (2015) 123–131, <https://doi.org/10.1016/j.jmatprotec.2014.07.030>.
- [7] G. Mohr, S.J. Altenburg, K. Hilgenberg, Effects of inter layer time and build height on resulting properties of 316L stainless steel processed by laser powder bed fusion, *Addit. Manuf.* 32 (2020), <https://doi.org/10.1016/j.addma.2020.101080>.
- [8] R.J. Williams, A. Piglion, T. Rønneberg, C. Jones, M.S. Pham, C.M. Davies, P.A. Hooper, In situ thermography for laser powder bed fusion: Effects of layer temperature on porosity, microstructure and mechanical properties, *Addit. Manuf.* 30 (2019), <https://doi.org/10.1016/j.addma.2019.100880>.
- [9] M.A. Toema, *Physics-Based Characterization of Lambda Sensor Output to Control Emissions From Natural Gas Fueled Engines*, Kansas State University, 2010.
- [10] E. Hryha, L. Nyborg, Process control system for delubrication of PM steels, *Acta Metall. Slovaca.* 18 (2012) 60–68.
- [11] C. Pauzon, K. Dietrich, P. Forêt, S. Dubiez-Le Goff, E. Hryha, G. Witt, Manuscript: Control of residual oxygen in the L-PBF process atmosphere for Ti-6Al-4V, *Addit. Manuf.* 38 (2021), <https://doi.org/10.1016/j.addma.2020.101765>.