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Influence of focus offset on the microstructure of an intermetallic gamma-TiAl based alloy produced by electron beam powder bed fusion / Ghibaud, C; Wartbichler, R; Marchese, G; Clemens, H; Ugues, D; Biamino, S. - In: JOURNAL OF MANUFACTURING PROCESSES. - ISSN 1526-6125. - ELETTRONICO. - 89:(2023), pp. 132-141.  
[10.1016/j.jmapro.2023.01.061]

*Availability:*

This version is available at: 11583/2979631 since: 2023-06-27T13:31:49Z

*Publisher:*

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*Published*

DOI:10.1016/j.jmapro.2023.01.061

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# Influence of focus offset on the microstructure of an intermetallic $\gamma$ -TiAl based alloy produced by electron beam powder bed fusion

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## ARTICLE INFO

### Keywords:

Additive manufacturing  
electron beam powder bed fusion  
Focus offset  
Intermetallics  
Titanium aluminides

## ABSTRACT

It is well established in literature that, when processing intermetallic  $\gamma$ -TiAl components by electron beam powder bed fusion, a banded microstructure is frequently formed because of an inhomogeneous Al distribution since more pronounced evaporation of Al occurs at the top of the melt pool.

This feature is particularly promoted when highly energetic process parameters (high beam currents, slow beam speeds, narrow line offsets) are used. Therefore, an approach already suggested in the literature to reduce the Al loss is to minimize the energy level of the process parameter during production. However, there is a limit to such kind of approach: minimizing the beam current or increasing the beam speed, or increasing the line offset will, at a certain point, results in not being able to achieve a completely dense material and thus some process-induced porosity, the so-called lack-of-fusion defects, starts to occur in the produced parts.

In this study, the effect of an additional parameter of the electron beam powder bed fusion process is taken under consideration: the focus offset (FO), i.e. the distance between the focusing plane of the electron beam with respect to the powder bed. The effect of the FO on the residual porosity, microstructure, phase composition, hardness as well as chemical composition is investigated, thus having the possibility to demonstrate that also the FO can affect the Al loss and play a fundamental role in the generation of a homogenous microstructure, contributing to mitigate the appearance of a banded microstructure.

## 1. Introduction

Intermetallic  $\gamma$ -TiAl-based alloys are interesting structural materials because of their low density (around 4 g/cm<sup>3</sup>) and outstanding properties, such as high specific tensile strength, high specific Young's modulus as well as excellent creep resistance for components working at elevated temperatures (600–750 °C), especially when compared with the heavier (about 8 g/cm<sup>3</sup>) Ni-base superalloys [1–6]. For instance, in 2006, GE announced the implementation of the  $\gamma$ -Ti-48Al-2Nb-2Cr alloy (in at.%, unless stated otherwise) for low-pressure turbine (LPT) blades for the 6th and 7th stages of the GENx™-1B aircraft engines powering the Boeing 787 aircraft [7]. The utilization of TiAl alloys allows the reduction of fuel consumption, NO<sub>x</sub> emissions, and noise when compared with former engines [8]. Intermetallic  $\gamma$ -TiAl-based alloys also find application in the automotive sector, where they are employed as

the constituent material for turbine wheels in turbochargers to increase engine efficiency and decrease harmful emissions. An example can be found in the Lancer cars model of Mitsubishi, which is equipped with a  $\gamma$ -TiAl turbocharger [9]. Furthermore, at the beginning of the late 1990s, in Formula 1 racing engines, the use of  $\gamma$ -TiAl-based alloys as valve material was revolutionary and provided a significant performance advantage that has fundamentally changed the engine designer's concept for light and heat-resistant valves [10].

The good mechanical properties of  $\gamma$ -TiAl based alloys stem from their microstructures and constituent phases [4,11,12]. The main constituent phases are the  $\gamma$ -TiAl phase (L1<sub>0</sub> ordered tetragonal face-centered structure) and the  $\alpha_2$ -Ti<sub>3</sub>Al phase (DO<sub>19</sub> ordered hexagonal structure). Moreover, it is possible to find traces of  $\beta_0$ -phase (B2 ordered body-centered structure) [8,13,14]. The general mechanical properties associated with the respective microstructure are reported in the work of

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<https://doi.org/10.1016/j.jmapro.2023.01.061>

Received 14 September 2022; Received in revised form 12 December 2022; Accepted 23 January 2023

Available online 2 February 2023

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Recina et al. [15]. The microstructures of  $\gamma$ -TiAl-based alloys are commonly divided into four types: i) fully lamellar, ii) nearly lamellar, iii) duplex, and iv) equiaxed. The lamellar grains are responsible for high creep resistance and toughness, although a high amount of lamellar colonies is detrimental to ductility at room temperature and to fatigue resistance. On the contrary, the equiaxed microstructure enables the improvement of the ductility at room temperature as well as fatigue resistance [3,4,8,15,16]. For this reason, the desired microstructure must be carefully tailored for the envisaged structural component through the optimization of both its processing route and its subsequent heat treatment, thus matching the mechanical properties required for its application.

At present, the state of art for the production of  $\gamma$ -TiAl-based alloy components such as LPT blades is precision casting, despite some issues [4,11,12,16]. Firstly, the crucible used in the casting process may be the source of possible contaminations, and this is particularly critical in the case of  $\gamma$ -TiAl-based alloys due to their high reactivity in the melted state [17]. In particular, if the oxygen content exceeds 0.1 wt%, the plastic fracture strain under tensile loading, that it is known not to be very high for these alloys and it is always the critical point to face for their industrial applications, is drastically reduced [4]. Secondly, preheated moulds are necessary to avoid an incomplete filling due to the high viscosity of the melt, thus resulting in a slow cooling rate and thus to the formation of a coarser microstructure or segregation of the heavier alloying elements.

Thanks to the possibility of overcoming the above mentioned casting drawbacks, electron beam-powder bed fusion (EB-PBF) started to be considered a very attractive additive manufacturing technique to process  $\gamma$ -TiAl-based alloys. The high preheating temperature, i.e. over the brittle-to-ductile transition temperature of TiAl alloys (around 700 °C), allows the production of crack-free components, while the controlled vacuum ( $2 \times 10^{-3}$  mbar, adjusted by backfilling He flux) limits concentration of interstitial elements in the component [18–21]. So, since 2010, the processability of  $\gamma$ -TiAl-based alloys by EB-PBF has been studied, where the initial works of Biamino et al. [19] and Murr et al. [20] have mainly paid attention to the achievement of dense samples and components. More details on the process steps of EB-PBF can be found in [22,23].

However, the vacuum environment and the high local energy employed in the EB-PBF process lead to easy evaporation of some light elements, for example, Al in  $\gamma$ -TiAl-based alloys. This Al evaporation occurs from the top of the melt pool, so if it is not controlled, it may generate a banded microstructure consisting of alternating Al-depleted zones and Al-rich zones as reported in [13,18,24–26]. For  $\gamma$ -TiAl-based alloys, this microstructure feature is typical of the EB-PBF process, and the response in terms of mechanical properties was, for instance, studied by Todai et al. [26] resulting in a high anisotropy of the tensile properties. Such high anisotropy in the tensile properties is caused by the preferential orientation (perpendicularly to the building direction) of the banded microstructure originated by a not homogeneous Al evaporation. No anisotropy caused by the solidification direction in each layer or due to the thermal gradient in the building direction have ever been reported for  $\gamma$ -TiAl-based alloys processed by EB-PBF. This behavior is different than observed in Ni-superalloys [27,28] or Ti alloys [29] where, in the as-built condition, grains are typically elongated in the building direction due to the thermal gradient in the building direction.

Regarding the EB-PBF process parameters, there are already investigations that deal with the effect of the current, the speed, and the line offset of the electron beam on the Al loss, proposing the reduction of the energy input given to the material as a route to limit the Al loss, thus increasing the microstructural homogeneity [11,15,22]. However, it was already reported that low energy densities can lead to not complete densification of the built specimens with process-related residual porosities, the so-called lack-of-fusion (LoF) defects [25,30–32].

Despite the various studies on the effect of the process parameters on

**Table 1**

Chemical composition (in atomic percentage, at.%) of the starting powder determined by OES-ICP analysis.

	Al	Cr	Nb	Fe	Ti
Powder	48.73	1.92	2.00	0.03	Bal.

the processability of  $\gamma$ -TiAl-based alloys, there is a parameter whose role in Al loss and microstructure modification has never been investigated so far: the focus offset (FO). The FO is a current that shifts the focal plane, thus modifying the dimensions of the electron beam spot size on the powder bed [33–37]. So, the present paper aims to study the effect of the FO on the residual porosity, microstructure, chemical composition, phases, and hardness using a Ti-48Al-2Nb-2Cr alloy. Furthermore, subsequent heat treatments were performed to compare the microstructure evolution of samples produced with two different FOs.

## 2. Materials and methods

In this study, gas atomized Ti-48Al-2Nb-2Cr powder with particle sizes between 40 and 150  $\mu\text{m}$  was supplied by Arcam EBM (GE Additive). Table 1 lists the chemical composition of the powder determined through optical emission spectrometer-inductively coupled plasma (OES-ICP) analysis.

Cuboid samples with a side of 18 mm and a height of 35 mm were built using an A2X Arcam EBM (GE Additive) machine at an accelerating voltage of 60 kV. The melting parameters were kept constant (beam current,  $I = 10$  mA, beam speed,  $v = 1600$  mm/s, line offset,  $L_{\text{off}} = 0.2$  mm), except for the FO, varied from 5 to 30 mA. The samples will be hereafter identified by the FO used, where the value of the current, expressed in mA, will be reported as a subscript (example: FO<sub>+5</sub> means that a current of 5 mA was used for the FO parameter). A scanning strategy with a rotation of 90° between consecutive layers was used. A preheating temperature of 1050 °C was set using the defocused beam atop each newly spread powder layer of 90  $\mu\text{m}$  during the preheating step. The preheating temperature was measured by a thermocouple located below the building platform, and, consequently, the registered temperature slightly decreased to around 1010 °C when reaching a total height of 35 mm.

For metallographic preparation, the samples were cut along the building direction and ground using SiC sandpapers up to 2500 grit. Finally, the samples were polished down to 0.03  $\mu\text{m}$  with OP-S suspension. The polished samples were analyzed to determine the residual porosity by means of a light optical microscope (LOM, Zeiss AXIO Imager.M2 equipped with an Axio-CamHRC.5 camera). A total of 20 LOM images per sample at 50 $\times$  were taken and subsequently post-processed with ImageJ software. For the microstructural analyses, a scanning electron microscope (SEM Versa 3D Dual Beam from Thermo Fischer, formerly FEI, operating at 10 kV) was used in backscattered electron (BSE) mode. In all the micrographs, the building direction Z is upwards.

In order to evaluate the mechanical performance, Vickers hardness tests HV0.05 and HV10 were carried out on polished samples using a Qness 60 A+ microhardness tester from ATM Qness. The Vickers hardness was conducted with 10 indentations per sample. Moreover, for some selected conditions, a micro Vickers hardness map was produced by executing 900 indentations (30  $\times$  30 with a distance of 50  $\mu\text{m}$  between each indentation), covering an area of 1.5  $\times$  1.5 mm<sup>2</sup>.

X-Ray Diffraction (XRD) analyses were performed on the samples to quantitatively determine the phases by Rietveld refinement using TOPAS 4.1 software. XRD analyses were performed utilizing a D8 Advanced diffractometer from Bruker AXS, using Cu-K $\alpha$  radiation and an angular range of 11–82° with a step size of 0.02° in a Bragg-Brentano geometry.

Thermomechanical analysis (TMA - Setaram Setsys Evolution) was performed on 5  $\times$  5  $\times$  10 mm<sup>3</sup> samples extracted from the 18  $\times$  18  $\times$  35

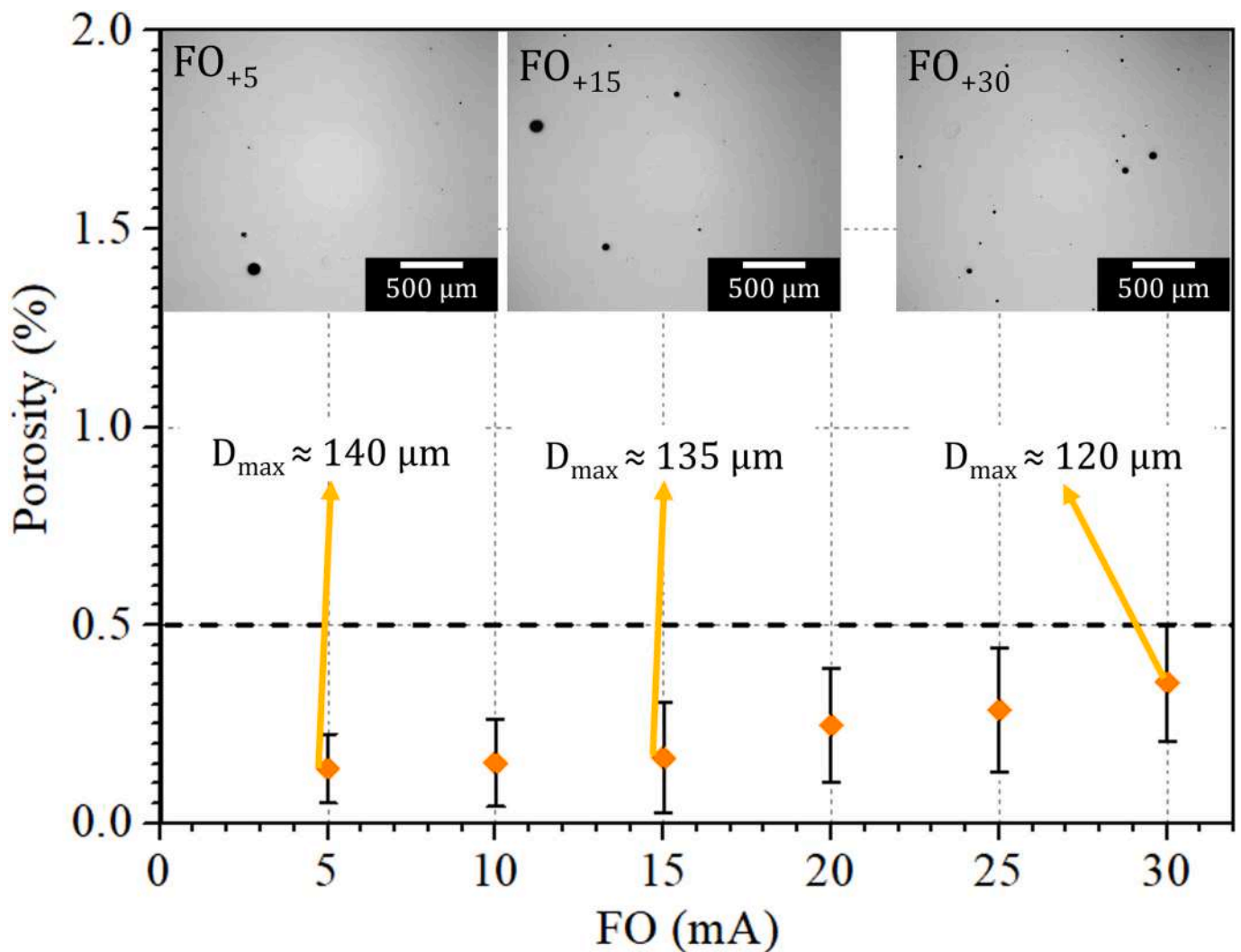


Fig. 1. Residual porosity of the samples built using different FOs. LOM images showing the residual defects of the samples FO<sub>+5</sub>, FO<sub>+15</sub>, and FO<sub>+30</sub> with the maximum diameter ( $D_{max}$ ) of the detected circular pore.

mm<sup>3</sup> specimens produced with different FOs. TMA was used to determine the phase transition temperatures in the range of 1250 and 1400 °C using a heating rate of 5 °C/min under an Ar flow of 20 ml/min to prevent oxidation. The chemical content of selected specimens was obtained by X-ray fluorescence (XRF) analysis.

Afterward, two conditions with low (FO<sub>+5</sub>) and high FO (FO<sub>+30</sub>) were subjected to subsequent heat treatments to compare their microstructure evolution.

These samples were heat-treated at 1050 °C for 24 h followed by air cooling and then analyzed by means of electron backscattered diffraction (EBSD using FEI system) to evaluate the distribution of the phases and their morphology. The samples were vibro-polished for 1 h using 0.03  $\mu\text{m}$  OP-S suspension. The analysis was carried out at 30 kV with a spot size of 7  $\mu\text{m}$  and a step size of 500–1000 nm. The software Team 4.3 and TSL OIM Analysis 7 were employed for performing the measurement. Considering that literature reports the challenge to properly detect  $\gamma$ -TiAl as a tetragonal phase due to the  $c/a$  ratio close to 1,  $\gamma$ -TiAl phase was measured as a cubic phase [13,38,39].

Moreover, the microstructure of the heat-treated samples at 1310 °C for 2 h followed by a cooling rate of around 125 °C/min was characterized by means of SEM in order to compare the generated microstructure from two different FOs.

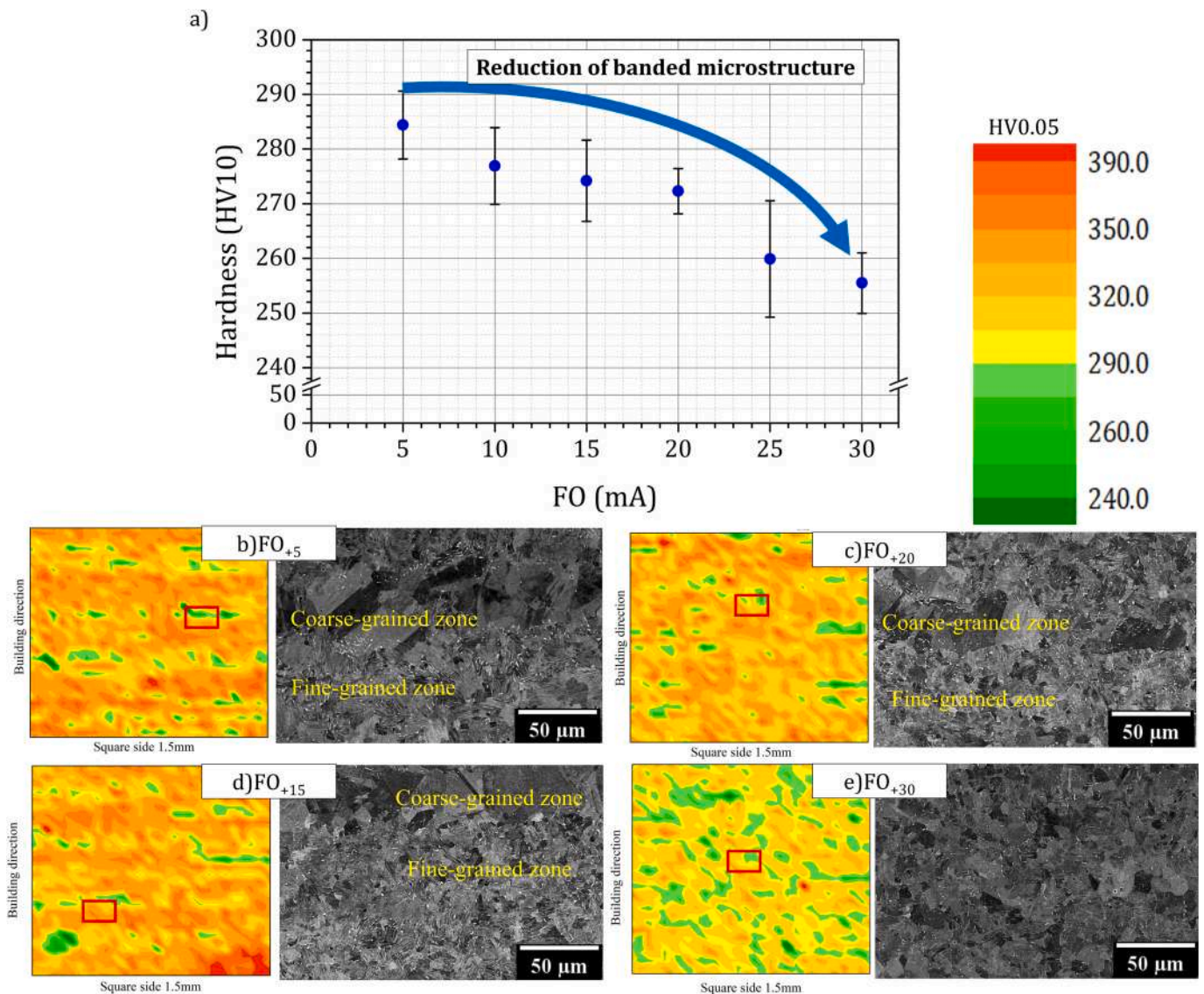
Finally, the phases' transformation was observed and recorded using a laser scanning confocal microscope (LSCM, VL2000DX-SVF17SP

Yonekura HT) under heating at high temperature and then cooling down to room temperature. The heating parameters for the LSCM were selected to generate a duplex microstructure. The parameters were: i) from 100 to 1100 °C heating with 500 °C/min, ii) from 1100 to 1310 °C with a heating rate of 50 °C/min in order to observe the eutectoid phase transition  $\alpha_2 + \gamma \rightarrow \alpha + \gamma$ , iii) isotherm at 1310 °C for 10 min, iv) cooling down to 1000 °C with a cooling rate of 100 °C/min to observe the lamellar precipitation, and v) finally a fast cooling down to room temperature. A flow of Ar prevented the oxidation of the sample during all the steps.

### 3. Results and discussion

Fig. 1 shows that, regarding the experimental conditions investigated in this paper, the porosity grew only marginally with the increment of the FO and was always below 0.5 %, which is a value in the same range reported in the literature for other  $\gamma$ -TiAl alloys [40–42]. Looking at the cross-sections of all the specimens, only scattered circular pores with diameters always less than 150  $\mu\text{m}$  have been identified and can be in the insert of Fig. 1.

However, the authors would like to point out that this finding is factual only for the present experimental conditions. Nevertheless, more important changes of the FO or investigating different I and v conditions or different materials than TiAl alloys it may happen that FO changes do



**Fig. 2.** a) HV10 hardness of samples produced with different FOs; b-e) HV0.05 maps reporting the microhardness distribution with a SEM image at high magnification showing the presence or absence of banded structures. The SEM-BSE micrographs represent the areas pointed out by the red rectangle on the HV0.05 maps. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

play a decisive role in residual porosity. For instance, Lee et al. [35] reported the effect of the FO on the Inconel 718 alloy produced by EB-PBF. They observed that a highly defocused beam above a certain FO threshold involved the formation of LoF defects. Indeed, increasing the FO increases the beam spot size and the melt pool size. A wider and shallower melt pool size (with the same process parameters) means that the same energy is distributed on a larger area, thus reducing the energy input per area delivered into the melt pool. Consequently, too low area energies can result in a high level of process-derived LoF defects.

However, no LoF defects were detected for all the investigated samples, indicating that a variation of FO from 5 to 30 mA still allowed, at the actual I and v conditions, the production of specimens without large irregular porosities that can be referred to as process-induced defects, thus confirming that all the investigated FOs are eligible for successful production of specimens.

Afterward, the variation of the HV10 with the different FOs employed was characterized, and it is provided in the upper part of Fig. 2. An increment of the FO involved a gradual hardness reduction from FO<sub>+5</sub> to FO<sub>+30</sub>. In order to better investigate this aspect, similarly to what was done in a previous paper [13], a mapping of the hardness

HV 0.05 distribution of the samples produced with various FOs was conducted and is displayed in the bottom part of Fig. 2, coupled with a SEM micrograph.

The sample with FO<sub>+5</sub> exhibited a remarkable banded microstructure made of fine-grained zone alternated with coarse-grained ones. These two interpenetrating microstructures are not a novelty for  $\gamma$ -TiAl based alloy processed by EB-PBF and were deeply investigated in a previous paper [13]. The fine-grained zones developed from an Al-depleted region are constituted of a fine duplex-like microstructure to small lamellar colonies where the accumulation of the  $\alpha_2$ -Ti<sub>3</sub>Al phase exhibited a Zener pinning effect, thus inhibiting grain growth [43,44]. The grains are fine and there is this accumulation of  $\alpha_2$ -Ti<sub>3</sub>Al phase, which generally is harder than  $\gamma$ -TiAl phase in the Ti-48Al-2Cr-2Nb alloy [45]. Therefore, these bands exhibited a higher hardness with respect to the coarse-grained zones, as confirmed by the hardness maps in the bottom part of Fig. 2.

A similar degree of banded microstructure could also be detected for FO<sub>+15</sub> and FO<sub>+20</sub> conditions. In contrast, the FO<sub>+30</sub> specimen was characterized by a more homogenous microstructure and the corresponding hardness map that did not show banded structures. In

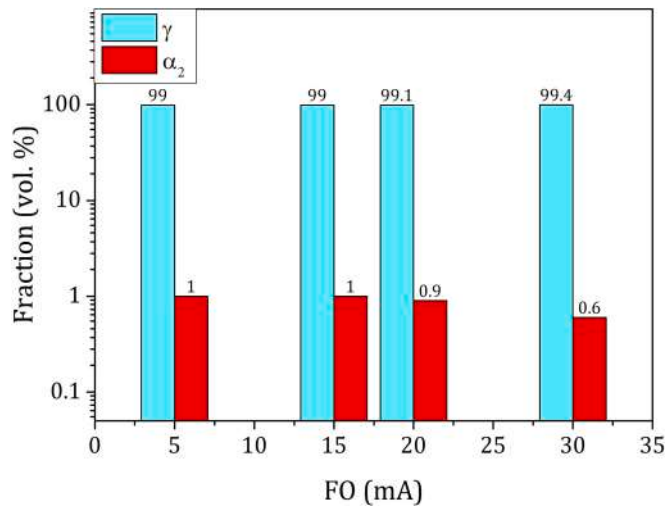


Fig. 3. Results of Rietveld phase quantification for Ti-48Al-2Cr-2Nb samples produced with different FOs.

particular, the fine-grained zones were severely reduced in the resulting microstructure of the FO<sub>+30</sub> specimen, thus determining a general decrease in the overall hardness (see the upper part of Fig. 2).

The phase quantification using the Rietveld method performed on the samples (Fig. 3) confirmed that an increment of the FO involved a gradual reduction of  $\alpha_2$ -phase content from FO<sub>+5</sub> to FO<sub>+30</sub>.

Since  $\alpha_2$ -phase may be underestimated by XRD analysis due to its small dimensions, the extreme conditions (FO<sub>+5</sub> and FO<sub>+30</sub>) were heat-treated at 1050 °C for 24 h. Subsequently, the specimens were cut and XRD measurements were performed. This temperature is well below any transformation temperature of the material, so the heat treatment just increased the dimensions of the  $\alpha_2$ -phase without altering the banded structures of the FO<sub>+5</sub> sample (Fig. 4.a and b) or the more homogeneous microstructure of the FO<sub>+30</sub> sample (Fig. 4.c and d) thus allowing the

possibility to measure  $\alpha_2$  content by means of EBSD analysis.

In the EBSD analysis, the high-angle grain boundaries (HAGBs) are indicated by black lines, while the  $\alpha_2$  and  $\gamma$ -phases are identified by red and light blue, respectively, The lower  $\alpha_2$  content for FO<sub>+30</sub> is confirmed clearly.

The Al-depleted regions that cause the banded structures are caused by a zone-by-zone differentiated Al evaporation. In particular, more pronounced evaporation of Al is revealed at the top of the melt pool, thus leading to inhomogeneous content of  $\alpha_2$ -grains, which lock the grain growth of the  $\gamma$ -grains where there is an  $\alpha_2$  accumulation [13,25]. This feature is particularly marked when highly energetic process parameters (high beam currents, slow beam speeds, and narrow line offsets) are used [25,46]. However, this feature can also be influenced by the FO, as demonstrated by the current work.

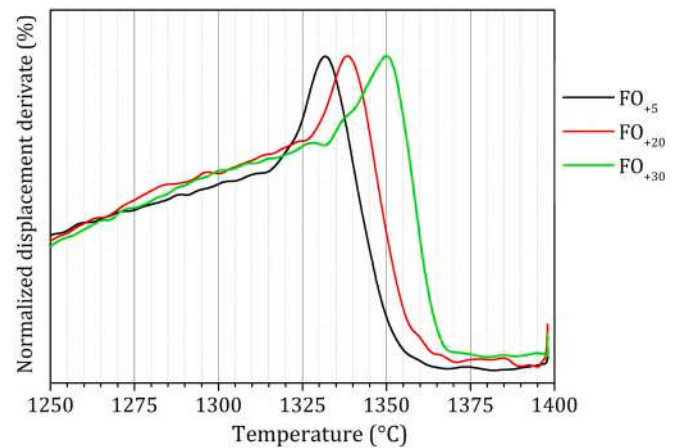


Fig. 5. Normalized displacement derivative curve over the temperature for samples produced with different FOs. The peaks indicate the  $\alpha$ -transus temperature, see text.

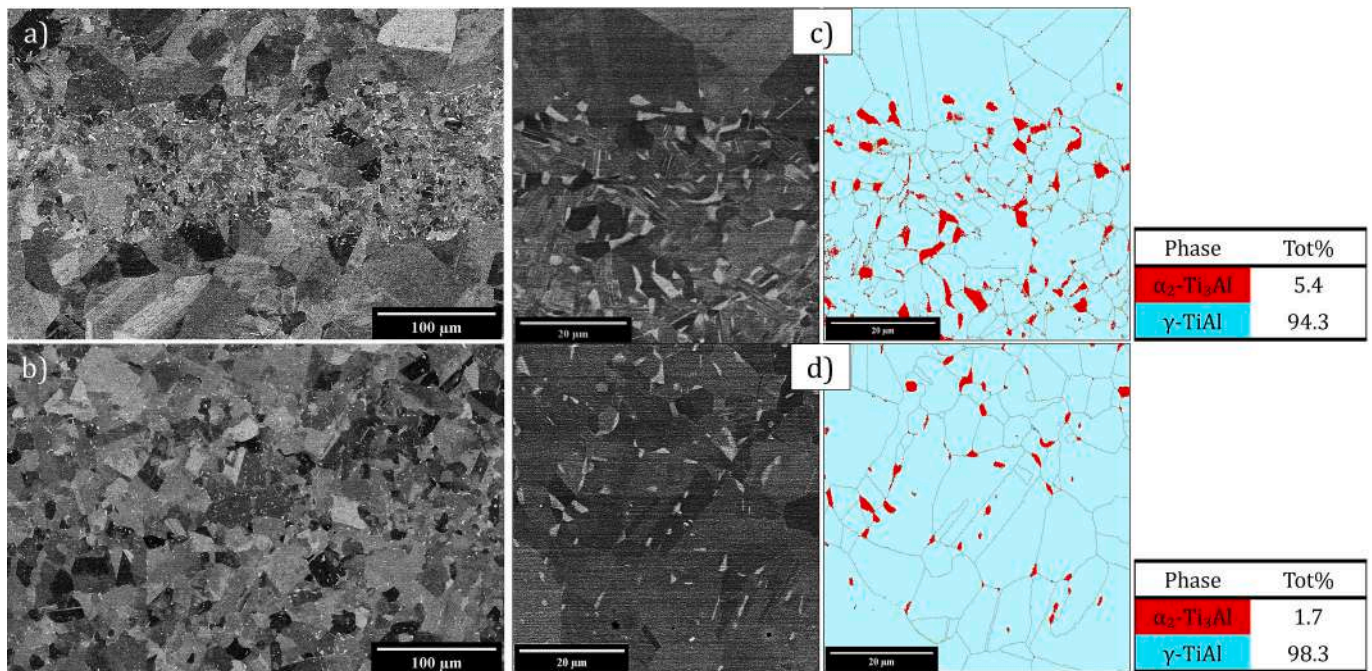


Fig. 4. SEM-EBSD images of samples heat-treated at 1050 °C for 24 h: a) low magnification view of the sample FO<sub>+5</sub>; b) SEM and EBSD phase map of sample FO<sub>+5</sub>; (c) low magnification view of the sample FO<sub>+30</sub>; d) SEM and EBSD phase map of sample FO<sub>+30</sub>. Note that in the EBSD phase maps, the HAGBs are indicated by black lines, while the  $\gamma$ -TiAl phase and the  $\alpha_2$ -Ti<sub>3</sub>Al phase are indicated in light blue and red, respectively. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

**Table 2**

Chemical compositions (at.%) of the powder and bulk samples as determined by OES-IPC and XRF methods. The Al loss (at.%) for the bulk specimens was obtained by subtracting the Al value of the powder.

Sample	Al	Cr	Nb	Ti	Al loss
Powder	48.73	1.92	2.00	Bal.	–
FO <sub>+5</sub>	47.48	1.92	2.03	Bal.	1.25
FO <sub>+20</sub>	47.60	1.95	2.02	Bal.	1.13
FO <sub>+30</sub>	48.04	1.95	1.99	Bal.	0.69

Consequently, it was also important to investigate the effects of the FOs on the Al evaporation. For this reason, both TMA and XRF chemical analyses were performed on the FO<sub>+5</sub>, FO<sub>+20</sub>, and FO<sub>+30</sub> conditions. The derivate of the displacement of the different samples determined by TMA is shown in Fig. 5. The peak identified the  $\alpha$ -transus temperature ( $T_{\alpha-\alpha+\gamma-\alpha}$ ) [47], which resulted to be influenced by the FO. In detail, the  $T_{\alpha}$  was 1332 °C, 1339 °C, and 1350 °C for FO<sub>+5</sub>, FO<sub>+20</sub>, and FO<sub>+30</sub>, respectively.

These results were confirmed by the XRF analysis (Table 2), where gradual mitigation of the Al evaporation with the increment of the FO was measured. In detail, comparing the Al content of the powder with the samples produced at different FOs, an Al loss of 1.25 and 0.69 at.% occurred at FO<sub>+5</sub> and FO<sub>+30</sub>, respectively, thus confirming that by changing the FO it is possible to modify the distribution of the energy density.

### 3.1. Correlation of the focus offset with the porosity, microstructure, Al loss, and hardness

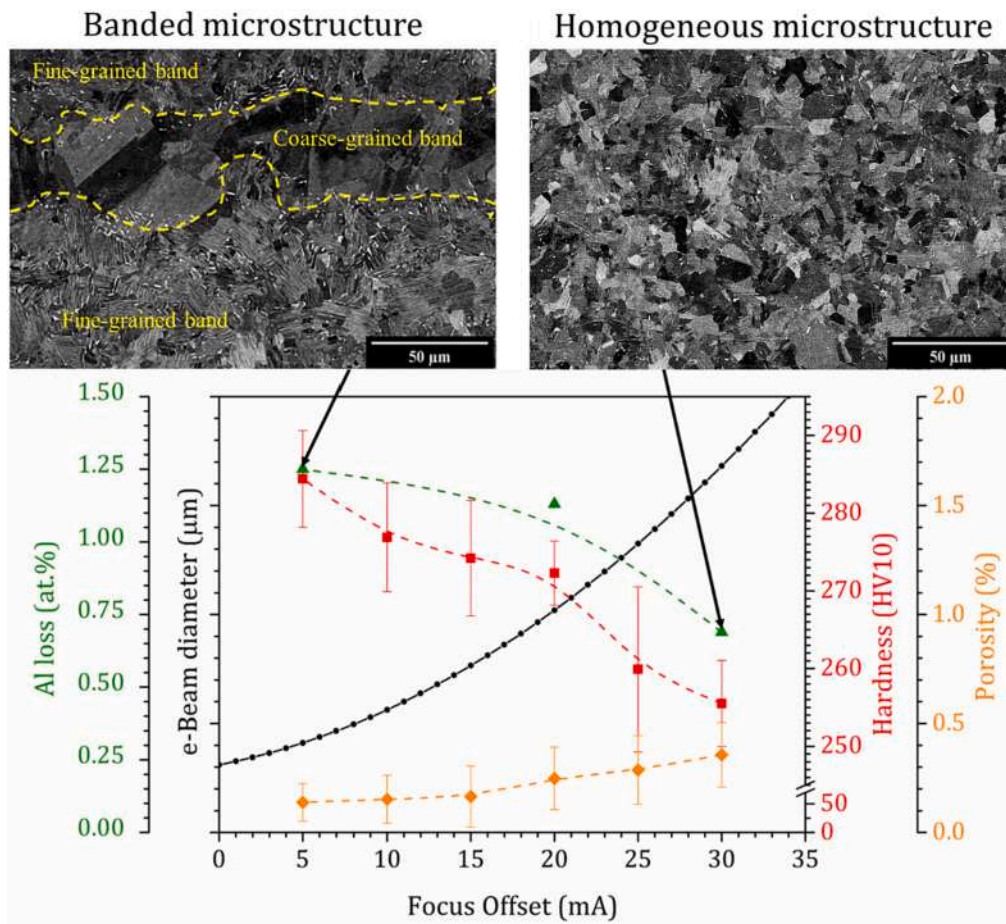
The increment of the FO enlarges the spot size following a parabolic trend that is influenced by the beam current used during the process. The machine suppliers did not completely disclose such kind of relationship, however, the only information we were able to find in literature about this topic, is the paper of Wadea et al. [33], in which the following equation in order to calculate the beam spot size from the FO was reported.

$$D_{\text{beam}} = 0.000102(FO)^2 + 0.00151(FO) + 0.131, \quad (1)$$

where  $D_{\text{beam}}$  is the diameter of the electron beam spot size in mm.

Fig. 6 shows that the trend of spot size variation caused by FO changes as calculated according to Eq. (1), well fits with the observed trends of our experimental measurements (residual porosity, hardness and Al loss). Note that, we did not display the spot size values on the related axis because the beam spot size must be considered qualitatively since the coefficients of Eq. (1) change when using different beam currents. Actually, in Wadea et al. [33] it is not defined at which beam current the given coefficients are associated. However, according to the authors' opinion, Fig. 6 is a useful way to relate the effect of FO variations to the resulting material.

A wider electron beam spot size generates a larger melt pool size, and consequently, the energy input delivered is distributed on a more extensive area, thus lowering the local energy that reaches the powder bed. So the FO can play a fundamental role in generating a homogenous



**Fig. 6.** Representation of the variation of the electron beam spot size (in black), Al loss (in green), hardness (in red), and porosity (in orange) with the FO. In addition, the microstructural features are shown. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

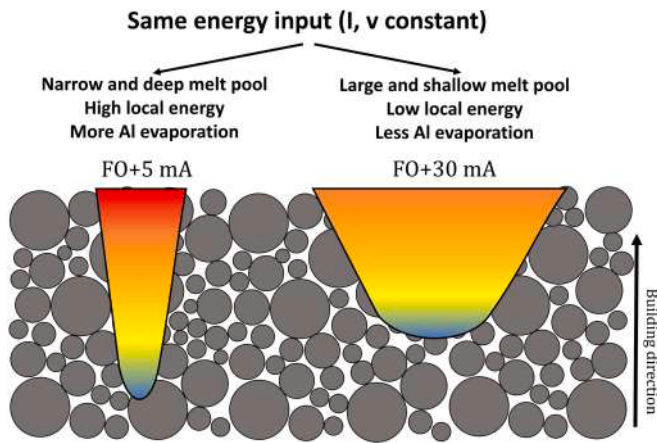


Fig. 7. Schematic illustration of shape and size of the melt pool with respect to focus offset in the EB-PBF process.

microstructure, mitigating the occurrence of a banded microstructure. A schematic illustration is presented in Fig. 7 showing the Gaussian beam and how it affects the melt pool size and, consequently, the Al evaporation.

Actually, the initial homogenous microstructure is a fundamental target for  $\gamma$ -TiAl based components since an inhomogeneous microstructure will result in undesirable and not uniform mechanical properties [48]. Moreover, the inhomogeneous microstructure cannot be homogenized through simple heat treatments, but will require high-temperature annealing followed by oil quenching [13]. This might induce cracks and metastable transformation unsuitable for industrial applications, especially for large components.

The parabolic trend of the spot size with respect to the FO explains the reason why the FO<sub>+15</sub> and FO<sub>+20</sub> still have a banded microstructure being more similar to FO<sub>+5</sub> than FO<sub>+30</sub>. In fact, going far from the minimum of the parabolic curve, the effect of increasing FO of the same quantity produces a wider spot size. Thus, FO<sub>+30</sub> was revealed to be a condition that was successful for having homogeneous microstructure, with just a slight increase of spherical porosity without the presence of LoF.

### 3.2. Microstructural evolution under heat treatments

The heat treatments are mandatory for  $\gamma$ -TiAl-based components in order to reach the desired mechanical properties. For this reason, the influence of heat treatment on the sample FO<sub>+5</sub> composed of a banded microstructure and the sample FO<sub>+30</sub> with homogenous microstructure was investigated.

The heat treatment consisted of annealing at 1310 °C for 2 h, followed by cooling to room temperature at about 125 °C/min, as reported in previous works on Ti-48Al-2Cr-2Nb material which was processed by EB-PBF [13,19]. The temperature was chosen to bring the material to the  $\alpha + \gamma$  phase-field region, thus obtaining a duplex microstructure after cooling. The same temperature was applied intentionally for both conditions (FO<sub>+5</sub> and FO<sub>+30</sub>) despite the different overall chemistry since the aim is not to reach a target microstructure after heat treatment, but to compare the microstructural homogeneity.

Fig. 8 shows the microstructures of the samples heat-treated at 1310 °C for 2 h for sample FO<sub>+5</sub> (Fig. 8.a and b) and sample FO<sub>+30</sub> (Fig. 8.c and d). The sample FO<sub>+5</sub> presented wide lamellar colonies formed from the previous fine grains duplex  $\gamma/\alpha_2$  bands. These zones of the sample are the weakest point of the microstructure, leading to poor mechanical properties, as reported in the works of Todai et al. [24,26].

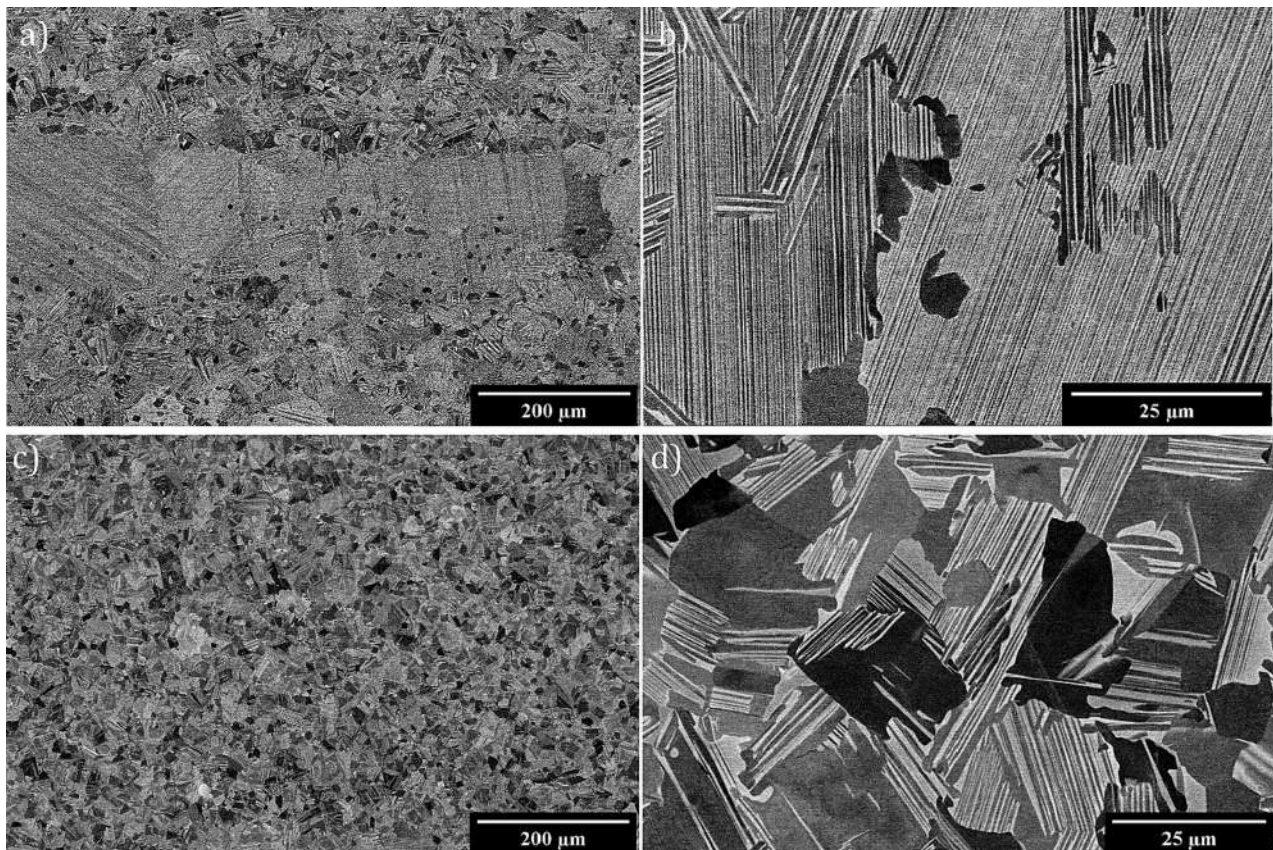
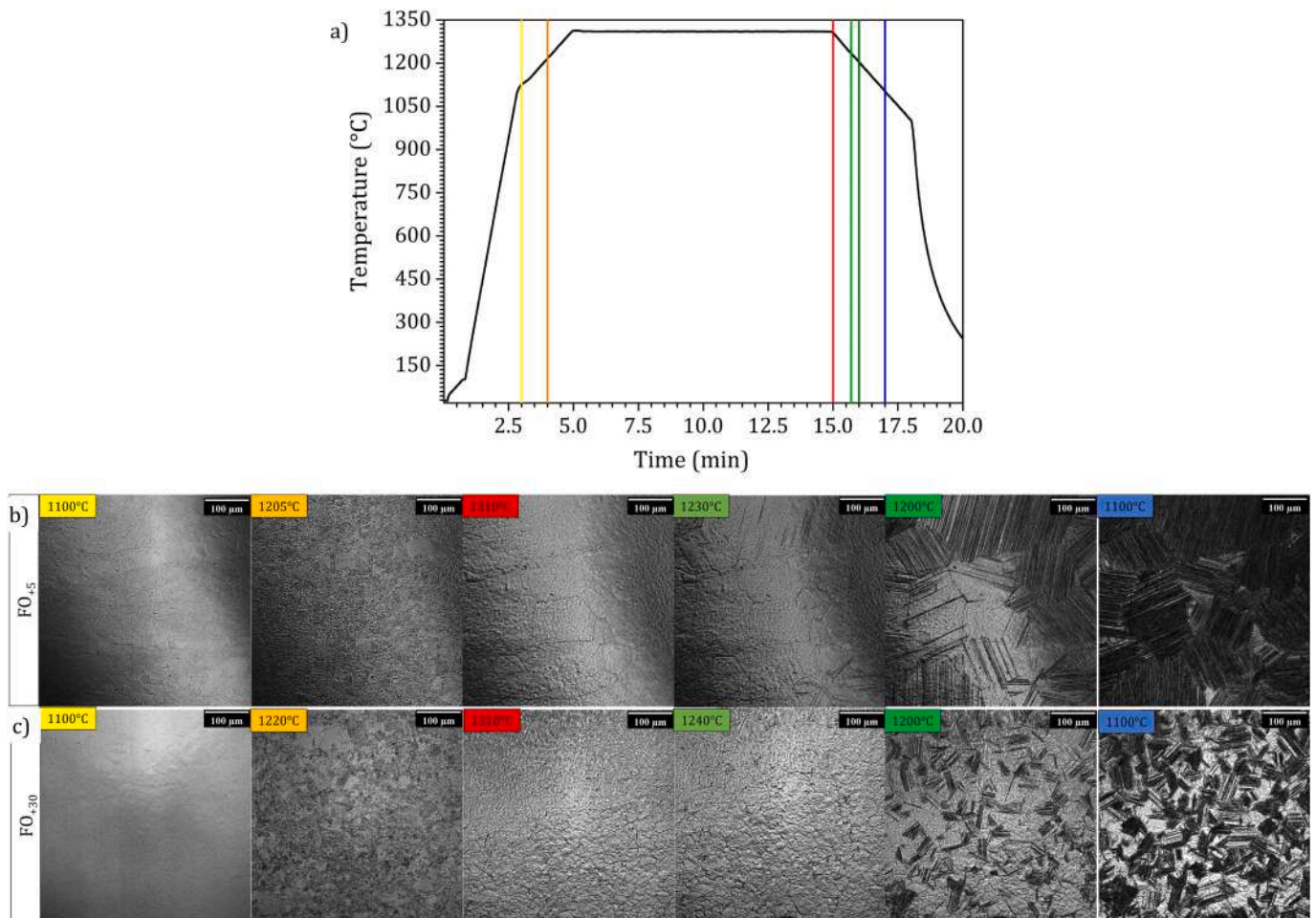


Fig. 8. a-b) SEM-BSE images of the heat-treated sample FO<sub>+5</sub> at different magnifications; c-d) SEM-BSE images of the heat-treated sample FO<sub>+30</sub> at different magnifications. Note that the heat treatment was performed at 1310 °C for 2 h, followed by a cooling rate of around 125 °C/min.



**Fig. 9.** LSCM results: a) temperature profile of the heat treatment: in b) images of the microstructure evolution for sample  $FO_{+5}$  c); images of the microstructure evolution for sample  $FO_{+30}$ . Note that the lines in a) point out where the microstructures at different temperatures were taken (see text).

On the contrary, the heat-treated sample  $FO_{+30}$  resulted in a more homogeneously duplex microstructure. The content of the lamellar phase was inferior because of the higher  $T_{\alpha}$  with respect to  $FO_{+5}$ , but what was more important, the lamellar colonies were homogeneously distributed within the material (Fig. 8.c and d). If required by the application, the lamellar content can be increased by selecting a higher temperature for the heat treatment. This means, the microstructure can be tuned for the desired mechanical properties without risking the formation of large lamellar bands.

In order to directly observe the microstructure evolution during the heat treatment, LSCM analysis was performed. This technique allows video-recording of microstructural changes which occur with increasing of temperature. With LSCM, not only the different temperatures for the phase transformations due to the different Al-loss can be seen, but especially it was detected that the transformations appear contemporary and homogeneously in the sample with  $FO_{+30}$ , while take place at different times and temperatures for different zones of the sample processed with  $FO_{+5}$ . Microstructure images were extracted from the video recorded during the analysis at different temperatures, as pointed out by the lines in Fig. 9.a. In the supplementary data, the complete video of the measurement is available.

For sample  $FO_{+5}$ , the  $\gamma$  dissolution in the  $\alpha$ -phase starts to be visible at around 1205 °C (Fig. 9.b). The dissolution initiated from fine  $\gamma$ -grains and  $\alpha_2/\gamma$  regions and finished in the coarse  $\gamma$ -grains band. At 1310 °C (Fig. 9.b), the  $\alpha$ -grains appeared coarse with a maximum dimension of around 200  $\mu\text{m}$ . The lamellar colonies of  $\gamma/\alpha_2$ -phases started to form at the  $\alpha$ -phase grain boundaries during the cooling at around 1230 °C,

following the steps reported by Appel et al. [49]. In detail, the formation of  $\gamma$  and  $\alpha_2$  lamellae follow the sequence  $\alpha \rightarrow \alpha + \gamma \rightarrow \alpha_2 + \gamma$  with the applied cooling rate (100 °C/min). For the formation of the  $\gamma$ -lamellae, the stacking fault of the hcp lattice of the  $\alpha$ -phase is the essential initial step. Then the formation can be subdivided into three phenomena; i) lattice transformation, ii) diffusion processes to adjust the composition of the  $\gamma$ -phase, and iii) ordering of the tetragonal face-centered  $\gamma$ -phase.

For sample  $FO_{+30}$ , the  $\gamma$  dissolution in the  $\alpha$ -phase started to be visible at around 1220 °C (Fig. 9.c). The higher temperature is consistent with the higher Al content of this sample with respect to the  $FO_{+5}$ . In this case, the dissolution was uniformly distributed in the overall investigated area. At 1310 °C, the microstructure mainly consisted of homogeneously dispersed  $\alpha$  grain with a maximum dimension of 50  $\mu\text{m}$ . For the  $FO_{+30}$  condition, the lamellar precipitation started at around 1240 °C, so about 10 °C degrees higher when compared to the  $FO_{+5}$  state, again in agreement with the overall higher Al content.

For sample  $FO_{+5}$ , almost all the  $\alpha$ -phase was transformed in lamellar grains of  $\gamma$  and  $\alpha_2$ -phase at 1100 °C, while only a few regions remained  $\gamma$ -phase at the grain boundaries at room temperature. On the contrary, for the sample  $FO_{+30}$ , the final microstructure (below 1100 °C) appeared homogeneously duplex.

Therefore, the Al-depleted regions were characterized by early growth of the  $\alpha$ -phase, which could grow enormously in the fine duplex bands locked only by the coarse  $\gamma$ -phase bands, which are the Al-rich regions. On the other hand, a homogeneous microstructure led to homogeneous nucleation of  $\alpha$ -phase and uniform grain growth within the material.

## 4. Conclusions

An approach to contain the Al-loss typical of the EB-PBF process for intermetallic  $\gamma$ -TiAl components is to minimize the energy level of the process parameter for the production. However, there is a limit to such kind of approach because minimizing the beam current or increasing the beam speed, or increasing the line offset will, at a certain point, turn in not being able to have a completely dense material, but some process-induced porosity, the so-called lack-of-fusion defects, starts to form.

In this paper, the FO parameter is revealed to be another key parameter that can be optimized for generating a homogenous microstructure in an engineering Ti-48Al-2Cr-2Nb alloy because modifying the spot size can contribute to lower the energy level delivered in the powder bed.

The following conclusions can be drawn from the present study:

- 1) The increment of the FO mitigated the Al-loss as demonstrated by TMA and XRF analyses and a FO of 30 mA produced a homogenous microstructure.
- 2) Among the explored FOs, the porosity slightly increased with the FO increment. The porosity level was below 0.5 % for all the conditions, and what is more important, the investigations reveal only gas porosity defects, while no LoF defects were detected.

For the first time, to the authors' knowledge, in this work the microstructural evolution and phase transformations of a Ti-48Al-2Cr-2Nb alloy was recorded by means of LSCM analysis. The LSCM analysis revealed the phase transition  $\alpha_2 + \gamma \rightarrow \alpha + \gamma$  during heating at different temperatures for the FO<sub>+5</sub> and the FO<sub>+30</sub> conditions. The growth of the  $\alpha$ -phase became visible at 1205 °C and 1220 °C for FO<sub>5</sub> and FO<sub>+30</sub>, respectively. For the FO<sub>+5</sub> condition, the  $\alpha$ -phase nucleates from the  $\alpha_2/\gamma$  duplex band. Moreover, for this condition, the  $\alpha$ -phase reached larger sizes than the FO<sub>+30</sub> condition. During cooling, for the FO<sub>+30</sub>, the lamellar formation started at a higher temperature (1240 °C) than the FO<sub>5</sub> (1230 °C). This resulted in a banded microstructure with large lamellar colonies for the FO<sub>+5</sub> state, while the FO<sub>+30</sub> state exhibited a homogenous finer duplex microstructure.

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.jmapro.2023.01.061>.

## 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.

## Acknowledgment

This project has received funding from the Clean Sky 2 Joint Undertaking under the European Union's Horizon 2020 research and innovation program under grant agreement No. 821274. The authors want to acknowledge also the Integrated Additive Manufacturing Centre at Politecnico di Torino (IAM @PoliTo), where the specimens were produced.

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