



Relationship between slag layer and in-situ nanoparticle formation in PBF–LB using reactive atmospheres

Christian Felber¹ · Erin Yule¹ · Eric A. Jägle¹

Received: 30 March 2026 / Accepted: 6 July 2026
© The Author(s) 2026

Abstract

Oxide dispersion strengthened (ODS) materials are typically processed using powder metallurgy routes or additive manufacturing to ensure homogeneous nanoparticle distribution in the materials. Processing routes where the nanoparticles are formed during manufacturing (in-situ) can effectively prevent problems occurring in ex-situ routes (e.g. powder blending) such as nanoparticle agglomeration. One of the approaches to manufacture in-situ ODS materials is the use of reactive process gases during powder bed fusion–laser beam (PBF–LB). Processing with carbon dioxide (CO₂) causes O uptake, which promotes nanoparticle formation. Manufacturing in CO₂ is accompanied by the formation of surface slag layers on the samples, whose effect on nanoparticle formation is yet unclear. In this work, the role of the slag layer on the formation of nanoparticles, as well as the layer formation mechanism, is studied. For this purpose, a model alloy, consisting of pure Fe alloyed with 4.3 wt% Ti is processed in CO₂, and the influence of the powder layer height, sample height, and re-melting on slag layer and nanoparticle formation is investigated. Results show that nanoparticles do not originate from the slag layer. Instead, the slag layer is formed by liquid nanoparticles floating to the surface of the melt pool. The slag layer accumulates from layer to layer until the slag layer thickness converges to a steady value. This work contributes to the understanding of particle formation mechanisms occurring in melt pools in additive manufacturing processes, which is necessary to define the requirements for alloy design of advanced in-situ particle reinforced ODS materials.

Keywords Material-gas interaction · Slag layer · In-situ particle formation · Process atmosphere · Powder bed fusion–laser beam

1 Introduction

Oxide dispersion strengthened (ODS) materials offer excellent high temperature properties, e.g. high temperature strength and creep resistance, which makes them ideal candidates for applications in turbine engines and aerospace engineering [1]. Fe-based ODS materials stand out from this broad group of materials as they additionally offer high radiation tolerance for applications in nuclear power reactors and

similar environments [2]. Additive manufacturing (AM) is nowadays frequently used to manufacture components from these materials, as processing nanoparticle-containing powder blends is difficult by conventional manufacturing routes, e.g. casting. There are ex-situ and in-situ approaches when introducing nanoparticles. Ex-situ approaches include ball milling and powder blending [3–5]. In-situ approaches are routes where the nanoparticles form during AM processes. Oxygen (O) is either introduced during gas atomization [6, 7], by oxide particles with low stability [8, 9] or during manufacturing with O-containing process atmospheres [10–14]. Nanoparticles form in the materials by reactions between O and the alloying elements in the melt pool. High levels of O in the materials are desired to increase particle formation.

Instead of using small O additions in an argon (Ar) atmosphere in the process chamber, more reactive atmospheres such as pure nitrogen (N₂) or carbon dioxide (CO₂) are potential candidates for in-situ nitride and oxide formation in AM processes [15–19]. The interaction between material

✉ Christian Felber
christian.felber@unibw.de

Erin Yule
erin.yule@unibw.de

Eric A. Jägle
eric.jaegle@unibw.de

¹ Institute of Materials Science, University of the Bundeswehr Munich, Neubiberg, Germany

and process gas using an N_2 atmosphere for Fe-based materials is low compared with CO_2 and often requires alloying to increase the reaction with N_2 [20], hence, CO_2 might be a better choice to process in-situ particle reinforced materials in PBF-LB.

Regardless of the chosen approach to manufacturing ODS materials in AM, most authors concentrate on the properties of the bulk microstructure and neglect the formation of slag and slag layers at the sample surface. The formation of oxide-rich slag layers is especially relevant when manufacturing in reactive atmospheres. Yin et al. [13] processed pure yttrium (Y) decorated 316L powder under Ar- O_2 atmospheres with O_2 fraction of 1–2 wt% and observed the formation of a Y_2O_3 slag layer at the sample surface. Ikehata et al. [18] processed Fe-Ti alloys in pure N_2 atmosphere, causing the formation of a Ti(O,N) layer at the samples' surface. The formation of slag layers in AM processes is also shown in simulations. It is predicted for low O contents originating from residual O from the process chamber or from the powder itself [21–23]. However, the influence of slag layers on nanoparticle formation has not been studied in the literature to the best of our knowledge.

Several authors [13, 24–26] observed large slag inclusions entrapped in their samples, similar to work of Felber et al. [27]. These micrometer-sized oxide particles originated from the slag layer and were caused by insufficient heat input (E_V) during printing. Consequently, the question emerges as to whether nanoparticles also emanate from the slag layer. Moreover, it is relevant to investigate whether the slag layer can be used to augment nanoparticle formation. For that purpose, it is necessary to understand how slag layers form and how they can be manipulated. It is known that the slag layer thickness, δ_{ox} , increases with increasing energy input (E_V) [27]. The volume fraction and size of the nanoparticles increase simultaneously.

In this work, the formation of slag layers and their influence on nanoparticle formation of pure Fe alloyed with 4.3 wt% Ti (designated as FeTi4.3) manufactured by PBF-LB in a reactive CO_2 atmosphere is studied. For this purpose, four different sets of experiments are conducted, investigating the effect of the powder layer thickness, build height, re-melting, and atmosphere on slag layer and nanoparticle formation. The manufacturing parameters, P and v , were kept constant through all individual experiments as their effect on O uptake, oxide fraction and microstructure evolution was elucidated in [27]. The slag layer thickness and particle fraction are determined by optical and scanning

electron microscopy and correlated with chemical analysis. The results obtained provide a fundamental basis for understanding the role of the slag layer in nanoparticle formation in additively manufactured ODS alloys.

2 Methodology

2.1 Materials

Experiments were conducted with a powder blend consisting of Fe with 99.95% purity mixed with 4.3 wt% Ti Grade 1 using a Turbula (Willy A. Bachhofen) tumble mixing device. The tumble mixer operated at 70 rpm for four hours, ensuring a homogeneous powder blend. Fe powder was purchased by metals for printing (m4p) with a PSD of 15–45 μm , and Ti powder from Eckart TLS with a PSD of 20–63 μm . The composition of the elemental powders is given in Table 1. CO_2 with a purity of 99.5% and Ar with a purity of 99.96%, supplied by Linde, were used for printing.

2.2 Manufacturing

An Aconity3D Midi+ was used to print samples. It is equipped with an IR ($\lambda = 1064$ nm) fiber laser with a Gaussian beam profile, a spot diameter of 80 μm , and a maximum power of 1 kW. Samples were manufactured with $P = 200$ W and $v = 800$ mm s^{-1} to achieve minimum porosity. The powder layer thickness, t , was set to 30 μm (“reference”), if not mentioned otherwise, and a 90° rotation strategy in between layers was chosen in each experiment. The hatch distance, h , was chosen to be equal to the laser spot diameter using a bi-directional scanning strategy. The residual oxygen concentration in the process chamber was set to 0.04 wt% in each experiment. Residual O concentration is measured with a lambda probe. The gas flow was set automatically to approximately 13–16 l min^{-1} , which was necessary to keep an excess pressure of 50 mbar in the process chamber. Simple cuboids with varying sample geometry, depending on the experiment, were printed in CO_2 or Ar atmosphere. A summary of all samples for each print is shown in Table 2.

The first experiment, “Powder layer height” (Fig. 1a), consisted of three separate build jobs where the powder layer thickness, t , was varied using 15 μm , 30 μm , and 60 μm . One cube ($10 \times 10 \times 5$ mm³) for microstructure analysis and 10 cubes for chemical analysis ($3 \times 3 \times 5$ mm³) were printed in each build job. As the sample geometry is identical for

Table 1 Chemical composition of Fe powder and Ti Grade 1 powder according to the certificate provided by the suppliers

Element (wt%)	Fe	C	O	S	P	
Fe	Bal	0.003	0.09	0.0038	<0.01	
Element (wt%)	Ti	Fe	O	N	C	H
Ti Grade 1	Bal	0.044	0.08	0.006	0.012	0.001

Table 2 Overview and description of samples of the four experimental series

Designation	<i>t</i>	Atm	
Layer height			Number of layers
t=15	15 μm	CO ₂	334
t=30	30 μm		167
t=60	60 μm		84
Build height			Sample height
1 layer	30 μm	CO ₂	0.03 mm
5 layers			0.15 mm
10 layers			0.30 mm
30 layers			0.90 mm
50 layers			1.50 mm
100 layers			3.00 mm
200 layers			6.00 mm
400 layers			12.0 mm
600 layers			18.0 mm
Re-melt			Number of re-melting cycles
0×	30 μm	CO ₂	No re-melting
1×			1 × re-melting
2×			2 × re-melting
3×			3 × re-melting
Multi atmosphere			Sequence
100 CO ₂	30 μm	CO ₂	3 mm sample printed in CO ₂
100 CO ₂ +R _{CO2}		CO ₂	Surface re-melted in CO ₂
100 CO ₂ +R _{Ar}		Ar	Surface re-melted in Ar
100 CO ₂ +1Ar	30 μm	Ar	Printing 1 layer in Ar on top of CO ₂
100 CO ₂ +10Ar	30 μm	Ar	Printing 10 layers in Ar on top of CO ₂
100 CO ₂ +50Ar	30 μm	Ar	Printing 50 layers in Ar on top of CO ₂
100 CO ₂ +50Ar+1CO ₂	30 μm	CO ₂	Printing 1 layer in CO ₂ on top of Ar
100 CO ₂ +50Ar+10CO ₂	30 μm	CO ₂	Printing 10 layers in CO ₂ on top of Ar
100 CO ₂ +50Ar+50CO ₂	30 μm	CO ₂	Printing 50 layers in CO ₂ on top of Ar

all three build jobs, the number of layers required to manufacture the samples depends on *t*, which results in different total laser exposure times in the CO₂ atmosphere. The slag layer was removed mechanically for half of the samples for chemical analysis by grinding until all surfaces were bare metal, to reveal the O level in the bulk. Samples that were ground are designated as “w/o surface” and those that were not post-processed as “As build—AB”.

The second experiment, “Build Height” (Fig. 1b), consisted of ten cubes with a dimension of 10×10 mm² and variable sample height (Table 2). The tenth sample was used for chemical analysis and is identical to the one with the largest height. For this purpose, the sample was cut into five blocks (10×10 × 3.6 mm³) from which three samples each were cut for chemical analysis (4×3 × 3 mm³). As all samples for chemical analysis were cut from one large cube, they are all in w/o surface state.

The third experiment, “Re-melt” (Fig. 1c), covered four sets of samples. Each of the sets consisted of one cube (10×10 × 5 mm³) for microstructure analysis and 8 for chemical analysis (4 in AB, 4 in w/o surface state, 3×3 × 5 mm³). The sets differed in the number of re-melting cycles (0x, 1x, 2x, and 3x) applied to each layer. Re-melting was done directly after powder consolidation for each sample. For example, the one-time re-melting sample was exposed twice without interruption before the laser moved to the next sample.

The last experiment, “Multi-Atmosphere” (Fig. 1d), consisted of nine identical cubes (10×10 × 3 mm³) that were printed in CO₂. After reaching a sample height of 3 mm (=100 layers), the cubes were given 10 min to cool down. The first cube was the reference specimen (100 CO₂) and was not exposed to the laser anymore. The 2nd one was once re-melted without powder in CO₂. Afterwards, the chamber was opened to remove the CO₂ from the build chamber and flooded with Ar for 90 min. Reaching an O level of 400 ppm in the chamber, the 3rd cube was once re-melted in Ar without powder. Before printing on top of the 4th–9th cubes was resumed, the platform was lowered by 30 μm , and the first powder layer was manually distributed on top of the samples. Printing was then continued under Ar atmosphere for the remaining six cubes. One layer was printed on top of the 4th cube (100 CO₂+1Ar), 10 layers on the 5th (100 CO₂+10Ar), and 50 layers on the 6–9th cube (100 CO₂+50Ar) before the job was paused again to switch to the CO₂ atmosphere, following the same procedure as switching from CO₂ to Ar. Subsequently, printing was continued in CO₂ on top of the three remaining cubes 7–9. This time, no re-melting was performed, and one layer was directly printed in CO₂ on top of the 7th cube (100 CO₂+50Ar+1CO₂), 10 layers on top of the 8th cube (100 CO₂+50Ar+10CO₂), and 50 layers on top of the 9th cube (100 CO₂+50Ar+50CO₂).

2.3 Chemical and microstructure analysis

Samples were cut from the baseplates using a hand saw to avoid unnecessary heat input and contamination/corrosion by cooling liquids, both avoiding an increase in the O level during preparation. The oxygen content of the samples was determined using inert gas fusion analysis with an Inductar ONH (Elementar Analysensysteme GmbH). Three samples for each state were measured, and an average value was calculated.

A Zeiss Ultra 55 FEGSEM equipped with an EDAX EDS Octane 9 Super Elite detector was used to capture images for particle analysis and analyze the composition of the oxide particles and slag layer. Particle analysis for each sample was done using 8 images, captured using the

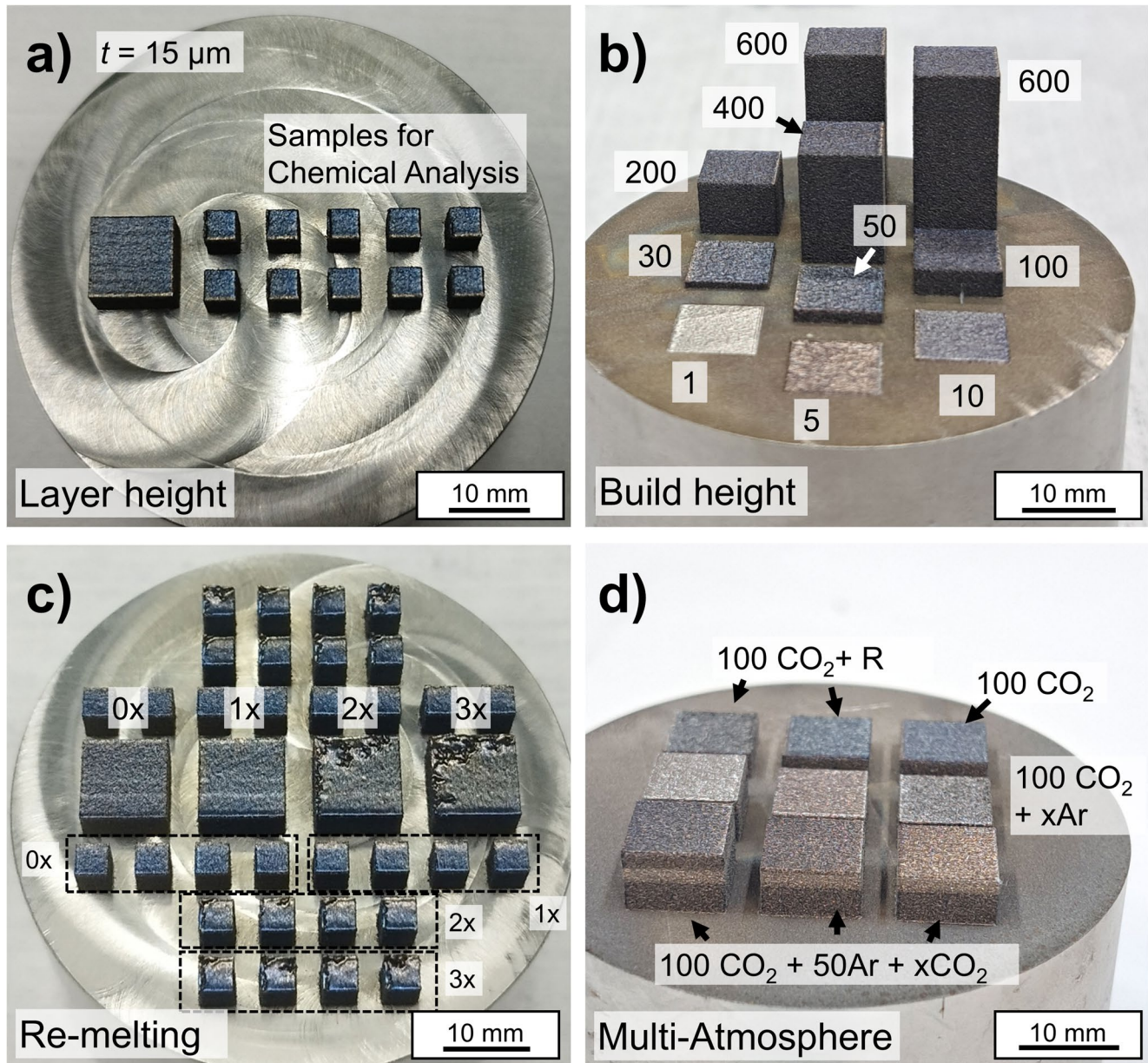


Fig. 1 Experimental series 1–4: **a** Samples for microstructural analysis ($10 \times 10 \times 5 \text{ mm}^3$) and chemical analysis ($3 \times 3 \times 5 \text{ mm}^3$) to study the influence of powder layer height ($t = 15 \text{ μm}$). The print jobs were repeated with $t = 30 \text{ μm}$ and $t = 60 \text{ μm}$. **b** Samples consisting of different numbers of layers to study the influence of build height (1–600 printed layers). Samples for chemical analysis are extracted from the second cuboid, consisting of 600 layers. **c** Samples for microstructure and chemical analysis were manufactured with integrated re-melting. The number of re-melting cycles after consolidation of each layer varies

from 0 to 3. **d** Samples were printed by alternating the process atmosphere. The base of each of the nine cuboids is the 100 CO₂ sample, which is the starting point for the eight other cuboids. +R displays samples that are once re-melted in Ar and CO₂. +xAr displays samples with x ($x = 1, 10, 50$) layers printed in Ar on top of the 100 CO₂ sample. +xCO₂ displays samples with x ($x = 1, 10, 50$) layers printed in CO₂ on top of the 100 CO₂ + 50Ar sample. More details on each sample are given in Table 2

ies from 0 to 3. **d** Samples were printed by alternating the process atmosphere. The base of each of the nine cuboids is the 100 CO₂ sample, which is the starting point for the eight other cuboids. +R displays samples that are once re-melted in Ar and CO₂. +xAr displays samples with x ($x = 1, 10, 50$) layers printed in Ar on top of the 100 CO₂ sample. +xCO₂ displays samples with x ($x = 1, 10, 50$) layers printed in CO₂ on top of the 100 CO₂ + 50Ar sample. More details on each sample are given in Table 2

this work to determine the deviations of each data point. For the Multi-Atmosphere experiments, images were taken close to the surface to ensure analyzing the top-most melt pools. For all other samples, images were captured at various locations within the bulk of the samples.

The fraction of inclusions originating from the slag layer, designated as “slag particles”, was determined using one cross-section of a remaining sample for chemical analysis or, if not available, a corresponding sample from microstructure analysis. Images were taken with a Keyence VK-3000 confocal microscope, and slag particle content was determined with the software ImageJ.

The slag layer thickness, δ_{ox} , was measured at the surface and the sides separately, as both can be different. For that purpose, δ_{ox} was calculated based on SEM images at low magnification of the whole cross-sectional area from a cube printed for chemical analysis ($3 \times 3 \times 4 \text{ mm}^3$ after cutting from the baseplate). 5–15 images (n) were taken from the surface and each of the two sides, depending on the magnification, which is necessary to cover the full length of the respective side. The area of the oxide, $A_{Ti-oxide}$, on the respective surface and its length, $l_{i,surface}$, is determined for each SEM image, and δ_{ox} is calculated by:

$$\delta_{ox} = \frac{1}{n} \times \sum_{i=1}^n \frac{A_{i,Ti-oxide}}{l_{i,surface}} \quad (1)$$

This procedure was required as δ_{ox} locally strongly fluctuates.

3 Results and discussion

3.1 Reference state

Felber et al. [27] showed that processing FeTi4.3 (4.3 wt% Ti) in CO_2 leads to the formation of an approximately $5 \mu\text{m}$ thick slag layer (Fig. 2a) on top and the sides of the samples as well as Ti-oxide nanoparticles (Fig. 2b) with an average diameter of approximately 35 nm . The nanoparticle composition is assumed to be mainly TiO . Slag particles (Fig. 2d) were incorporated in the bulk from the surface layer (Fig. 2c), resulting from manufacturing with low heat input (E_V). The oxygen in the samples is located in three forms: the slag layer, the slag particles, and nanoparticles.

3.2 Chemical analysis

The O content of the samples was measured in two states — AB and w/o surface. The difference between the two values is an approximation for the O content stored in the slag layer. Samples that are processed in CO_2 show a process gas uptake during manufacturing, compared to the powder level. Samples processed in Ar do not show process gas uptake and their oxygen level amounts to ca. $0.115 \text{ wt\%}$, independently of manufacturing parameters or strategy. The overall O uptake

does not appear to be significantly affected by the powder layer thickness used during printing (Fig. 3a). A lower powder layer thickness increases the number of layers that are exposed during manufacturing for the same sample height. The O stored in the slag layer decreases with increasing t , especially visible for the sample with $t = 60 \mu\text{m}$.

The O concentration at different locations within the samples, measured in the five blocks along the build direction, is constant (Fig. 3b). Note, no AB condition was measured for this experimental series, as the samples for chemical analysis were cut from the five blocks, which in turn were cut from a single cube with a dimension of $10 \times 10 \times 18 \text{ mm}^3$.

The re-melted samples show a strong increase in the oxygen level in AB and w/o surface condition with an increasing number of re-melting cycles (Fig. 3c). The increase in AB is larger than in the w/o surface condition, indicating that mostly the slag layer grows by re-melting. The increase in the w/o surface condition is either due to an increase in the nanoparticle fraction or the fraction of slag particles. Microscopy analysis is used in the following to break down the O distributions depending on the oxide forms and to explain the evolution of O concentration in the w/o surface condition.

3.3 Slag layer thickness

The slag layer thickness, δ_{ox} , decreases with decreasing t and hence decreasing number of printed layers per sample (Fig. 4a). However, δ_{ox} strongly varies along the surface, which complicates interpretation. The deviations increase with increasing δ_{ox} .

The slag layer on top of the samples quickly grows with increasing number of printed layers from $\delta_{ox} = 400 \text{ nm}$ for the first layer, until reaching a maximum of $\delta_{ox} \approx 4.5 \mu\text{m}$ after ca. 50 layers (1.5 mm) (Fig. 4b). This result shows that the slag layer accumulates upon printing until a certain constant thickness is reached.

δ_{ox} for re-melted samples (Fig. 4c) is highly affected by re-melting cycles, as already determined from chemical analysis (Fig. 3c). The slag layer at the sides strongly increases, whereas it decreases at the top of the samples. The decrease in δ_{ox} at the top surface is counterintuitive, as it was expected that increasing the contact duration with the atmosphere would increase O uptake and hence δ_{ox} . The reason for this behavior is elucidated in the discussion part.

The reference sample for the Multi-Atmosphere experiments, consisting of 100 layers printed in CO_2 (100 CO_2), is covered with a $3.4 \pm 0.3 \mu\text{m}$ thick slag layer on top. The build for this experiment consists of 9 of these samples, which serve as the basis for the Multi-Atmosphere experiments. Re-melting of the sample surface leads to an increase of δ_{ox} to $4.1 \pm 0.9 \mu\text{m}$ under CO_2 and $4.7 \pm 1.0 \mu\text{m}$ under Ar (Table 3).

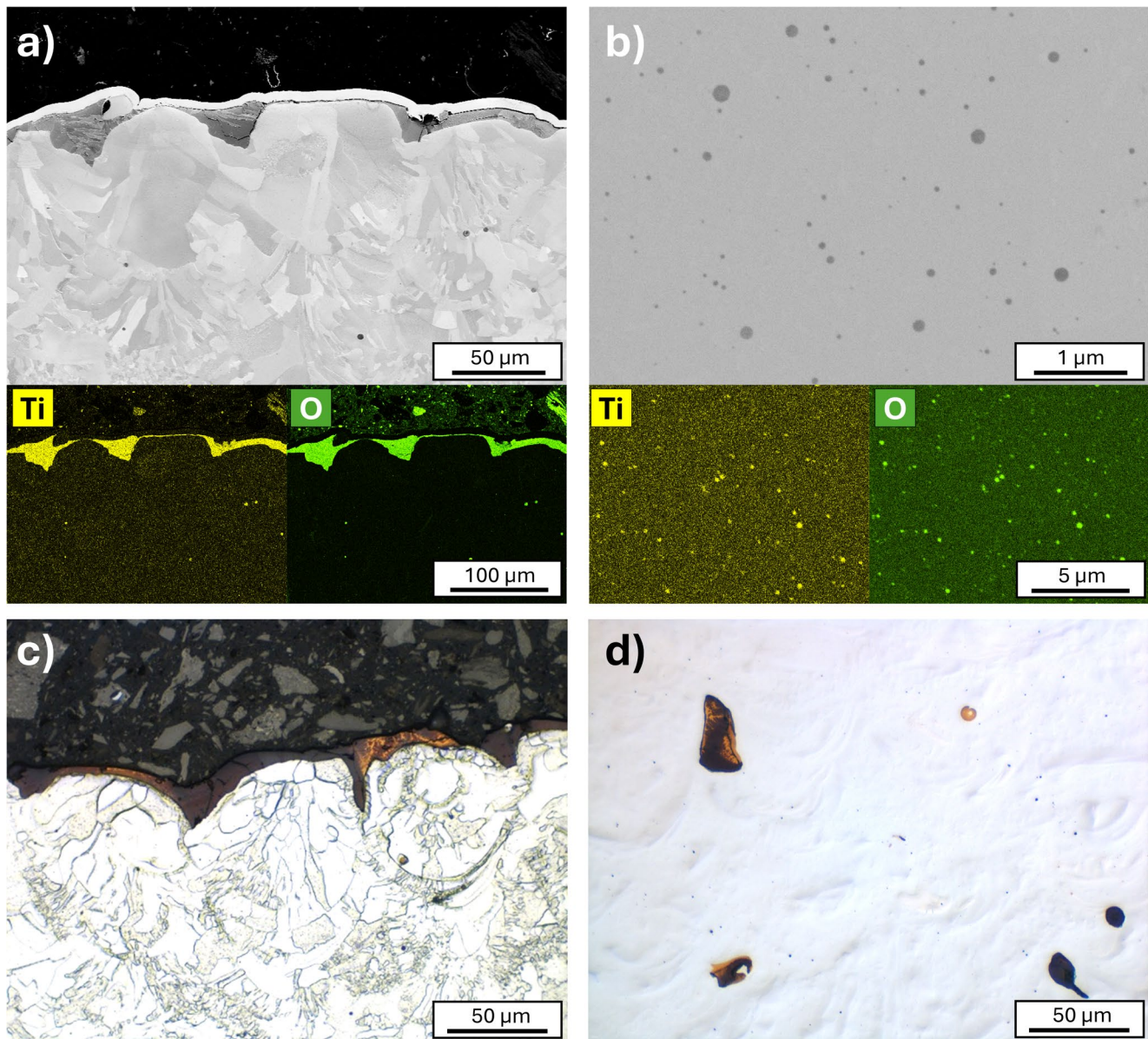


Fig. 2 Processing FeTi4.3 in CO₂ atmosphere causes the formation of **a** Ti-oxide slag layers on top of the samples and their sides and **b** Ti-oxide nanoparticles in the bulk. **c** The slag layer consists of two

phases—Ti₂O₃ (blueish-grey) and TiO (golden) as determined in XRD [27] from which **d**) micrometer size slag particles originate

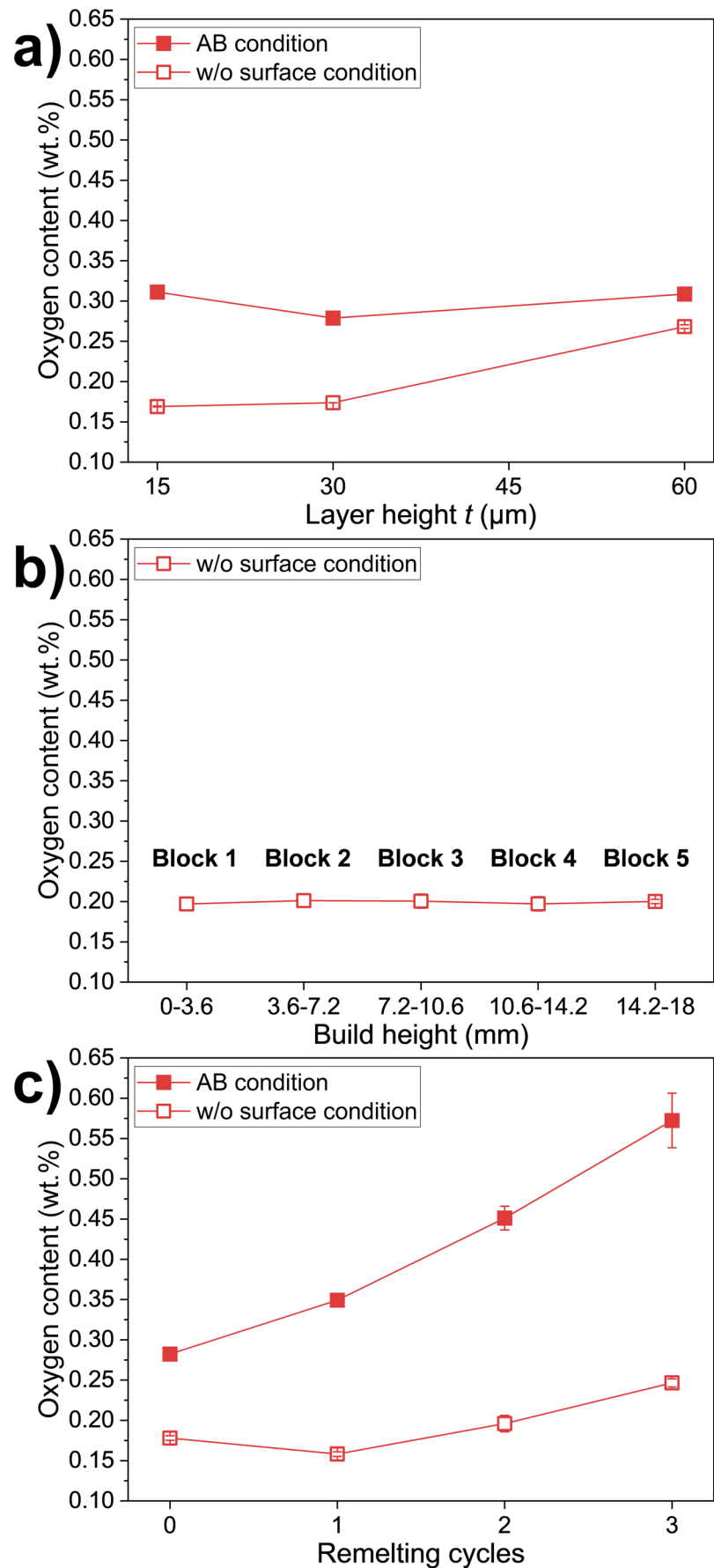
Printing is continued on the remaining samples in Ar, causing a drop in δ_{ox} , which proceeds with printing further layers until the slag layer nearly disappears. The opposite behavior is observed when printing is continued under CO₂ on top Ar-processed samples (Fig. 4d). The slag layer thickness increases similarly to that observed in the Build Height experiments (Fig. 4b).

3.4 Nano particle analysis

The powder layer thickness (Fig. 5a), build height (Fig. 5b), and re-melting (Fig. 5c) do not affect the particle size (d_p),

volume fraction (f_p), and number density (n_p) in the first two experiments. A slight trend towards larger particles and a lower number of particles might be visible for the re-melted samples with an increasing number of re-melting cycles (Fig. 5c), probably due to heat up of the samples by the longer laser exposure time. Increased melt pool temperatures favor growth and accelerate Ostwald ripening of nanoparticles [28]. The sample manufactured with $t=60\ \mu\text{m}$ (Fig. 5a) and the one $3\times$ re-melted in each layer (Fig. 5c) seem to be affected by process irregularities (e.g. recoating problems) as visible in Fig. 1a and c, which could have influenced nanoparticle formation.

Fig. 3 Chemical analysis of samples with **a** variable powder layer height, **b** along the build direction of the 18 mm high sample cut into 5 blocks, and **c** for samples using re-melting strategies. The composition is given for AB state (with the slag layer) and w/o surface state to measure the O stored in the bulk. The difference between the measurements corresponds to the O in the slag layer



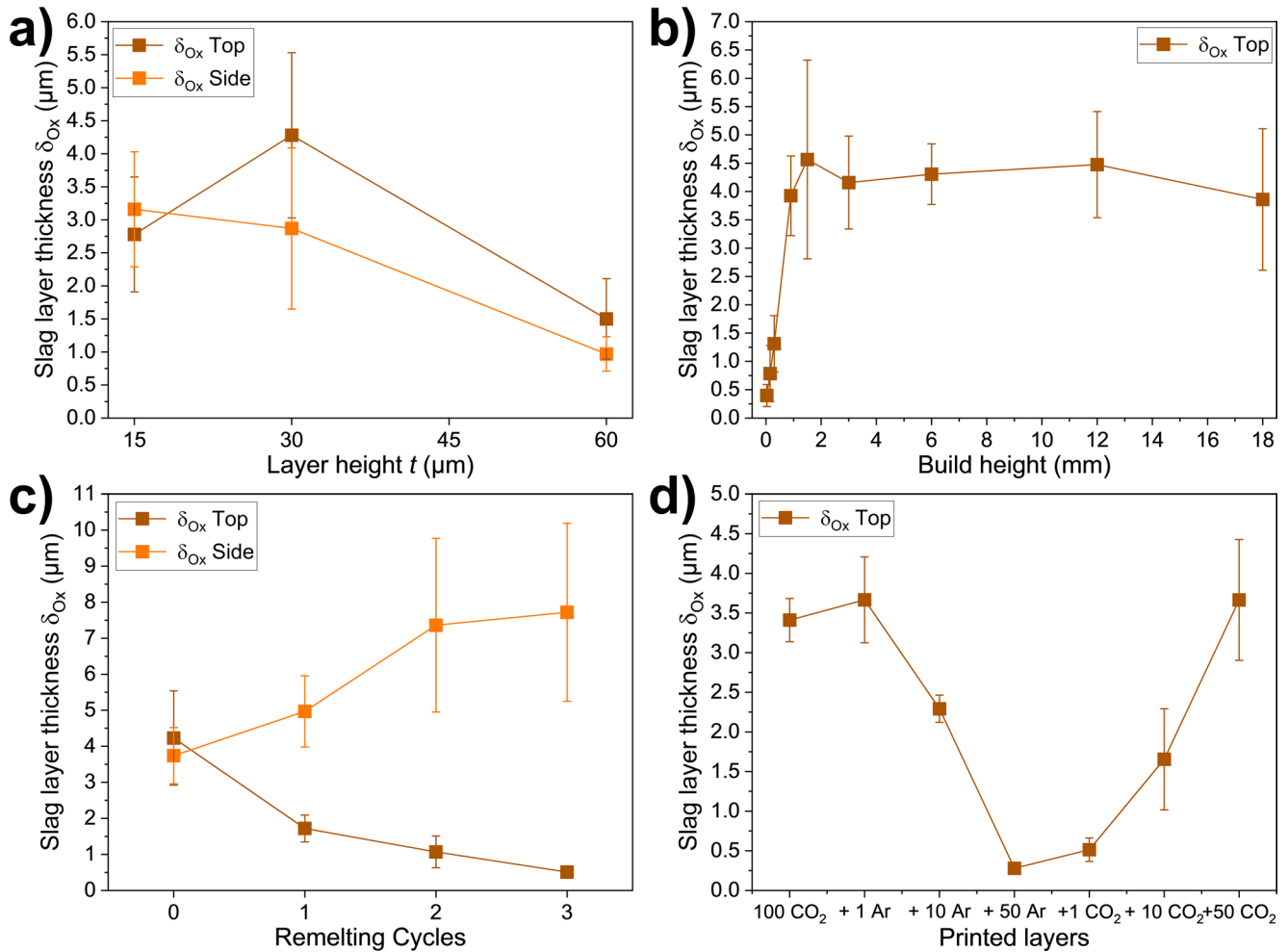


Fig. 4 Slag layer thickness δ_{ox} at the top (brown) and at the sides (orange) of the cubes dependent on **a** the powder layer thickness used for printing, **b** the build height of the printed samples and **c** the number

of re-melting cycles during processing. **d** shows the evolution of the slag layer when the atmosphere is switched from CO₂ to Ar and from Ar to CO₂ within one sample

Table 3 Slag layer thickness of 100 CO₂+R samples in CO₂ (R_{CO2}) and Ar (R_{Ar}), as well as for the reference sample (100 CO₂) from the multi-atmosphere experiments

	Slag layer thickness δ_{ox}
100 CO ₂	$3.4 \pm 0.3 \mu\text{m}$
100 CO ₂ +R _{CO2}	$4.1 \pm 0.9 \mu\text{m}$
100 CO ₂ +R _{Ar}	4.7 ± 1.0

In contrast, f_p and n_p are highly affected by the process atmosphere used in the Multi-Atmosphere experiment (Fig. 5d and Table 4). d_p is slightly increased for samples printed in CO₂ compared to those printed in Ar. Particle analysis for these samples was done close to the sample surface to ensure probing in the upper-most layer, which is especially important for samples that consist of only one layer printed in the changed atmosphere or the re-melted ones. The reference sample of the Multi-Atmosphere experiments, consisting of 100 layers printed in CO (100 CO₂), shows $f_p = 0.94 \pm 0.18 \text{ vol\%}$.

Re-melting in CO₂ (100 CO₂+R_{CO2}) does not change f_p significantly, whereas f_p drops for re-melting in Ar (100 CO₂+R_{Ar}) (Table 4). The nanoparticle concentration further drops when printing is continued in Ar from $f_p = 0.62 \pm 0.11 \text{ vol\%}$ for the first layer printed in Ar to $f_p = 0.40 \pm 0.13 \text{ vol\%}$ after only ten more printed layers in Ar.

When switching back to CO₂, the first printed layer in CO₂ contains a similar f_p than under Ar, which is explained by dilution of the probed layer (100 CO₂+50Ar+1CO₂) with the material printed in Ar below. This is the case as the melt pool depth is larger by a factor of 3–4 than the chosen layer height, t , of 30 μm . After 10 layers printed in CO₂ on top of 100 CO₂+50Ar, a comparable particle fraction and number density ($f_p = 1.04 \pm 0.24 \text{ vol\%}$, $n_p = 0.90 \pm 0.03 \times 10^{19} \text{ m}^{-3}$) are achieved as for the reference (100 CO₂) fully printed in CO₂.

3.5 Slag particle analysis

The third form of oxide, besides in the slag layer and nanoparticles, which occurs in the materials, are slag particles. Slag particles are micrometer-sized oxide particles that are randomly distributed in the materials. As elucidated in [27], these slag particles originate from the slag layer, hence their name. Slag particles result when the energy input and thus the melt pool lifetime are insufficient. The slag layer breaks up upon laser exposure and causes large oxide particles to float in the melt pool. These large particles tend to float to the surface again to merge with the newly formed slag layer, driven by their lower density. Insufficient time to reach the surface (limited by the melt pool lifetime) causes their incorporation in the bulk [27].

The slag content amounts to 0.2–0.4 vol% in most samples not affected by manufacturing problems, which seems to be typical for the parameters used and is constant along the build direction (Fig. 6a). Increasing the powder layer thickness, t , reduces E_V and results in porous samples that contain high slag contents (Fig. 6b).

Re-melting strongly affects the slag particle content in the materials. One-time re-melting is favorable to reduce the slag particle fraction, whereas multiple re-melting (2x, 3x) increases the slag particle content again. However, it is not the re-melting itself that causes the slag but the associated overheating of the surfaces, which causes increased surface roughness and hinders the deposition of a clean powder layer that fully covers the samples.

Samples that are affected by recoating issues are often porous. The slag often sits at the edges of the pores (Fig. 6d), which makes a proper analysis of the slag particle concentration by image analysis challenging and leads to overestimation of the real slag particle volume fraction. Avoiding these process irregularities comes hand in hand with limiting slag concentration in the samples.

3.6 Oxygen distribution

The data from microscopy, e.g. the slag layer thickness at the top and sides, the nanoparticle and slag particle fraction, can be used to calculate the corresponding fraction of O stored in the respective oxide forms (Fig. 7). This requires certain assumptions that are taken as follows:

- The slag layer composition used for calculation is Ti_2O_3 . The limited amount of TiO contained in the layer is neglected for simplicity [27].
- Slag particles are incorporated from the surface slag layer due to insufficient heat input during manufacturing. For this reason, the same composition, Ti_2O_3 , as for the slag layer, is assumed.

- Nanoparticles have a composition of TiO according to the Fe-0.2O-Ti phase diagram (Fig. 7) and [18].

The O content in TiO is 25 wt% compared to Ti_2O_3 with 33 wt%. Calculation is done for samples with a dimension of $3 \times 3 \times 4 \text{ mm}^3$, as used for chemical analysis. Details on the calculation are given in Appendix 1.

Figure 8 summarizes the evolution of O in the slag layer (top and sides), slag particles, and nanoparticles, with the change of a certain parameter in one single graph. The O fraction resulting from chemical analysis (C) and based on microscopy analysis (M) is further compared. Determination of O levels in the materials based on image analysis (M) and chemical analysis (C) show good agreement in most cases. Only samples that contain a high content of slag particles show larger deviations between the two methods. The content of slag particles seems to be systematically overestimated.

Figure 8a clearly shows that the powder layer thickness, t , is not affecting oxide fractions unless the energy input, E_V , is high enough to result in a stable printing process. The nanoparticle fraction, f_p , and the slag particle concentration are constant along the build direction (Fig. 8b). O levels determined by image analysis are slightly larger than those determined by chemical analysis, likely due to the overestimation of the slag concentration by image analysis. Re-melting causes an increased O uptake from the atmosphere, leading to enhanced oxide formation in the materials. However, mainly the O fraction of the slag layer and the slag particles is increased by re-melting, the O fraction of the nanoparticles is constant.

3.7 Correlation of slag layer and nanoparticle fraction

δ_{ox} and f_p of the Multi-Atmosphere experiment are plotted together to determine whether the nanoparticle concentration and slag layer thickness are correlated (Fig. 9). Both curves show a similar evolution, but δ_{ox} lags behind f_p when starting to print in Ar and also when printing is continued in CO_2 .

The reference state (100 CO_2) shows $f_p = 0.94 \pm 0.18 \text{ vol\%}$ and $\delta_{ox} = 3.4 \pm 0.28 \text{ }\mu\text{m}$, which are both comparable with the values from the previous three experimental series. Similar values are achieved for the 100 $\text{CO}_2 + 50\text{Ar} + 50\text{CO}_2$ sample. After 50 layers printed in Ar on top of samples processed in CO_2 (100 $\text{CO}_2 + 50\text{Ar}$), the slag layer nearly vanishes, and the nanoparticle concentration is approximately halved ($f_p = 0.49 \pm 0.09 \text{ vol\%}$ and $\delta_{ox} = 0.28 \pm 0.07 \text{ }\mu\text{m}$). After printing 50 layers in each of the two atmospheres, the values for f_p and δ_{ox} can be assumed to be representative of the respective atmosphere, Ar and CO_2 . f_p is adapting

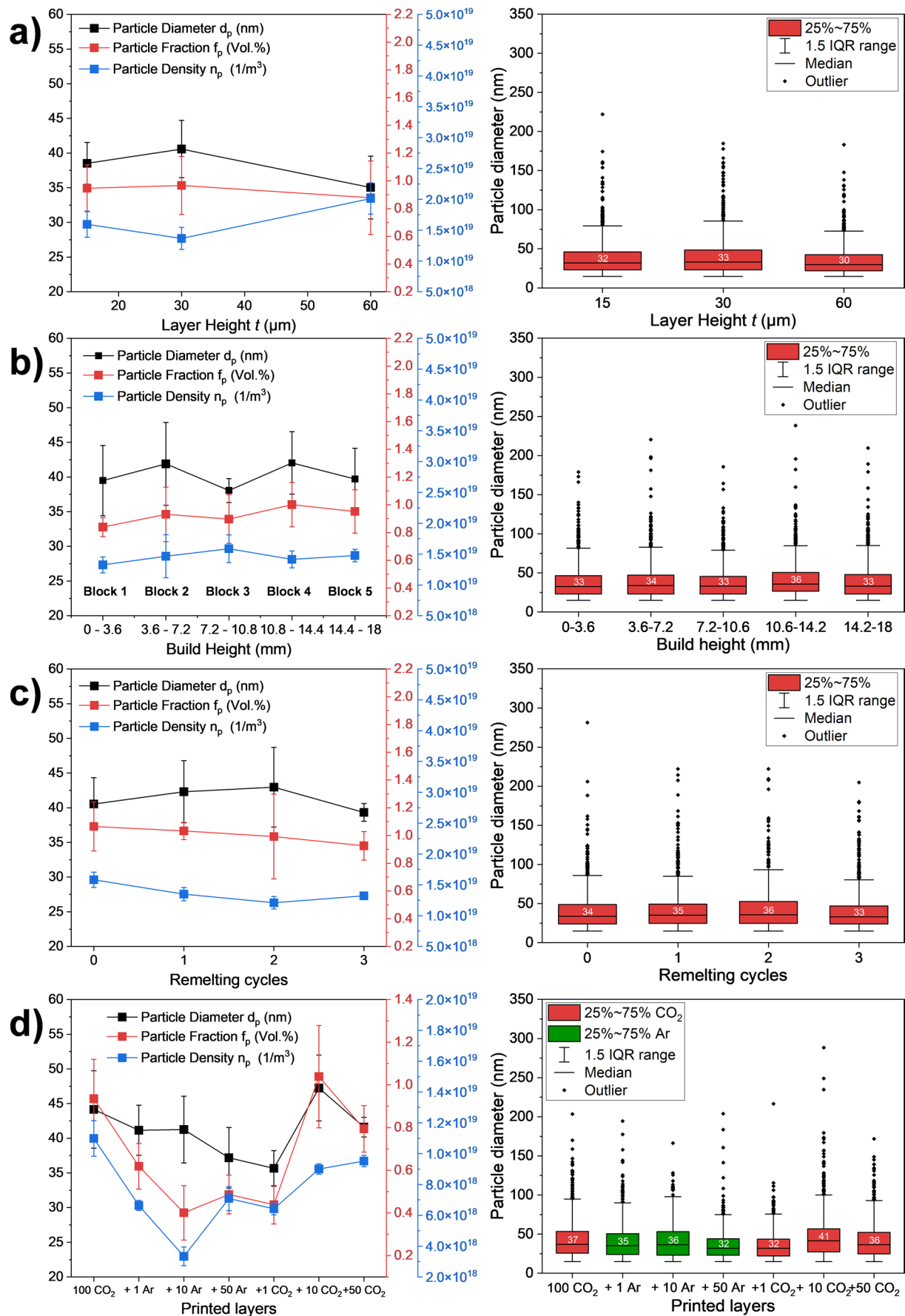


Fig. 5 Ti-oxide nanoparticle mean diameter d_p , particle fraction f_p , particle number density n_p , and particle distribution dependent on **a** the powder layer thickness used for printing, **b** the build height of the printed samples, and **c** the number of re-melting cycles during processing. **d** shows the evolution of the particle size, fraction, and number density when the atmosphere is switched from CO₂ to Ar and from Ar to CO₂ within one sample. Note the different scale of (**a–c**) compared to (**d**)

faster to the respective atmosphere than δ_{ox} . 100 CO₂+10 Ar even shows a smaller f_p than the one after+50Ar, despite the sample being covered with a thick slag layer ($\delta_{ox}=2.29\pm0.17\ \mu\text{m}$). The opposite holds for+10 CO₂ on top of 100 CO₂+50 Ar, which is only covered with a thin slag layer ($\delta_{ox}=1.66\pm0.64\ \mu\text{m}$). This is a strong indication that nanoparticle formation is not affected by an existing slag layer on top of the samples.+1Ar and+1CO₂ samples are affected by dilution with the material printed in the other atmosphere before. As mentioned before, this is the case as the melt pool depth is 3–4 times larger than the layer thickness, t .

4 Discussion

4.1 Manipulation of the slag layer thickness and its influence on nanoparticle concentration

Manufacturing in CO₂ causes a process gas uptake during manufacturing and hence increases the O level in the materials. CO₂ dissociates in two steps: CO₂ → CO+O and CO → C+O. Process gas dissociation is induced by laser exposure and occurs in the vicinity of the laser plume and causes an increased O partial pressure close to the liquid–gas interface [19]. The short laser exposure times and confinement close to the plasma plume complicate giving a value for the O partial pressure due to process gas dissociation. In-situ optical emission spectroscopy (OES) was currently applied in literature to investigate composition of the process atmosphere in AM [29, 30], and could be used in future work to define the chemical composition of the atmosphere in reactive atmospheres. Besides O partial pressure, chemical composition of the materials and temperature affects gas uptake in the materials. Further details are given in corresponding literature on high pressure casting [31–33], which is believed to be applicable to our processes in AM. O concentrations reach values of up to 0.60 wt% in AB, and 0.15–0.25 wt% in w/o surface condition (Fig. 3). Measurements of the O level in the powder result in a concentration of ca. 0.105 wt%, which is slightly higher than specified in the supplier's certificate. As a reference, FeTi4.3 samples processed in Ar have O levels around 0.115 wt%. The difference in O concentration compared to the feedstock powder is likely due to oxidation by the residual oxygen

in the process chamber, which was kept below 0.04 wt% (400 ppm) in all experiments.

The O fraction determined by chemical and microscopy analysis shows, in general, good correlation (Fig. 8). But chemical analysis cannot differentiate between O stored in nanoparticles and slag particles, unlike microscopy analysis.

However, chemical analysis is more appropriate for obtaining a value for the slag particle concentration, due to their statistical occurrence, as explained in the following. Chemical analysis probes the full volume of the sample. In contrast, the determination of the slag particle content by microscopy is limited to a finite number of cross-sections. The quantification of slag particle content by chemical analysis, on the other hand, requires knowledge of the amount of O stored in nanoparticles. In this work, it was shown that, independently of the used strategy and for identical manufacturing parameters, the nanoparticle fraction in samples processed in CO₂ is constant. Thus, the amount of O stored in the nanoparticles remains constant and can be assumed to be roughly 0.15 wt% (c.f. 2×re-melting samples with lowest slag particle fraction of 0.09 Vol%). The O excess above this value can be directly attributed to the O stored in slag particles (Figs. 3 and 6).

Variation of the powder layer thickness, t , does not significantly influence the slag layer thickness, δ_{ox} , at the top and side, as long as E_V is sufficiently high (Fig. 4). E_V sinks with increasing t , which causes the incorporation of slag particles from the slag layer when a certain t is overcome, which increases the O level in w/o surface state (Fig. 3a). Thus, the incorporation of slag particles also reduces δ_{ox} of the top surface (Fig. 8a). Slag particle incorporation and process irregularities are strongly connected and can both be avoided when using optimum process parameters. The formation of slag in ODS materials manufactured by AM is only occasionally reported in literature, but often with an insufficient explanation for their origin [13, 23–26].

Re-melting is beneficial for decreasing the slag particle concentration in the materials (Fig. 6). Performing a single re-melting after each consolidated layer distinctly reduces the slag particle content. Re-melting most probably leads to a redistribution of slag particles from the bulk to the slag layer. The samples 100 CO₂+R_{CO2} and 100 CO₂+R_{Ar} from the Multi-Atmosphere experiments were only once re-melted and might also show an increase in δ_{ox} compared to the reference, 100 CO₂, for this reason (Table 3). However, overheating the samples by re-melting several times should be avoided, as this can lead to the opposite result, evident from the 2× and 3×re-melted samples. Overheating causes rough surfaces and even build-ups (c.f. Fig. 1c) on which the powder cannot be deposited uniformly anymore [34, 35]. Re-melting increases the surface temperature of the samples with increasing number of cycles. This pre-heating

Table 4 The mean particle diameter d_p , volume fraction f_p , and number density n_p of 100 CO₂+R samples in CO₂ (R_{CO2}) and Ar (R_{Ar}), as well as for the reference sample (100 CO₂) from the Multi-Atmosphere experiments

	d_p (nm)	f_p (vol%)	n_p ($1 \times 10^{19} \text{ m}^{-3}$)
100 CO ₂	22.1 ± 2.8	0.94 ± 0.18	1.10 ± 0.12
100 CO ₂ +R _{CO2}	21.9 ± 1.6	0.92 ± 0.16	0.99 ± 0.08
100 CO ₂ +R _{Ar}	21.8 ± 1.2	0.78 ± 0.13	0.83 ± 0.07

effect causes a higher heat input and hence apparent value of E_p . Heat input by the chosen parameters for re-melting are not optimal anymore caused by the additional heat, which increases surface roughness and causes the recoating issues that lead to slag particle incorporation. The increased slag particle concentration in the $2 \times$ and $3 \times$ re-melted samples aligns with the decreasing δ_{ox} at the top of the samples. Part of the oxide from the top slag layer is incorporated in the bulk as slag particles.

δ_{ox} at the sides is, in contrast, steadily increasing with increasing number of re-melting cycles. Slag at the sample's side originates from the slag layer, which is dragged along the melt pool boundaries [21–23, 27, 36] and is not re-melted during PBF–LB in case of the outer-most tracks. The driving force for material flow is mainly the marangoni effect, which is dependent on the surface tension and hence surface temperature distribution. Marangoni convections transports the slag towards the sides of the melt pool. Note, slag layers do not originate from solid state oxidation as they are too heterogeneous in thickness and their thickness is too high. Since the melt pool width typically exceeds the laser spot size, the laser would not directly expose the sides of the cubes during re-melting experiments. Consequently, slag at the sample's sides accumulates with increasing number of re-melting cycles. Furthermore, repeated re-melting increases the surface temperatures and may promote evaporation of Ti-oxide due to its high volatility [37, 38]. This evaporation could explain the decreasing δ_{ox} observed at the top surface, where the laser irradiates the surface. In contrast, the sample sides are not directly exposed to the laser and are not affected by evaporation for this reason.

4.2 The relationship of slag layer and nanoparticle formation

The increased O uptake from the atmosphere, e.g. by re-melting, promotes oxide formation in the materials. But the nanoparticle fraction, f_p , and number density, n_p , are in the same range for all samples processed in CO₂ (Fig. 5). Only samples that were re-melted show a slight increase in the particle size, which might be explained by the aforementioned pre-heating effect and higher apparent E_p . Instead, the increased oxide formation occurs via an increase in the slag layer thickness (Figs. 4 and 8). f_p seems to be physically

limited in the material for a certain set of manufacturing parameters, and excess oxide is always transferred to the slag layer.

The slag layer thickness accumulates with increasing number of layers until reaching a saturation value (constant thickness), when printing is performed in CO₂, whereas an existing slag layer decreases upon printing in Ar, until it nearly vanishes (Figs. 4b and 9). The formation and growth of slag layers is caused by O uptake from the CO₂ atmosphere, their reduction and disappearance by the absence of O uptake under Ar. The O uptake promotes nanoparticle formation. Thus, f_p and n_p are distinctly larger for samples processed in CO₂ compared to those processed in Ar.

Nanoparticle formation and slag layer formation are correlated, and both are enhanced due to O uptake during printing in CO₂. However, the response of the slag layer is slow compared to nanoparticle formation when the process gas is changed (Fig. 9). An existing slag layer does not influence nanoparticle formation. In the performed experiments, f_p and n_p are solely dependent on the used atmosphere. Conversely, nanoparticle formation does influence slag layer formation, which is explained as follows:

The O solubility in Fe–Ti alloys is limited both in the solid and in the liquid state (Fig. 7), as Ti is a strong deoxidizer [39–42]. This is the driving force for the formation of Ti-oxides in Fe–Ti alloys. During PBF processing, initially, Fe and Ti powder melt due to laser exposure and are mixed in the melt pool. Ti reacts with O from both powders and from the atmosphere. O uptake causes O supersaturation in the melt pool, which promotes (presumably liquid) nanoparticle formation, as absorbed O instantaneously reacts with Ti during manufacturing to decrease the O level in solution. This indicates that O supersaturation in the melt pool controls nanoparticle nucleation. Our model agrees with existing literature, describing nanoparticle formation and evolution during additive manufacturing processes [24, 28]. Nanoparticles grow in the melt pool and float towards the surface (negative sedimentation), once they reach a critical size, due to their lower density compared to the Fe–Ti melt (Stokes' law) [43]. The still-liquid nanoparticles merge at the melt pool surface and form the slag layer. Nucleation and growth of nanoparticles, as well as negative sedimentation occurs within t_{MP} (quasi steady state) and stop upon solidification. Rapid solidification freezes in the instantaneous microstructural state, including smaller nanoparticles levitating in the melt. Increasing t_{MP} by re-melting strategies or decreasing layer height, t , merely prolongs the time available for these nanoparticle-removal processes but cannot increase f_p and n_p in the melt pool, as both quantities are limited by the negative sedimentation of the nanoparticles.

Kinetics of O uptake and oxide nanoparticles are fast considering melt pool temperatures distinctly above 2000 °C

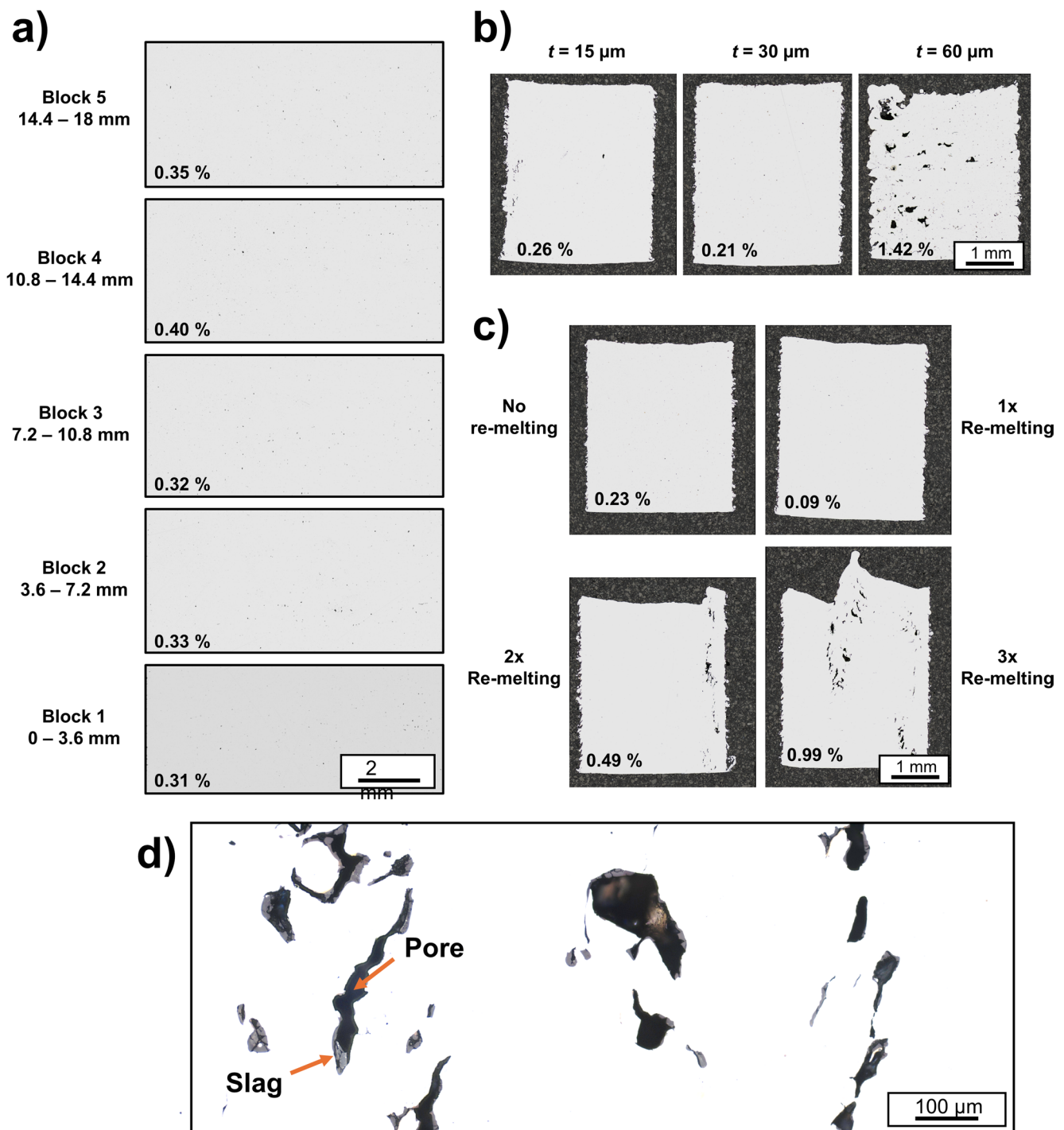


Fig. 6 Sample overview at low magnification and their corresponding slag particle concentration (Vol%) measured for **a** the 5 blocks along the build direction corresponding to the area where samples for chemical analysis were extracted, **b** samples manufactured with different powder layer thicknesses, and **c** for samples exposed to re-melting.

[44] and do not limit nanoparticle fraction. Previous work showed that oxygen uptake and slag layer thickness strongly and nanoparticle fraction slightly increase with E_{J} , explained by the increased time for gas uptake due to an increased

d shows a representative high magnification image of a highly porous area. The image is intentionally overexposed to make differentiation between slag and pore visible. Slag particles partially sit at the edges of pores

melt pool lifetime [27]. Considering the low melt pool lifetimes of ca. 0.3 ms [27] in PBF, there is enough time for nanoparticles to float to the surface and form a slag layer with a thickness of 5 μm . Hence, kinetics are not limiting

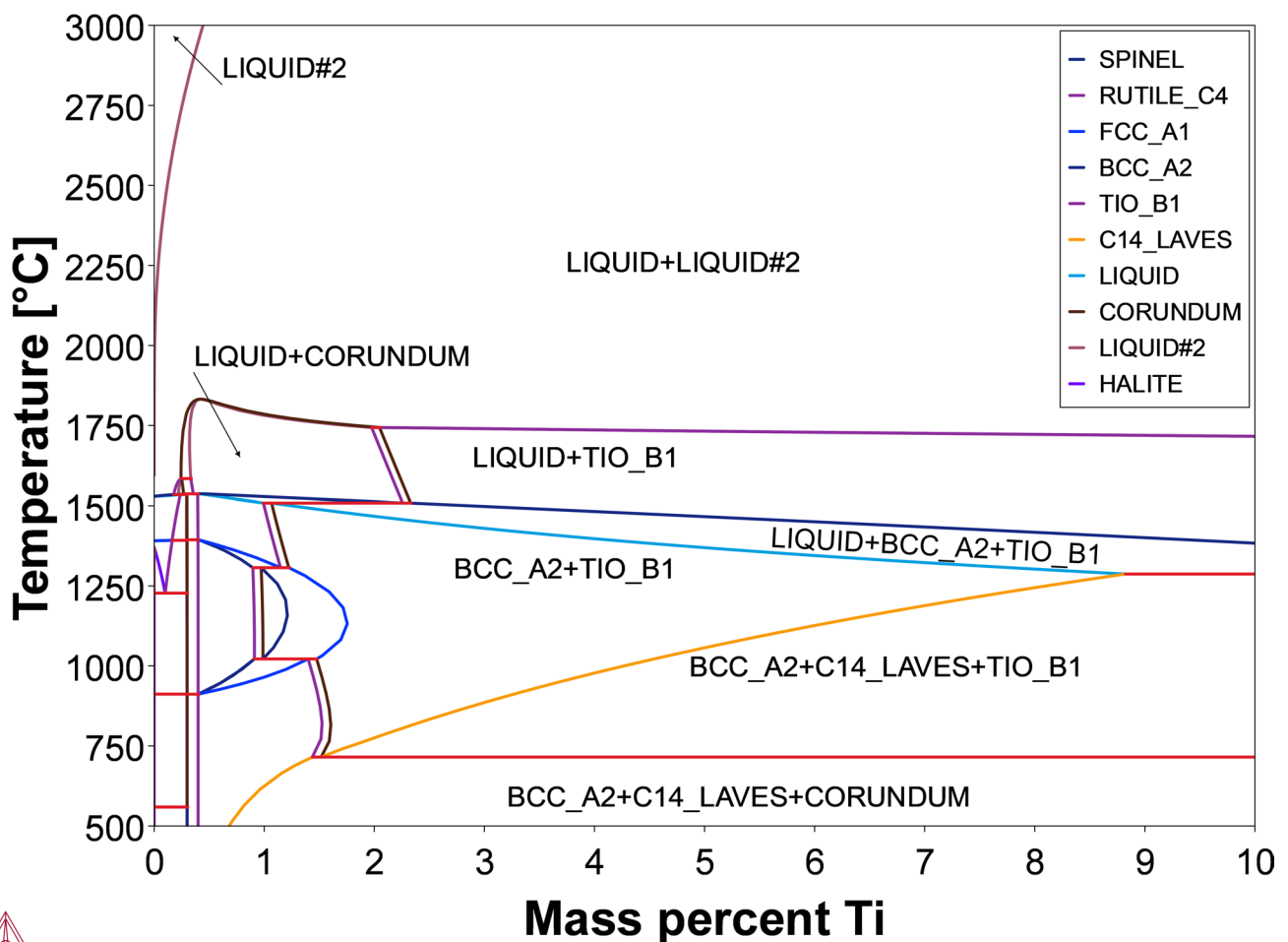


Fig. 7 Phase diagram of Fe_{0.2}wt%O—Ti in the relevant temperature range for PBF—LB

nanoparticle fraction but thermodynamics, e.g. O supersaturation in the melt pool depend on the used atmosphere, does.

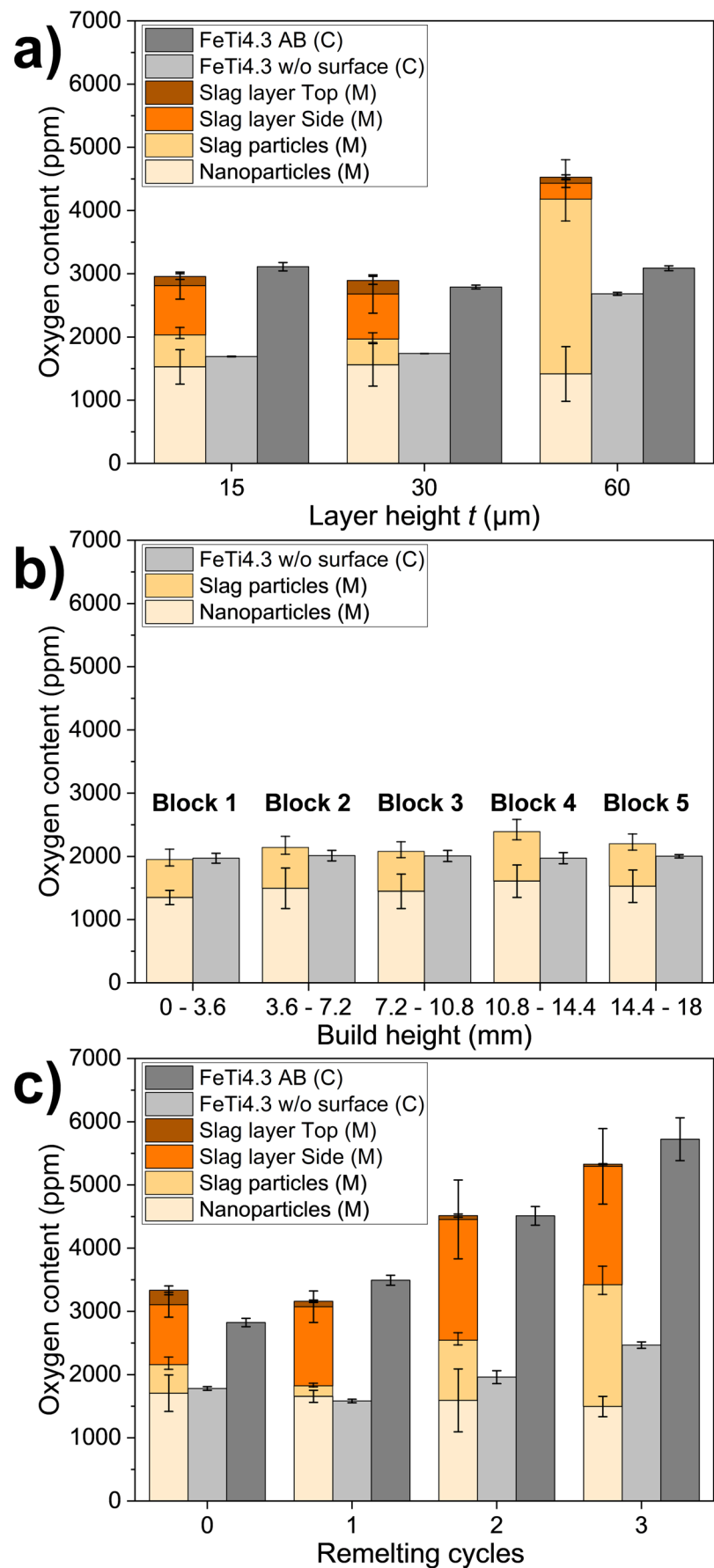
Although the evidence by the performed Multi-atmosphere experiments is indirect, the description of nanoparticle behavior in the melt pool is reasonable. More direct evidence would be possible using synchrotron experiments [45]. However, resolution in synchrotron experiments is often too low to resolve the ca 40 nm small nanoparticles and monitor their behavior in the melt pool. Furthermore, nanoparticle tracing by computational fluid dynamics simulations could be used to predict nanoparticle behavior but requires an accurate model describing processes in the melt pool and experimental validation [46].

The laser breaks up the slag layer at the top of the samples when printing the subsequent layer, which results in micrometer-sized oxide particles (slag particles). The sedimentation velocity scales with the particle size according to Stokes' law [43]. Therefore, slag particles (μm range) float faster to the surface to form the new slag layer than the nanoparticles (10 nm range), which reached the critical

size [27]. Slag particle incorporation results when the time required to reach the surface is smaller than t_{MP} . Ti-oxide cannot dissolve during melting, considering the low O solubility in Fe—Ti, and hence accumulates at the top of the samples in each layer. This explains the growth of the slag layer from each printed layer until a constant thickness is reached. The nanoparticle fraction and slag layer thickness reached a constant value after approximately 50 layers as shown in the build height experiments. The nanoparticle fraction reaches a constant value already after 10 layers. Both values, δ_{ox} and f_p remained constant when printing up to 600 layers (20 mm). This result indicates that if parameters remain optimal during the printing, δ_{ox} and f_p remain constant when printing parts of bigger size.

The liquid slag layer on top of the melt pool might shield the melt pool from the atmosphere, prevent further gas uptake, and restrict its further growth [47]. The reduction of the slag layer upon printing in Ar might be explained by the incorporation of small slag particles (c.f. Fig. 6a) in the bulk, which reduces δ_{ox} after each printed layer. Measurement of the slag particle content showed that even under optimal

Fig. 8 Comparison of calculated O fraction divided into the slag layer at the surface, the slag layer at the sides, slag particles and nanoparticles based on microscopy analysis (M) and O fractions determined in chemical analysis (C): **a** variation of the powder layer thickness, **b** for different build heights and **c** for different number of re-melting cycles



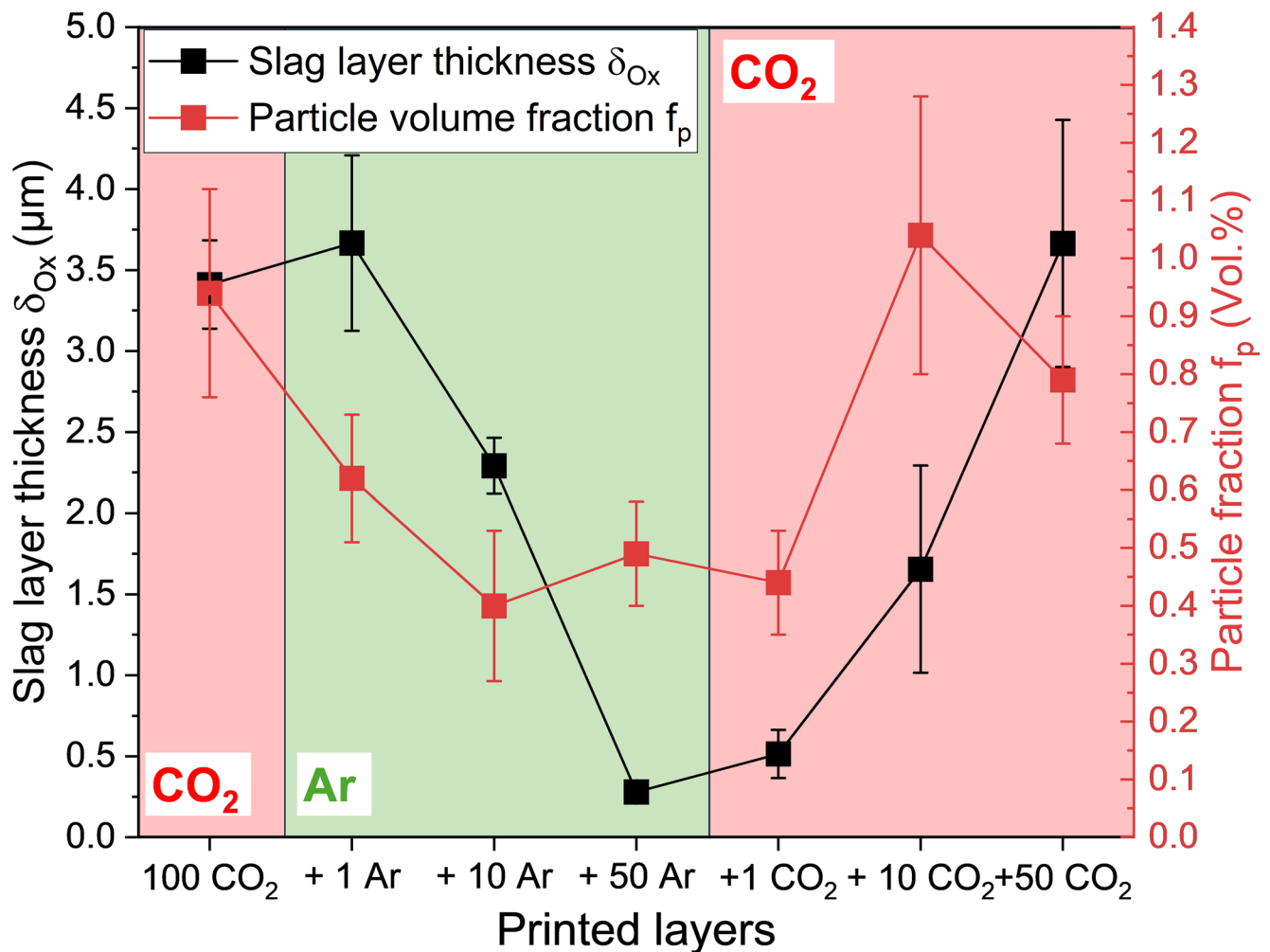


Fig. 9 Correlation of the average layer thickness at the surface with the particle fraction in the melt pool below. The red shaded area marks areas that are printed in CO₂ and the green ones in Ar

parameters, slag particles (0.2–0.4 vol%) are observed in the materials. Promoted by higher O concentrations close to the liquid–gas interface and at the melt pool surface, further oxidation of the slag layer is likely [21, 22]. This might explain the occurrence of slag layers with a composition of mainly Ti₂O₃ [27], whereas the nanoparticles are believed to have a composition of TiO, as predicted by the equilibrium phase diagram for FeTi4.3 (Fig. 7) and found in literature [18].

The nanoparticle fraction in additively manufactured Fe–Ti alloys is determined by processes forming nanoparticles, which are strongly dependent on the O supersaturation in the melt pool, and processes removing particles from the melt pool, via coarsening, floating up, and formation of the slag layer. While the latter processes are difficult to prevent, the former may be enhanced by increasing the activity of O at the atmosphere/melt pool surface. Thus, increasing the O partial pressure in the CO₂ atmosphere could be a potential

strategy to increase particle formation, but it remains difficult to achieve, as most machines work under ambient pressures. Alternatively, more highly reactive atmospheres could be used to increase O uptake in the melt pool to increase particle formation. However, this strategy carries the risk of powder bed ignition or other safety risks [20].

For commercially available ODS steels, the oxide load is typically around 0.3–0.5 wt% and hence a little lower than achieved by the FeTi4.3 model alloys fabricated under CO₂ in this work [1]. The presented approach could therefore be an alternative to commonly applied ex-situ routes in AM of ODS materials. The work presented is furthermore relevant for ex-situ fabricated ODS alloys, as the melting point of the most commonly used oxides is overcome during AM [44]. Thus, nanoparticle coarsening as well as slag layer and slag particle formation by their floating to the surface are relevant for the fabrication of all types of ODS alloys in AM.

5 Conclusions

In this work, the formation mechanism of the slag layer and in-situ nanoparticle formation, as well as their relationship, are studied for PBF–LB in a reactive CO₂ atmosphere. The following conclusions can be drawn:

- The formation of slag layers in PBF–LB is a cumulative process. The slag layer grows with an increasing number of printed layers until a constant thickness is reached, dependent on the used parameters. We argue that the slag layer forms from Ti-oxide droplets that are too large to levitate in the liquid melt pool, float up, and merge to form a liquid slag layer on top of the liquid melt pool. As manufacturing under CO₂ promotes particle formation (high f_p), slag layers are growing under CO₂, whereas existing slag layers are shrinking upon printing in Ar (low f_p).
- Slag particles from the slag layer are incorporated into the materials when the heat input (E_p) during manufacturing is not high enough or when process disruptions (e.g. recoating issues) occur upon manufacturing. The incorporation of slag particles reduces the slag layer thickness at the top of the samples.
- The nanoparticle fraction remaining in the melt pool is limited by nanoparticle flotation to the slag layer at the melt pool surface. Increasing the contact duration of the melt pool with the atmosphere thus only affects the thickness of the slag layer but does not assist in increasing the nanoparticle fraction.
- The nanoparticle fraction and number density are dependent on the process atmosphere only, for a certain set of parameters, and hence on the O supersaturation in the melt pool during processing, resulting from the respective atmosphere. Higher O supersaturation by O uptake during processing in CO₂, enhances deoxidation reactions in the melt pool, causes more nanoparticle droplets to nucleate, and increases the nanoparticle fraction in the materials.

Appendix 1: Calculations to determine oxygen levels based on microscopy (Fig. 8)

Nanoparticles

Composition: TiO.
Density_{TiO}: 4.95 g cm⁻³
Density_{FeTi4.3}: 7.74 g cm⁻³
Wt% O in TiO: 25 wt%

$$wt.\%_{TiO} = \frac{vol.\%_{TiO} * Density_{TiO}}{vol.\%_{TiO} * Density_{TiO} + (1 - vol.\%_{TiO}) * Density_{FeTi4.3}} * 100$$

$$O_{Nanoparticles} (wt.\%) = wt.\%_{TiO} * 0.25$$

Slag particles

Composition: Ti₂O₃.
Density_{Ti2O3}: 4.49 g cm⁻³.
Density_{FeTi4.3}: 7.74 g cm⁻³.
Wt% O in Ti₂O₃: 33 wt%

$$wt.\%_{Ti2O3} = \frac{vol.\%_{Ti2O3} * Density_{Ti2O3}}{vol.\%_{Ti2O3} * Density_{Ti2O3} + (1 - vol.\%_{Ti2O3}) * Density_{FeTi4.3}} * 100$$

$$O_{Slagparticles} (wt.\%) = wt.\%_{Ti2O3} * 0.33$$

Slag layer

Composition: Ti₂O₃.
Density_{Ti2O3}: 4.49 g cm⁻³.
Density_{FeTi4.3}: 7.74 g cm⁻³.
Wt% O in Ti₂O₃: 33 wt%.

The length of the top surface, l_{Top} , and the height of the sides, h_{Side} , is measured for a cross-section with area, A_{Cube} , of a sample printed for chemical analysis. Note, the third dimension, which is identical to l_{Top} , cancels out in the calculation of

$$vol.\%_{Ox} .$$

$$A_{Ox} = A_{TopOxideLayer} + 4 * A_{SideOxideLayer}$$

$$A_{TopOxideLayer} = \delta_{Ox,Top} * l_{Top}$$

$$A_{SideOxideLayer} = \delta_{Ox,Side} * h_{Side}$$

$$vol.\%_{Ox} = \frac{A_{Ox}}{A_{Cube}} * 100$$

$$wt.\%_{Ox} = \frac{vol.\%_{Ox} * Density_{Ti2O3}}{vol.\%_{Ox} * Density_{Ti2O3} + (1 - vol.\%_{Ox}) * Density_{FeTi4.3}} * 100$$

$$O_{Ox} (wt.\%) = wt.\%_{Ox} * 0.33$$

Acknowledgements This research is part of the project FLAB-3Dprint and funded by dtcc.bw—Digitalization and Technology Research Center of the Bundeswehr, which we gratefully acknowledge. dtcc.bw is funded by the European Union—NextGenerationEU. Furthermore, the authors acknowledge financial support from the Universität der Bundeswehr München, for Open Access Publishing.

Author contribution CF: Conceptualization, Methodology, Formal Analysis, Investigation, Writing—original draft, Writing—review & editing, Visualization, Project administration. EY: Conceptualization, Investigation, Writing—original draft, Writing—Review & Editing. EAJ: Conceptualization, Methodology, Formal Analysis, Writing—original draft, Writing—review & editing, Supervision, Project administration, Funding acquisition.

Funding Open Access funding enabled and organized by Projekt DEAL.

Data availability Data will be made available on request.

Declarations

Conflict of interest The authors declare no conflict of interest.

Open Access This article is licensed under a Creative Commons Attribution 4.0 International License, which permits use, sharing, adaptation, distribution and reproduction in any medium or format, as long as you give appropriate credit to the original author(s) and the source, provide a link to the Creative Commons licence, and indicate if changes were made. The images or other third party material in this article are included in the article's Creative Commons licence, unless indicated otherwise in a credit line to the material. If material is not included in the article's Creative Commons licence and your intended use is not permitted by statutory regulation or exceeds the permitted use, you will need to obtain permission directly from the copyright holder. To view a copy of this licence, visit <http://creativecommons.org/licenses/by/4.0/>.

References

- Wilms MB, Rittinghaus S-K, Goßling M et al (2023) Additive manufacturing of oxide-dispersion strengthened alloys: materials, synthesis and manufacturing. *Prog Mater Sci* 133:101049. <https://doi.org/10.1016/j.pmatsci.2022.101049>
- Ukai S, Ohtsuka S, Kaito T et al. (2017) Oxide dispersion-strengthened/ferrite-martensite steels as core materials for generation IV nuclear reactors. In: *Structural materials for generation IV nuclear reactors*. Elsevier, pp 357–414
- Shi Y, Lu Z, Xu H et al (2019) Microstructure characterization and mechanical properties of laser additive manufactured oxide dispersion strengthened Fe–9Cr alloy. *J Alloys Compd* 791:121–133. <https://doi.org/10.1016/j.jallcom.2019.03.284>
- Vasquez E, Giroux P-F, Lomello F et al (2019) Elaboration of oxide dispersion strengthened Fe–14Cr stainless steel by selective laser melting. *J Mater Process Technol* 267:403–413. <https://doi.org/10.1016/j.jmatprotec.2018.12.034>
- Wilms MB, Pirch N, Gökce B (2023) Manufacturing oxide-dispersion-strengthened steels using the advanced directed energy deposition process of high-speed laser cladding. *Prog Addit Manuf* 8:159–167. <https://doi.org/10.1007/s40964-022-00319-1>
- Horn T, Rock C, Kaoumi D et al (2022) Laser powder bed fusion additive manufacturing of oxide dispersion strengthened steel using gas atomized reaction synthesis powder. *Mater Des* 216:110574. <https://doi.org/10.1016/j.matdes.2022.110574>
- Han B, Feng K, Li Z et al (2024) Oxide dispersion strengthening in CrCoNi medium entropy alloy built by selective laser melting with oxygen-rich powder. *J Alloys Compd* 973:172798. <https://doi.org/10.1016/j.jallcom.2023.172798>
- Stegman B, Yang B, Shang Z et al (2022) Reactive introduction of oxide nanoparticles in additively manufactured 718 Ni alloys with improved high temperature performance. *J Alloys Compd* 920:165846. <https://doi.org/10.1016/j.jallcom.2022.165846>
- Chung S, Lee T, Jeong W et al (2023) Additive manufacturing of oxide dispersion-strengthened CoCrNi medium-entropy alloy by in situ oxide synthesis. *J Alloys Compd* 965:171340. <https://doi.org/10.1016/j.jallcom.2023.171340>
- Wang Y, Wang B, Luo L et al (2023) Effects of process atmosphere on additively manufactured FeCrAl oxide dispersion strengthened steel: printability, microstructure and tensile properties. *Mater Sci Eng A* 882:145438. <https://doi.org/10.1016/j.msea.2023.145438>
- Jia H, Zhou Z, Li S (2022) A new strategy for additive manufacturing ODS steel using Y-containing gas atomized powder. *Mater Charact* 187:111876. <https://doi.org/10.1016/j.matchar.2022.111876>
- Mirzababaei S, Ghayoor M, Doyle RP et al (2021) In-situ manufacturing of ODS FeCrAlY alloy via laser powder bed fusion. *Mater Lett* 284:129046. <https://doi.org/10.1016/j.matlet.2020.129046>
- Yin H, Wei B, Shmatok A et al (2023) On the nanoscale oxide dispersion via in-situ atmospheric oxidation during laser powder bed fusion. *J Mater Process Technol* 322:118191. <https://doi.org/10.1016/j.jmatprotec.2023.118191>
- Zhang S, Li K, Zhang W et al (2023) A novel additive manufactured reduced activation ferritic/martensitic steel enhanced by in-situ nanoparticles benefiting from oxygen addition. *Scr Mater* 235:115627. <https://doi.org/10.1016/j.scriptamat.2023.115627>
- Cakmak O, Chung SG, Lee S-H et al (2024) Effect of process atmosphere on microstructure, melt pool, texture, precipitate characteristics, and mechanical properties of laser powder bed fusion Fe–12Cr–6Al. *Addit Manuf Lett* 10:100219. <https://doi.org/10.1016/j.addlet.2024.100219>
- Cakmak O, Lee S-H, Chung SG et al (2024) In-situ synthesis of nitrides and oxides through controlling reactive gas atmosphere during laser-powder bed fusion of Fe–12Cr–6Al. *Mater Des* 240:112862. <https://doi.org/10.1016/j.matdes.2024.112862>
- Ikehata H, Jägle E (2021) Grain refinement of Fe–X alloys fabricated by laser powder bed fusion. *Mater Sci Forum* 1016:580–586. <https://doi.org/10.4028/www.scientific.net/MSF.1016.580>
- Ikehata H, Mayweg D, Jägle E (2021) Grain refinement of Fe–Ti alloys fabricated by laser powder bed fusion. *Mater Des* 204:109665. <https://doi.org/10.1016/j.matdes.2021.109665>
- Haines MP, Peter NJ, Babu SS et al (2020) In-situ synthesis of oxides by reactive process atmospheres during L-PBF of stainless steel. *Addit Manuf* 33:101178. <https://doi.org/10.1016/j.addma.2020.101178>
- Felber C, Köberl M, Jägle EA (2025) Powder bed fusion—laser beam in reactive atmospheres—ignition limits for Fe and Ti–6Al–4V powder blends in CO₂ and N₂. *Powder Technol* 456:120843. <https://doi.org/10.1016/j.powtec.2025.120843>
- Chia HY, Wang L, Yan W (2023) Influence of oxygen content on melt pool dynamics in metal additive manufacturing: high-fidelity modeling with experimental validation. *Acta Mater* 249:118824. <https://doi.org/10.1016/j.actamat.2023.118824>
- Chia HY, Zhang Y, Wang L et al (2024) Unveiling gas–liquid metal reactions in metal additive manufacturing: high-fidelity modeling validated with experiments. *Acta Mater* 275:120029. <https://doi.org/10.1016/j.actamat.2024.120029>
- Eo D-R, Chung SG, Jeon JM et al (2021) Melt pool oxidation and reduction in powder bed fusion. *Addit Manuf* 41:101982. <https://doi.org/10.1016/j.addma.2021.101982>
- Hou W, Stubbs T, DeBeer-Schmitt L et al (2024) Dissolution zone model of the oxide structure in additively manufactured

- dispersion-strengthened alloys. *Addit Manuf* 96:104554. <https://doi.org/10.1016/j.addma.2024.104554>
25. Stubbs T, Hou R, Leonard DN et al (2024) Dispersoid coarsening and slag formation during melt-based additive manufacturing of MA754. *Addit Manuf Lett* 9:100195. <https://doi.org/10.1016/j.adllet.2024.100195>
 26. Kenel C, de Luca A, Joglekar SS et al (2021) Evolution of Y₂O₃ dispersoids during laser powder bed fusion of oxide dispersion strengthened Ni–Cr–Al–Ti γ/γ' superalloy. *Addit Manuf* 47:102224. <https://doi.org/10.1016/j.addma.2021.102224>
 27. Felber C, Farquhar L, Jägle EA (2026) The influence of process parameters on in-situ oxide dispersion strengthened Fe–Ti–alloys in PBF–LB under reactive atmosphere. *Mater Des* 264:115696. <https://doi.org/10.1016/j.matdes.2026.115696>
 28. Wassermann NA, Li Y, Myers AJ et al (2024) Limits of dispersoid size and number density in oxide dispersion strengthened alloys fabricated with powder bed fusion-laser beam. *Addit Manuf* 81:104022. <https://doi.org/10.1016/j.addma.2024.104022>
 29. Gabriel P, Eibl F, Barcikowski S et al (2026) Toward real-time chemical mapping during laser powder bed fusion: robust in-situ spectroscopy and 3D reconstruction. *Addit Manuf* 119:105112. <https://doi.org/10.1016/j.addma.2026.105112>
 30. Ziefuss AR, Gabriel P, Streubel R et al (2024) In-situ composition analysis during laser powder bed fusion of Nd–Fe-based feedstock using machine-integrated optical emission spectroscopy. *Mater Des* 244:113211. <https://doi.org/10.1016/j.matdes.2024.113211>
 31. Jiang Z, Li H, Chen Z et al (2005) The nitrogen solubility in molten stainless steel. *Steel Res Int* 76:740–745. <https://doi.org/10.1002/srin.200506090>
 32. Satir-Kolorz AH, Feichtinger HK (1991) On the solubility of nitrogen in liquid iron and steel alloys using elevated pressure. *Int J Mater Res* 82:689–697
 33. Simmons JW (1996) Overview: high-nitrogen alloying of stainless steels. *Mater Sci Eng A* 207:159–169. [https://doi.org/10.1016/0921-5093\(95\)09991-3](https://doi.org/10.1016/0921-5093(95)09991-3)
 34. Rauner D, Beuerlein K-U, Zhang R et al (2025) Multi-scale simulation approach for the prediction of overheating under consideration of process parameters in powder bed fusion of metals using a laser beam. *J Mater Process Technol* 338:118769. <https://doi.org/10.1016/j.jmatprotec.2025.118769>
 35. Ranjan R, Chen Z, Ayas C et al (2023) Overheating control in additive manufacturing using a 3D topology optimization method and experimental validation. *Addit Manuf* 61:103339. <https://doi.org/10.1016/j.addma.2022.103339>
 36. Felber C, Diez JG, Jägle EA (2026) The influence of reactive process atmospheres on the microstructure evolution of additively manufactured CP titanium. *Mater Charact* 231:115829. <https://doi.org/10.1016/j.matchar.2025.115829>
 37. Lamoreaux RH, Hildenbrand DL, Brewer L (1987) High-temperature vaporization behavior of oxides II. Oxides of Be, Mg, Ca, Sr, Ba, B, Al, Ga, In, Tl, Si, Ge, Sn, Pb, Zn, Cd, and Hg. *J Phys Chem Ref Data* 16:419–443. <https://doi.org/10.1063/1.555799>
 38. Liu J, Wen P (2022) Metal vaporization and its influence during laser powder bed fusion process. *Mater Des* 215:110505. <https://doi.org/10.1016/j.matdes.2022.110505>
 39. Cha W-Y, Nagasaka T, Miki T et al (2006) Equilibrium between titanium and oxygen in liquid Fe–Ti alloy coexisted with titanium oxides at 1873 K. *ISIJ Int* 46:996–1005. <https://doi.org/10.2355/isijinternational.46.996>
 40. Cho Y-M, Kang Y-B (2024) Thermodynamic model for deoxidation of liquid steel considering strong metal–oxygen interaction in the quasichemical model framework. *Int J Miner Metall Mater* 31:988–1002. <https://doi.org/10.1007/s12613-023-2766-7>
 41. Jung I-H, Deckerov SA, Pelton AD (2004) A thermodynamic model for deoxidation equilibria in steel. *Metall Mater Trans B* 35:493–507. <https://doi.org/10.1007/s11663-004-0050-4a>
 42. Fruehan RJ (1970) Activities in liquid Fe–Al–O and Fe–Ti–O alloys. *Metall Trans* 1:3403–3410. <https://doi.org/10.1007/BF03037871>
 43. Tadros TF (2023) *Emulsionen*. De Gruyter
 44. Wang R, Garcia D, Kamath RR et al (2022) In situ melt pool measurements for laser powder bed fusion using multi sensing and correlation analysis. *Sci Rep* 12:13716. <https://doi.org/10.1038/s41598-022-18096-w>
 45. Ioannidou C, König H-H, Semjatov N et al (2022) In-situ synchrotron X-ray analysis of metal additive manufacturing: current state, opportunities and challenges. *Mater Des* 219:110790. <https://doi.org/10.1016/j.matdes.2022.110790>
 46. Wang L, Guo Q, Chen L et al (2023) In-situ experimental and high-fidelity modeling tools to advance understanding of metal additive manufacturing. *Int J Mach Tools Manuf* 193:104077. <https://doi.org/10.1016/j.ijmachtools.2023.104077>
 47. Ronay M, Nordlander P (1987) Barrier to oxygen penetration on metal and oxide surfaces. *Phys Rev B Condens Matter* 35:9403–9406. <https://doi.org/10.1103/PhysRevB.35.9403>

Publisher's Note Springer Nature remains neutral with regard to jurisdictional claims in published maps and institutional affiliations.