

The influence of alloying on properties of additively manufactured in-situ particle reinforced Fe-Ti alloys using reactive process atmospheres

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ABSTRACT

In powder bed fusion – laser beam (PBF–LB), the process gas has been used to fabricate in-situ oxide dispersion strengthened (ODS) steels. Processing in reactive atmospheres, e.g. CO₂ and N₂, causes process gas dissociation and absorption into the melt pool, thereby promoting nanoparticle formation. The intensity of the interaction depends on the atmosphere's reactivity and the alloy composition. A strong interaction with the atmosphere is favorable to enhance nanoparticle formation. In this work, Fe-Ti model alloys with varying Ti content are processed via PBF–LB. The influence of the Ti alloying content on process gas uptake, nanoparticle, and slag layer formation is studied using Ar, CO₂, and N₂ atmospheres. Based on thermodynamic simulations, the resulting oxides are, depending on the Ti concentration, TiO or Ti₂O₃. The fraction and number density of nanoparticles are mainly determined by the processing gas and resulting O supersaturation in the melt pool. In-situ alloying and Ti-oxide nanoparticles additionally affect the grain structure of the processed materials and hence the hardness and creep performance of the materials. This work provides the fundament for targeted alloy design for advanced in-situ particle reinforced materials using reactive atmospheres in AM.

1. Introduction

Powder bed fusion – laser beam of metals (PBF–LB) is typically conducted in protective atmospheres for process safety and to limit oxidation. For this purpose, argon (Ar), nitrogen (N₂), and helium (He) are most commonly used as process gases [1,2]. He and Ar are inert gases that do not interact with the materials during manufacturing, whereas N₂ dissociates during laser exposure and may be absorbed by the materials [3]. Similarly, residual oxygen (O₂) in the process chamber dissociates upon laser exposure and is picked up by the materials during printing [4]. The O level in the process chamber is typically adjusted to values below 2000 ppm, depending on the materials' requirements, e.g. the desired purity of the material, and its reactivity [5].

The interaction of the materials with the process atmosphere during additive manufacturing (AM) processes has recently been exploited to process in-situ particle reinforced materials by setting elevated levels of residual oxygen in the process chamber [6–11] or using reactive gases that dissociate during manufacturing [12–17]. The feedstock powder used in PBF–LB carries a certain amount of oxygen, which further contributes to oxide nanoparticle formation during manufacturing [16] and can be manipulated during atomization by controlling the O content in

the atomization atmosphere [13] and the amount of deoxidizers in the materials [18]. All these approaches aim to increase O and N levels in the melt pools to promote particle formation and achieve high nanoparticle concentrations.

In this work, two reactive process gases (carbon dioxide (CO₂) and N₂) are used to promote in-situ oxide and nitride particle formation during PBF–LB. O, C, and N uptake from the process atmosphere needs to be high for this purpose. The interaction of a material with the atmosphere, which controls the uptake, is mainly influenced by three factors: the reactivity of the process gas, the reactivity of the materials to be processed, and the process parameters.

First, regarding process parameters, increasing the heat input, e.g. volumetric energy density, E_V , by increasing laser power, P , and decreasing scan speed, v , increases the uptake of O from the process atmosphere [19]. We have previously shown that the evolution of the O level in additively manufactured samples requires distinguishing between the as-built (AB) condition and the bulk (w/o surface) condition due to the formation of surface slag layers. The total O level in Fe-based materials increases with E_V (AB), and the O level in the bulk (w/o surface) decreases with increasing E_V . The slag layer is often neglected in the discussion of the influence of processing parameters on the process

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gas uptake in the literature, and a proper description of sample preparation for chemical analysis is missing, which potentially causes contradictory findings in the literature [19].

Next, the reactivity of the process gas depends on the type of gas used and its concentration in the atmosphere. For all gases, the uptake increases with increasing partial pressure of the reactive gas species, e.g. O or N [14,20–22]. Although N₂ dissociates upon laser exposure and is absorbed by the materials, its reactivity is low, and hence, N uptake in Fe-based materials is limited [12,15–17,23–25]. CO₂ offers a higher reactivity than N₂ and causes high O uptake from the process gas in Fe-based materials, in contrast [14,19]. However, the high reactivity of CO₂ limits its application to unreactive materials such as Fe-based ones, in order to not risk ignition of the powder in the machine. Previous work showed that ignition is prevented when the Ti concentration in Fe-Ti is below 23 wt% [26].

Last, the interaction with the process atmosphere is dependent on the alloy composition of the materials. Whereas Fe- and Ni-based materials can readily be processed in pure N₂ atmospheres, Ti-based materials are so reactive that processing is only possible in diluted Ar-N₂ atmospheres [27–29]. Even at low N₂ partial pressure in Ar-N₂ atmospheres, N uptake in Ti is intense and clearly exceeds the O uptake of Fe-based materials processed under CO₂, a much more reactive gas than N₂ [14,20].

When it comes to alloy design for additively manufactured oxide and nitride dispersion strengthened (ODS/NDS) alloys by reactive atmospheres, the combination of all three components (atmosphere, material, and parameters) needs to be considered to adjust the interaction of the material with the atmosphere. The material-gas interaction needs to be maximized to enhance in-situ particle formation in the alloys without risking process safety. As Fe-based alloys are not very reactive, a highly reactive atmosphere, e.g. CO₂, should be used during manufacturing. Alloying can assist in increasing the reactivity of the alloy and controls the composition of in-situ formed oxides [23,24]. However, the effect of alloying on process gas uptake, nanoparticle and slag layer formation, microstructure evolution and mechanical properties has not been studied thoroughly yet, to our best knowledge. This is important to understand the requirements for alloy design of in-situ particle reinforced materials to promote particle formation in additively manufactured ODS and NDS alloys.

In this work, the influence of Ti in-situ alloying on properties of Fe-based ODS and NDS materials manufactured in reactive atmospheres via PBF-LB is investigated. For this purpose, powder blends consisting of Fe and Ti in three concentrations (1.8 wt%, 4.3 wt%, and 8.7 wt%) were created by tumble mixing and processed under Ar (reference), CO₂, and N₂ (only for 4.3 wt% Ti). Chemical and microscopy analyses are used to study the effect of the Ti alloying content and atmosphere on process gas uptake and microstructure evolution, including the formation of oxide nanoparticles and slag layers. Thermodynamic (Thermo-Calc) simulations are conducted to support the explanation of the microstructure evolution in the materials. The material hardness at room temperature and the creep lifetime are determined and correlated with the microstructures.

2. Methodology

2.1. Materials

Fe-Ti alloys with three different Ti fractions were created by tumble mixing of the two elemental powders, Fe and Ti Gd. 1, purchased from m4p and Eckart TLS, respectively, using a Turbula tumble mixing device (Willy A. Bachhofen). Pure Fe was used as a reference in this work. The powder size distribution (PSD) of Fe powder is slightly smaller (15–45 µm) than that of Ti Gd. 1 (20–63 µm) according to the suppliers' certificates. The composition of the two powders is listed in Table 1. FeTi1.8, FeTi4.3, and FeTi8.7 powder blends consisting of 1.8 wt% (2 at %), 4.3 wt% (5 at %), and 8.7 wt% (10 at %) Ti, were prepared by weighing in the powders and blending for 4 h. These levels were chosen

Table 1

Chemical composition of Ti Grade 1 powder and Fe powder according to the certificates 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

to cover a wide range within the solubility limit of Ti in Fe and to avoid the formation of unwanted Laves phases. Fig. 1 shows an SEM image of the FeTi4.3 powder blend and a corresponding EDS mapping at lower magnification. The powders, Fe and Ti Gd. 1, are easily distinguished in SEM, as Fe is a non-spherical, water atomized powder, whereas Ti Gd. 1 powder is spherical and gas atomized. The mixing duration was sufficient to create a homogeneous blend, as confirmed by EDS.

2.2. Manufacturing

Samples were manufactured using an Aconity3D Midi + PBF-LB-machine on a 316 L stainless steel build plate with a diameter of 55 mm. The machine is equipped with an IR fiber laser ($\lambda = 1064$ nm) with a Gaussian intensity profile and a spot size of 80 µm. A powder layer thickness, t , of 30 µm and a 90° rotation strategy were used to print the samples. The hatch distance, h , was set equal to the spot size. The baseplate is filled with cubic samples of different geometries (Fig. 2), with the majority of samples printed for chemical analysis.

Samples with a dimension of $3 \times 3 \times 6$ mm³ were used for the measurement of the O and N content, and $3 \times 3 \times 10$ mm³ for the C content. Note that the height difference is due to different requirements of the sample mass for inert gas fusion (O/N/H) and combustion analysis (C/S). Furthermore, the first 2 mm of the samples were support structures, which were printed to facilitate the removal from the baseplate with a hand hacksaw. This procedure allows removing the samples without risking much contamination and damage to the surface caused by other removal techniques. Samples for chemical analysis were prepared in two conditions, as built (AB) and w/o surface to reveal O, C, and N levels in the bulk without measuring the surface layer. W/o surface samples in the middle of the platform were printed without support, as these samples were ground still attached to the platform, using a metal file, before removal. Approximately 1 mm was removed from each side until the bare metal surface appeared and no more oxide (blueish color) was visible. This method approved to be suitable in previous work [19]. Samples for microstructure analysis with a dimension of $10 \times 8 \times 10$ mm³ were printed supportless and were removed using a band saw.

Manufacturing under CO₂ slightly decreases process stability, e.g. causing more spatter particles to be ejected and a bigger laser plume compared to manufacturing under Ar and N₂. However, the samples' part qualities (e.g. part density) are not negatively affected.

Four different parameter sets were selected to cover a wide range of E_V . All cubes are sorted depending on their purpose and their parameters (Fig. 2). Samples for O/N/H analysis were at the bottom of the baseplate, close to the inert gas flow inlet, to reduce the risk of being affected by spatter particles. Samples destined for C/S analysis are positioned at the other end of the baseplate close to the gas flow outlet, as spatter particles are not expected to affect C measurements significantly. Although a gradient in gas composition (e.g. CO₂) along the gas flow and built plate might exist, its influence is expected to be low and disappear in the measurement error, as manufacturing is carried out in pure CO₂ and N₂ atmosphere, with each gas available in excess. The cubes were placed on the platform in blocks consisting of four samples (parameters 1 to 4) to achieve a better distribution of samples across the built plate and to reduce the effect of the position on the baseplate, which could be relevant for chemical analysis. The different parameter sets 1–4 can be distinguished readily for samples manufactured under CO₂ due to

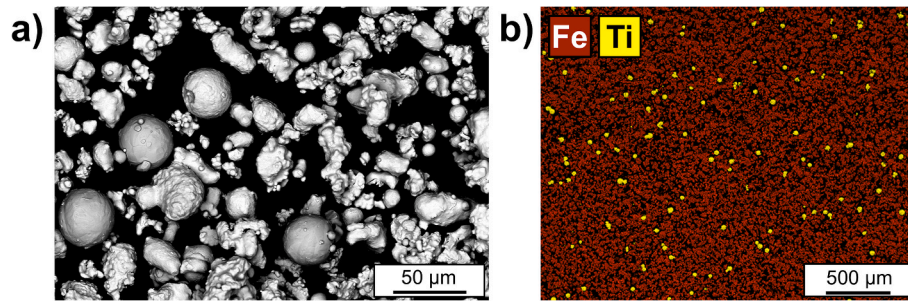


Fig. 1. FeTi4.3 powder blend used for experiments: (a) SEM image at high magnification of the powder blend consisting of spherical Ti particles (dark) and non-spherical Fe particles (bright) captured with an AsB detector. (b) EDS image of the powder blend at low magnification shows homogeneous distribution of Ti particles in the FeTi4.3 blend.

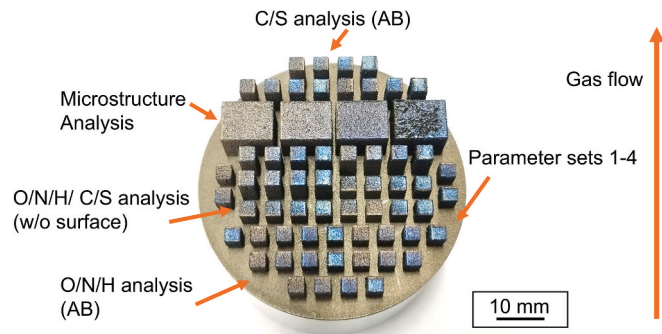


Fig. 2. Sample overview of one build job, exemplary shown for FeTi8.7 samples printed in CO₂ atmosphere. Cubic samples with different dimensions are printed for chemical analysis (small cubes) and microstructure analysis (large cubes). Samples are printed with four different E_V . Coloration of the sample surface due to the formation of a surface slag layer, which increases with E_V .

different coloration caused by different degrees of oxidation (Fig. 2). The four different parameter sets are shown in Table 2. For microstructure analysis, only parameter set 3 is used as the corresponding samples have the lowest porosity, and the effect of different parameters on the microstructure has already been studied in our previous work [19].

2.3. Chemical and crystallographic analysis

O and N levels were determined by inert gas fusion analysis (O/N/H) using an Inductar ONH cube device (Elementar). The geometry of the samples was chosen to achieve a sample mass of 250–300 mg. The C content was measured using combustion analysis with an Inductar CS cube (Elementar). Cubes with a mass of 500–600 mg were printed for this purpose.

X-ray diffraction (XRD) was performed using a Bruker Discover D8 in Bragg-Brentano geometry. The device was operated at 40 kV and 40 mA using a copper cathode, nickel absorber, polycapillary lens, and 1 mm collimator in point mode. The step size was set to 0.01° with a speed of 1° min⁻¹ in between 10 and 100° (2θ). XRD was used to reveal the composition of the oxide in the slag layer. XRD was assisted by electron backscatter diffraction (EBSD) for the same purpose and to spatially resolve the phases in the slag layer. EBSD was done using an EDAX Velocity Super installed in a Zeiss Ultra 55 FEGSEM (field emission gun

scanning electron microscopy). Measurements were performed with acceleration voltage of 15 kV, an aperture of 120 μm, at an exposure time of 3 ms, and a step size of 0.1 μm. Postprocessing of EBSD images, e. g. phase selection and clean-up, was done using OIM analysis software version 8.6.

Thermodynamic modelling was done using the software ThermoCalc 2023a with the database TCFE12 (Steels/Fe-Alloys v12.0).

2.4. Microstructure analysis

Microscopy was performed using a Zeiss Axiophot (Optical microscopy) and Zeiss Ultra 55 FEGSEM. Chemical analysis of microstructural elements was done using an EDAX Octane Elite Super EDS (energy dispersive X-ray spectroscopy) at 5 kV to improve sensitivity for O detection. The 70 mm² chip and the thin Si₃N₄ detector window further improved detection of low-weight elements such as O, N, and C.

The surface slag layer thickness, δ_{ox} , of FeTi4.3 was determined using $n = 5$ –15 images for each sample along the sample's top surface, depending on the used magnification. Magnification was chosen as small as possible to capture a large area without sacrificing measurement accuracy. Due to the strongly heterogeneous layer thickness along the surface, the average δ_{ox} was calculated using the area of the oxide at the surface, A_{Oxide} , divided by its length, $l_{Surface}$:

$$\delta_{ox} = \frac{1}{n} \times \sum_{i=1}^n \frac{A_{i,Oxide}}{l_{i,Surface}} \quad (1)$$

Particle analysis was done using eight high-magnification secondary-electron images (at 50k x), capturing different areas across the sample. A threshold method was applied with the software ImageJ to determine the grand mean values of the particle diameter, d_p , fraction, f_p , and number density, n_p . The mean average error ($\frac{1}{n} \sum_{i=1}^n |x_i - \bar{x}|$) was calculated for each data point and used to describe the uncertainty of each measurement throughout this work.

EBSD maps of the grain structures were captured using an acceleration voltage of 15 kV and a 120 μm aperture at an exposure time of 3 ms, and step sizes dependent on the chosen magnification and microstructure. Grain size evaluation was done using the software OIM Analysis v8.6 with a grain tolerance angle of 15°, a minimum grain size of 5 pixels, and a confidence index ≥ 0.1 (filter). Data points with a confidence index < 0.1 were neglected in the analysis as they might have been misindexed.

2.5. Mechanical testing

Hardness testing is performed using a Qness 60 A + EVO micro hardness tester from QATM with a load of 0.5 kg, measuring 15 indents across the sample to determine an average value.

Creep testing was performed by small punch creep testing (SPC) according to EN 10371 using an SPC1000 device from UTM. In this

Table 2

Parameter for the sets 1–4 used for the experiments.

	P (W)	v (mm s ⁻¹)	σ (μm)	t (μm)	E_V (J mm ⁻³)
1	260	1400	80	30	77
2	210	1000			88
3	200	800			105
4	260	800			135

method, a constant load is transferred with a punch to a Si_3N_4 ball with a diameter of 2.8 mm in contact with the specimen and the punch displacement is measured until a deflection of 2.3 mm is reached. Cylinders with a diameter of 10 mm were printed in the Aconity3D Midi+, removed from the baseplate, and machined to a diameter of 8.0 ± 0.01 mm. Disks with a thickness of roughly 0.8 mm were cut from the cylinders using a Brilliant 220 cutting machine from QATM. Both sides of the disks were ground in two steps (P320 and P1200) to the final SPC sample thickness of $0.5 \text{ mm} \pm 0.005 \text{ mm}$. Creep testing was performed at 600°C with a load of 450 N. Two samples for each condition were measured to ensure repeatability.

3. Results

3.1. Chemical analysis

Fig. 3 shows the O, C, and N concentrations of pure Fe and Fe-Ti alloys with different Ti fractions, manufactured in Ar, CO_2 , and N_2 atmospheres. The two different states, AB and w/o surface, are depicted using solid symbols and lines for AB, and outline symbols and dashed lines for w/o surface in the figure. The difference between the two values corresponds to the amount of O, C, and N stored in the slag layers.

Samples processed in Ar keep an O level slightly higher than in the feedstock powder ($0.11\text{--}0.12 \text{ wt}\%$). The increase in O concentration is likely due to oxidation by residual O in the process chamber. In contrast, samples that are processed in CO_2 show distinctly larger O levels than those manufactured in Ar due to O uptake from the process gas. As no significant O uptake occurs during printing in Ar for all Fe-Ti alloys, O, C, and N levels are shown for FeTi4.3 only. The O uptake from the CO_2 atmosphere increases linearly with E_V and with a similar gradient for all material compositions (Fig. 3a and b). Increasing the Ti content from 0 wt% to 8.7 wt% in the materials shifts the curves towards higher O concentration without significantly changing the slope of the curves

(Fig. 3b).

Although pure Fe and Fe-Ti show a similar interaction with CO_2 and Ar atmospheres, some differences between Ti alloyed and pure Fe materials should be noted. Pure Fe shows nearly identical O concentrations in AB and w/o surface condition, indicating that there is no pronounced slag layer on the samples' surface (Fig. 3a). In contrast, Fe-Ti alloys show a clear difference between AB and w/o surface state, which increases with increasing E_V as elucidated in a previous work [19] (Fig. 3b). The difference between AB and w/o surface state increases with increasing tendency for slag layer formation, as the Ti content increases. Furthermore, no interaction with C from CO_2 or N from N_2 is observed for pure Fe, as these samples keep C and N levels as in the raw powders when being processed in CO_2 and N_2 . For this reason, the evolution of C and N levels is plotted for Fe-Ti alloys only.

The C uptake from the atmosphere (Fig. 3c) follows the same trends as observed for O uptake in AB state and increases with increasing Ti fraction in the powder blend. However, C uptake is much lower compared to O uptake, and the difference between AB and w/o surface condition is small. C uptake indicates that a full dissociation of CO_2 to C and O has taken place, at least to some extent.

N contents evolve similarly to C contents (Fig. 3d) when replacing the CO_2 atmosphere with N_2 . N contents in AB and w/o surface conditions do not significantly differ, as observed for C. Note that only FeTi4.3 samples were processed in N_2 , as similar dependencies as for CO_2 processed samples were expected and measurements were omitted for this reason. Thus, N uptake is assumed to increase with increasing Ti fraction, as it mirrors the behavior of C uptake under CO_2 . For example, pure Fe manufactured under N_2 does not show N uptake, as it does not show C uptake when manufactured under CO_2 . Alloying with 4.3 wt% Ti causes N uptake and thereby approximately triples the N content in the samples. Hence, alloying Ti strongly promotes O, C and N uptake, reflecting the high reactivity of Ti. Pure Ti is very reactive with N_2 and therefore, in the literature, it is usually only processed in Ar- N_2 atmospheres. Even for N

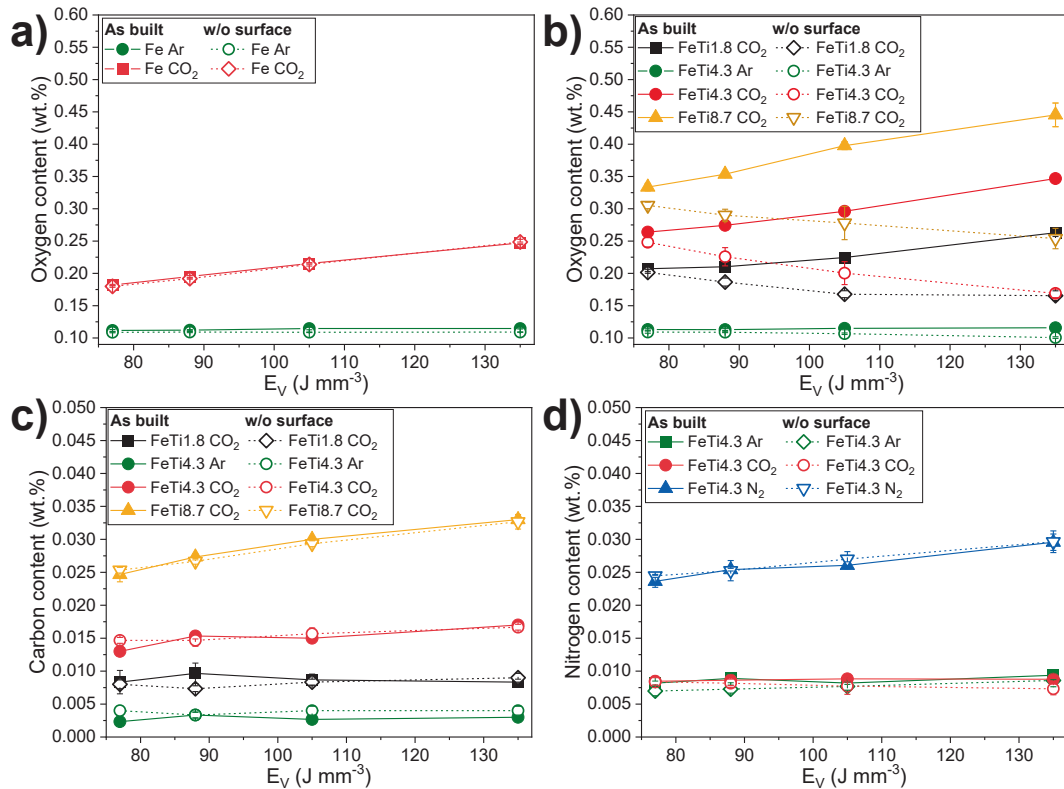


Fig. 3. Evolution of (a) O content in pure Fe samples manufactured in Ar and CO_2 , (b) O content of Fe-Ti samples manufactured in Ar and CO_2 , (c) C content of Fe-Ti samples manufactured in Ar and CO_2 (d) N concentration for FeTi4.3 manufactured in N_2 in AB (solid line) and w/o surface (dashed line) condition dependent on the used E_V .

concentrations as small as 3 vol%, N uptake is in the range of several thousand ppm [20]. In comparison, N uptake for FeTi4.3 (4.3 wt% Ti) under 100 vol% N₂ atmosphere is only in the range of 200 ppm in this work.

3.2. Slag layer

Chemical analysis indicated the formation of slag layers, especially for specimens processed in CO₂. Fig. 4 shows the evolution of δ_{ox} dependent on the Ti content in the samples and the process atmosphere used. δ_{ox} increases substantially with increasing Ti content for samples manufactured under CO₂ and begins to saturate at high Ti levels. The slag layer on samples manufactured in Ar and N₂ is thin and less affected by the Ti content in the materials.

Pure Fe specimens manufactured in CO₂ show only a small slag layer at the surface with a thickness of $\delta_{ox} = 0.28 \pm 0.9 \mu\text{m}$. No slag layer was observed for pure Fe processed in Ar or N₂.

The Ti content in the materials further affects oxide composition, as shown in Fig. 5. Analysis of the slag layer composition is done for CO₂ processed samples only due to their large δ_{ox} in contrast to Ar or N₂ processed Fe-Ti samples, and as the composition is expected to be identical for Ar and N₂ processed ones, based on optical microscopy (OM). OM shows two phases with two distinct colors in the slag layer – one blueish-grey and the other golden. The fraction of the golden phase increases with increasing Ti content in the samples, as visible from Fig. 5a–c. XRD is used to determine their composition, but no attempt is made to exactly quantify relative fractions. The XRD diffractogram contains four different phases: Ti_{1.67}O₃, Ti₂O₃, TiO, and α -Fe. α -Fe peaks result from the material below the slag layer. Their peak intensities correlate inversely with δ_{ox} in Fig. 5, as the expected penetration depth of x-rays for the material is roughly 5 μm and hence in the range of δ_{ox} . Furthermore, the surface is not covered with a uniform slag layer, e.g. slag often accumulates at the melt pool edges (Fig. 5b and c).

FeTi1.8 samples (Fig. 5a) depict a slag layer that is mostly blueish-grey. The XRD spectrum of FeTi1.8 shows a large peak at a 2θ angle of 31.6° , which is the strongest reflex {222} related to Ti_{1.67}O₃. This peak distinctly decreases for FeTi4.3 and FeTi8.7 (Fig. 5b and c). The golden phase is identified as TiO (rock salt structure). Strong reflexes related to TiO occur at $2\theta = 37.2^\circ$ {031} and $2\theta = 37.6^\circ$ {211} in FeTi8.7. However, the slag layer of FeTi8.7 samples still consists of a high fraction of Ti_{1.67}O₃. FeTi4.3 shows a spectrum more similar to FeTi1.8, as it consists of a high fraction of Ti_{1.67}O₃, but with a lower peak

intensity due to the presence of TiO in the slag layer.

In contrast, previous work conducted on the same materials composition showed a slag layer consisting of Ti₂O₃ and TiO for FeTi4.3 manufactured in Ar [19]. Likely, the composition of the slag layer is not homogeneous, consists of several phases as detected in XRD, and therefore, the composition can deviate from the composition of stoichiometric phases. Furthermore, local differences in the phase constitution are expected in the slag layer consisting of Ti_{1.67}O₃, Ti₂O₃, and TiO.

EBSD analysis (Fig. 5d and e) of the surface slag layer reveals, in comparison with optical microscopy, that the golden phase is TiO. Ti₂O₃ is shown in EBSD analysis for the blueish-grey phases, as Ti_{1.67}O₃ could not be selected for indexing.

The formation of Ti-slag layers at the sample surface causes Ti depletion in the bulk. EDS area scans show a reduction of the Ti concentration in the bulk between Ar and CO₂-processed samples (Table 3). FeTi4.3 samples processed in N₂ show the same Ti concentration as those manufactured in Ar.

3.3. Particle analysis

SEM and EDS of FeTi4.3 show the formation of homogeneously dispersed Ti-oxide nanoparticles. EDS measurements (Fig. 6) indicate similar particle compositions for Ti contents in between 1.8 wt% and 8.7 wt%. However, the exact oxide form (TiO, Ti₂O₃, ...) cannot be revealed by EDS or EBSD due to their small size and limited accuracy. Ti-oxide nanoparticles form in Fe-Ti samples manufactured in Ar by a reaction between Ti and O from the powders (Fig. 6a). Pure Fe shows the formation of Fe-oxide particles, as shown in previous work [19].

Although Ti-carboxide nanoparticles were expected for Fe-Ti samples manufactured under CO₂ due to O and C uptake from the atmosphere, no C could be detected in the particles (Fig. 6b). This might be due to the difficulties in detecting C via EDS and the small size of the particles. The formation of Ti-carboxides in similar alloys was reported in the literature using higher resolution analysis techniques [9,14].

Processing Fe-Ti samples in N₂ leads to the formation of Ti-oxynitrides, although similar difficulties concerning the ability to detect N by EDS exist (Fig. 6c). A further obstacle is the peak overlap between Ti L and N K lines in the EDS spectrum, making differentiation between the two elements challenging. However, the formation of Ti-oxynitride particles is reasonable as reported by several researchers processing Fe-based alloys by PBF-LB [12,16].

Fig. 7 shows the evolution of the nanoparticle fraction, f_p , particle diameter, d_p , and particle number density, n_p , in dependence on the Ti fraction in the feedstock and the atmosphere. Independent of the Ti level in the powders, f_p and n_p are strongly increased when manufacturing in CO₂ compared to Ar. The particle diameter, d_p , is slightly increased in CO₂-processed samples. Manufacturing under N₂ leads to a slightly higher fraction of nanoparticles, with a larger diameter but lower number density than in Ar. The fraction and size of the nanoparticles show the same trends as the overall O level in the materials. Apparently, O uptake from the atmosphere promotes particle formation in the alloys.

On the other hand, the Ti level in the powder does not seem to significantly change f_p , d_p , and n_p . A slight trend towards larger particles and a lower number density with increasing Ti content might be visible in the data.

3.4. Grain size and mechanical properties

The grain size and its distribution are strongly affected by the Ti level added in the powder blends. Note that the images in Fig. 8 show only a quarter of the used area to determine the average grain size based on the area fraction, D_A , for better visibility of the fine grains in the figure. Pure Fe shows grains elongated in the build direction. D_A in Fe decreases upon manufacturing in CO₂, i.e., increased O levels in pure Fe due to the process gas uptake cause grain refinement (Fig. 8a).

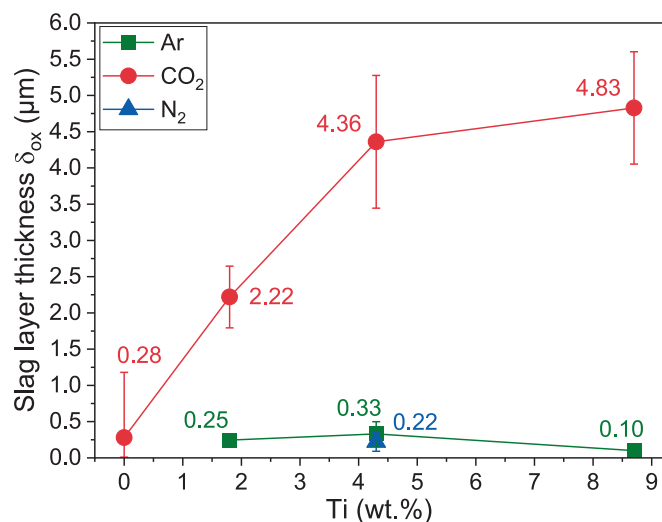


Fig. 4. Slag layer thickness measured for Fe-Ti alloys with different Ti contents after processing in Ar (green), CO₂ (red) and N₂ (blue) atmosphere with parameter set 3 (200 W, 800 mm s⁻¹).

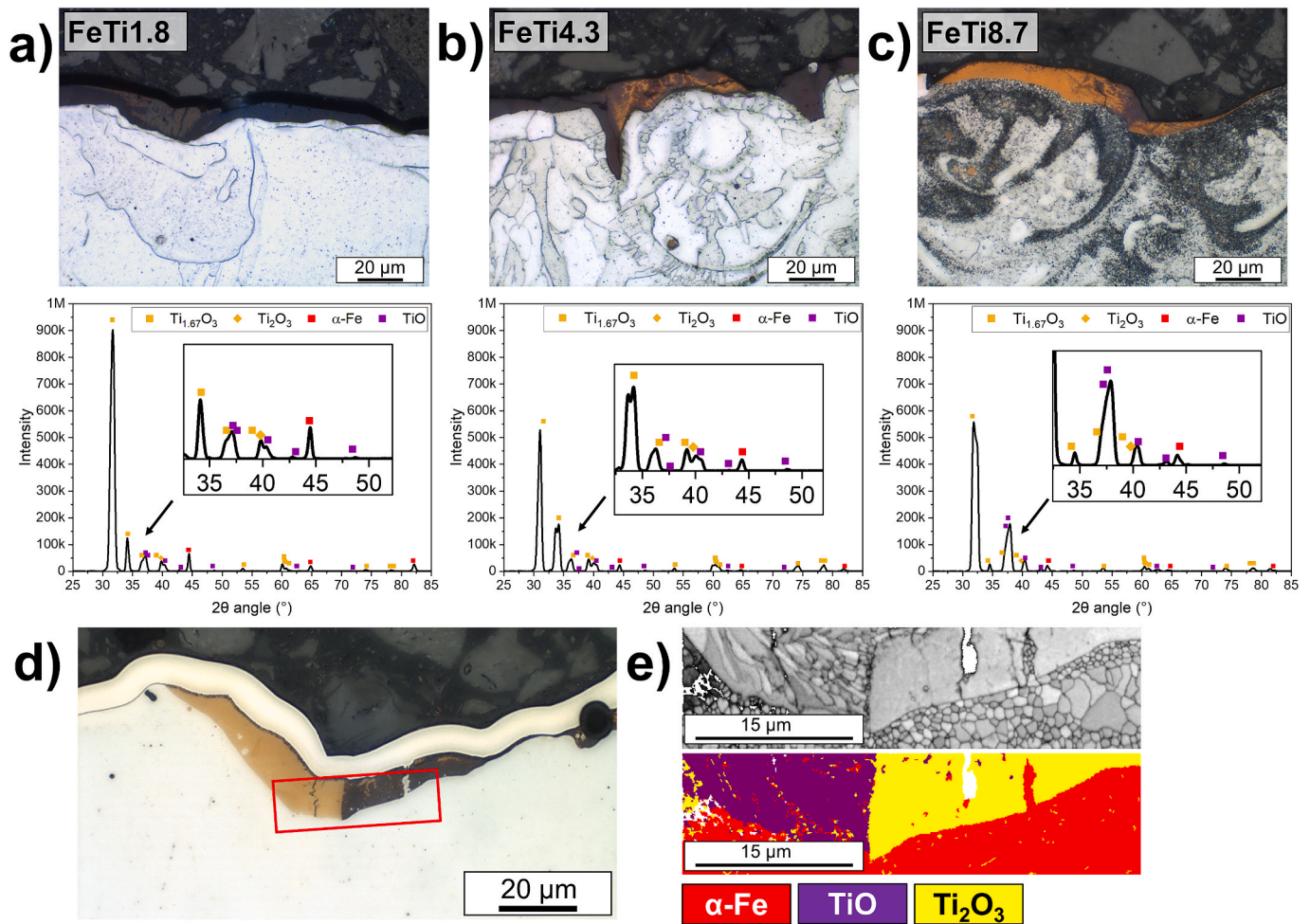


Fig. 5. Optical microscopy images of the surface slag layer of (a) FeTi1.8, (b) FeTi4.3 and (c) FeTi8.7 and their corresponding XRD diffractograms. According to the XRD diffractograms, the blueish grey phase is identified as $Ti_{1.67}O_3$ and the golden phase as TiO. (d) Mixed surface slag layer on top of FeTi8.7 samples depicts the golden phase as TiO and the blueish-grey phase as Ti_2O_3 as determined via (e) EBSD phase analysis. Note, $Ti_{1.67}O_3$ was not available for indexing in EBSD.

Table 3

Titanium concentration measured in the bulk via EDS area scans of samples with different nominal Ti concentration (powder blend) dependent on the process atmosphere used. Ti is transferred from the bulk to the slag layer, causing the reduction.

(wt% Ti)	Nominal	Ar	CO ₂	N ₂
FeTi1.8	1.8	1.4 ± 0.1	1.1 ± 0.1	
FeTi4.3	4.3	3.7 ± 0.2	3.4 ± 0.2	3.7 ± 0.2
FeTi8.7	8.7	7.9 ± 0.4	6.7 ± 0.2	

The addition of 1.8 wt% Ti to Fe causes coarser grains after processing in Ar and CO₂ compared to pure Fe specimens. Epitaxial growth is also observed (cf. Fig. 13 Appendix A.1). D_A is even larger for FeTi1.8 samples manufactured in CO₂ compared to Ar (Fig. 8b). Note that the dark areas are caused by Ti powder particles that did not properly melt and were not distributed in the melt pools (c.f. Fig. 14 Appendix A.2).

In contrast, FeTi4.3 samples depict grain refinement, and, different from FeTi1.8, grain size decreases by manufacturing in CO₂ compared with Ar and even more so when manufacturing in N₂ (Fig. 8c). Furthermore, a bimodal grain size distribution is observed in the microstructure, consisting of many very small grains and some large grains. The same bimodal grain size distribution is observed for FeTi8.7 (Fig. 8d). However, the fraction of very small grains is even larger in FeTi8.7 than in FeTi4.3 and the small grains are even smaller.

The hardness of Fe-Ti samples increases with increasing Ti level due

to the finer grain size, improving Hall-Petch strengthening, as well as solid solution hardening by Ti dissolved in Fe (Fig. 9). Particle reinforcement seems to have a minor influence on hardness.

Pure Fe samples fail almost immediately upon loading during creep testing (Fig. 10). The time to failure, t_f , increases with increasing Ti fraction. The highest t_f is observed for Ar-processed FeTi8.7 and FeTi4.3. Samples processed under CO₂ and N₂ show lower creep resistance than Ar-processed samples. FeTi8.7 samples have a comparable lifetime to FeTi4.3 ones despite the higher minimum creep rate, $\frac{d\epsilon_c}{dt}$, explained by lower creep rates at low displacements, ϵ_c (Table 4). The creep rate and time to failure are affected by solid solution hardening (Ti content), grain size, and nanoparticle fraction. Their respective contribution cannot be separated quantitatively from each other from the current results. Qualitatively, solid solution hardening seems to have the highest effect, and grain size to a lesser extent, as elucidated in the discussion part. Samples and corresponding microstructures show nearly defect-free samples after testing, as shown in Fig. 16 in Appendix A.4.

4. Discussion

4.1. Effect of Ti content on gas uptake

The material-gas interaction during melting in reactive atmospheres in PBF-LB depends mainly on two reactions: The equilibrium between dissociated gas in the laser plume and the liquid melt pool (absorption), which will be discussed in this section, and the equilibrium inside the

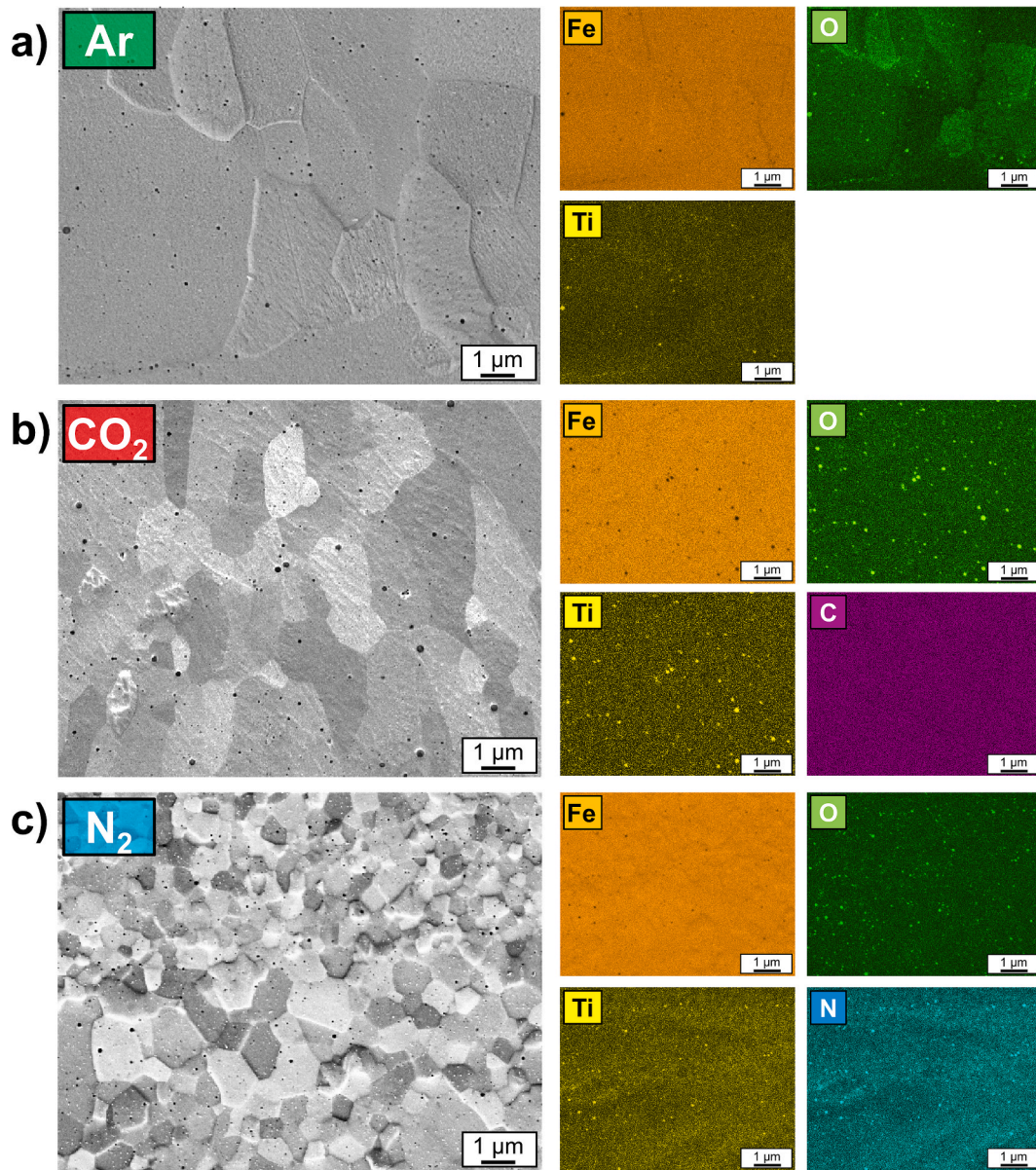


Fig. 6. Chemical composition of nanoparticles for FeTi4.3 samples manufactured in Ar, CO₂ and N₂ atmosphere: (a) Ti-oxide particles form in FeTi4.3 processing in Ar and (b) CO₂ due to a reaction of Ti with O in the powder and from the atmosphere. No C is detected in the particles resulting from processing in CO₂. (c) Ti-oxyntitride particles are observed in FeTi4.3 after processing in N₂ by a reaction with O in the powder and N from the atmosphere.

melt pool (deoxidation), which will be discussed in [section 4.2](#). Both processes occur simultaneously during PBF-LB and affect each other.

The interaction of Fe-based materials with the process gas during manufacturing increases with increasing Ti content in the powder feedstock. The O content is increased in the samples manufactured in CO₂ ([Fig. 3](#)), compared to those manufactured in Ar and N₂, due to gas uptake. Absorption of C and N is observed for materials alloyed with Ti only, but not for pure Fe. The O uptake from the CO₂ atmosphere is distinctly larger than for C, or for N from the N₂ atmosphere.

Before O, C, and N can be absorbed by the material, CO₂ and N₂ must dissociate, as the solubility of CO₂, carbon monoxide (CO), and N₂ in the liquid metal is low [[14,30,31](#)]. The dissociation energy of CO₂ → CO + O amounts to 526 kJ mol⁻¹ and of full dissociation CO₂ → C + 2O to 1598 kJ mol⁻¹ [[32](#)]. Full dissociation of CO₂ must occur during processing at least to a certain degree due to the observed C uptake and the insolubility of CO in liquid Fe [[30](#)]. The dissociation energy of N₂ → 2 N is higher than for the first dissociation step of CO₂, with 942 kJ mol⁻¹ [[33](#)]. The different dissociation energies might explain the larger O uptake

compared to N, which is approximately twice the energy that is necessary for the first dissociation step of CO₂. Full dissociation of CO₂ requires approximately three times the energy of dissociation into CO and O, which explains lower C uptake compared to O, in addition to the lower molar fraction of C in CO₂.

The gas uptake after dissociation can be described based on an equilibrium reaction between the atmosphere and liquid melt pool and depends on the partial pressures p_{CO_2} , p_{CO} , and p_{N_2} of the respective gas [[31,34,35](#)]. Note that, although it is clear that these reactions do occur, the short melt pool lifetimes and rapid cooling rates inherent in PBF-LB might prevent achieving equilibrium. The equilibrium constants for each atmosphere are given by Eqs. 2–4.

$$K_{CO,O} = \frac{f_O \times [\%O]}{\left(\frac{p_{CO_2}}{p_{CO}}\right)} \quad (2)$$

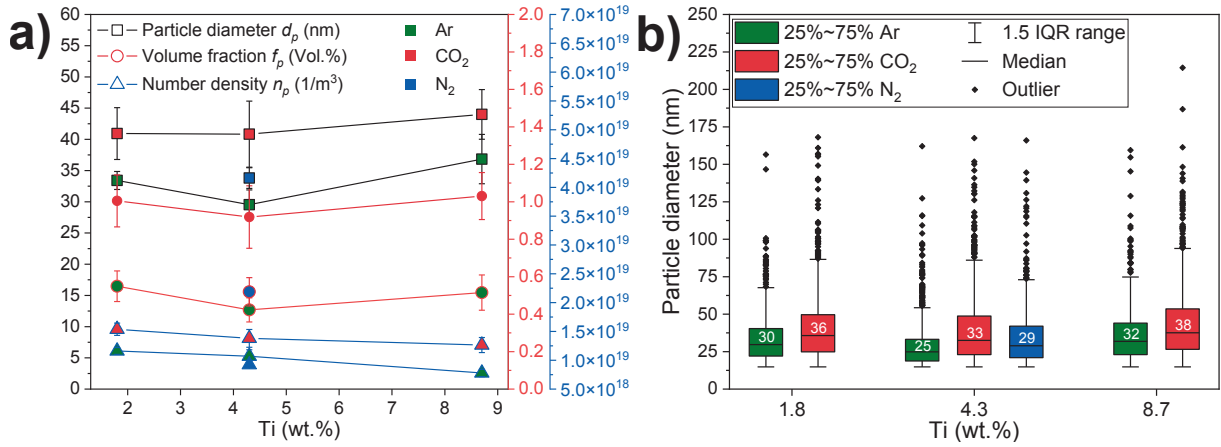


Fig. 7. (a) Particle size (black square), volume fraction (red circle) and number density (blue triangle) of nanoparticles. The filling of the symbols depicts the the atmosphere used to print the samples, which is green for Ar, red for CO₂ and blue for N₂. (b) Boxplot diagram showing the particle size distribution dependent on the Ti level and the atmosphere, with the same color designation as in (a) and the median value in the box.

$$K_{C,O} = \frac{(f_C \times [\%C]) \times (f_O \times [\%O])^2}{\left(\frac{p_{CO_2}}{p_0}\right)} \quad (3)$$

$$K_N = \frac{f_N \times [\%N]}{\left(\frac{p_{N_2}}{p_0}\right)^{1/2}} \quad (4)$$

The partial pressures of each gas are normalized using the atmospheric pressure p_0 of 1 atm. $[\%O]$, $[\%C]$, and $[\%N]$ denote the fraction of the respective element that is absorbed in the melt pool, according to the model. f_O , f_C , and f_N are the temperature and composition-dependent activity coefficients of the elements dissolved in the melt. The degree of dissociation, hence the ratio between CO₂ and CO in the laser plume, affects O uptake in the melt pool as can be seen in Eqs. (1) and (2).

In conventional AM machines, the gas pressure in the process chamber is only slightly above atmospheric level, and an increase in the partial pressure of CO₂ and N₂ beyond ca. 1100 mbar cannot be realized to increase the O, C, and N uptake. However, the process gas uptake can be increased by alloying. The activity coefficients f_O , f_C , and f_N are mainly dependent on the fraction of alloying elements and their first and second-order interaction parameters.

Several authors studied the dependence of alloy composition and temperature on the N uptake of iron-base alloys in N₂ atmospheres under high pressures [31,34–37]. Especially, nitride-forming elements have been found to increase the N uptake. Among these elements, Ti shows the highest effect by more than one order of magnitude. The high affinity of Ti to O and C allows drawing the same conclusions concerning an increased O and C uptake by alloying with Ti as for N. Accordingly, increased O, C, and N absorption with increasing Ti content is observed in this work (Fig. 3).

The calculated FeTi4.3-0.2 – N phase diagram shows a N solubility limit of ca. 0.15–0.20 wt% for FeTi4.3 (4.3 wt% Ti) (Fig. 11d), which is distinctly higher than for oxygen. 0.2 wt% O was included in the calculation to address the high reactivity of O with Ti and to predict phase stabilities more realistically. At 1873 K (1600 °C), N solubility is low, and high N concentrations would cause the formation of TiN, as stated by [38]. The solubility in the melt decreases with decreasing temperature in accordance with [34–37]. TiN (FCC_A1#2) is the first phase that solidifies in the system when the temperature decreases below 2000 °C.

The FeTi4.3–0.2O – C phase diagram (Fig. 11c) is similar to the one for N but shows a high C solubility in liquid Fe. TiC (FCC_A1#2) solidifies at $T < 1400$ °C and hence after TiO, in contrast to the FeTi4.3–0.2N system. TiC formation is not observed in the current experiments,

likely prevented by the short melt pool lifetimes and high solidification rates in PBF–LB.

4.2. Deoxidation reactions in the melt pool

In contrast to C and N, O is practically insoluble in Fe–Ti alloys, as depicted in Fig. 11b. The O solubility for a Fe–Ti alloy with 4.3 wt% Ti is less than 0.001 wt% [39]. Instead, an emulsion of two liquids, labelled LIQUID (liquid Fe–Ti) and LIQUID#2 (liquid Ti–oxide), forms at elevated temperatures for O concentrations above the solubility limit, i.e., in a supersaturated state. Extensive research has been done on understanding processes in metallurgy concerning deoxidation reactions in steel melts [39–42].

The oxide composition changes with Ti and O content, as depicted in the calculated phase diagrams (Fig. 11a and b). With increasing Ti and decreasing O content, the oxides formed are Ti₃O₅ → Ti₂O₃ → TiO. Ti₃O₅ is not predicted by Thermo-Calc for the compositional range used in this work, as it forms for Ti contents < 0.1 wt% [39].

The deoxidation equilibrium reactions for Ti₂O₃ and TiO in the melt pools are:



Chemical and microscopy analysis (Figs. 3 and 4) showed the formation of slag layers whose thickness increases with increasing Ti and O content in the materials. The O uptake during manufacturing in CO₂ promotes layer formation and explains the larger δ_{ox} compared to Ar and N₂ (Fig. 4).

XRD reveals a composition of the slag layers of Ti_{1.67}O₃, which deviates from Ti₂O₃ as predicted in the phase diagram, and TiO, as predicted by the phase diagram. The fraction of TiO in the XRD spectra increases with increasing Ti content in the materials according to the calculated phase diagram and as reported in the literature [39]. The deviation between expected Ti₂O₃ and experimentally determined Ti_{1.67}O₃ may result from further oxidation due the presence of higher O concentration close to the melt pool surface as simulated by Chia et al. [43,44]. Hence, slag layer composition might be less homogeneous as they consist of a mixture of different oxides and are prone to further oxidation at the melt pool surface where the process gas uptake occurs. Previous work, in contrast, showed that the slag layer consists mainly of Ti₂O₃ [19]. Current XRD results are supported by EBSD phase analysis detecting Ti₂O₃ and TiO (Fig. 5).

Deoxidation reactions, as described in the literature [39–42], typically involve the formation of solid oxides, due to the temperatures at

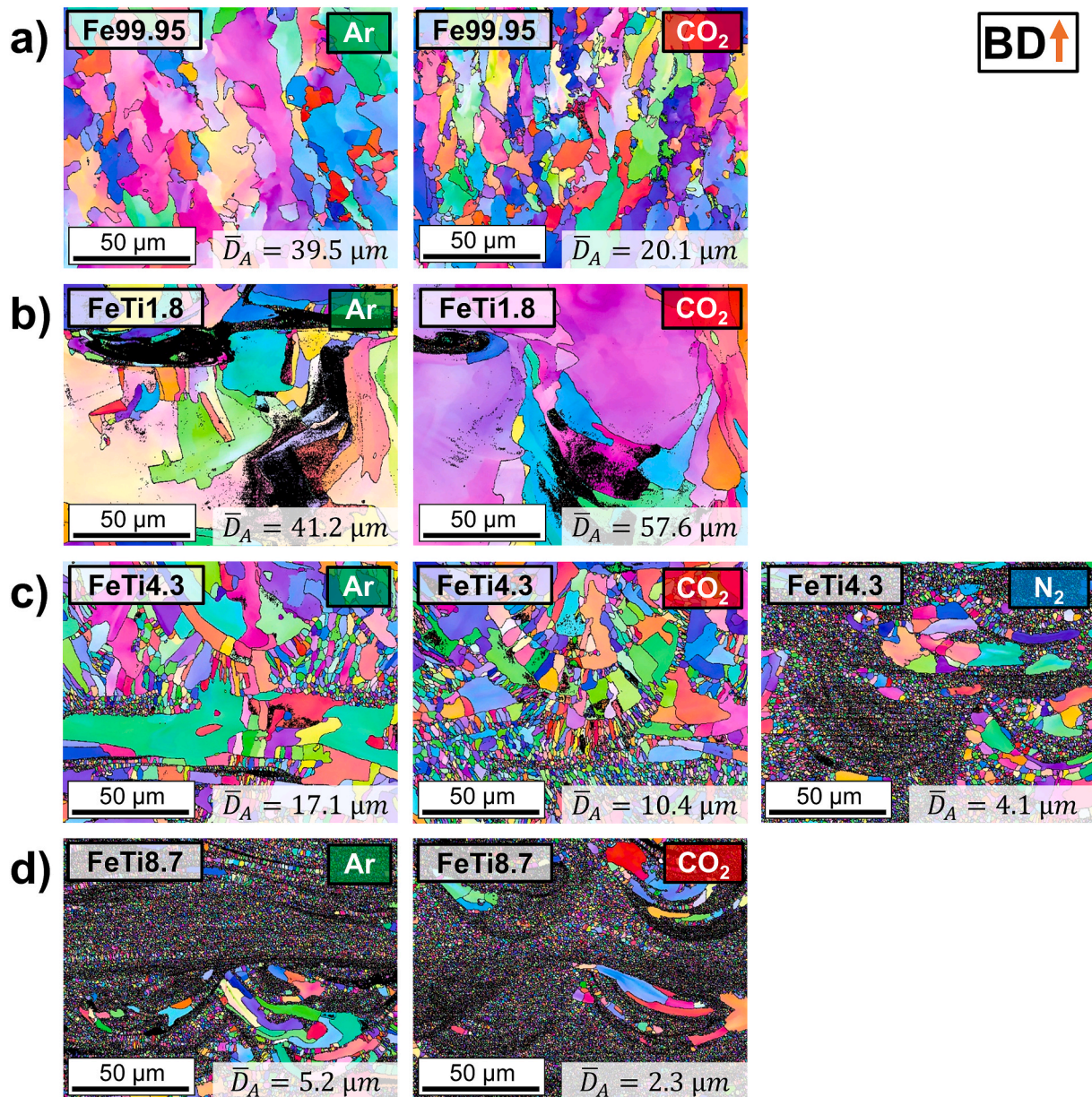


Fig. 8. Grain structure of the processed materials in Ar, CO₂, and N₂ along the build direction: (a) pure Fe, (b) FeTi1.8, (c) FeTi4.3, and (d) FeTi8.7. The number in the box, $\bar{D}_A$, denotes the average grain size determined for an area which is four times larger than the area of the image. Note, the dark areas in (b) are caused by Ti powder particles that did not properly melt and were not distributed in the melt pools (c.f. Fig. 14 Appendix A.2).

which these calculations were performed, which is typically at 1600 °C. Melt pool temperatures in PBF-LB can reach distinctly higher temperatures, up to 3000 °C, and differ across the melt pool [45]. Hence, deoxidation reactions need to be considered with liquid oxides (LIQUID#2) floating in/on the melt pool with similar O fractions as in the solid ones, as given in Eqs. 5 and 6.

Despite the low O solubility in Fe-Ti alloys, a strong O uptake from the atmosphere is measured in chemical analysis (Fig. 3b), which increases with increasing Ti fraction. Deoxidation removes O from the melt (LIQUID → LIQUID#2) and hence decreases O in solution. The depletion of the melt causes an increased O uptake from the atmosphere to compensate for the reduced O concentration according to the equilibrium reaction between atmosphere and melt pool.

For pure Fe, on the other hand, where there is a significant O solubility in the liquid state, the decomposition of O-rich melts occurs upon cooling below $T < 2000$ °C and for $O > 0.15$ wt% (Fig. 15 Appendix A.3). C and N are not absorbed by pure Fe.

4.3. Nanoparticle formation by deoxidation reactions

Considering the deoxidation reaction for particle formation, a higher amount of Ti and O on the educt side increases their activity and hence leads to the formation of more oxides on the product side. EDS analysis (Fig. 6) shows that the fraction of nanoparticles is distinctly increased by higher O and slightly increased by higher N supersaturation in the melt pool, as achieved by printing under CO₂ and N₂, but not by higher Ti levels in the materials (Fig. 7). In fact, increasing the Ti content in the materials also increases oxide formation, but the oxide is found in the slag layer rather than in nanoparticles. A strategy for achieving even higher nanoparticle fractions hence needs to promote the deoxidation reaction without increased slag layer formation.

Ti-oxide nanoparticles nucleate in the melt pool during PBF-LB, as long as it is liquid, grow, and coarsen within this time. Nanoparticles start to float to the surface to agglomerate in the slag layer when their size overcomes a critical value, as described by Stokes' law [46] and as

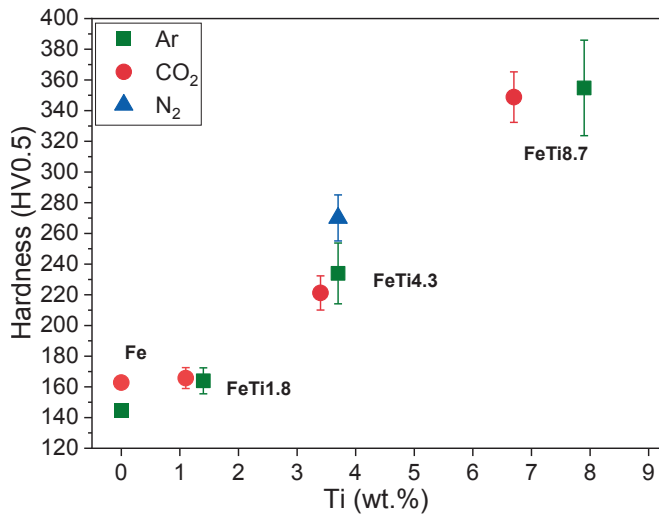


Fig. 9. Hardness evolution for pure Fe and Fe-Ti samples with different Ti content processed under Ar (green), CO₂ (red), and N₂ (blue) atmosphere.

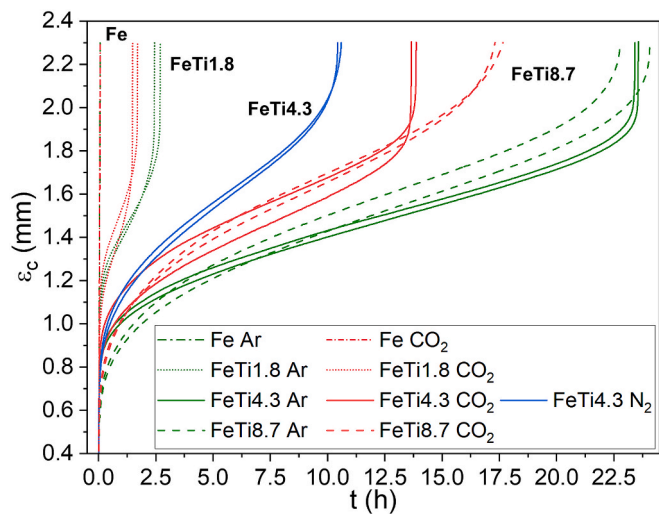


Fig. 10. Creep resistance and lifetime (SPC) of the processed samples at 600 °C and 450 N. The process gas is color coded (green: Ar, red: CO₂, blue: N₂) and the different compositions are distinguished with different line types (dash-dot: Fe99.95 Ar, dash-dot: Fe99.95 CO₂, dotted: FeTi1.8, solid: FeTi4.3, dashed: FeTi8.7).

Table 4
Minimum creep rate of the processed samples.

$\text{Min} \frac{d\epsilon_c}{dt} (\mu\text{m h}^{-1})$	Ar	CO ₂	N ₂
Fe99.95	13,200	30,300	
FeTi1.8	194 ± 10	306 ± 27	
FeTi4.3	30 ± 1	48 ± 3	78 ± 2
FeTi8.7	38 ± 1	44 ± 4	

discussed in our previous work [19]. TiO (4.95 g cm⁻³) and Ti₂O₃ (4.53 g cm⁻³) have a lower density than Fe (7.81 g cm⁻³), which is the main driving force for negative sedimentation of liquid Ti-oxide. In fact, negative sedimentation prevents achieving a higher nanoparticle concentration in the materials despite enhanced oxide formation. According to phase diagram calculations (Fig. 11c and d), TiC and TiN (FCC_A1#2) precipitate from the melt as solids during cooling at temperatures of about 1800 °C for TiN and 1350 °C for TiC. In contrast, the O solubility

in liquid Fe-Ti is extremely low, so liquid Ti-oxide (LIQUID#2) forms as a separate phase already in the liquid state. In contrast, TiN and TiC formation occurs at lower temperatures only present in the melt pools during cooling. The formation of a TiN and TiC rich shell around the oxide nanoparticles would have been expected for this reason, as reported in literature [9,14]. On the other hand, C and N contents in the materials are low (Fig. 3c and d) and cooling rates are high in PBF-LB which might have caused solute trapping. Hence, in the current work we cannot ascertain in which phase C and N are located in the microstructure, so this statement remains speculative. Thus, future work is planned to investigate the role of C and N during manufacturing in reactive CO₂ and N₂ atmospheres in specimens with increased melt pool lifetimes and thus decreased cooling rates.

The size of nanoparticles, d_p , is not significantly affected by Ti content but slightly increases due to O and N uptake in the melt pool (Fig. 7). It has been reported previously that manufacturing parameters (P , v), e.g., determining the melt pool lifetime, have a distinct effect on d_p [19,47,48]. Interestingly, their effect is larger than that of the composition of the melt pool investigated here.

4.4. Effect of particle formation on microstructure

The in-situ formed nanoparticles affect the grain structure in the material depending on their composition and crystallography. TiO (sodium chloride structure) acts as a grain refiner in ferritic alloys, as opposed to Ti₂O₃ (corundum structure) [16], which leads to coarse grains. This behavior is also observed for other particle compositions, e.g. Al₂O₃, with a corundum structure [15,17]. According to phase diagrams, the nanoparticle composition depends on the Ti fraction in the materials. The formation of Ti₂O₃ particles for FeTi1.8 and TiO particles for FeTi4.3 and FeTi8.7 is expected from the simulated phase diagrams (Fig. 11a) and was experimentally verified in previous work, using high resolution microscopy analysis [15,16].

The microstructure of FeTi1.8 samples shows large columnar grains that solidified epitaxially along the built direction (Fig. 13 Appendix A.1), which is due to the formation of Ti₂O₃ nanoparticles. The grain structure is distinctly refined in FeTi4.3 and even more so in FeTi8.7 due to the formation of TiO nanoparticles (Fig. 8). However, the stronger grain refinement for FeTi8.7 compared to FeTi4.3, despite similar f_p and n_p in both materials, suggests that not all particles have a composition of TiO. Instead, both particle compositions, TiO and Ti₂O₃, form in FeTi4.3 and FeTi8.7, but with a higher TiO/Ti₂O₃ ratio in FeTi8.7 than in FeTi4.3.

Using powder blends in PBF-LB in combination with low melt pool lifetimes and high cooling rates could explain the deviation in particle compositions from the prediction of the equilibrium phase diagrams. Short melt pool lifetimes limit the homogenization of Ti across the melt pool, leading to areas that are enriched or depleted in Ti [19]. This might also explain the observed bimodal grain structure in FeTi4.3 and FeTi8.7 (Fig. 8). Areas in which Ti barely mixed are occasionally observed in FeTi1.8, which led to unsuccessful indexing in EBSD. This is considered a manufacturing artefact caused by the in-situ alloying of blended powders (Fig. 14 Appendix A.2) and could be avoided by using pre-alloyed powder [49]. Note that these badly indexed areas were filtered out before grain size measurements. Deviations from equilibrium microstructures are common for additively manufactured materials due to the inherent high cooling rates and low liquid lifetimes [50–52].

Manufacturing in a reactive CO₂ atmosphere resulted in higher f_p and n_p compared to manufacturing in Ar (Fig. 7). The effect of the respective nanoparticles on the microstructure is therefore enhanced in CO₂-processed samples. Accordingly, a larger average grain size, D_A , is observed for FeTi1.8 and a lower one for FeTi4.3 and FeTi8.7 when processing in CO₂ compared to Ar. FeTi4.3 processed under N₂ shows the smallest grain size for this Ti content. Ti(O,N) nanoparticles apparently are more efficient grain refiners than the pure oxide, TiO.

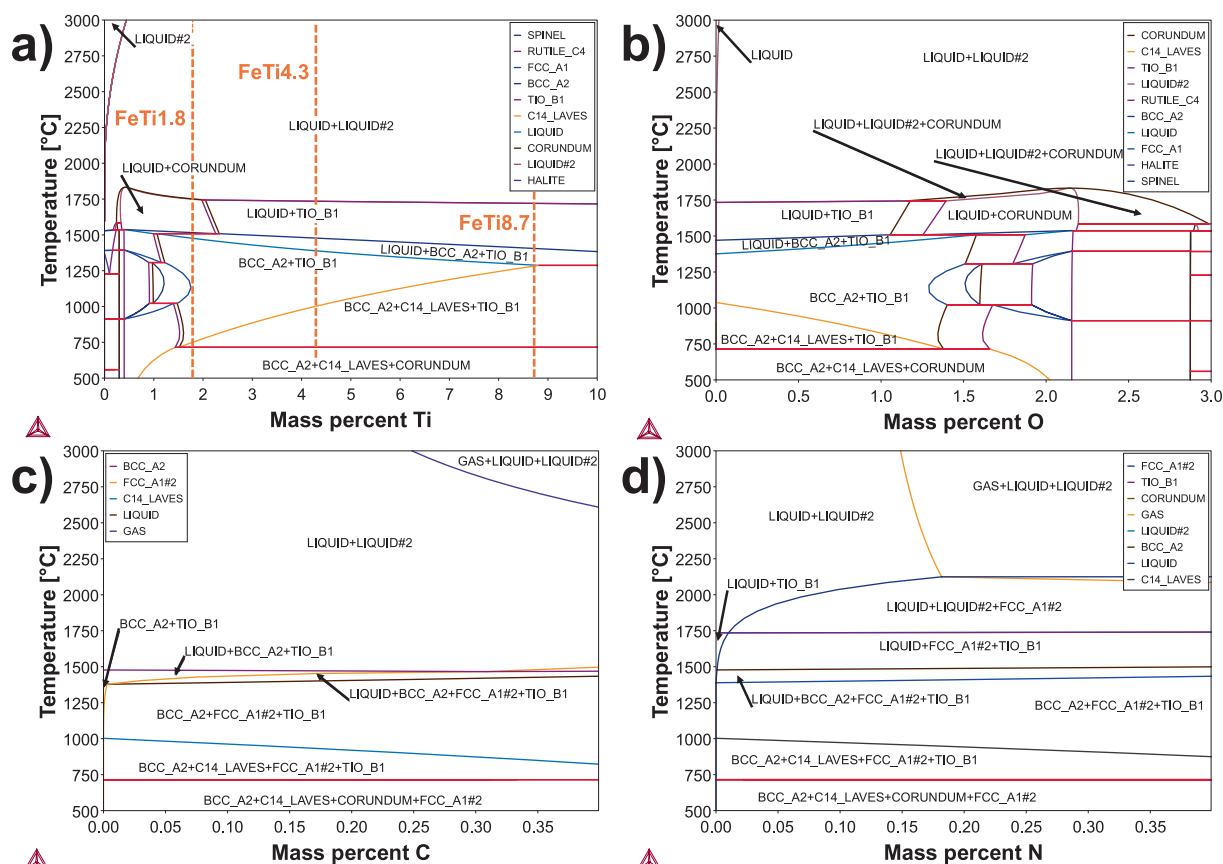


Fig. 11. Phase diagrams calculated with Thermo-Calc: (a) Fe-0.2 – Ti, (b) Fe-Ti4.3 – O, (c) Fe-Ti4.3-0.0.2 – C, and (d) Fe-Ti4.300.2 – N. Thermo-Calc version 2023a and the TCFe12:Steels/Fe-Alloys v12.0 are used for simulations.

4.5. Mechanical properties

Mechanical properties of the in-situ nanoparticle reinforced materials are affected by solid-solution strengthening, particle-induced grain refinement/coarsening, and nanoparticle reinforcement. This work aimed to study the effect of f_p and n_p on material properties. However, solid solution hardening and grain refinement dominate material properties in Fe-Ti alloys, and the effect of nanoparticles on hardness and creep strength vanishes. For this reason, the effect of the enhanced particle formation under CO₂ on material properties cannot be verified for the selected Fe-Ti materials system.

Ti concentration in the bulk of the samples as determined by SEM/EDS (Table 3) provides an approximation for the amount of Ti in the bulk material. A differentiation between Ti stored in nanoparticles and in solid solution is not obtained. However, simple calculations (Table 5) reveal that the fraction of Ti stored in nanoparticles and slag layer is low

and that it can be assumed that the values determined by SEM/EDS ($Ti_{SEM/EDS}$) approximately correspond to Ti in solid solution (Ti_{SS}).

Calculations assume that O has zero solubility in the materials (cf. Fig. 11b), and thus is found in oxides only. According to previous work [19], a composition of TiO (75 wt% Ti – 25 wt% O) for the nanoparticles and Ti₂O₃ (67 wt% Ti – 33 wt% O) for the slag layer is assumed for the calculations. For FeTi1.8, nanoparticle composition is assumed to be Ti₂O₃ according to Fig. 11b. O concentrations are provided by chemical analysis (Fig. 3). The O stored in the nanoparticles (O_{Nanoparticles}) corresponds to the O concentration in w/o surface state. The O concentration in the slag layer (O_{Slag layer}) is approximated by the difference between AB and w/o surface state. The amount of Ti in solid solution remains after subtracting the amount of Ti stored in nanoparticles (Ti_{Nanoparticle}) and the slag layer (Ti_{Slag layer}) from the nominal Ti content (Ti_{Nominal}) in the powder blends.

Hardness increases with increasing Ti fraction for Fe-Ti alloys due to

Table 5

Calculation of Ti concentration in solid solution (Ti_{SS}), assuming zero O solubility in the materials. The amount of O in the slag layer ($O_{Slag\ layer}$) and nanoparticles ($O_{Nanoparticles}$) was determined by chemical analysis (Fig. 3) of samples manufactured with $P = 200\ W$ and $v = 800\ mm\ s^{-1}$, such as for the samples used for creep testing and hardness measurements. Ti fraction stored in the nanoparticles ($Ti_{Nanoparticles}$) and the slag layer ($Ti_{Slag\ layer}$) are calculated assuming a composition of TiO (nanoparticles) and Ti_2O_3 (slag layer) [19]. Nanoparticle composition in FeTi1.8 is assumed to be Ti_2O_3 due to the low Ti fraction in the materials. Calculated values of Ti_{SS} are slightly above measured ones ($Ti_{SEM/EDS}$).

(wt%)	O _{Slag layer}	O _{Nanoparticle}	Ti _{Nominal}	Ti _{Slag layer}	Ti _{Nanoparticle}	Ti _{SS}	Ti _{SEM/EDS}
FeTi1.8 Ar	0.012	0.098	1.800	0.024	0.196	1.58	1.4
FeTi1.8 CO ₂	0.057	0.168	1.800	0.114	0.336	1.35	1.1
FeTi4.3 Ar	0.008	0.107	4.300	0.016	0.321	3.96	3.7
FeTi4.3 CO ₂	0.096	0.200	4.300	0.192	0.600	3.51	3.4
FeTi4.3 N ₂	0.009	0.101	4.300	0.018	0.303	3.98	3.7
FeTi8.7 Ar	0.005	0.108	8.700	0.010	0.324	8.37	7.9
FeTi8.7 CO ₂	0.120	0.278	8.700	0.240	0.834	7.63	6.7

enhanced solid-solution strengthening and grain refinement (Fig. 9). Fe-Ti samples processed under CO₂ show a slightly lower hardness compared to the ones fabricated in Ar with the same composition, although the average grain size is smaller for FeTi4.3 and FeTi8.7, and the particle concentration is higher. A possible reason for the lower hardness of samples processed in CO₂ is the depletion of Ti in the bulk due to the formation of surface slag layers, as measured in EDS (Table 3). This would further explain the high hardness of FeTi4.3 processed in N₂, as no significant slag layer forms at the surface, so no depletion of Ti in the bulk is expected, but strong grain refinement is observed.

Creep strength is dominated by solid solution hardening, which shows the greatest influence on creep strength followed by the grain size (Fig. 12). The minimum creep rate and creep lifetime of FeTi4.3 and FeTi8.7 are similar, as strong grain refinement in FeTi8.7 compensates for the increase in creep strength by the higher Ti fraction (Fig. 10). FeTi1.8 shows distinctly lower creep lifetime than the other two compositions, despite the large average grain size. Consequently, samples manufactured in CO₂ show lower creep strength than the ones manufactured in Ar, explained by the loss of Ti in the bulk due to the transfer to the slag layer (Table 3).

The Ti distribution in the materials is slightly inhomogeneous due to the powder blending approach. However, differentiation between compositional inhomogeneities is required: Partially unmelted Ti particles are an artefact, only occurring in FeTi1.8 microstructure samples. Whereas slight fluctuations of the Ti concentration occur in all Fe-Ti alloys, are expected to locally influence nanoparticle composition and cause the bimodal grain size distribution. However, these fluctuations are so small that they cannot be detected in EDS. Using pre-alloyed powders would eliminate these problems, as a homogeneous Ti distribution is expected after printing. This is the recommended approach for processing in a production setting.

In this work, the effect of inhomogeneous Ti distribution caused by using a powder blending approach was tried to be mitigated as much as possible: In hardness measurement, 15 positions across each sample were measured and each position was checked in advance to not hit an unmelted Ti particle or slag particle. Furthermore, unmelted Ti particles occurred in the microstructure samples only but were not observed in the samples for SPT testing, as visible in Fig. 16 in Appendix A.4, showing representative sections of the microstructure at low magnification. We therefore consider the mechanical testing results to be reliable. Additionally, here we are interested mainly in the different properties due to different Ti contents between the alloys, which can be reliably estimated from the current experiments, as difference in Ti concentrations are large between the three different Fe-Ti alloys.

The materials processed in this work show a homogeneous nanoparticle distribution and high particle fraction of ca. 0.9 vol% (0.6 wt%) under CO₂, which is comparable to conventional ODS alloys with oxide fractions in between 0.3 and 0.5 vol% [53]. Hence, using the process atmosphere to manufacture in-situ Ti-oxide nanoparticle reinforced

materials could be an alternative to ex-situ reinforced ODS alloys. The oxide composition of the in-situ nanoparticles can be tuned by adjusting the Ti alloying content, which gives further control of the grain size in the materials and mechanical properties. However, suitable alloy compositions and processing conditions would need to be found in order to avoid Ti loss due to slag layer formation while keeping the beneficial effects of the nanoparticles.

5. Conclusions

In this work, the influence of different Ti alloying contents on the properties of additively manufactured Fe-Ti model alloys fabricated by PBF-LB under reactive CO₂ and N₂ atmosphere was studied. The following conclusions can be drawn:

- Fe-Ti alloys processed under CO₂ and N₂ show O and C uptake, and N uptake from the process atmosphere. Only Ti alloyed materials absorb C and N, whereas pure Fe does not show an increase in both elements compared to the powder feedstock, even when manufactured in CO₂ or N₂ atmosphere. The interaction with the atmosphere increases with increasing Ti level in the materials, causing higher process gas uptake. Samples processed in Ar show a slight O uptake, likely from residual O₂ in the process chamber.
- Fe-Ti alloys show the formation of Ti-oxide slag layers on the sample surface, which is promoted by increased levels of O, due to the process gas uptake and by increasing the Ti fraction in the powder feedstock. Pure Fe does not show significant slag layer formation, in contrast. The slag layer is mainly composed of Ti_{1.67}O₃ and TiO, and their fraction depends on the Ti level in the powder blend. High Ti contents favor the formation of TiO at the cost of Ti_{1.67}O₃.
- The size, fraction, and number density of the nanoparticles are not significantly influenced by the Ti alloying content, but by O supersaturation in the melt pool. Using a reactive CO₂ atmosphere for manufacturing instead of Ar increases O supersaturation and promotes particle formation.
- The composition of the nanoparticles is determined by the level of Ti in the feedstock powder. High Ti contents favor the formation of TiO compared to lower Ti contents, resulting in the formation of Ti₂O₃.
- The in-situ formed TiO particles primarily solidify upon cooling and act as a heterogeneous nucleation site for ferrite grains, resulting in grain refinement. Ti₂O₃ nanoparticles cause the opposite effect. The hardness of the materials increases with increasing Ti content due to higher solid solution strengthening and more grain refinement. The creep resistance is mainly improved by solid solution hardening but reduced by the accompanying grain refinement.

CRedit authorship contribution statement

C. Felber: Writing – review & editing, Writing – original draft,

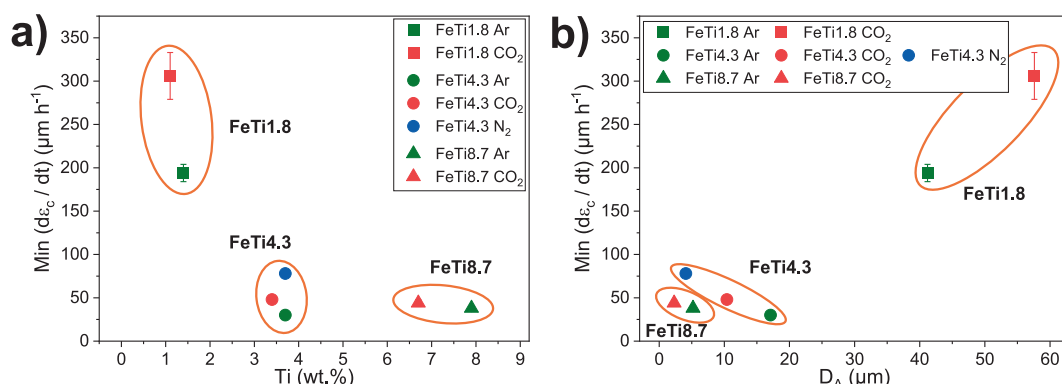


Fig. 12. Contribution of (a) Ti content and (b) average grain size to the minimum creep rate.

Visualization, Project administration, Methodology, Investigation, Formal analysis, Conceptualization. **E.A. Jäggle:** Writing – review & editing, Writing – original draft, Supervision, Project administration, Methodology, Funding acquisition, Formal analysis, Conceptualization.

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.

Appendix

A.1 FeTi1.8 microstructure

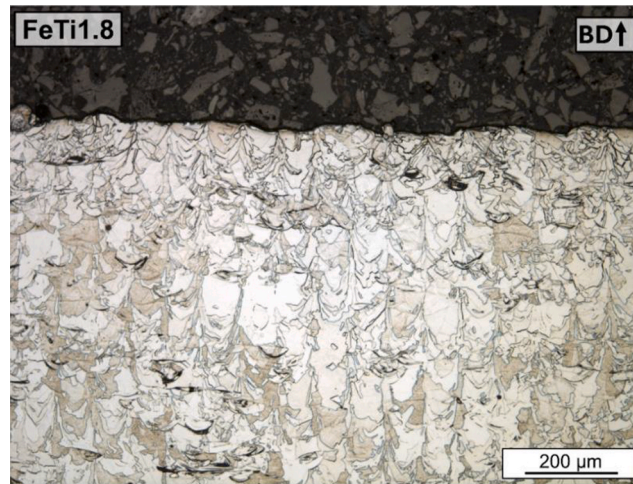


Fig. 13. Microstructure of FeTi1.8 in optical microscopy showing the formation of elongated grains in build direction.

A.2 Ti distribution in Fe-Ti alloys

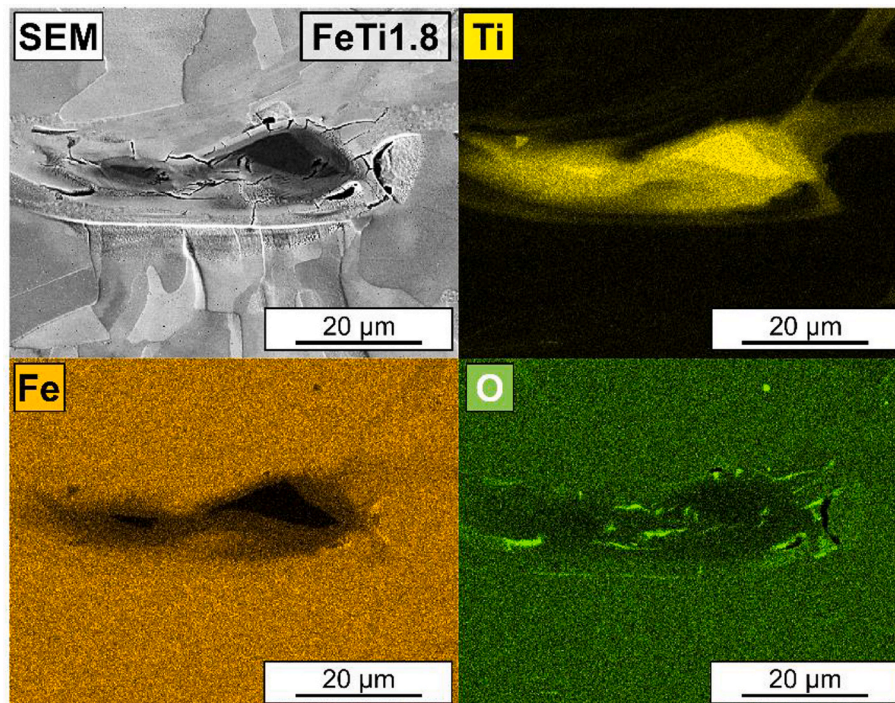


Fig. 14. Ti enrichment in the bulk microstructure of FeTi1.8 caused by insufficient melting of Ti powder particles during PBF–LB.

A.3 Fe–O phase diagram

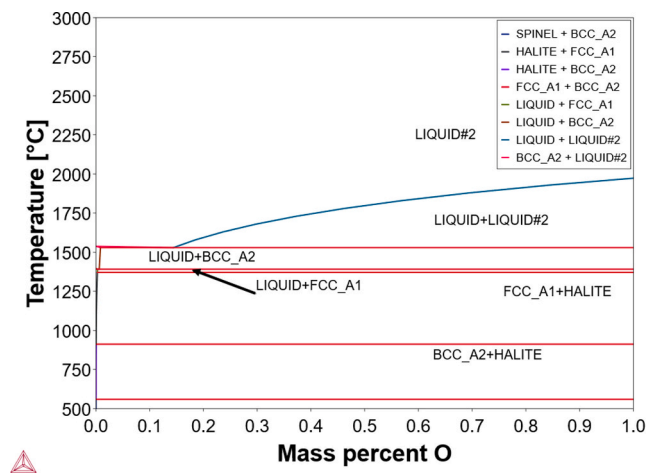


Fig. 15. Phase diagram of Fe-O showing high O solubility at elevated temperatures and the precipitation of liquid Fe-oxide (LIQUID#2) for high O contents and temperatures above the melting point of Fe.

A.4 SPCT specimens after testing and their microstructure

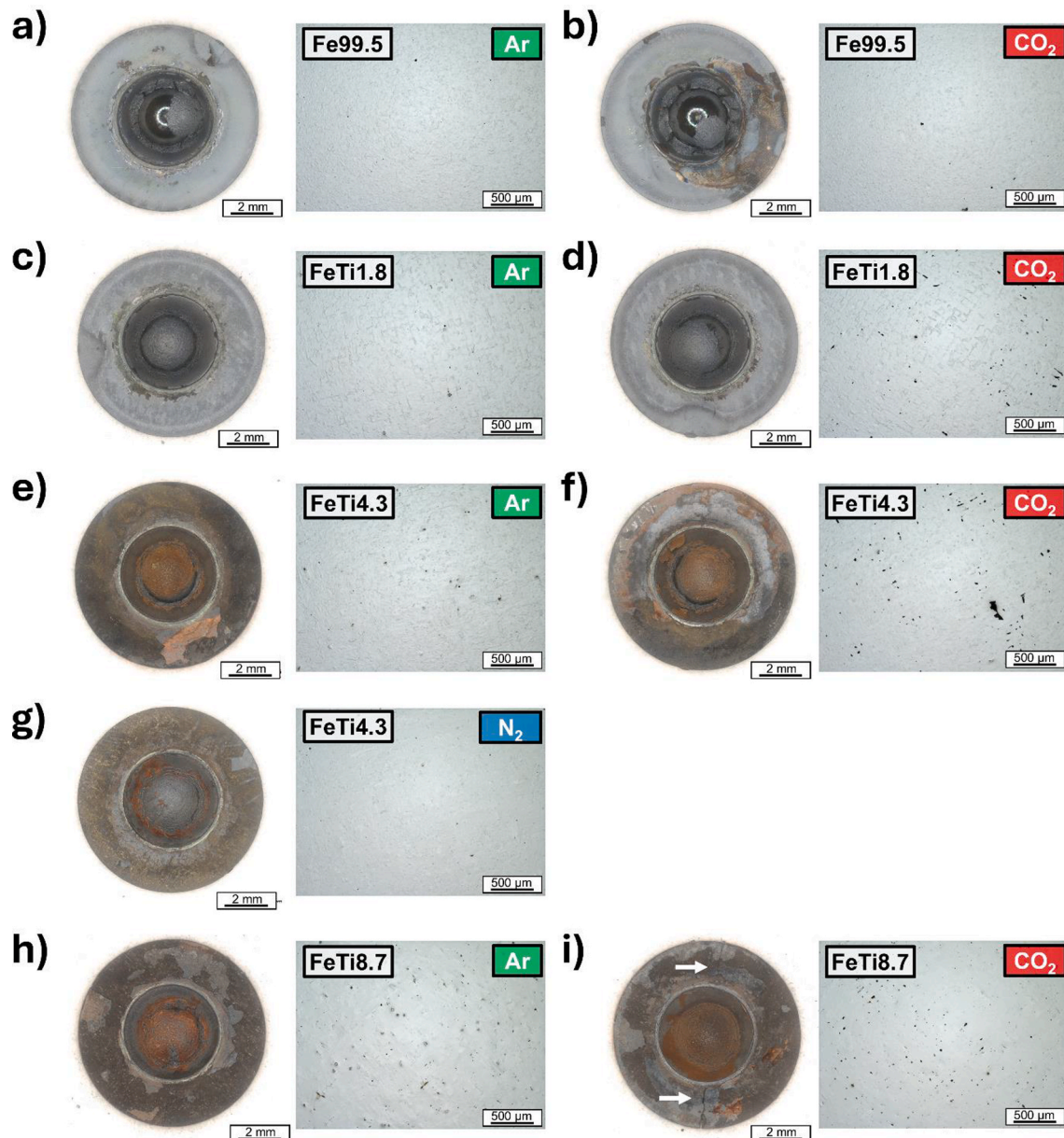


Fig. 16. SPT samples after testing (upside down) and corresponding microstructure: (a) Fe Ar, (b) Fe CO₂, (c) FeTi1.8 Ar, (d) FeTi1.8 CO₂, (e) FeTi4.3 Ar, (f) FeTi4.3 CO₂, (g) FeTi4.3 N₂, (h) FeTi8.7 Ar, (i) FeTi8.7 CO₂. Samples processed in CO₂ show higher slag particle fraction than under Ar. FeTi8.7 samples also show large amount of slag particles under Ar. The high Ti concentration in FeTi8.7 causes cracking during testing due to increasing embrittlement with increasing Ti fraction (white arrows).

Data availability

Data will be made available on request.

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