Oxidation response of additively manufactured eutectic Mo-Si-B alloys

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Abstract. Previous studies showed the technical feasibility of additive manufacturing (AM) of Mo-Si-B alloys using the laser powder bed fusion (L-PBF) process. The competitiveness of the properties of AM alloys was demonstrated by a comparison to literature data from conventionally processed Mo-Si-B materials. In addition to the excellent mechanical properties at ambient and elevated temperatures, the oxidation resistance in a wide temperature range is another decisive aspect for the potential use of AM Mo-Si-B alloys. The present work shows investigations on the cyclic oxidation performance of a eutectic AM Mo-16.5Si-7.5B alloy. Depending on the temperature (800 °C, 1100 °C, 1300 °C), the oxidation mechanisms are different, which is due to different reactions at the surface of the alloys accompanied with mass changes of samples. These mass changes can be explained on the basis of microstructural investigations. However, compared to a directionally solidified (DS) alloy, the AM alloy shows improved oxidation resistance.

1 Introduction

Recent research has demonstrated that Mo-Si-B alloys from the three-phase field Mo₇ₓ-Mo₅ₓSi-Mo₅ₓSiB₂ are promising candidates for high temperature applications, e.g. in gas or aircraft turbines or solar receivers [1–4]. This class of materials combines different key capabilities, like excellent ultra-high temperature creep resistance, sufficient fracture toughness and good oxidation resistance. It is also known that all properties, specifically the oxidation resistance, depend strongly on the phase fraction and size of the Mo solid solution (Mo₇ₓ) phase [5].

In general, there are some established production routes that result in dense and crack-free Mo-Si-B bulks, which are powder metallurgical methods based on solid-solid reactions [6,7] and techniques based on melting and subsequent cooling. Examples for the latter are drop-casting and annealing [8] or directional solidification [9]. These processes lead to either typical ultra-fine-grained and homogeneous microstructures in case of using powder metallurgy, typical solidification features, like dendrites of the primarily solidified phase, in case of cooling from a molten phase, or elongated constituents in case of directional solidification. Furthermore, depending on the processing route and the alloy composition,
the materials behavior may be tailored in terms of balancing the ambient and high temperature properties and providing isotropic or anisotropic properties.

In the present approach we use the current knowledge on the solidification paths of Mo-Si-B alloys and a new additive processing method. Additive manufacturing (AM) allows the production of various geometries as well as the integration of complex cooling structures. This combination of a promising type of material and improved manufacturing technique will result in more efficient parts, e.g. for modern turbine blades. However, additive manufacturing of Mo-Si-B alloys is very challenging due to the ultra-high melting temperatures of this type of alloys ($T_M > 2000 \degree C$), the requirement of spherical powders for providing a good flowability in the AM process, and the affinity for oxygen. Oxygen is known to embrittles Mo alloys significantly, even if present in ppm concentrations [10].

Additionally, there is a complex temperature-dependent oxidation mechanism working in multi-phase Mo-Si-B alloys. This is schematically drawn in Figure 1. While heating a Mo-Si-B sample in air there will be a slight mass gain at temperatures $> 500 \degree C$, which is due to the formation of a B-Si-Mo-O scale at the surface. A MoO$_3$ phase starts to form at these comparatively low temperatures. Around 700 $\degree C$ the MoO$_3$ phase begins to evaporate from the surface, which then results in a porous B-Si-O scale. In this temperature window a catastrophic mass loss may lead to entire degradation of the Mo alloy, which is called the “pesting” phenomenon. At 800 $\degree C$ MoO$_3$ transiently evaporates and permeates out, which results in a significant mass loss as shown in Figure 1. At higher temperatures above 1000 $\degree C$ the mass change is then reduced due to the formation of a borosilicate glass layer, which protects the surface from further oxidation. With increasing the temperature up to 1300 $\degree C$ the evaporation of B$_2$O$_3$ will result in a B-lean silica scale [5,11–13].

![Figure 1. Schematic representation of the temperature-dependent oxidation behavior of Mo-Si-B alloys.](image-url)

In order to use Mo-Si-B alloys as structural materials at ambient and high temperatures, a good oxidation resistance at elevated temperatures is essential. Cyclic oxidation tests provide an excellent imitation of real conditions including heating and cooling comparable to start and shut-down processes in high temperature turbines. In the present work it will be shown how a AM Mo-Si-B alloy behaves during cyclic oxidation treatment at three different temperatures: (1) 800 $\degree C$, which is within the “pesting” regime, (2) 1050 $\degree C$ - 1100 $\degree C$, which is a typical application temperature of state-of-the-art Ni-based superalloys used in gas turbines and (3) 1300 $\degree C$ as a potential application temperature of Mo-Si-B alloys. For this study a eutectic AM alloy Mo-16.5Si-7.5B is used, which is selected on the basis of a recently published liquidus projection identifying the eutectic point [14]. The oxidation response of this material is compared to a directionally solidified (DS) alloy with a very similar chemical composition of Mo-17.5Si-8B.
2 Materials and Methods

The eutectic alloy composition of Mo-16.5Si-7.5B (in at. %) was chosen due to the lowest melting point of the Mo-rich Mo-Si-B materials, which is slightly above 2000 °C. This may be beneficial for the gas atomization process and laser additive processing as compared to Mo-Si-B alloys with even higher Mo concentrations, since the melting point of Mo is ~2600 °C. The details of gas atomization yielding in spherical pre-alloyed powders are described elsewhere [15]. For additive manufacturing the process of laser powder bed fusion (L-PBF) is used, which allows to build complex geometries by using high pre-heating temperatures. The latter is important since the brittle-to-ductile transition temperature of this type of materials is around 1000 °C and heating is essential to avoid cracks during cooling [16]. For comparison, a directionally solidified (DS) alloy Mo-17.5Si-8B processed via a crucible-free float-zone technique was used. DS samples with a typical diameter of 6 mm were grown in a high purity He atmosphere using a solidification rate of 1 mm/min [17].

Microstructural investigations of the L-PBF and DS materials in their as-processed state and after oxidation were carried out on samples cut via electrical discharge machining (EDM), subsequently embedded in a hot mounting polymer (Struers PolyFast). After wet-grinding from 500 down to 1200 grit, the specimens were polished with a 3 µm and 1 µm diamond suspension and finished by a colloidal silica suspension (OPS) [16,17]. The microstructural observations were accomplished by scanning electron microscopy (SEM - FEI ESEM XL30 FEG and Zeiss EVO 15 equipped with EDS). For the AM Mo-16.5Si-7.5B alloy, electron backscatter diffraction (EBSD) analysis was performed to identify the phases using a Zeiss Merlin SEM equipped with a Nordlys EBSD camera and Aztec software package (Oxford Instruments) provided by the Forschungszentrum Jülich, Germany.

Samples for cyclic oxidation tests were cut from the bulks in dimensions of 3 mm x 3 mm x 5 mm for L-PBF and 3 mm in diameter and 5 mm in height for the DS material. For cyclic oxidation certain temperature regimes and exposure times were chosen: (i) 800 °C/ 500 h as well as 800 °C/ 100 h on pre-oxidized (pre-oxidation at 1300 °C/ 1 h, according to [13]) samples, (ii) 1050 °C/ 500 h and 1100 °C/ 100 h and (iii) 1300 °C/ 100 h.

To study the oxidation kinetics, the exposure of samples in static air at certain temperatures was typically accomplished at defined cycles. For the AM L-PBF Mo-16.5Si-7.5B alloy, the intermediate weighing of samples was performed every 10 minutes during the first hour, every 60 minutes up to 8 h and finally every 24 hours until the total exposure time (100 h or 500 h, respectively) was reached. The experimental work was done at the IfKorr (Institut für Korrosions- und Schadensanalyse, Magdeburg, Germany) using a LF15/14 Nabetherm furnace. The exposure times per cycle for alloy DS Mo-17.5Si-8B were 10 minutes during the initial oxidation stage (< 1 h), subsequently increased to 1 h cycles up to 20 h, followed by 5 h cycles up to 50 h and 10 h cycles until the final 100 h of total oxidation exposure was reached. The experiments were performed under static air in a box furnace at 1100 °C (Thermconcept KLS 15/14).

Before placing the AM L-PBF and DS samples within the pre-heated furnace, their initial weight and dimensions were exactly determined. At the end of each cycle the samples were removed from the furnace, cooled down to room temperature under atmospheric conditions and weighed carefully to determine their weight loss after certain exposure times. Afterwards the samples were re-exposed to the particular temperature regime described above. Finally, the macroscopic sample morphology and shape retention after finishing the total exposure time was recorded using stereomicroscopes (Olympus SZX7 and Zeiss Stemi 2000 C).

The resulting weight change curves were plotted vs. the exposure time, representing the predominant oxidation mechanisms. Additionally, in order to gain a better understanding of the temperature-dependent oxidation mechanisms, the microstructural features of the oxidized samples were studied via SEM. Therefore, the samples were prepared as described above.
3 Results and Discussion

3.1 Microstructures after processing

The microstructure of alloy AM L-PBF Mo-16.5Si-7.5B is depicted in Figure 2a, representing Mo5si dendrites embedded in a silicide matrix. The SEM-BSE image reveals that during AM processing, the liquid melt solidifies extremely fast which leads to the very fine microstructure in the lower µm scale. Additional EBSD analysis enabled the identification of the corresponding phases. The resulting EBSD phase map, illustrated in Figure 2b, shows the homogeneous distribution of the three phases Mo5si (blue), Mo3Si (yellow) and Mo5SiB2 (red). With 48 vol. % the Mo3Si phase is dominantly represented whereas the phases Mo5si and Mo5SiB2 occur with 30 vol. % and 22 vol. %, respectively.

In contrast, the DS Mo-17.5Si-8B alloy shows a cellular growth of ternary eutectic cells with a well-aligned microstructure orientation parallel to the growth direction. Figure 2c and d illustrate the DSed microstructure in which the typical eutectic cells can be observed. Ternary eutectic colonies with typical cell sizes ≤ 100 µm are clearly visible. The appearance of the ternary eutectic is similar to that prepared via AM, consisting of ~ 50 vol. % Mo3Si eutectic matrix phase and small amounts of Mo5si (~ 20 vol. %) and Mo5SiB2 (~ 30 vol. %).

![Figure 2. SEM-BSE micrographs of the near eutectic alloys (a) AM Mo-16.5Si-7.5B with (b) EBSD phase mapping of Mo5si (blue), Mo3Si (yellow), Mo5SiB2 (red) and DS Mo-17.5Si-8B in (c) longitudinal and (d) transversal direction.](image)

3.2 Cyclic oxidation response at 800 °C

The following sections concentrate on the description of the oxidation properties of the eutectic alloy AM Mo-16.5Si-7.5B compared with the DS Mo-17.5Si-8B alloy having a very similar chemical composition. The mass loss curves and corresponding microstructures of the oxide scales are shown and discussed in order to understand the predominant oxidation mechanisms.

The temperature regime around 800 °C is the most challenging for Mo-Si-B alloys due to the occurrence of the “pesting” phenomenon characterized by catastrophic oxidation due to MoO3.
evaporation. The mass loss curve of alloy AM Mo-16.5Si-7.5B, being typical for Mo-rich alloys according to Parthasarathy et al. [13], is represented by the blue curve in Figure 3a.

![Mass loss curve of alloy AM Mo-16.5Si-7.5B](image)

**Figure 3.** Mass change curves (a) of as-built and pre-oxidized AM Mo-16.5Si-7.5B oxidized at 800 °C and macroscopic cross sections of (b) the pre-oxidized sample after 100 h and (c) the sample without pre-treatment after 500 h.

This typical pesting behavior is characterized by significant weight change right from the beginning of exposure. After approximately 30 h almost no further mass loss than ~263 mg/cm² can be observed, which means that catastrophic oxidation failure occurred by nearly entire oxidation of the sample. After the total exposure time of around 500 h, the sample is nearly fully oxidized, as depicted in Figure 3c.

In order to improve the oxidation resistance at 800 °C, the pre-treatment concept after Parthasarathy et al. [13] was used. Significantly slower oxidation kinetics was reported, when pre-oxidizing the samples (1300 °C for 1 h) allowing the formation of thermodynamically stable silica scales, which prevents the sample from MoO₃ formation and evaporation. Accordingly, the alloy AM Mo-16.5Si-7.5B was initially oxidized at 1300 °C for 1 h before the sample had been tested under cyclic conditions at 800 °C for a total exposure time of 100 h.

During the relatively short pre-oxidation time a protective SiO₂ should form relatively fast on the material’s surface. After its formation MoO₃ volatilizes nearly instantaneously at this temperature, the materials surface is quickly saturated with Si and B forming the silica scale. Since also B₂O₃ slowly evaporates at 1300 °C, the B₂O₃-lean glass layer is highly viscous. As can be seen in Figure 3b, a protective SiO₂-B₂O₃ scale could be formed, resulting in a significantly improved oxidation resistance at 800 °C compared to the as-built AM Mo-16.5Si-7.5B alloy, depicted in Figure 3c. However, after approximately 60 h, significant local oxidation occurs.

An EDS element mapping of an area with beginning oxidation, depicted in Figure 3, shows the formation of a porous B-lean SiO₂ scale enabling further oxygen diffusion. It can be assumed, that due to the high fractions of Mo₅Si + Mo₅p phase in this alloy, but only 22 % of Mo₅SiB₂ phase, no significant reservoir for the formation B₂O₃ is present. Hence, the viscosity of the glass layer is reduced, impeding the formation of a continuous protective scale at intermediate temperatures. Accordingly, pre-oxidizing the sample just offers a temporary protection against oxidation at 800 °C as long as the previously formed borosilica scale is completely intact.
Figure 4. SEM-SE micrograph of the pre-oxidized AM Mo-16.5Si-7.5B alloy oxidized at 800 °C for 100 h combined with EDS element mapping for B, O and Si.

3.3 Oxidation resistance at 1050 °C and 1100 °C

The temperature regime around 1100 °C is very important since it displays a typical application temperature of current state-of-the-art Ni-based superalloys used in gas turbines. In contrast to the oxidation mechanism at 800 °C accompanied with a high mass loss, the oxidation behavior changes when the samples are exposed and oxidized at higher temperatures.

As depicted in Figure 5a, after an initial mass loss during the first hour at 1050 °C and 1100 °C, there is no further change of the mass of the samples in case of AM Mo-16.5Si-7.5B. Even if the exposure time is extended to 500 h, no further mass loss can be observed at 1050 °C. However, for alloy DS Mo-17.5B-8B, Figure 5a shows a higher initial mass loss compared to AM Mo-16.5Si-7.5B until a plateau-like behavior is reached. This slightly higher oxidation rate might be due to the larger sized Mo phases in the DS alloy in comparison to the AM alloy (Figure 2), as it is known from Rioult et al. [5] that the microstructure size scale affects the mass losses in the transient stage of oxidation.

In good correlation with the initial sample geometry, the present alloys maintained their geometry reasonably well (Figure 5b, c, d), due to the formation of the protective SiO$_2$-B$_2$O$_3$ scale. However, it is assumed, that these protective scales exhibit different “qualities” since in contrast to the AM alloys, further slight mass loss over longer exposure time is observed for alloy DS Mo-17.5B-8B.

Figure 5. Oxidation response at 1050 °C and 1100 °C: (a) mass change curves of AM and DS materials and macroscopic images of the AM Mo-16.5Si-7.5B alloy after (b) 100 h, (d) 500 h and (c) DS Mo-17.5Si-8B after 100 h.
This can be attributed to the different microstructures after AM and DS processing which have a significant influence on the formation of the protective layer. As illustrated in Figure 2, the ternary eutectic Mo₃-Mo₃Si-Mo₅SiB₂ microstructure in the AM samples is much finer than the DS material. Consequently, the diffusion paths for the reaction of O₂ with the constituents Mo₃, Mo₃Si or Mo₅SiB₂ are much smaller due to the fine microstructure, so a protective borosilicate layer can be formed very rapidly, as can be seen in Figure 6a and c. Hence, with scale thicknesses of ~10 µm after 100 h at 1100 °C and ~20 µm after 500 h at 1050 °C, thin but continuous layers formed.

Furthermore, the microstructure scale effect explains the higher specific mass loss of alloy DS Mo-17.5Si-8B relative to its AM processed counterparts. Even if the eutectic structures may have similar phase scales, the directionally grown microstructure is much coarser in the direction of crystal growth compared to the AM alloys. Hence, the boundaries between the different eutectic cells are much larger having coarser phases of Mo₃, Mo₅SiB₂, and Mo₃Si and thus, lead to a slightly increased oxidation rate in the initial stage of the reaction. Furthermore, this leads to varying scale thicknesses between ~20 µm and 50 µm in dependence on the individual phase located near the surface. As visualized in Figure 6b, the surface scale of the DS alloy exhibits a higher porosity compared to the ones of AM Mo-16.5Si-7.5B. Due to the evaporation of B₂O₃, MoO₃ may partially volatilize through small pores in the protective glass layer. Therefore, a small mass loss over longer exposure time is observed within the steady-state period for alloy DS Mo-17.5B-8B at 1100 °C.

![Figure 6. SEM micrographs of the oxidized alloys after exposure; (a) AM Mo-16.5Si-7.5B and (b) DS Mo-17.5Si-8B after 100 h at 1100 °C, respectively, and (c) AM Mo-16.5Si-7.5B after 500 h at 1050 °C.](image)

3.4 Oxidation performance at 1300 °C
The AM Mo-16.5Si-7.5B showed promising oxidation behavior at 1100 °C, so in the next step the material is exposed at 1300 °C since this temperature demonstrates a potential application temperature of Mo-Si-B alloys. The mass loss curve shown in Figure 7a represents an initial mass loss of ~10 mg/cm² which is even smaller than the initial mass loss at 1100 °C (~20 mg/cm²). The fluctuations in the initial stage of the curve might be explained by increased diffusion processes at 1300 °C, which enable a rapid surface coverage. The minimum thickness of the protective SiO₂-B₂O₃ scale was about 5 µm. With increasing exposure time, the mass loss reached a steady level. At the end of exposure, after 100 h, the sample geometry is still in good correlation with the initial sample geometry, as depicted in Figure 7b. In Figure 7c the typical surface layers are observed: a boron-lean interdiffusion zone (comparable to the oxidation mechanism at 1100 °C, Fig. 4), which is covered by a continuous borosilica scale.
Figure 7. Mass change curve of AM Mo-16.5Si-7.5B at 1300 °C (a) and macroscopic image of the AM Mo-16.5Si-7.5B alloy after 100 h of cyclic oxidation (b) as well as a cross section (c) showing the protective glass layer.

4 Summary and Conclusions
The cyclic oxidation tests on the eutectic alloys AM Mo-16.5Si-7.5B and DS Mo-17.5Si-8B demonstrated the influence of different temperature regimes and microstructures on the oxidation performance of Mo-Si-B alloys. To this end, the following conclusions can be drawn:

1. Despite of the similar composition of the AM Mo-16.5Si-7.5B and DS Mo-17.5Si-8B, the respective processing routes created different microstructures. The alloy AM Mo-16.5Si-7.5B showed a finer and more homogeneous microstructure compared with DS Mo-17.5Si-8B, which exhibits an anisotropic microstructure with larger MoSS regions.

2. At 800 °C, a “pesting” behavior was observed for the AM Mo-16.5Si-7.5B alloy as it is typical for Mo alloys. A pre-oxidation procedure at 1300 °C for 1 h before starting the exposure at 800 °C helped to enhance the oxidation resistance of the sample temporarily. After ~60 h exposure time, again oxidation failure accompanied with a higher mass losses was observed.

3. Due to the much finer microstructure and homogeneity, the AM Mo-16.5Si-7.5B alloy showed an improved oxidation performance compared with the DS Mo-17.5Si-8B alloy. This was attributed to the much finer microstructure which offers much smaller diffusion paths for the reaction of O₂ with either MoSS, Mo₃Si or Mo₅SiB₂. So, the protective SiO₂-B₂O₃ glass formed very rapidly, which resulted in a high stability of sample geometry and small mass losses (~20 mg/cm²) over exposure time. Even if the exposure time is extended to 500 h at 1050 °C, no further mass losses could be observed.

4. The kinetics for the formation of the protective borosilicate layer is temperature-dependent, i.e. the layer forms faster at 1300 °C, significantly reducing the evaporation at the initial experimental procedure. This finally lead to a very promising oxidation performance in case of AM Mo-16.5Si-7.5B providing a high stability after slight initial mass losses of ~10 mg/cm².

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