SCP 10.3:
Microstructure Coarsening of Additively Manufactured Mo-9Si-8B at Elevated Temperatures

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Introduction

Molybdenum-silicon-boron (Mo-Si-B) alloys are promising candidates for high-temperature applications due to their excellent oxidation resistance and mechanical stability. Among these, Mo-9Si-8B exhibits a favorable combination of properties for structural components operating under extreme conditions. However, the stability of its microstructure at elevated temperatures remains a crucial factor influencing its long-term performance.

This work investigates the microstructural evolution of Mo-Si-B alloys produced via Laser Powder Bed Fusion (L-PBF) and Electron Beam Melting (EBM), as well as their modification during heat treatment. The objective is to analyze grain growth kinetics and to provide a fundamental understanding of how thermal exposure affects the microstructure. Particular focus is placed on the influence of grain and phase size, as both have a critical impact on the creep properties of the material. The obtained insights will serve as a basis for further research on mechanical behavior and the optimization of heat treatment strategies.

Materials and Methods

Mo-Si-B specimens were fabricated using the Laser Powder Bed Fusion (L-PBF) in an Aconity3D machine with an inductive preheating system to achieve temperatures of up to 1200 ◦C in the substrate as well as the Electron Beam Melting (EBM) system (Freemelt® ONE, Freemelt AB, Gothenburg, Sweden). The additively manufactured material was analyzed in the as-built state to determine the microstructural constituents and parameters. The additively manufactured materials were then sectioned into 5×5×5 mm^3 cubes using Wire Electrical Discharge Machining (Wire EDM) to ensure precise and damage-minimized sample preparation. To investigate the effect of high-temperature exposure on microstructure evolution, a design of experiments (DOE) was conducted, varying heat treatment temperatures from 1200°C to 1600°C and dwell times up to 50 hours. This approach allowed the observation of both short-term and long-term coarsening effects. Following thermal exposure, the specimens were metallographically prepared using sequential grinding and polishing steps to obtain high-quality surfaces for microstructural analysis. Each sample was examined in two cross-sectional orientations - one perpendicular (Z-plane) and the other parallel to the build plane (XY-plane). Microstructural characterization was carried out using Scanning Electron Microscopy (SEM) in Backscattered Electron (BSE) mode to assess grain morphology and phase distribution. Electron Backscatter Diffraction (EBSD) was employed to measure grain sizes, phase fractions, and crystallographic orientations. Additionally, X-ray Diffraction (XRD) analysis was conducted to identify and quantify the present phases.

Results and Discussion

The microstructural analysis indicates a temperature-dependent coarsening behavior. Fine-microstructures in as-built L-PBF and EBM samples coarsen progressively with increasing heat treatment temperature and holding time, which is in good agreement with the literature [1]. Existing studies indicate that larger grain sizes reduce the contribution of grain boundary sliding to deformation [2], [3]. In conventionally processed Mo-Si-B alloys, increasing grain size by a factor of 5 to 10 has been shown to reduce creep rates by an order of magnitude [4]. *Figure* shows the microstructural difference between as-built and heat-treated condition of a L-PBF processed near-eutectic Mo-Si-B alloy. The microstructure of as-built L-PBF samples consists of fine molybdenum solid solution (Mo_{ss}) dendrites surrounded by a silicide matrix (*Figure*). According to EBSD analysis, the phase composition of as-built L-PBF samples consists of 29.7% Mo_{ss} phase, 22.2% Mo_5-SiB_2-phase, and 48.1% Mo_3-Si phase. After heat treatment at 1600 °C for 10 hours, the microstructure coarsens slightly while maintaining a similar phase composition [1].

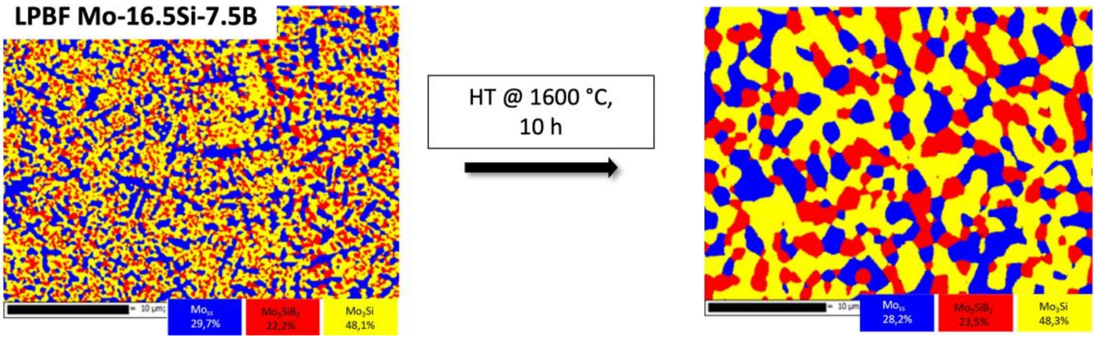

Figure 1 Microstructure of a Mo-Si-B sample manufactured with L-PBF; left: EBSD image of the "as-built" sample with Mo_{ss} (blue), Mo_3Si (yellow) and Mo_5SiB_2 (red) and right: EBSD image of a sample heat treated at 1600 °C for 10h – more details are given in **[1]**

Due to similar process characteristics (rapid cooling rates, layer wise process, etc.) between EBM and L-PBF, a comparable microstructural evolution is expected for EBM-processed samples. The observed microstructural changes provide essential groundwork for future studies on the relationship between grain size and high-temperature performance.

Fichtner et al. [1] evaluated creep properties of L-PBF Mo-Si-B alloys and compared them to those processed by other manufacturing routes. *Figure* illustrates the creep behavior of different Mo-Si-B alloys and a nickel-based superalloy demonstrating that additively manufactured (AM) samples exhibit superior creep resistance. This improved performance can be attributed to the distinct microstructure of L-PBF materials, particularly the higher fraction of intermetallic phases, which form a silicide matrix during solidification. The increased silicon and boron content in L-PBF samples promotes the formation of silicides, resulting in enhanced creep resistance compared to powder metallurgically (PM) processed alloys, which primarily contain a Mo_{ss} matrix. The annealing procedure will affect the creep response of AM samples significantly, as depicted by the arrows in *Figure 2*. Directional solidification (DS) processed Mo-17.5Si-8B alloys display even better creep performance than AM samples. This behavior is due to processing effects, which lead to the formation of elongated grains with specific orientations, as well as a coarser microstructure compared to the ultrafine-grained L-PBF material. Consequently, enhanced creep resistance can be attributed to both the specific phase composition and the morphological characteristics of grains and phases.

By leveraging the favorable phase constitution inherent to additively manufactured materials and applying targeted heat treatments to modify grain size and morphology, a further improvement in creep properties may be anticipated.

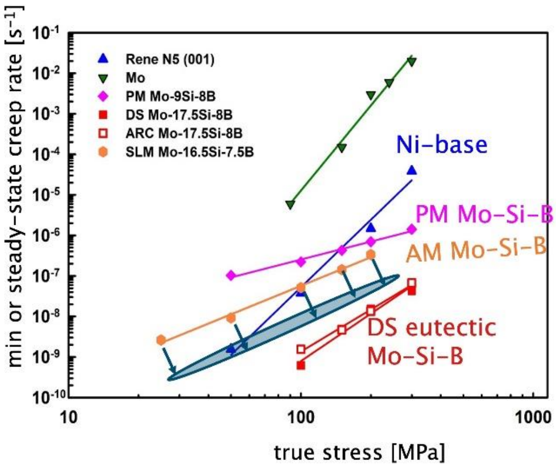

Figure 2 Creep properties of Mo–Si–B alloys manufactured by additive manufacturing (AM), powder metallurgy (PM) [5] and by directional solidification/zone melting technique (DS) [3] at 1093 °C.

In summary, the performed investigations demonstrate that the microstructure of additively manufactured Mo-Si-B alloys undergoes significant coarsening during heat treatment, with the extent of growth being strongly temperature-dependent. This study provides a foundation for further investigations into the relationship between grain size and mechanical properties, particularly creep resistance. These findings highlight the potential of microstructural engineering through optimized heat treatments to enhance the high-temperature performance of Mo-Si-B alloys, particularly in terms of creep resistance and phase stability.

References

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