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Improved fatigue properties of laser powder bed fusion of Al–4.74Mg–0.70Sc–0.32Zr alloy via hot isostatic pressing

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ABSTRACT

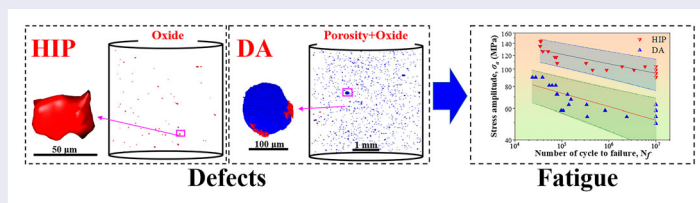
Laser powder bed fusion (L-PBF) processed components usually exhibit reduced fatigue performance owing to unavoidable defects at multiple scales, especially when the loading direction is parallel to the building direction. In this study, hot isostatic pressing (HIP) was applied to the L-PBFed Al–4.74Mg–0.70Sc–0.32Zr (wt.%) alloy to enhance the fatigue properties with a focus on the defect (porosities and inclusions) effects, compared with the direct-aged samples. The results demonstrated that HIP induced inconspicuous microstructural effects, but it healed the porosities and promoted oxidation. Therefore, the fatigue crack initiation transformed the oxide-associated porosity into an oxide, with a significant fatigue strength improvement.

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KEYWORDS

Al–Mg–Sc–Zr alloy; laser powder bed fusion; fatigue; hot isostatic pressing; 3D reconstruction



IMPACT STATEMENT

This study highlights the critical importance of oxides in fatigue performance.

Recently, microalloying Sc/Zr has been demonstrated as a type of modification for new-generation Al alloys fabricated by laser powder bed fusion (L-PBF) [1–3]. This is because the addition of Sc/Zr allowed the formation of primary $\text{Al}_3(\text{Sc}, \text{Zr})$ and secondary $\text{Al}_3(\text{Sc}, \text{Zr})$. The primary $\text{Al}_3(\text{Sc}, \text{Zr})$ formed during solidification can provide in-situ heterogeneous nucleation sites for the Al matrix. The secondary $\text{Al}_3(\text{Sc}, \text{Zr})$ diffusely precipitates during the ageing treatment, which can further strengthen the Al matrix [4–6]. Thus, the Al–Mg–Sc–Zr alloy takes advantage of its low sensitivity to hot cracking and its improved mechanical properties [7–9]. Numerous studies have been conducted on the tensile behavior, fracture, and toughness, which exhibit attractive strength-ductility-toughness

combinations for aerospace industrial applications [5,7,10,11].

However, the high cycle fatigue (HCF) properties of the L-PBFed Al–Mg–Sc–Zr alloy are still much lower than those of their wrought counterparts because of the presence of defects, especially when the loading direction is parallel to the building direction [12]. Various methods have been developed to improve the fatigue properties of L-PBFed components, including process parameter optimization, heat treatment, shot peening, and hot isostatic pressing (HIP) [13–17]. Among them, HIP can directly reduce the defect content of the entire part by simultaneously releasing the residual tensile stress [14,18–20]. In contrast, HIP leads to a change in the matrix/second phase and promotes oxidation

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[11,21,22]. Thus, here, we studied the fatigue properties, microstructure (α -Al grain and $\text{Al}_3(\text{Sc}, \text{Zr})$ precipitates), and defects (porosities and oxides) of the L-PBFed Al-4.74Mg-0.70Sc-0.32Zr (wt.%) alloy under HIP (325°C/4 h/100 MPa) conditions, compared with those of the direct-aged (DA, 325°C/4 h) conditions. The underlying mechanism is discussed based on the initiation of fatigue cracks associated with porosity/oxide defects.

The pre-alloyed powder (diameter: 15–53 μm) with a chemical composition of Al-4.74Mg-0.47Mn-0.70Sc-0.32Zr-0.10Fe-0.09Si (wt.%) being detected by inductively coupled plasma-atomic emission spectroscopy, was fabricated using a vacuum induction gas atomization (VIGA) process. The L-PBF preparations were conducted using a commercial EOS M280 machine with substrate preheating at 80°C in an Ar environment with an O concentration below 1000 ppm. All the samples were printed using a strip scan strategy with a scan direction rotation of 67° between two consecutive layers. Two cylindrical specimen types with a height of ~ 67 mm, a diameter of 11 mm, a height of 108 mm, and a diameter of 20 mm were fabricated to prepare tensile and fatigue specimens. The laser powder, hatch distance, scanning speed, and layer thickness were 370 W, 0.12 mm, 1000 mm/s, and 30 μm , respectively [23]. The hot isostatic pressing (HIP AIP-40H) treatment at 325°C for 4 h with 100 MPa pressure in a nitrogen atmosphere, and direct-aged (DA, Mxx1400-30) treatment at 325°C for 4 h was applied to the as-built specimens, respectively.

The metallographic specimens were sectioned parallel to the deposition direction and prepared by grinding and electropolishing (LectroPol-5) at a voltage of 23 V for 10 s using an electrolyte (10 vol.% perchloric acids in a 90-vol.% ethanol solution). The samples underwent microstructural observation using a scanning electron microscope (SEM, FEI Helios Nano-Lab) equipped with an electron backscatter diffraction (EBSD) system. The data was collected at a step size of 0.2 μm and analyzed using the CHANNEL5 software package. The precipitates were analyzed using a transmission electron microscope (TEM, FEI Themis). The specimens were polished and then subjected to twin-jet polishing.

The defects in the DA and HIP specimens were characterized by X-ray computed tomography (X-ray CT, Zeiss Xradia 520 versa) using a 120 kV voltage and 120 μA current. The data were recorded with a voxel size of 2.51 μm and analyzed using DRAGONFLY software.

Before the mechanical tests, the tensile and fatigue specimens were machined according to the schematic shown in Fig. S1. To minimize the surface roughness effects, these samples were polished to a high surface quality ($R_a < 0.4 \mu\text{m}$) using abrasive papers (2000 #)

mounted on the numerical control machine tool (K6530). The tensile properties were tested with three averaged results, using an Instron 3382 machine at a 1 mm/min crosshead displacement rate at room temperature (RT). The fatigue properties were measured at RT using an MTS Landmark 200-Hz machine. The sinusoidal waveform had a 0.1 stress ratio at 100 Hz. The samples were cycled until failure (failed) or stopped at 10^7 cycles (run-out).

Figure 1 shows the microstructure of the L-PBFed Al-Mg-Sc-Zr alloy under DA conditions along the building direction (the microstructure perpendicular to the building direction shown is in Fig. S2). A heterogeneous grain structure with an equiaxed grain (EG) band at the boundary and a columnar grain (CG) domain in the center was formed in the molten pool (Figure 1(a)). The orientation relationship between the Al matrix and $\text{Al}_3(\text{Sc}, \text{Zr})$ is as follows: $[011]\text{Al}||[011]\text{Al}_3(\text{Sc}, \text{Zr})$ and $(\bar{2}00)\text{Al}||(\bar{2}00)\text{Al}_3(\text{Sc}, \text{Zr})$ (Figure 1(b)). Thus, they are effective heterogeneous nucleation sites that induce the formation of fine EGs [24]. The high-temperature gradient without nucleation at the center of the melt pool caused elongated CGs [25]. After DA treatment, Sc and Zr that dissolved into the Al matrix precipitated diffusely as secondary $\text{Al}_3(\text{Sc}, \text{Zr})$, but their size was smaller than that of the primary $\text{Al}_3(\text{Sc}, \text{Zr})$ (Figure 1(c)) [26].

Figure 1(d) shows a similar microstructural feature after the HIP compared to the DA conditions. Note that some CGs underwent growth (Figure 1(d)), which sparsely occurred with only a few molten pools in one $10 \times 10 \text{ mm}^2$ plane (the detailed comparison shown in Fig. S3). It was due to grain boundary pinning by the precipitates in the L-PBFed Al-Mg-Sc-Zr alloy, such as some primary $\text{Al}_3(\text{Sc}, \text{Zr})$, Mn/Fe-rich, and Mg/Si-rich particles [5,7]. After HIP, the precipitation of secondary $\text{Al}_3(\text{Sc}, \text{Zr})$ was also evident inside the grains, and the size and distribution were not significantly different compared to that of DA (Figure 1(f)).

In general, L-PBFed Al alloys contain two types of defects: porosities (including gas pores and lack of fusion, LOF) and inclusions (mainly oxides in this study) [15,16,27,28]. In most previous studies, the effect of inclusions was neglected because they are difficult to quantitatively characterize [28–30]. In this study, we exploited the different densities among the inclusions (4.0 g/cm³ for Al_2O_3) [16], Al-matrix (2.67 g/cm³), and porosities (approximately 0 g/cm³) to distinguish them by X-ray CT technology and count them separately. As shown in Fig. S4, the density of oxide is the largest, so the contrast is the brightest; the density of porosity is the lowest, so the contrast is the darkest. The 3D views of defects can be obtained by selecting pixels with similar contrast on each slice and conducting 3D imaging.

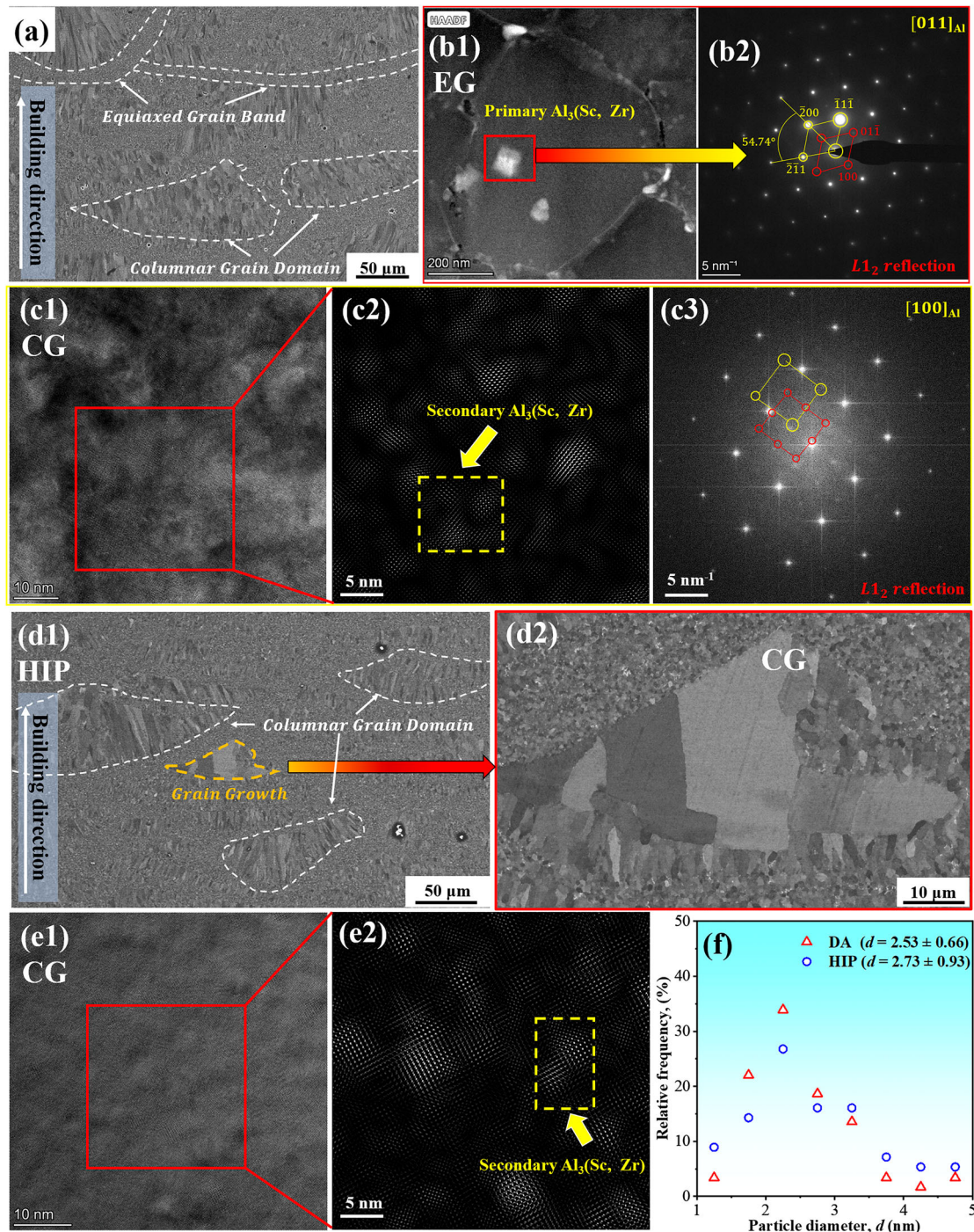


Figure 1. (a) BSE image showing the grain structure of the L-PBFed Al–Mg–Sc–Zr alloy under direct-aged conditions (DA). (b) TEM image showing an equiaxed grain (EG) with the primary $\text{Al}_3(\text{Sc, Zr})$. (c) High-resolution TEM (HRTEM) image (c1) and corresponding IFFT (c2), showing the secondary $\text{Al}_3(\text{Sc, Zr})$ in columnar grains (CGs). (d) BSE images showing the grain growth after hot isostatic pressing (HIP). (e) HRTEM image and corresponding IFFT showing the secondary $\text{Al}_3(\text{Sc, Zr})$ in CGs. (f) Particle distribution for the HIP and DA samples.

The 3D views of the defects are shown in Figure 2. In the DA specimens, porosities (shown in blue) were the main defects with a volume fraction of 0.44%, whereas the oxide content (shown in red) was low, accounting for 0.01%. The equivalent diameter distributions (d , defined as the spherical diameter with the same volume as the

defects) are shown in Figure 2(c). To avoid the effect of noise, defects smaller than $10\ \mu\text{m}$ were discarded. The porosities exhibited a larger range of sizes ($10\text{--}151\ \mu\text{m}$ for the range in d) compared to the oxides ($10\text{--}25\ \mu\text{m}$ for the range in d). Moreover, oxides were present and interconnected with the pores (Figure 2(a)). In the studies of

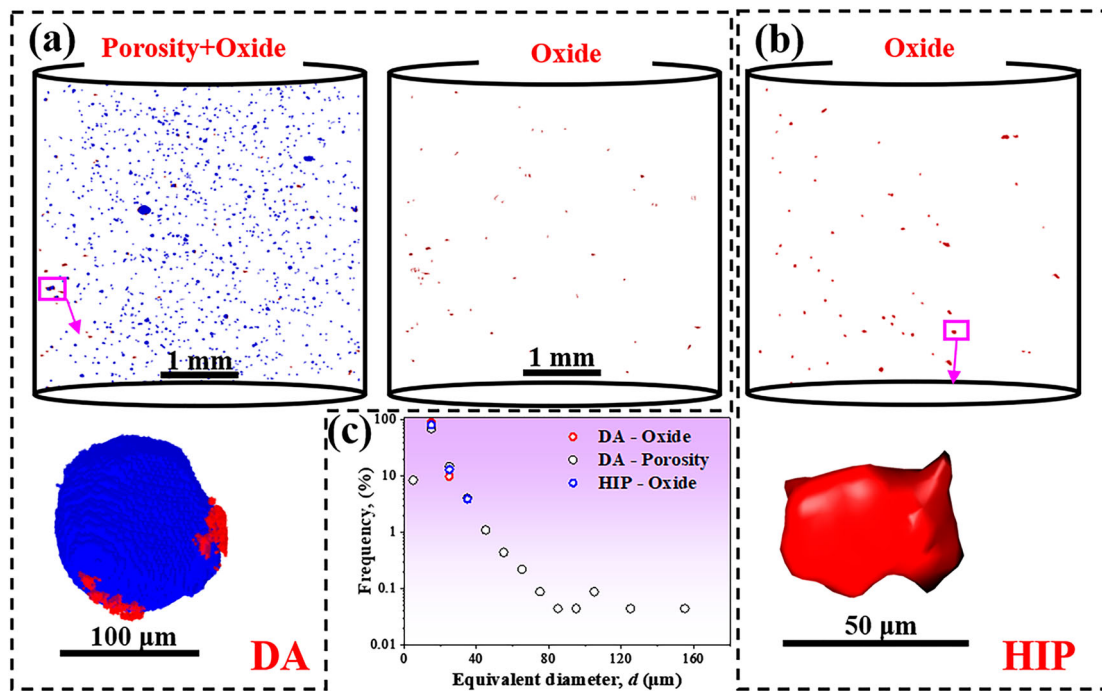


Figure 2. X-ray CT images of porosity/oxide defects under DA (a) and HIP (b). Blue represents porosities and red represents oxides. (c) Size distribution of the defects.

Tang et al. and Chu et al. [16,31], it was observed that the oxides, owing to the poor wettability with the aluminum melt, will lead to a void between the oxide surface and the melt, resulting in irregular pore formation [32]. These defects are usually larger than gas pores, which are similar to the LOF defects that are generated owing to insufficient energy input. In addition, their shape is irregular. The shape of the defects will affect the stress concentration factor. For porosities, the stress concentration factor of the spherical shape is the smallest. It increases with the irregularity of the shape. In the previous study [23], these irregular defects have a higher stress concentration factor than gas pores through numerical simulation, thus resulting in a greater negative effect on fatigue performance. In this work, to distinguish this type of defect, we call them oxide-associated porosities (Figure 2(a)) [33]. The oxides chiefly originate from the surface oxide layer of the powder or the residual oxygen in the Ar environment [16,34]. After HIP, the porosities were almost eliminated (smaller than the CT resolution of 2.5 μm). The oxide volume (accounting for 0.02%), as well as the number, slightly increased but remained below 40 μm.

The mechanical properties, including tensile and fatigue properties, are presented in Figure 3. The strengths (yield strength, YS; ultimate tensile strength, UTS) of the specimens in the DA and HIP conditions are similar, which is consistent with the microstructure approximation results in the two conditions mentioned

above. A slight improvement in the properties related to porosity (elongation to failure, ε_f ; Young's modulus, E) occurred, by 7.01% and 1.18%, respectively. This change is mainly due to the 0.44% porosity reduction. The fatigue strength of the HIP-treated samples calculated by the staircase method (Figure 3(c)) [35] is significantly improved compared to the DA-treated samples, from 57.0 to 99.8 MPa. The combination of UTS and fatigue strength (σ_f) of the L-PBFed Al-Mg-Sc-Zr alloy under the HIP and DA conditions are compared to those of 7075-T651 high-strength wrought Al alloy (the relevant fatigue data shown in Fig. S5) and L-PBFed AlSi10Mg alloys [36], as presented in Figure 3(d). The fatigue strength of the HIP-treated samples was close to the level of 7075-T651 high-strength wrought Al alloy.

For HCF, the fatigue crack initiation stage is very critical. Indeed, the fracgraphs of the specimens in two conditions exhibit different crack initiation features [30,37]. The defects at the crack initiation for all observed DA-treated specimens are porosities surrounded by oxides (oxide-associated porosity). This is unusual, as it indicates that such defects have a negative effect on the fatigue crack initiation over LOF and gas pores and are critical defects for controlling the fatigue performance. There are two possible reasons why these defects are more harmful: (i) they are generally large in size (oxides are present around the porosities with large volumes in Figure 2(a)). (ii) The weak interface between the oxides

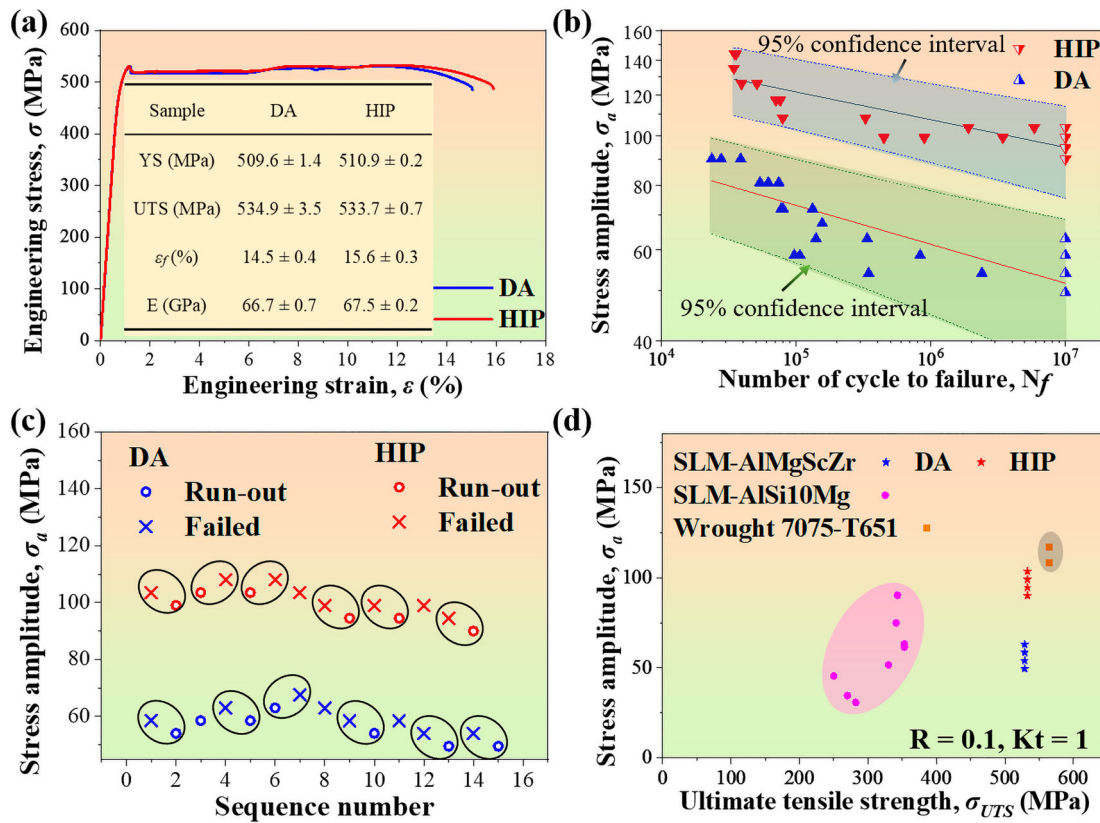


Figure 3. (a) Engineering stress-strain curves, (b) S-N curves of the L-PBFed Al–Mg–Sc–Zr alloy under DA and HIP conditions, (c) The staircase calculation process, the black circles indicating the six pairs for the calculation, (d) Plots comparing fatigue strength in terms of stress amplitude as a function of the ultimate tensile strength.

and the Al-matrix may be a favorable channel for fatigue crack propagation. Because a small oxide may also lead to the appearance of a large porosity (Figure 2(a)), the probability of crack propagation through the oxide from the porosity is not high enough. However, in almost all observed samples (except for No. 18, the detailed information shown in Table S2) at the DA condition in this study, the defects at crack initiation were oxide-associated porosity, implying a facilitative effect of oxide on fatigue crack initiation. Therefore, it is reasonable to assume that the oxide and porosity binding feature of oxide-associated porosity are the key reasons why such defects become crack initiation. In addition, as shown in Figure 4, enrichment of elemental Sc was also detected within the oxides. This may indicate that the Sc element promotes the oxidation of Al-matrix [38], which means the addition of the Sc element can damage the fatigue performance.

It is clear that controlling defects is a viable way to optimize the fatigue performance, but the complete elimination of defects is currently an uneconomic and difficult option. Therefore, it is important to reasonably predict the fatigue life of a component wherein the defect features are known. To quantify the effect of different defect types

on fatigue performance, the relationship between defect size and stress amplitude is plotted in Figure 5, where the curves are calculated by the following equation:

$$\Delta K_{th} = \sigma_a F_w \sqrt{\pi \sqrt{\text{area}}}, \quad (1)$$

where F_w is a geometric factor depending on the location of defects (0.5 for internal, 0.65 for surface), σ_a is the stress amplitude, and $\sqrt{\text{area}}$ is Murakami's parameter whose value approximates the equivalent radius of defects ($d/2$). ΔK_{th} is the long crack-growth threshold, which is unknown for this material. Because there was no significant change in the alloy microstructure under both conditions, the values were assumed to be the same. To evaluate the value of ΔK_{th} , two points were taken as the critical point: one failed sample (54 MPa, 92.3 μm) with the minimum value as shown in Supplements and one run-out sample (63 MPa, 80 μm) that defected by X-ray CT, such that $\Delta K_{th} = 0.649 \text{ MPa m}^{1/2}$ (the detailed information shown in Table S1).

The red and blue rectangles in Figure 5 represent the defect size distribution of the L-PBFed Al–Mg–Sc–Zr alloy under the two conditions detected by the X-ray CT technique, which are located exactly near the critical

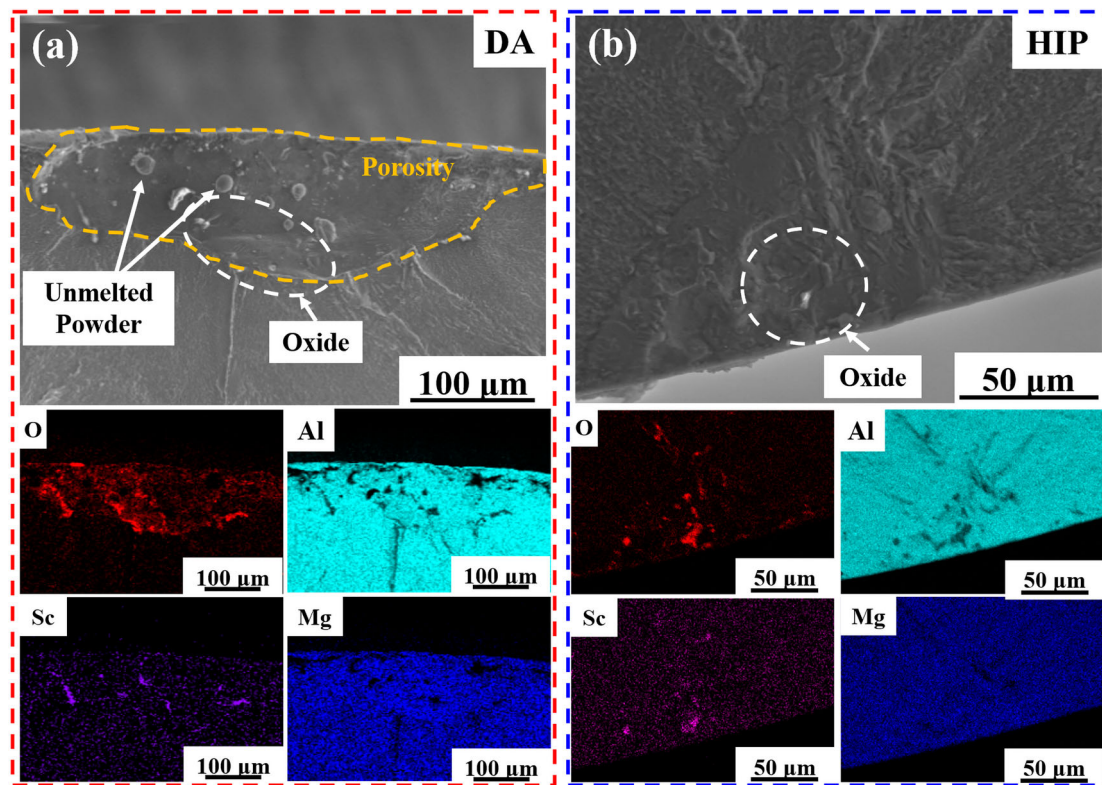


Figure 4. The fatigue crack initiation site with oxide (marked by the white dash line)/porosities (marked by the yellow dash line) and corresponding elemental mapping for O, Al, Sc, and Mg, (a) DA, (b) HIP.

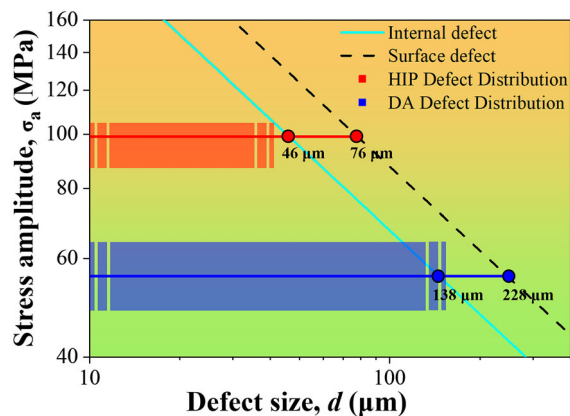


Figure 5. Kitagawa-Takahashi diagram of L-PBFed Al-Mg-Sc-Zr alloy in DA and HIP conditions showing the variation between equivalent (d) and stress amplitude (σ_a).

surface defect size corresponding to the fatigue strength under the two conditions. The increased fatigue strength of the HIP-treated specimens can be attributed primarily to the decrease in defect size.

In summary, the present work offered a feasible post-heat treatment (HIP) to improve the fatigue strength of the L-PBFed Al-Mg-Sc-Zr alloy to 99.8 MPa, which was compared to the wrought 7075-T651 alloy. The HIP treatment can heal the porosities without a significant change

in microstructure. However, it promoted oxidation (Al and Sc elements). Oxides damage the fatigue performance because they promote the formation of porosities and become the critical defects at the initiation of fatigue cracks.

Disclosure statement

No potential conflict of interest was reported by the author(s).

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