主　論　文　の　要　約

論文題目　**Additive manufacturing of near-eutectic Al–Fe binary alloy by laser powder bed fusion: Processing parameters, post-heat treatments, microstructure, and properties**
(レーザ粉末床溶融結合法による共晶組成近傍の Al-Fe 二元系合金の積層造形：プロセス条件、熱処理、組織および性質)

氏　　名　**QI Xing**

論　文　内　容　の　要　約

Laser powder bed fusion (L-PBF), one of the common additive manufacturing processes, has been widely utilized in the manufacture of various types of metallic materials with improved microstructure and properties. To develop the microstructure and critical properties of Al–2.5Fe (wt%) binary alloys potentially applied in high-performance heat sinks, the feasibility of introducing the L-PBF technique into the manufacture of the Al–2.5Fe alloys was validated in the present dissertation.

In Chapter 1, the conception and motivation of this study were systematically introduced by the review on the related social and academic backgrounds. The consistent pursuit of high-dissipation heat sinks requires the development of high-performance aluminum (Al) alloys, which are widely applied in various types of heat sinks. However, the poor balance of strength-thermal conductivity in the conventional Al alloy series is a key issue to resolve. Al–Fe alloys with relatively high specific strength and thermal conductivity were competitive candidate materials for high-performance heat sinks, though they are suffered from the formation of the coarsened Al–Fe intermetallic phase, especially the θ-$Al_{13}Fe_4$ phase that is brittle at ambient temperature and thus deteriorates their deformability during plastic working processes. Therefore, various processing routes, including the

severe plastic deformation and rapid solidification processes, were proposed to develop the microstructures of Al–Fe alloys, particularly in improving the size, distribution, and morphology of the Al–Fe intermetallic phases. Nonetheless, several disadvantages of these techniques restrict their practical application in the manufacture of heat sinks. L-PBF technique, featuring an extremely high cooling rate during solidification and high geometric flexibility, offers the possibility to manufacture complicated-shaped parts with effectively improved microstructure to be potentially applied to high-performance heat sinks. The prior targets of this study are to establishing the optimum processing windows for L-PBF processing of full dense Al–2.5Fe alloy parts, and then providing the fundamental data on the variabilities in the microstructural characteristics and associated properties as a function of laser conditions and post-heat treatments, clarifying the microstructure-properties correlation to contribute for further management of desirable mechanical and thermal properties.

In Chapter 2, the investigation on optimizing the L-PBF processing of Al–2.5Fe alloy parts was presented. The influence on part qualities of laser parameter sets of laser power (*P*) and scanning speed (*v*) was mainly examined, and the influence of powder characteristics was preliminarily investigated as well. The feasibility of applying L-PBF into manufacturing Al–2.5Fe alloys was confirmed, and the laser parameter sets of P = 204 W, $v \leq 800$ mm·s^{-1} were optimized to manufacture dense samples with relative densities > 99% through the systematical examinations. The correlation of relative densities and $P \cdot v^{-1/2}$ derived from the deposited energy density model (ΔH), which was described as a single curve, determined a critical value of the *P* and *v* set for manufacturing dense samples. Therefore, it is revealed that $P \cdot v^{-1/2}$ derived from ΔH, instead of the pervasively introduced volumetric energy density model (E), was proved to be a highly valid design parameter for L-PBF processing of Al–2.5Fe alloy samples. Under the same processing parameters, lack of fusion defect was occasionally observed in the near full dense sample manufactured using the irregularly shaped P1 powder with a large PSD, whereas the highly spherical-shaped P2 powder with a smaller particle size distribution (PSD) significantly promoted the elimination of lack of fusion defect within the samples, ensuring the manufacture of large-sized bulk samples of high quality for subsequent investigations.

In Chapter 3, the typical microstructural and crystallographic features of the L-PBF processed Al–2.5Fe alloy were characterized, and their corresponding changes induced by varying laser parameter sets of laser power (*P*) and scanning speed (*v*) were also systematically investigated. In the L-PBF processed Al–2.5Fe alloy samples, multiple half-cylindrical melt pools induced by laser irradiation were characterized for the macroscopic structure. Moreover, an inhomogeneous microstructure across the MPBs was typically characterized as well, in which the submicron-sized cellular structure was located along the MPBs, and the gradual refinement of Al–Fe intermetallic phase inwards the melt pool was identified with the presence of numerous nanosized particles of metastable Al_6Fe phase in the matrix. The supersaturated solid solution of Fe atoms in concurrent with random crystallographic orientation was found in the α-Al matrix, which was composed of many elongated and twisted columnar grains with an average size of ~20 μm along the building direction. The variation in Fe concentration within the matrix was also found in the studied samples, a far higher concentration of solute Fe atoms in the matrix was detected inside the melt pool. The development of Al–Fe intermetallic phases was revealed in the samples manufactured with the higher $P\cdot v^{-1/2}$, the promoted formation of stable θ phase might occur at the MPBs. The average size of the columnar α-Al grains gradually increased with the increasing laser energy density. Meanwhile, the progression of {001} texture formation was identified in the studied samples with the higher $P\cdot v^{-1/2}$ applied in L-PBF, and the clear prevalence of <001>-oriented grains was observed in the A4 sample manufactured with the highest energy density. The higher $P\cdot v^{-1/2}$ in L-PBF also led to a relatively lower concentration of solute Fe atoms in the matrix inside the melt pool. A fundamental understanding of laser parameter-microstructure correlation was established on the present results of changes in microstructural and crystallographic features induced by the varying laser parameters of $P\cdot v^{-1/2}$, enabling the feasibility of tailoring the desirable microstructure via altering the laser parameters.

In Chapter 4, the changes in microstructural features concerning the Al–Fe intermetallic phases and α-Al matrix during thermal annealing processes at elevated temperatures of 300 °C and 500 °C for different durations in the L-PBF processed A1* sample (P = 204 W, v = 600 mm·s^{-1}). Upon annealing at 300 °C, the sluggish microstructural changes were characterized. The macroscopic laser-induced melt pool morphologies were still observed in A1* samples after 1000 h. The formation of stable θ phases was initially found within the submicron-sized cellular structure along MPBs and at the grain boundaries after 10 h, whereas only slight growth of nanosized Al_6Fe phase by interconnecting was observed inside the melt pool. The small-sized needle- and plate-shaped θ phases were initially identified concurrently with the fine Al_6Fe phase within the melt pool after 100 h. Though further coarsening of these θ phases was characterized, a considerable number of nano-sized Al_6Fe phases was presented in the microstructure as well even after 1000 h. Upon annealing at 500 °C, dramatic changes in microstructural features were observed. The laser-induced melt pool morphologies were eventually eliminated after 10 h, and a comparatively homogenous but significantly coarsened microstructure was introduced into A1* sample after 100 h. The pronounced transformation at 500 °C led to the growth of stable θ phases into the size approaching micrometer order in the α-Al matrix and the much reduced the number density of Al–Fe intermetallic phases. The supersaturated solid solution of Fe atoms in the α-Al matrix was gradually reduced upon annealing at elevated temperatures. The strong pinning of finely distributed particles of Al–Fe intermetallic phases on grain-boundary motion was observed in the annealed samples, thus contributing to the slight grain growth of α-Al matrix structure during the annealing processes.

In Chapter 5, the fundamental understanding of the critical properties (tensile properties and thermal conductivities) of the L-PBF processed Al–2.5Fe binary alloys was obtained, and their corresponding changes induced by varying laser parameter sets of *P* and *v* were systematically evaluated, and the changes in these two critical properties of the standard A1* sample upon different annealing processes were examined as well. A high tensile strength of ~320 MPa was achieved in the A1* specimens of additively manufactured Al–2.5Fe alloy via L-PBF. The solid solution strengthening of supersaturated solute Fe atoms in the matrix and the dispersion strengthening (Orowan strengthening) by the numerous nanosized Al_6Fe phases within the melt pool predominantly contributed to the highly improved high strength level of the L-PBF processed specimens. The anisotropy in tensile ductility was identified in all studied specimens manufactured with varying laser energy density ($P \cdot v^{-1/2}$), the lower tensile elongation along the *Z* direction was obtained, which is mainly attributed to the local difference in hardness across the MPBs in the microstructure. A tradeoff between the tensile strength and ductility was also found in the studied specimens, in which the increased $P \cdot v^{-1/2}$ resulted in a reduced strength but an enhanced ductility. The changes in the tensile properties were closely associated with the variations in Fe concentration in the matrix induced by the varying laser energy density. A sustained high strength level of ~280 MPa was found in the A1* specimens annealed at 300 °C for a long duration of 1000 h, which was mainly contributed from the remained nanosized Al_6Fe phases and relatively fine θ phases within the stable α-Al matrix structure. However, the tensile strength was dramatically reduced upon annealing at 500 °C for 10 h due to the pronounced metastable-stable phase transformation accompanied with the greatly decreased number density of Al–Fe intermetallic phase in the microstructure. As a result of the decreased concentration of solute Fe atoms in the matrix, the thermal conductivities of the A1* samples were continuously improved upon the annealing at elevated temperatures for prolonged durations. Meanwhile, thermal conductivities of the A1* samples (~150 $W\ m^{-1}\ K^{-1}$) increased to ~190 $W \cdot m^{-1} \cdot K^{-1}$ with limited sacrificing strength after annealing at 300 °C for 100 h, enabling them the considerably improved strength-thermal conductivity balance for their potential application in heat sinks. Therefore, these results imply that the proper post-heat treatments could be effective in tailoring the tensile strength-thermal conductivity balance for improved performance by taking advantage of the optimized microstructural parameters. Moreover, it is prospective to achieve the desirable thermal and mechanical properties (high strength and adequate ductility) by concurrently optimizing the laser parameters and additional heat treatments.

In Chapter 6, the main findings of this study were summarized. The feasibility of applying L-PBF into the manufacture of full dense Al–2.5Fe binary alloy sample was validated by optimizing the laser parameters (laser power (*P*) and scanning speed (*v*)) based on the design parameter of $P \cdot v^{-1/2}$ derived from the deposited energy density (ΔH). A significantly refined microstructure with numerous nanosized Al_6Fe phases distributed was identified in the L-PBF processed Al–2.5Fe binary alloy, proving the capability of L-PBF in improving the microstructure of this alloy. The effective role of altering laser energy density in controlling the microstructural parameters was confirmed as well, especially the concentration of solute Fe atoms in the matrix. A sluggish microstructural evolution upon annealing at 300 °C was characterized, indicating high microstructural stability at this potential service temperature of heat sinks. It was also revealed that additional heat treatments could efficiently modify the microstructure with proper designs. A high tensile strength was prominently featured in the L-PBF processed Al–2.5Fe alloy specimens and a significantly improved tensile strength-thermal conductivity balance was also obtained by the proper annealing treatments, demonstrating the validity of the L-PBF technique in combination with the post-heat treatments in developing the desirable performance of the studied alloys. The prospective concerning the further development of the microstructure and performance of L-PBF processed Al–2.5Fe alloys was also proposed.