

報告番号	甲 第 13797 号
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主 論 文 の 要 旨

論文題目 **Microstructures and mechanical properties of Al–Si alloys additive-manufactured by laser powder bed fusion**
(レーザ粉末床溶融により積層造形された Al–Si 合金の微視組織と機械的性質)

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論 文 内 容 の 要 旨

Laser powder bed fusion (L-PBF) process is a burgeoning additive manufacturing (AM) technology, which utilizes the energy source of a laser beam to melt metal powders to print metal parts with desirable geometries. In the present study, the microstructures and mechanical properties of the Al–Si alloys printed by L-PBF process were systematically and thoroughly investigated. The present thesis was divided into 7 chapters and abstracted as follows.

In Chapter 1, the motivation of this research was introduced. The general backgrounds of AM technology, L-PBF process and Aluminium (Al) alloys and lattice structure (as a new catalog of cellular structure) were briefly introduced. The previous studies of L-PBF printed Al–Si alloys and lattice structures were reviewed in detail. The studies related to the Al–Si alloys and lattice-structured Al–Si alloys printed by L-PBF process are extensive and multitudinous, whereas there are still some gaps in scientific knowledge and technical issues for further understanding the L-PBF printed Al–Si alloys and using in practical application. To fill these gaps in the L-PBF printed Al–Si alloys, the main research objectives were defined.

In Chapter 2, the microstructure of the L-PBF printed AlSi12 alloy (one of the most widely used Al–Si alloys for L-PBF process) and its change after annealing treatments at elevated temperatures were systematically investigated. The typical microstructure of melt pools (locally

melted and solidified region) was observed in L-PBF printed AlSi12 alloy. The cellular microstructure consisting of α -Al decorated with eutectic Si phases can be observed within the melt pools. After annealing at temperatures ranging from 200 °C to 300 °C, the Si phases became larger and many fine particles dispersed in the α -Al phases, the cellular microstructure can be observed as well. After the 530 °C annealing, the Si particles became further large and the needle-like intermetallic phases of β -AlFeSi were observed. The supersaturated solid solution was confirmed in L-PBF printed AlSi12 alloy, whereas the contents of the solute Si were varied depending on the locations inside the melt pool and near the melt pool boundary. The content of the solute Si in the α -Al phase decreased significantly after annealing at high temperatures. The results provide a fundamental understanding of the microstructures in the AlSi12 alloy printed by L-PBF process.

In Chapter 3, the change in the tensile behavior after annealing was firstly investigated to fundamentally understand the mechanical properties of the L-PBF printed Al–Si alloys. The as-printed specimen exhibited a high strength (over 400 MPa) with low elongation (below 10 %). The remarkable anisotropic tensile properties were found depending on the tensile direction in the as-printed specimens. The strength decreased and elongation increased continuously with increasing the annealing temperature, indicating a trade-off relationship between strength and ductility. The anisotropic tensile properties can be weakened or even annihilated by applying annealing, which is due to the microstructural development during the annealing at high temperatures.

To further understand the deformation behavior, the effect of the strain rate on the tensile properties was also addressed in this chapter. An anomalous behavior of the negative strain rate sensitivity (m) was found in the as-printed specimens. The negative m value could be due to the dynamic precipitation which occurred resulting from the supersaturated solid solution with extremely high concentrated Si element (above 3 %) of the L-PBF printed Al–Si alloys during the deformation.

Then the in-situ XRD measurements during the tensile test were carried out on the L-PBF printed and annealed AlSi12 alloy specimens. The results were utilized to discuss the strengthening and deformation mechanism of the L-PBF printed Al–Si alloys. The as-printed specimen exhibited a relatively high dislocation density, which could enhance the strain hardening, resulting in the superior strength of the L-PBF printed Al–Si alloys. The high dislocation density could be contributed due to the cellular Si phases, supersaturated solid solution of α -Al matrix and dynamic precipitation behavior during the deformation. Based on the aforementioned results and discussion, it can be concluded that the superior strength of the L-PBF printed Al–Si alloys could be due to the comprehensive effect of the cellular morphology

of Si phases and supersaturated solid solution of α -Al matrix (associated to dynamic precipitation) during the deformation.

Combined with the aforementioned three main parts of Chapter 3, the results provided a deep and thorough understanding of the deformation behavior of the L-PBF printed Al–Si alloys.

In Chapter 4, an approach to introduce nano-sized precipitates by applying direct aging treatment was proposed to achieve the simultaneous enhancement in strength and ductility of the L-PBF printed Al–Si alloys. The nano-sized Si precipitates with sizes above 10 nm could play a critical role in the obstacles to dislocation motion and contribute to the enhanced strain-hardening, resulting in a simultaneous enhancement in the tensile strength and ductility.

In Chapter 5, to fundamentally understand the rapid solidification microstructure of L-PBF printed Al–Si alloys, the AlSi10Mg alloy powder (the most commonly used Al alloys for L-PBF process) was used to investigate the solidification reactions at various cooling rates controlled by using fast scanning calorimetry (FSC) technique. The microstructural characterization of the FSC measured AlSi10Mg alloy samples was performed. The conventionally casted alloy ingots were prepared as reference samples. Two exothermic peaks, which were responsible for the primary solidification of α -Al phases and the α -Al/Si eutectic reaction, were found in all samples solidified at different cooling rates controlled by FSC measurement. The two exothermic peaks appeared broader and shifted to lower temperatures at higher cooling rates. The samples solidified at low cooling rates (below $1\text{ }^{\circ}\text{C}\cdot\text{s}^{-1}$) showed the microstructure comprising of coarsened α -Al dendritic phases, eutectic Si phases, β -AlFeSi phases, π -Al₃Mg₃FeSi₆ and Mg₂Si phases, which corresponds well to the conventional cast Al–Si alloys. The same intermetallic phases (β phase and π phase) were observed in the sample solidified at a higher cooling rate of approximately $10^2\text{ }^{\circ}\text{C}\cdot\text{s}^{-1}$, whereas the α -Al dendritic phases and eutectic Si phases were much finer. In the sample solidified at a high cooling rate of $4\times 10^4\text{ }^{\circ}\text{C}\cdot\text{s}^{-1}$, fine microstructure consisting of primary α -Al phase decorated with lamellar-shaped Si phases was observed, and no intermetallic phase was found. The presence of solute Si in the primary α -Al phases has been confirmed at high cooling rates (above $10^2\text{ }^{\circ}\text{C}\cdot\text{s}^{-1}$). This result indicated the higher concentration of solute Si in α -Al phase solidified at higher cooling rate. The dendrite arm spacing of the primary α -Al phase became smaller in the samples solidified at higher cooling rate. Based on the comparison of the dendrite arm spacing of primary α -Al phase with the width of columnar α -Al phases in the L-PBF printed sample, the local cooling rates within the melt pool and near the melt pool boundary during the L-PBF process were estimated to be $2.2\times 10^5\text{ }^{\circ}\text{C}\cdot\text{s}^{-1}$ and $4.7\times 10^4\text{ }^{\circ}\text{C}\cdot\text{s}^{-1}$, respectively.

In Chapter 6, the microstructure and its change with annealing at different temperatures in the lattice-structured AlSi10Mg alloy were systematically investigated. The gradient microstructure (continuous change in microstructure) was observed in the node and strut parts of the as-printed lattice structure. The formation of gradient microstructure could be due to the local variation in the cooling rates caused by the unmelted alloy powder particles near the surface of printed parts. The gradient microstructure could lead to the continuous change in the micro-hardness in the node and strut parts of the lattice structure. After the 300 °C annealing, the Si phases were larger and many fine Si particles were precipitated inside the α -Al phase, whereas the gradient microstructure can be observed as well. After the 530 °C annealing, the homogenous microstructure comprising of coarsened Si particles and β -AlFeSi intermetallic phases were observed. The formation of the homogeneous microstructure could also lead to a constant hardness in the lattice structure.

The thesis was summarized in Chapter 7. The present study provided a deeper and more thorough understanding for the Al–Si alloys printed by L-PBF process. The understanding and knowledge obtained from the present study can be used to not only the other Al-based alloys but also other fcc alloys (e.g. Cu alloys). For example, the correlation between the microstructures and mechanical properties discussed in the present study can be effectively used for the development of novel high performance Al-based alloys. Furthermore, it can be expected that the discussion based on the novel findings and results obtained in the present study can accelerate the development in not only L-PBF printed Al–Si alloys but also other Al-based alloys for using in practical applications in automotive and aerospace fields.