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Change in microstructure of selectively laser melted AlSi10Mg alloy with heat treatments

Naoki Takata * , Hirohisa Kodaira 1 1 , Keito Sekizawa 1 , Asuka Suzuki, Makoto Kobashi

Department of Materials Process Engineering, Graduate School of Engineering, Nagoya University, Furo-cho, Chikusa-ku, Nagoya 464-8603, Japan

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ABSTRACT

In the present study, we examined changes in the microstructure and mechanical properties of AlSi10Mg alloy, initially fabricated using selective laser melting (SLM) combined with a powder-bed system, by applying heat treatments at temperatures of either 300 or 530 °C. The as-fabricated samples exhibited a characteristic microstructural morphology and {001} texture. Melt pools corresponding to the locally melted and rapidly solidified regions were found to be composed of several columnar α-Al grains surrounded by fine eutectic Si particles. A fine dislocation substructure consisting of low-angle boundaries is present within the columnar α-Al grains. At elevated temperatures, fine Si phase precipitates within the columnar α-Al phase and coarsening of the eutectic Si particles occurs. These fine Si particles inhibit grain growth in the α-Al matrix, resulting in the microstructural morphology and [001] texture observed in the heat-treated samples. The dislocation substructure disappears in the columnar α-Al grains. Furthermore, the formation of a stable intermetallic phase occurs, reaching microstructural equilibrium after long-term exposure. The as-fabricated specimen exhibits a high tensile strength of approximately 480 MPa. The strength is independent of the tensile direction, that is, normal and parallel to the building direction. In contrast, the tensile ductility is found to be direction-dependent, and is therefore responsible for a fracture preferentially occurring at a melt pool boundary. The directiondependence of the tensile ductility was not found in the specimen that had been heat-treated at 530 °C. The present results provide new insights into the control of the direction-dependence of the tensile properties of AlSi10Mg alloys fabricated by SLM.

1. Introduction

Porous materials can present unique physical properties like a low apparent density, a high impact-energy absorption, low thermal conductivity, gas permeability, and a high specific stiffness [1–[5\].](#page--1-0) This endows these materials with a wide range of potential applications in the areas of thermal insulation, shock damping/absorption, acoustic absorption, catalyst support, and biomedical implants. Porous aluminum (Al) alloys have been intensively studied $[6,7]$ in terms of their compressive absorption properties and simple production process. These porous Al alloys have attracted considerable attention due to their deformability and low density, making them promising candidates for application to the crumple zones of automobiles [\[3\].](#page--1-2) To improve the energy absorption capability of such alloys, it is necessary to control the factors affecting the porous structure, such as the porosity, pore size, pore shape, and pore distribution.

One potential processing route for fabricating open-cell porous materials with a controlled porous structure is powder bed fusion (PBF) additive manufacturing [\[8\].](#page--1-3) Powder bed fusion processes use either laser or electron beams to melt and fuse powdered metals and/or alloys. These processes include the commonly used selective laser melting (SLM), selective laser sintering (SLS), direct metal laser sintering (DMLS), electron beam melting (EBM), and selective heat sintering (SHS) [\[9,10\].](#page--1-4) New technological developments have recently been applied to the SLM process [\[9,10\]](#page--1-4) to enable the fabrication of cellular lattice structures using a range of metals and alloys [\[11\]](#page--1-5). A porous Al alloy with a cellular lattice structure fabricated by SLM exhibits an unstable compressive stress with a series of peaks and troughs [\[12,13\]](#page--1-6). These compressive properties cannot satisfy the requirements of the structural parts of crumple zones, since this application requires a stable and highly reliable compressive strength (plateau-stress) to achieve high energy absorption [\[7\].](#page--1-7) Preliminary experiments revealed that subsequent heat treatments influence the deformation behavior of porous Al alloys fabricated by SLM, leading to a more stable deformation under compression. These results give rise to the possibility of fabricating a porous Al alloy with superior impact energy absorption

⁎ Corresponding author.

 $^{\rm 1}$ graduate student.

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E-mail address: takata.naoki@material.nagoya-u.ac.jp (N. Takata).

through the application of the SLM process together with additional heat treatments. To optimize the compressive properties by the application of heat treatment, it is essential to understand the development of the microstructures of Al alloys fabricated by SLM during the heat treatment, together with the associated mechanical properties. However, most previous studies have focused on the effect of T6 heat treatments (solution treatment and subsequent aging) on the mechanical properties of Al alloys fabricated by SLM [\[14](#page--1-8)–16] since AlSi10Mg alloy (the most commonly used alloy for additive manufacturing [\[17\]\)](#page--1-9) is recognized as being an age-hardenable alloy. Therefore, the microstructural changes in SLM-fabricated AlSi10Mg alloys caused by heat treatment at various temperatures are not yet fully understood.

In the present study, to better understand the microstructure development of SLM-fabricated AlSi10Mg alloy during heat treatment at elevated temperatures, the microstructure and texture of SLM-fabricated AlSi10Mg alloy, heat-treated at different temperatures, was examined and compared with the constitute phases determined by thermodynamic equilibrium calculations. The mechanical properties of the heat-treated SLM AlSi10Mg specimens were examined by conducting tensile tests. The results were then utilized to discuss two issues: (1) the microstructure development process of the SLM AlSi10Mg alloy during heat treatment at elevated temperatures, (2) the effects of the microstructural characteristics on the tensile properties of the SLM AlSi10Mg alloy heat-treated at elevated temperatures.

2. Experimental procedure

In the present study, AlSi10Mg alloy powder [\[17\]](#page--1-9) with a particle size ranging from 1 µm to 34 µm was used. SEM images of the powder are shown in [Fig. 1\(](#page-1-0)a). Details of the selective laser melting (SLM) process used to produce the AlSi10Mg alloy samples are described in the literature $[9,10,18]$. The nominal compositions of the alloy powder and measured compositions (as analyzed by inductively coupled plasma-atomic emission spectrometry (ICP-AES)) are listed in [Table 1](#page--1-10). Note that the proportions of the major alloy elements (Si, Mg, and Fe) in the initial powder are almost the same as those in the bulk sample fabricated using SLM. The SLM processing for fabricating the cube samples (45 mm in length) was carried out at room temperature using an EOSINT M 280 additive-manufacturing system, equipped with an Yb laser operating at 380 W (EOS GmbH, Germany). The parameters applied to the fabrication of the samples were as follows; the thickness of the bedded-powder layers was 30 µm, the hatch spacing between the adjacent laser-scanning tracks was approximately 100 µm, and the angle of rotation between the bedded-powder layers was 67°. The laserscanning track applied in this study is shown schematically in [Fig. 1](#page-1-0)(b). Hereafter, the directions normal and parallel to the bedded-powder

layer are designated the Z direction and X/Y direction, respectively. High-purity Ar gas was used in the SLM processing to prevent oxidation of the fabricated sample. The as-fabricated bulk sample was held at 300 °C for 2 h (annealing) or at 530 °C for 6 h (solution-treatment), followed by quenching in water. To enable a comparison with the SLM sample, the studied powder was melted by high-frequency induction and then solidified to prepare an as-cast ingot of the studied AlSi10Mg alloy. Note that the cooling rate in solidification (experimentally measured by K-type thermocouples) is approximately 0.3 °C/s.

Various methods were used to prepare the samples used for the observations. The bulk samples to be observed by optical microscopy were both mechanically and electro-polished with a solution of perchloric acid and ethyl alcohol, at a volume ratio of 1:9 at room temperature. Cross-sectional samples (observed from the X/Y direction) were ion-polished by a cross-section polisher at 5 V. The microstructures were observed by using a scanning electron microscope (SEM) operating at 30 kV. An orientation analysis was carried out by electron backscatter diffraction (EBSD) using step sizes of 0.1 µm and 2 µm. The hardness (HV) of these samples was measured using a Vickers indenter at a constant load of 9.8 N at room temperature. Tensile tests using plate specimens with a gauge length of 14 mm and a thickness of 2 mm were carried out at a strain rate of 1.2 \times 10⁻³/s (corresponding to a cross-head speed of 1 mm/min) at room temperature. In the present study, tensile test specimens along the X/Y direction were cut out from the center part of the fabricated cube samples. The gage portion of the prepared tensile test specimens along the Z direction was located around the center of the fabricated cube samples. SEM was used to observe the fracture surfaces of the tested specimens.

3. Thermodynamic assessment of AlSi10Mg alloy

A thermodynamic equilibrium calculation for the Al–Si–Mg–Fe quaternary system was carried out using a CALPHAD approach [19–[21\]](#page--1-11), based on a thermodynamic database for an Al-based multicomponent system (PanAl) [\[22\]](#page--1-12) for the measured alloy composition of Al–10.8Si–0.2Mg–0.4Fe (wt%). [Fig. 2](#page--1-13) presents (a) the composition of the studied AlSi10Mg alloy on a vertical section of the Al–x-Si–0.2Mg–0.4Fe (wt%) of the Al–Si–Mg–Fe quaternary system and (b) the calculated mol fractions of the constituent phases in equilibrium at various temperatures. The calculated phase diagram ([Fig. 2\(](#page--1-13)a)) indicates the possible solidification path. The liquidus is located at approximately 600 °C and the initial solid phase is α -Al (fcc) for the studied alloy composition. A three-phase region of α Al + Si(diamond) + β-AlFeSi (τ_6 -Al₉Fe₂Si₂) [\[23,24\]](#page--1-14) is below the solidus temperature of approximately 540 °C. A four-phase region of α Al + Si (diamond) + β-AlFeSi + Mg₂Si appears at temperatures lower than 400 °C, whereas

> Fig. 1. (a) SEM images of initial AlSi10Mg alloy powder used in this study and (b) schematic showing laser scanning tracks with 67° rotation on each powder layer.

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