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Short communication

Contribution of Mg_2Si precipitates to the strength of direct metal laser sintered AlSi10Mg

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A B S T R A C T

Microstructure of deformed DMLS-AlSi10Mg under quasi-static loading was studied using TEM to elaborate the strengthening mechanisms. In addition to Orowan (due to presence of Si precipitates), Hall-Petch (due to eutectic Si walls), and dislocation hardening (due to pre-existing entangled dislocations) mechanisms, Mg_2Si precipitates (colonies) contributed to the strength of alloy by impeding dislocation motion. The level of this contribution was evaluated as ~13 MPa by comparing the modeled and measured yield strength.

1. Introduction

Enhancing the strength of metallic alloys is an ongoing challenge since developing the next generation of lightweight structures for energy conversion goals is of particular interest. Amongst the available lightweight structural metallic materials, aluminum possesses unique characteristics that make it an interesting candidate in automotive, marine and aerospace sectors. Improvement of the strength of metallic alloys, including aluminum alloys, has been mainly achieved by impeding dislocation motion [1]. In non-heat-treatable aluminum alloys, this goal is accomplished via solid solution strengthening, existence of particles (intermetallic second phase), and grain size (Hall-Petch) effect [2]. In addition, introducing high dislocation density in the matrix (during some manufacturing processes) can effectively result in further strength enhancement through dislocation hardening [3]. Therefore, controlling the microstructural characteristics during manufacturing of metallic materials to obtain desirable strength is essential.

Revolutionary laser-based metal additive manufacturing (AM) processes have the capability of fabricating near-net shaped parts with tailored microstructures [4]. Laser powder bed fusion (L-PBF) technique that is also known as direct metal laser sintering (DMLS) or selective laser melting (SLM) is among the most commonly used AM processes [5]. L-PBF of aluminum alloys has been under investigation in recent years to expand the knowledge on the microstructure-properties relationship in these alloys. In particular, Al-Si-Mg alloying system has been of particular interest due to its low density, superior corrosion resistance, high specific strength, and low propensity to solidification cracking [6]. The strengthening mechanisms of L-PBF-AlSi10Mg under quasi-static uniaxial tensile loadings was studied, where very fine cell-like structures of eutectic Si and presence of fine Si precipitates were reported as inhibitors of dislocation motion through Orowan strengthening mechanism [7] and Hall-Petch effect [8]. Moreover, networks of dislocations were reported in AlSi10Mg microstructure fabricated through L-PBF, which contributed to strengthening of the material through dislocation hardening [9,10]. In a recent study, Hadadzadeh et al. [11] showed that Orowan mechanism, Hall-Petch effect, and dislocation hardening are the main strengthening mechanisms in DMLS-AlSi10Mg under quasi-static uniaxial tensile loading. In addition to these mechanisms, precipitation of Mg_2Si in AlSi10Mg alloy has been identified as another strengthening mechanism, specifically in heat treated alloys [12]. Presence of Mg_2Si in the as-fabricated L-PBF-AlSi10Mg has been reported in some studies (e.g. [13]), while in some other studies (e.g. [14]) precipitation of Mg_2Si in the as-fabricated L-PBF-AlSi10Mg was not reported.

Despite the available studies on the strengthening mechanisms in L-PBF-AlSi10Mg alloy, details of the effect of magnesium on the strength of the alloy, specifically in the form of Mg_2Si precipitates in the as-fabricated material has not been studied. To elaborate the level of contribution of each strengthening mechanism on the strength of DMLS-AlSi10Mg, samples of AlSi10Mg were additively manufactured using DMLS process and subjected to uniaxial tests under quasi-static loading. The microstructures of the as-built and deformed samples were studied using advanced electron microscopy techniques. The strengthening mechanisms (including the effect of Mg_2Si precipitates) were revealed and the effect of dislocations' motion on the microstructural characteristics of the material was investigated in detail.

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Table 1
EOS M290 machine specifications and DMLS process parameters used for AlSi10Mg.

Specification/Parameter	Type/Value
Laser type	400 W Yb-fiber laser
Atmosphere	Ar (with maximum 0.1%O_2)
Beam spot size (μm)	100
Building plate dimensions (mm × mm × mm)	250 × 250 × 325
Preheat temperature (°C)	200
Laser power (W)	370
Scan speed (mm/s)	1300
Hatch distance (μm)	190
Layer thickness (μm)	30

2. Experimental procedure

Rod shaped samples with dimensions of ϕ 12 mm × L 120 mm were additively manufactured through DMLS process in horizontal configuration, as their longitudinal axis was perpendicular to the building direction. The powder used in the bed was AlSi10Mg provided by EOS GmbH with a size distribution of 10–45 μm and a chemical composition of Al-10%Si-0.33%Mg-0.55%Fe (in wt%). The samples were fabricated using an EOS M290 machine with specifications listed in Table 1. In addition, the standard process parameters employed in the core of the material yielded the least porosity are shown in the same table.

Uniaxial tensile cylindrical samples (dogbones) were designed according to ASTM E8–15a standard [15] in a way to avoid the process parameters used to manufacture the bottom layers (Downskin) and top layers (Upskin) of the rods. These samples possessed a gauge length of 24 mm and gauge diameter of 6 mm, where machined out of the as-built DMLS-AlSi10Mg rods using a CNC machine. The tensile tests were performed using an Instron Model 1332 universal testing machine at a strain rate of 9×10^{-4} s^{-1}. The tests were repeated three times to ensure the repeatability of the trials.

Details of the microstructure of as-built and deformed samples were investigated using transmission electron microscopy (TEM). TEM studies were conducted using an FEI Tecnai Osiris TEM with X-FEG gun operating at 200 keV. The super-EDS X-ray detection system combined with a high current density electron beam in the scanning mode (STEM) was employed to study precipitates. Using a sub-nanometer electron probe, spatial resolutions in the order of 1 nm were obtained in EDS elemental mapping. Details of TEM sample preparation can be found in [11].

3. Results and discussion

The microstructure of the as-built DMLS-AlSi10Mg sample was studied inside the melt pools, since the intra-pool characteristics dominate the overall microstructure of L-PBF samples [11]. Fig. 1 shows the STEM bright field (STEM-BF) microstructure of DMLS-AlSi10Mg. Very fine primary α-Al cells developed as solidification began and continued, followed by evolution of continuous networks of eutectic Si at the end of the solidification process. Such a fine microstructure is typical for L-PBF-AlSi10Mg alloy [7,13]. In addition to the cell-like structure, networks of entangled dislocations developed inside the cells as a result of rapid solidification [16]. These dislocations interact both with eutectic Si walls and Si precipitates, as seen in Fig. 1(b) [9]. $Al_8FeMg_3Si_6$ and Al_3FeSi intermetallic phases also formed over the cell boundaries [9]. In addition to the Si precipitates, the dislocations were in interaction with fine Mg_2Si precipitates, as seen in Fig. 1(b) and (c). The Mg_2Si precipitates observed as small precipitates colonies, mostly with needle-shape morphologies, in small quantities [17]. Evolution of Mg_2Si precipitates was not predicted using computational thermodynamics (FactSage™ with the FTlite database [18]) under both equilibrium and non-equilibrium solidification conditions [9]. It seems heating cycles experienced by the material during DMLS process led to diffusion of Mg and Si atoms through the supersaturated α-Al matrix and formation of Mg_2Si colonies. Formation of Mg_2Si zones or colonies has been reported during early stages of age hardening of Al-Mg-Si alloys [19]. In these alloys, the migration of solute atoms is controlled by the thermal energy (during aging treatment) and assisted by quenched-in vacancies [20]. In fact, fine Mg_2Si colonies form by migration of vacancy-solute pairs [21]. Moreover, quenched-in dislocations have been also identified as critical sites for nucleation and growth of Mg_2Si [22]. Quenched-in vacancies can form in DMLS-AlSi10Mg alloy due to the ultrafast cooling rates (10^3–10^8 K/s) achieved during the process.

Characteristics of Si precipitates in the aluminum matrix were studied using high-resolution TEM (HRTEM) and one typical HRTEM image is shown in Fig. 1(d) along with fast Fourier transform (FFT) patterns of Al-matrix, Si precipitate, and matrix/precipitate interface. FFT patterns indicate an orientation relationship between the matrix and the precipitates; however, a semi-coherent interface developed at matrix/precipitate boundaries. Evolution of semi-coherent interface was a result of thermal conditions experienced by the material during the DMLS process. Characteristics of the microstructure are summarized in Table 2, where the dislocation density was calculated using the method reported in literature [23]. It is noted that, the dislocation density in the as built microstructure was calculated as 2.15×10^{14} m^{-2}.

A typical engineering stress-strain curve of DMLS-AlSi10Mg under quasi-static uniaxial tensile test is shown in Fig. 2(a). Yield stress (YS) (using 0.2% strain method), fracture strain and ultimate tensile strength (UTS) of the material was evaluated as 235 ± 5 MPa, 7.5 ± 1% and 386 ± 10 MPa, respectively. The microstructure of the deformed DMLS-AlSi10Mg was studied and a typical STEM-BF image is shown in Fig. 2(b). In general, deformation of DMLS-AlSi10Mg led to the generation of new dislocations and increase of the dislocation density to 2.4×10^{14} m^{-2} as a result of dislocation motion and pile up [7,9]. Considering the EDS elemental map, the network of dislocations is in interaction with both the Si walls (eutectic phase) and Si precipitates. In other words, these barriers impeded the dislocation motion [16]; therefore, both Hall-Petch effect and Orowan hardening mechanism are active during the deformation of DMLS-AlSi10Mg [24]. Moreover, the dislocation hardening mechanism is also an active mechanism, since the dislocations are in mutual interaction [16].

Details of dislocation-Si precipitate interactions are shown in Fig. 3. The dislocations bypassed the Si-precipitates by bowing around and penetrating through them due to the semi-coherency characteristics of the precipitates [25]. Referring to the STEM-BF image and Si EDS map in Fig. 3, the majority of the dislocations do not possess a straight configuration, after bypassing the Si-precipitates. This is mainly due to the misfit strain field around the semi-coherent precipitates that makes the dislocations to bow toward or away from the precipitates after bypassing [25].

In addition to the Si precipitate-dislocation interaction, the dislocations were observed in interaction with fine Mg_2Si precipitates, as seen in Fig. 3. Fig. 4 shows an EDS elemental map of a nanometric needle-shaped Mg_2Si precipitate along with a corresponding EDS spectrum from the core of the precipitate, which confirms existence of Mg_2Si. As seen in Fig. 3, the dislocations were pinned by the Mg_2Si colonies and their further motion seemed to be constrained. Therefore, it appears that Mg_2Si colonies did in fact contribute to the strength of the DMLS-AlSi10Mg.

To elaborate on the level of Mg_2Si precipitates contribution to the strength of DMLS-AlSi10Mg, the strength of this alloy was calculated considering well-known strengthening mechanisms. These mechanisms include Orowan mechanism (due to Si precipitates) [7,8], Hall-Petch effect (due to eutectic Si walls as they act as grain boundaries) [26], and dislocation hardening (due to pre-existing entangled dislocations) [27]. The yield strength (σ_y) of DMLS-AlSi10Mg can be calculated as follows [8,26]: