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Selective laser melting of nano-TiB_2 decorated AlSi10Mg alloy with high fracture strength and ductility

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ABSTRACT

An in-situ nano-TiB_2 decorated AlSi10Mg composite (NTD-Al) powder was fabricated by gas-atomisation for selective laser melting (SLM). Fully dense and crack-free NTD-Al samples were produced using SLM. In contrast to the NTD-Al powder without cell-like microstructure, the SLMed NTD-Al had a textureless microstructure, consisting of fine grains and cells, with well dispersed nano-TiB_2 particles inside the grains and rod-like nano-Si precipitates inside the cells. Both nano-TiB_2 particles and nano-Si precipitates exhibited a highly coherent interface with the Al matrix, indicative of a strong interfacial bonding. The formation of this microstructure was attributed to the sequential solidification of non-equilibrium and eutectic Al-Si upon rapid cooling during SLM. As a result, the SLMed NTD-Al showed a very high ultimate tensile strength ~530 MPa, excellent ductility ~15.5% and high microhardness ~191 $HV_{0.3}$, which were higher than most conventionally fabricated wrought and tempered Al alloys, previously SLMed Al-Si alloys and nano-grained 7075 Al. The underlying mechanisms for this strength and ductility enhancement were discussed and a correlation between this novel microstructure and the superior mechanical properties was established. This study provides new and deep insights into the fabrication of metal matrix nanocomposites by SLM from in-situ pre-decorated composite powder.

1. Introduction

As an emerging powder bed additive manufacturing technique, selective laser melting (SLM) has enjoyed a rapid development in the past decade [1–3]. Its characteristic layer-wise fabrication process enables the rapid production of complex geometry components without the time-consuming mould design process [4,5]. In addition, the fast cooling rate (10^3–10^6 °C/s) also offers a promising alternative to fabricate components with unique and highly tailorable microstructures, e.g. ultrafine, gradient, metastable or amorphous microstructures [6–14]. All of these have rendered SLM one of the frontier manufacturing techniques across a broad range of industries, including aerospace, automotive, biomedical and other niche industries [5]. However, one of the key hurdles impeding further applications of SLM is the current severe limitation on materials suitable for SLM processing. Up till now, only a very limited number (less than 20) of metal powders including certain Al-, Ti-, Ni-alloys and steels possess acceptable processability and can be used to produce dense components by SLM [2–5]. This is far from adequate for both current and future applications and industrial take-up of SLM. To eliminate this bottleneck, it is crucial to design and develop suitable materials with acceptable SLM processability. Therefore, more and more efforts have recently been made to expand the materials palette for SLM and metal matrix composites are of major focus [15,16].

Recently, fully dense Al-, Ti- and Fe-based metal matrix composites with secondary metal or ceramic particles as additions have been successfully fabricated using SLM with improved mechanical properties [17–23]. For example, Gu et al. reported on the fabrication of Al nanocomposites with a novel ring-structured TiC

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reinforcement by SLM of AlSi10Mg with nano-TiC ceramic particles [17]. The fabricated nanocomposites exhibited improved tensile strength (486 MPa) and high microhardness (188 $HV_{0.1}$). In addition, they also reported on the fabrication of Ti composites by SLM of a mixture of pure Ti and micrometer-sized SiC particles [21]. The microstructure showed a unique network of distributed TiC and a sub-micron dendritic morphology, which resulted in high microhardness (980 $HV_{0.2}$) and a low friction coefficient. Song et al. successfully SLMed Fe-based nanocomposites with micrometer-sized Fe and nano-SiC particles [18]. The fabricated parts showed a microstructure with homogeneously distributed nano-SiC particles, which resulted from the decomposition of the micrometer SiC starting powder, resulting in a very high tensile strength (753 MPa). Apart from this, similar attempts were also made to fabricate other metal matrix composites via SLM including in-situ Ti-TiB_2 composites [19], a novel β-Ti composites from Ti6Al4V ELI and micrometer-sized Mo [20], in-situ TiC reinforced Ti-Al composites from pure Ti, Al and graphite [22] and TiB reinforced near α-Ti metal composites from pure Ti and micrometer-sized CrB_2 [23].

As mentioned above, current SLM fabrication of metal matrix composites largely relies on a mechanical mixing process (e.g. ball milling) to ensure a homogenous mixture of the starting metals and ceramic particles before SLM. As such, the drawbacks of this process are apparent: (1) in most cases, the added fine secondary particles (diameter $\leq$ 5 μm) would deteriorate the flowability of the matrix powder [2]; (2) due to different laser absorptivity of the ceramic particles, an apparent difference in thermal history between the ceramic particles and the metals would be inevitable, which can also affect or complicate the melt pool behavior [24]; and (3) although some in-situ reaction can be introduced to improve the interfacial bonding between the reinforcement particles and the matrix [16], this improvement is limited because the wettability of the metal matrix and secondary metal or ceramic particles is difficult to control. These challenges all together would pose significant difficulty in optimising the SLM process of the metal matrix composites to achieve fully dense and crack-free components. As a consequence, it becomes also difficult to tailor the microstructure and hence obtain the desired mechanical properties of the metal matrix composites.

In this study, a nano-TiB_2 decorated AlSi10Mg composite (NTD-Al) powder was gas-atomised for SLM. The microstructures and mechanical properties of the NTD-Al powder and SLMed NTD-Al were systematically characterised. The SLMed NTD-Al exhibited very high tensile strength, excellent ductility and high microhardness. The underlying reason was attributed to the novel microstructure consisting of fine grains and cells, strengthened by well-dispersed nano-TiB_2 particles and nano-Si precipitates.

2. Experimental procedures

2.1. Fabrication of nano-TiB_2 decorated AlSi10Mg powder

The nano-TiB_2 decorated AlSi10Mg (NTD-Al) powder used in this study was fabricated in two steps. Firstly, nano-TiB_2 was introduced into pure Al (99.99%, CHALCO) in a high-purity graphite crucible in air using an electrical resistance furnace at 900 °C via the in-situ reaction method. Reactive salts of K_2TiF_6 and KBF_4 with high purity (≥99.0%, Taian Health Chemical Co., Ltd.) were slowly added into the molten Al metal during melt stirring at 600 rpm for 15 min. After slag removal, the molten composite was cast into an iron mould to obtain a nano-TiB_2 reinforced pure Al composite master alloy. Secondly, Mg (99.99%, CHALCO) and Al-Si master (99.99%, CHALCO) alloys were subsequently added into the re-molten pre-synthesized nano-TiB_2 reinforced Al composite master alloy and homogenized for 10 min. NTD-Al powder was produced by an in-house built gas-atomiser using He gas. The chemical composition of the powder is 9.81 wt% Si, 0.32 wt% Mg, 11.6 wt% TiB_2 (~7 vol %) with Al balance, measured through inductively coupled plasma atomic emission analysis (ICP-AES). The average diameter of the nano-TiB_2 particles is ~100 nm, as reported elsewhere [25] and the sieved powder fraction between 15 and 45 μm was used for the SLM process.

2.2. Selective laser melting process

NTD-Al specimens (10 × 10 × 6 mm) and tensile bars were produced on an in-house built selective laser melting machine which is equipped with a fibre laser, with a wavelength of 1.06 μm and maximum power of 300 W on the part bed. An inert, high purity (≥99.99%) argon gas atmosphere flow was used during the whole process to minimise oxidation. Different laser powers (200–300 W) and scan speeds (800–2000 mm/s) were used. The powder layer thickness was fixed at 30 μm, the scan spacing at 105 μm, without pre-heating. The density of the SLMed NTD-Al was measured using the Archimedes method (a theoretical density ~2.81 g/cm^3 was used).

2.3. Microstructure characterisation

The microstructure of the NTD-Al powder and SLMed materials was characterised using a FEI-Nova NanoSEM 450 scanning electron microscope (SEM, acceleration voltage 10 kV, working distance 5 mm) and a FEI Tecnai G2 transmission electron microscope (TEM, operated at 200 kV). A state-of-the-art FEI Titan Themis 300 TEM, equipped with a probe aberration corrector and a highly efficient (4 quadrant) energy dispersive X-ray (EDX) system and operated at 200 kV, was further used for atomic and nano-scale characterization in the scanning TEM (STEM) mode. The probe size was set to 0.1 nm with a convergence semi-angle of 22.5 mrad. The collection angle of the high angle annular dark field (HAADF) detector was in the range of 80–150 mrad. The size and morphology of the grain structure were investigated by image analysis software Image Pro Plus. At least ten different regions on each sample were taken for image analysis. The TEM samples from the powder were prepared using focused ion beam (FIB). The TEM samples from the SLMed NTD-Al materials were mechanically polished and finally thinned by a Precision Ion Polishing System (Gatan PIPS™) where low-angle and low-current polishing conditions were used in conjunction with a liquid nitrogen cold stage. Texture and grain size of SLMed NTD-Al material were characterised using electron backscattered diffraction (EBSD) using a TSL orientation imaging microscope system mounted on a FEI-Nova NanoSEM 450 SEM. A step size of 0.1 μm was used in all EBSD measurements.

The phase formation in the powder and SLMed NTD-Al materials were characterised using X-ray diffraction (XRD, Siemens D500 powder diffractometer, Cu Kα target, operated at 40 kV and 40 mA with a step size of 0.02° and scanning speed 2°/min, all XRD scans were done on the plane parallel to the SLM building direction).

2.4. Mechanical properties

Tensile tests were carried out on SLMed and machined specimens (gauge length ~4 × 2 × 16 mm, prepared by electrical discharge machining, EDM), using an Instron 5982 machine at a constant strain rate of 1 mm/min. Strain was measured with a 12.5 mm gauge length extensometer. Samples were aligned perpendicular to the build direction and a total of 8 samples were tested. The microhardness of the SLMed material was measured on a Zwick-3202 Vickers hardness tester with a load of 0.3 kg and a