Microstructure characteristics and its formation mechanism of selective laser melting SiC reinforced Al-based composites

Xuan Zhao\textsuperscript{a,b}, Dongdong Gu\textsuperscript{a,b,*}, Chenglong Ma\textsuperscript{a,b}, Lixia Xi\textsuperscript{a,b}, Han Zhang\textsuperscript{a,b}

\textsuperscript{a} College of Materials Science and Technology, Nanjing University of Aeronautics and Astronautics, Yudao Street 29, Nanjing, 210016, Jiangsu Province, PR China
\textsuperscript{b} Jiangsu Provincial Engineering Laboratory for Laser Additive Manufacturing of High-Performance Metallic Components, Nanjing University of Aeronautics and Astronautics, Yudao Street 29, Nanjing, 210016, Jiangsu Province, PR China

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\textbf{A B S T R A C T}

SiC ceramic reinforced Al-based composites were successfully synthesized by selective laser melting (SLM) of 8.5 vol % SiC/AlSi10 Mg powder mixtures. The mechanism of microstructure evolution in an individual molten pool of the SLM processing was investigated. Three characteristic zones were well distinguished, including center and boundary areas of the molten pool as well as the heat-affected zone. The results revealed that the microstructure changing from columnar dendrite growth to cellular growth along the building direction could be attributed to the change of G/R (G is temperature gradient and R is solidification velocity). Phase identification of the SLM-fabricated parts was performed by X-Ray Diffraction (XRD) and Energy Dispersive Spectroscopy (EDS) analysis, further demonstrating the in-situ formed phases in the molten pool. Al4SiC4 crystals were formed in situ around the partially melted SiC ceramic particles. The nanoindentation tests conducted on different areas of the SLM-fabricated Al-based composites exhibited the high nanohardness (2.27 GPa and 2.15 GPa) and the elastic modulus (78.94 GPa and 75.46 GPa) in the center and boundary of molten pool, respectively, which were much higher than the reported value of conventional materials.

1. Introduction

In recent years, demands of high-performance aluminum matrix composites (AMCs) in the automotive and aerospace industries are drastically increasing due to their high specific strength and stiffness, excellent wear resistance and low coefficient of thermal expansion [1,2]. AMCs combined with favorable properties of aluminum matrix and reinforcing phases, have also received considerable research interest [3,4], in which a variety of ceramic reinforcements, such as SiC, Fe2O3, TiC, Al2O3 and TiB2 [5–9], have been often used due to their high hardness and excellent corrosion resistance. Notably, large amounts of functional components with complex geometries, such as air, screw, oil tube, etc., are involved in the automotive and aerospace fields. These complex configurations along with relatively low ambient ductility of AMCs in comparison with aluminum alloys, remain challenging for precisely shaping of the complex components through traditional processing routes [10].

Selective laser melting (SLM), as one of rapidly developed additive manufacturing techniques, has received much attention in terms of its great potential to near net shaping of metallic components with complex structures [11–16]. Based on complete melting philosophy, SLM process is characterized by selectively melting of the powder layer and subsequently rapid solidification of the mesoscale molten pool layer by layer [17,18]. The complex non-equilibrium physical and chemical metallurgical nature of SLM processing can cause a homogenous dispersion of reinforcements in the matrix and induce complex interactions at the interface between the Al matrix and the reinforcements [19,20], thereby tailoring microstructures and performances of the manufactured AMC components appropriately. Dadbakhsh et al. investigated the influence of materials and processing windows during SLM on fabrication of Al/Fe2O3 composite parts from powder mixtures [6]. An in-situ reaction occurred by modifying the visual surface and altering material characteristics in the SLM processing windows and improved mechanical properties were obtained with addition of Fe2O3 [6]. Gu et al. fabricated novel TiC/AlSi10 Mg nanocomposites parts with ring-structure nano-TiC reinforcements uniformly distributed along the Al grain boundaries by SLM [7]. These nanocomposite parts demonstrated a remarkable improvement in both tensile strength and microhardness. Obviously, addition of the reinforcements not only complicates the physical/chemical reaction at the interface between the
Al matrix and the reinforcements, but also results in unfavorable bonding due to poor wettability of the reinforcements by molten Al alloys. Due to rapid melting/consolidation nature of SLM, the molten material tends to experience a distinctively physical and chemical metallurgical process during high-energy non-equilibrium processing, indicating complicated modes of heat and mass transfer and forming a unique microstructure in the AMCs [21]. Limited work has been performed on quantitatively thermodynamic investigation of AMCs during SLM processing, which has an important influence on the microstructure evolution. Owing to rapid melting/solidification of powder material and resultant small molten pool dimension, it is difficult to directly obtain transient temperature and velocity profiles through experiments [22,23]. Hence, numerical simulation is essential to analyze temperature evolution within an individual molten pool during SLM.

This work has investigated the microstructural evolution of the SLM-processed Al-based composite parts within an individual molten pool. Based on a numerical simulation, the solidification nucleation and growth mechanism of the local molten pool were discussed. The formed phases and interaction mechanism of the SLM produced SiC/AlSi10Mg during SLM processing were analyzed in detail. The structure-dependent nanohardness measured within an individual molten pool were also discussed.

2. Experimental procedures

2.1. Materials

Spherical AlSi10Mg powder (99.8% purity) with a particle size ranging from 20 to 63 μm and poly-angular SiC ceramic powder (99.6% purity) with a mean particle size of 3.5 μm were used in this study. A QM series Planetary Mill (Nantai instrument co. LTD., NanJing) was employed to achieve a homogeneous powder mixture, which was composed of 91.5 vol % AlSi10Mg and 8.5 vol % SiC ceramic powder. The powder mixture was prepared in an argon atmosphere in a glove box. Then the milling process was carried out under a condition of a ball-to-powder weight ratio of 1:1 and a rotation speed of 200 rpm. The milling time was performed in a mode containing a 15-min milling and 5-min pause and was set to 2 h. Subsequently, mixed powder with a weight of 2 kg was prepared for the SLM process.

2.2. Selective laser melting process

An independently developed SLM system is equipped with a YLR-500-WC ytterbium fiber laser (IPG Laser GmbH, Germany) with a spot size of 70 μm and a maximum power of 500 W, an automatic powder spreading device, and a computer system for process control. Before the SLM processing, an aluminum substrate was fixed on a building platform (174.50 mm × 174.50 mm × 14.50 mm) and leveled precisely. Then, the processing chamber was pumped to high vacuum conditions and then filled with high-purity argon at a pressure of 200 Pa to carefully control the O2 content below 20 ppm and minimize oxidation during the overall SLM process. During SLM, the mixed powder was spread homogeneously on the substrate by the powder spreading device and the laser beam scan the pre-spread powder selectively based on the slice profile of computer-aided design (CAD) model. An inter-layer stagger scanning strategy was applied in this work. According to the previous experiments, the processing parameters were set as follows: laser power p = 350 W, scan speed v = 2500 mm/s, hatch space h = 50 μm and layer thickness ℓ = 50 μm. Finally, the samples with dimensions of 11 mm × 11 mm × 5 mm were fabricated accordingly.

2.3. Material characterization

The cross sections of the SLM samples along the building direction were grounded and polished according to the standard procedures. For microstructure observations, the polished surfaces were then etched with Keller’s reagent containing HF (1.0 mL), HCl (1.5 mL), HNO3 (2.5 mL), and distilled water (95 mL) for 20 s. The microstructure of the SLM-processed part was characterized using an S-4800 field emission scanning electron microscope (FE-SEM, Hitachi, Tokyo, Japan) at a voltage of 5.0 kV. The chemical compositions were determined by an EDAX energy dispersive X-ray spectroscopy (EDX) (EDAX, Inc., Mahwah, NJ), using a super-ultrathin window sapphire detector. The phases of the specimens were identified by a Bruker D8 Advance X-ray diffractometer (XRD) (Bruker AXS GmbH, Karlsruhe, Germany) with Cu Kα radiation at 40 kV and 40 mA, using a continuous scan mode of 4°/min. Nanohardness tests were performed at room temperature using DUH-W201S nanoindentation tester (Shimadzu Corporation, Tokyo, Japan). Before the nanoindentation tests, the surface of the sample was polished according to the standard procedures. A loading-unloading test mode was used, and the maximum load applied was 1000 μN with a loading/unloading rate of 250 μN/s, holding time for 5 s at the maximum load. During the nanohardness measurements, the load and indentation depth were recorded, and then used to construct loading-unloading plots. The uncertainty of nanohardness and elastic modulus values is about 3% and 4%, respectively.

2.4. Numerical simulation

In order to study the thermal evolution behavior and velocity field within the molten pool during the SLM processing of AlSi10Mg/SiC composites, the simulation was conducted using the commercial computational fluid dynamics software FLUENT. The model used in this study has been described elsewhere [19,22]. The laser heat source with a scan speed of 2500 mm/s followed a Gaussian distribution is mathematically defined as a heat flux in this model. Furthermore, the powder bed is regarded as the homogeneous and continuous media. The fluid motion in the molten pool is considered to be Newtonian and incompressible fluid. Flow in the molten pool is considered to be laminar [23]. Meanwhile, thermal conductivity (k) and specific heat (Cp) of the used materials are temperature-dependent in the melting process. The as-used material properties and SLM processing parameters are listed in Table 1. The variation of thermal-physical parameters of AlSi10Mg and SiC with temperature are shown in Fig. 1.

3. Results and discussion

3.1. Microstructural evolution in different regions of molten pool

Fig. 2 shows the microstructural features in different regions of a molten pool. The adjacent melting tracks after solidification were obvious and boned well without pores observed, thus indicating that the SiC/AlSi10Mg composites possessed good processability as well as good wettability between the SiC ceramic particles and Al matrix. Fig. 2a shows the cross section of the molten pool with three typical characteristic zones of SLM-processed AMCs [27]: center zone (zone I), boundary zone of a molten pool (zone II), and heat-affected zone (HAZ) (zone III). It is observed that the remaining SiC ceramic particles were randomly distributed in the molten pool (as seen in Fig. 2a). The characteristic morphologies of molten pool in three different regions

<table>
<thead>
<tr>
<th>Parameters</th>
<th>Values</th>
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<tbody>
<tr>
<td>Laser absorptivity of the AlSi10Mg powder</td>
<td>0.09 [16]</td>
</tr>
<tr>
<td>Laser absorptivity of the SiC powder</td>
<td>0.78 [24]</td>
</tr>
<tr>
<td>Ambient temperature, T0</td>
<td>300 K</td>
</tr>
<tr>
<td>Powder layer thickness, ℓ</td>
<td>50 μm</td>
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<tr>
<td>Hatching space, h</td>
<td>50 μm</td>
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<tr>
<td>Radius of the laser beam, ω</td>
<td>70 μm</td>
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are shown in Fig. 2b–d. In the center of molten pool (zone I), fine cellular microstructure dominated and the average cell size reached $\sim 0.3 \mu m$ (Fig. 2b). A relatively clear interface between adjacent $\alpha$-Al grains was observed (Fig. 2b), which could be attributed to the precipitation of primary Si particles along the grain boundaries. But the interface became vague and fiber-like microstructure with typical eutectic microstructure formed along the $\alpha$-Al grain boundaries close to the zone II (Fig. 2b). The microstructure located at the boundary of molten pool (zone II) was transformed from cellular to dendritic morphology (Fig. 2c), simultaneously showing an apparent orientation which could be ascribed to great temperature gradients. This is in good agreement with the results reported by Kimura et al. [28]. A small amount of SiC ceramic particles and newly formed phases with a strip structure were observed in the zone II (Fig. 2c). It is noted that the fiber-like microstructure extended largely in this zone, which is ascribed to a relatively small cooling rate and coarsening growth at the boundaries. For the heat-affected zone (zone III), a number of dispersed particles were formed in the matrix, inferred to Si precipitates (Fig. 2d). Similarly, Thjis et al. found dispersed particles in the heat-affected zone of the SLM-prepared AlSi10 Mg, demonstrating that these particles were precipitated eutectic Si phase [29].

It is found that the microstructure of molten pool was non-uniform and showed a significant change at different regions. This is expected to be induced by various temperature gradients in the small-sized molten pool during the high-energy laser irradiation. This is in good agreement with the results reported by Liu et al. [21], where the cooling rate induced discrepancy in microstructural gradient and element distribution during SLM. The temperature at the boundary was much lower than that inside the molten pool because of the heat flux based on a Gaussian distribution. The temperature distribution plots of melt pool from top and cross-section view are shown in Fig. 3. The melt pool exhibited an elongated shape along the scanning direction (as marked by white arrow). The appearance of the ellipse shape instead of circular shape can be attributed to the effect of the movement of heat source. Fig. 3b exhibits the transient temperature distribution at the cross section of the molten pool as the laser interacts with the powder bed. The white dash line corresponds to the isotherm of the melting temperature of the AlSi10 Mg powder, the region inside which had higher temperature than the melting temperature of AlSi10 Mg, resulting in a small molten pool in this area. As the maximum temperature of molten pool obtained from simulation was 1150 K, much lower than 2827 °C (the melting point of SiC ceramic [30]), it is supposed that the SiC ceramic particles were not melted completely under the experimental condition.

**Fig. 1.** Variation of thermal conductivity and specific heat of (a) AlSi10 Mg and (b) SiC with temperature [25,26].

**Fig. 2.** FE-SEM image showing the cross sections of characteristic melt pool of the SLM-processed parts (a) and magnified structure located in different zones: (b) Center zone I; (c) Molten pool boundary zone II; (d) Heat-affected zone III.
However, with a high-energy laser the SiC ceramic particles would in situ react with aluminum melt partially, thereby forming a strip-structured phases close to the unmelted SiC. As reported by D. Gu et al. [31], reinforcing phases included unmelted micron-sized SiC particles, in-situ formed micron-sized Al₄SiC₄ strips and in-situ produced submicron Al₄SiC₄ particles in the SLM of AlSi10Mg/SiC (20 wt.%). It can be preliminarily inferred that the newly formed strip-structured phases are Al₄SiC₄.

Fig. 3c shows the Marangoni flow with a radially circulating flow pattern, transferring heat and mass within the molten pool. As the laser-induced molten pool exhibits large temperature gradients due to introduce of Gaussian laser heat source, this will make contributions to surface tension gradients accordingly and cause a Marangoni flow from low surface tension to high surface tension area on the surface of the melt pool. During the SLM of SiC/AlSi10Mg composites, migration of SiC ceramic reinforcements is affected by two different forces. The SiC ceramic particles were dragged by the friction force resulting from the viscosity of the melt despite the thermal capillary force driving the migration of SiC ceramic particles. The SiC ceramic particles can circulate several times by the motion of fluid in the molten pool. The intensity of the fluid flow plays a significant role in the dispersion of the SiC ceramic reinforcements in the Al matrix. This explains why the SiC ceramic particles were randomly distributed in the molten pool (see Fig. 2a).

The profiles of temperature distribution versus distance from the top surface and cooling rate versus time calculated by finite element analysis method are shown in Fig. 4 in order to reveal the solidification features of zones I, II and III in the molten pool. The cooling rate varied from a negative value to a positive value when the laser beam scans approach and leave away from the detected point, corresponding to the respective melting and solidification processes of the SLM-induced melt pool. The calculated maximum temperature of zones I, II and III was 1149 K (Figs. 4b), 1060 K (Fig. 4c) and 879 K (Fig. 4d), respectively and the corresponding maximum cooling rate was $2.55 \times 10^6$ K/s, $1.80 \times 10^6$ K/s and $0.33 \times 10^6$ K/s, respectively. This indicates that the maximum cooling rates decrease with distance from the top surface within an individual molten pool. As reported by Rosenthal et al. [32], the solidification of Al alloy in the SLM processing depends on the temperature gradient($G$) and on the solidification velocity ($R$) in the molten pool. A relative low value of $R$ at a constant $G$ contributed to formation of a stable planar consolidation front, whereas with an increasing solidification velocity the consolidation front changes from a cellular to dendritic structure. Similarly, for the different morphologies in the molten pool of the SLM-processed Al-based composite parts, $G$, solute concentration ($C$) and $R$ ($R = V/G$, $V$ is cooling rate) are the three factors affecting the growth mode of structure.

According to the data obtained directly from the simulation results, the $G/R$ in the center region ($3.56 \times 10^6$ K/s/m²) was almost one order magnitude smaller than that at the boundary ($6.72 \times 10^5$ K/s/m²) of the individual molten pool. As a result, the combined effects of relatively low $G/R$ and low solute concentration of Si lead to development of a cellular structure in the center of the molten pool. On the contrary, columnar dendrites is well developed in the boundary region (Fig. 2). This explains that the microstructure of the SiC/AlSi10Mg composites changed from a cellular structure to a dendritic structure with an increasing distance from the center of molten pool.
Due to the layer processing strategy of SLM, remelting process is another possible factor contributing to formation of non-uniform microstructure, which occurs between two adjacent molten pools. The regions at the boundary of the molten pools can melt twice because of the hatch overlap, and the grains developed along the positive temperature gradient. Si phases can precipitate from Al matrix sufficiently and even globular Si particles grow coarsely. As a result, the remelting effect leads to the precipitation of large Si particles in the heat-affected zone (the light-grey particles as seen in Fig. 2d).

3.2. Phase identification

Fig. 5 shows the XRD spectra of the SiC/AlSi10 Mg composite powder (black curve) and SLM-processed SiC/AlSi10 Mg parts (red curve). Strong diffraction peaks corresponding to Al were detected. Besides, weak SiC diffraction peaks were identified, indicating that a small amount of unmelted SiC particles remained in the matrix after solidification. Meanwhile, additional diffraction peaks from Al4SiC4 were observed in the XRD spectra of the SLM-processed SiC/AlSi10 Mg parts, which could be ascribed to in-situ reaction between the Al melt and the SiC ceramic particles in laser-induced molten pool. At high temperature of SLM processing, Si and C from SiC reinforcements tended to dissolve in molten aluminum alloys and the in-situ reaction between the Al melt and the SiC ceramic particles occurred forming new Al4SiC4 phase. A small quantity of Si phases was also found, consistent with the microstructure observation in microstructure section. Although aluminum alloys have a high affinity to oxygen element, the characteristic peaks of aluminum oxide were not detected in the SLM-processed Al-based composites in the limit of XRD detection. This implies that the atmosphere for preparation and SLM processing of the AlSi10Mg/SiC specimens could avoid the oxidation behaviors of powder and as-fabricated samples.

The high-magnification FE-SEM images, as shown in Fig. 6 and Fig. 7a, present characteristic morphologies of the remaining SiC ceramic particles and the newly formed strip-structured Al4SiC4 in the molten pool. The morphology of the SiC ceramic particles significantly depends on their locations in the molten pool. As the heat is accumulated greatly in the center of the molten pool at a high-energy irradiation of laser beam, it is reasonable to expect that the SiC ceramic particles located in the central region were melted partially and the in-situ reaction of SiC ceramic particles with Al melt occurred at the...
boundary of SiC particles. The SiC particles became very smooth instead of the poly-angular shape used as starting powder. While at the boundary of the molten pool, the energy absorbed by the powder is somewhat lower than that in the center of the molten pool, the SiC ceramic particles have more tendency to remain sharp and rough (as seen in Fig. 6).

Fig. 7 illustrates the EDS results of the chemical compositions collected from the SLM-processed composites in Fig. 7a. Three different phases are measured, including the large-sized SiC ceramic particles, strip-structured Al₄SiC₄, and eutectic Al-Si. As shown in Fig. 7b, EDS point detected in the grey area (point 1) revealed that the fibrous phase showed a proportion of Al (88.03 at. %) and Si (11.97 at. %), corresponding to eutectic Al-Si. The Al matrix with a cell microstructure (point 2) exhibited a small amount of Si element (8.85 at. %), inferred to α-Al matrix. As revealed in Fig. 7(d), the Al, Si, and C elements with an approximate Al:Si:C atom ratio of 4:1:4 was detected in the strip-structured reinforcements (point 3). It demonstrated that Al₄SiC₄ phase was in situ formed during SLM via the reaction between molten aluminum and SiC, in good agreement with the XRD results.

Due to the relatively high mass fraction of SiC (8.5 vol %) in the starting powder and incomplete in-situ reaction during the laser-induced rapid melting/solidification process, it is reasonable to expect that there existed primary SiC phase after SLM. The in-situ formation of Al₄SiC₄ can be explained based on the Al-Si-C ternary alloy phase diagram [33], as given in Fig. 8. The starting component used in this study is the Al:Si:C mole ratio of 78:15:7, which is far below the content for formation of Al₄SiC₄. In such situation, the SiC firstly reacts with molten aluminum to form Al₄C₃ in the temperature range of 940–1620 K according to the previous study [31]. With dissolution of partial SiC ceramic particles in the melt, the reactions P and M will thermodynamically occur accompanied with reaction Q in the molten pool [33]. Therefore, Al₄SiC₄ phase are formed close to the SiC ceramic particles. This finding is in good agreement with the results reported by Walter et al. [34].

P: L + C + Al₄C₃ → Al₄SiC₇

![Fig. 6. The morphologies of the SiC particles in the SLM-processed SiC/AlSi10 Mg composite.](image)

![Fig. 7. (a) The SiC particles in the molten pool center of SLM-processed SiC/AlSi10 Mg composite and EDS results showing the chemical compositions collected from the SLM-processed composites: (b) fibrous area (point 1); (c) cellular area (point 2); (d) strip-structured reinforcement (point 3).](image)
M: $L + C + Al_3SiC_2 \rightarrow Al_4SiC_4$

Q: $L + C \rightarrow Al_4SiC_4 + SiC$

3.3. Nanohardness

The nanoindentation tests were performed on three different areas: reinforcing structure, center of molten pool and boundary of molten pool. The hardness is defined as a ratio of the maximum indentation load ($F_{\text{max}}$) to the projected area of hardness impression ($A$). The nanohardness ($H$) can be calculated as follows [35]:

$$H = \frac{F_{\text{max}}}{A}$$  \hspace{1cm} (1)

where $F_{\text{max}}$ is the maximum load and $A$ is the projected area of the hardness impression.

According to Oliver and Pharr method [35], the reduced elastic modulus $E_R$ can be determined by the data of a complete loading/unloading cycle. The reduced elastic modulus is expressed as:

$$E_R = \frac{\sqrt{\pi S}}{2} \frac{S}{A}$$  \hspace{1cm} (2)

where $E_R$ is the reduced elastic modulus, $S$ is the stiffness and $A$ is the projected area of the hardness impression.

Load-depth curves and the corresponding nanohardness and elastic modulus values of the SLM processed SiC/AlSi10Mg composites are shown in Fig. 9. The nanoindentation depth and the deformation recover behavior varied with different zones of molten pool in the SLM-processed SiC/AlSi10Mg composites. The nanohardness significantly depends on the microstructure and phases developed in the molten pool [36]. The respective nanohardness and elastic modulus values calculated from measurements are 2.27 GPa and 78.94 GPa in the center, 2.15 GPa and 75.46 GPa at the boundary of molten pool. Moreover, unmelted SiC particles and newly formed Al$_4$SiC$_4$ phases exhibited much higher nanohardness (7.21 GPa) and elastic modulus (197.13 GPa) than that of the molten pool. It has to be mentioned that the measured nanohardness of the composites is coincident with the measured microhardness across the molten pool along the building direction.

4. Conclusions

This work has investigated the microstructure evolution and nanohardness of the SiC/AlSi10Mg composite fabricated by SLM technology. The following conclusions are drawn:

1. The microstructure in the center of the molten pool was dominated by fine cellular structure and the average size of cell reached $\sim 0.3 \mu m$, while the main microstructure at the boundary of molten pool was columnar dendrites. The solidification in the SLM processing of SiC/AlSi10Mg alloy depends on three factors of the temperature gradient ($G$), the solidification velocity ($R$), and the concentration of solute Si in the micro-sized molten pool, suggesting that a variation of $G/R$ and the concentration of Si contributes to a structural change from columnar dendrite growth to cellular growth.

2. The in-situ reaction between aluminum melt and SiC ceramic
particles led to the formation of Al5SiC3 reinforcements close to the partially melted SiC ceramic particles. Three different phases including the unmelted micron-sized SiC ceramic particles, the in situ formed Al5SiC3 phase, and the eutectic Si phase, are observed in the as-fabricated SiC/AlSi10Mg composites.

(3) The hardness and elastic modulus of the SLM-produced SiC/AlSi10Mg parts are 2.27 GPa and 78.94 GPa in the center, 2.15 GPa and 75.46 GPa at the boundary of molten pool. The nanohardness values of SLM-prepared composites is more than twice than that of as-cast AlSi10Mg alloy, while it is comparable to the reported nanohardness of laser melted AlSi10Mg counterparts. The SiC ceramic particles and in-situ formed phase Al5SiC3 play a significant role in the structure development and nanohardness change in different regions of micro-sized molten pool.

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