Investigation of melt-growth alumina/aluminum titanate composite ceramics prepared by directed energy deposition

Yunfei Huang, Dongjiang Wu, Dake Zhao, Fangyong Niu and Guangyi Ma

Highlights:

- The shaping quality, microstructure and mechanical properties of Al$_2$O$_3$ /Al$_6$Ti$_2$O$_{13}$ (6 mol% TiO$_2$) composite ceramics were systematically investigated as a function of energy input in an extensive process window.
- The formation mechanism of solidification defects and the evolution process of microstructure for Al$_2$O$_3$ /Al$_6$Ti$_2$O$_{13}$ (6 mol% TiO$_2$) composite ceramics were revealed.
- The optimized process parameters of Al$_2$O$_3$ /Al$_6$Ti$_2$O$_{13}$(6 mol% TiO$_2$) composite ceramics were determined.

View online: https://iopscience.iop.org/article/10.1088/2631-7990/abf71a

Article Download: https://iopscience.iop.org/article/10.1088/2631-7990/abf71a/pdf

Citation: Huang Y F, Wu D J, Zhao D K, Niu F Y, Ma G Y. Investigation of melt-growth alumina/aluminum titanate composite ceramics prepared by directed energy deposition. Int. J. Extrem. Manuf. 3, 035101(2021).

Related articles:

Effects of laser scanning strategies on selective laser melting of pure tungsten
Dongdong Gu, Meng Guo, Hongmei Zhang, Yixuan Sun, Rui Wang and Lei Zhang
Citation: Gu D D, Guo M, Zhang H M, Sun Y X, Wang R et al. Effects of laser scanning strategies on selective laser melting of pure tungsten. Int. J. Extrem. Manuf. 2, 025001 (2020).

A review on microstructures and properties of high entropy alloys manufactured by selective laser melting
Chen Zhang, Junkai Zhu, Huai Zheng, Hui Li, Sheng Liu and Gary J Cheng
Citation: Zhang C, Zhu J K, Zheng H, Li H, Liu S et al. A review on microstructures and properties of high entropy alloys manufactured by selective laser melting. Int. J. Extrem. Manuf. 2, 032003(2020).

An overview of laser-based multiple metallic material additive manufacturing: from macro- to micro-scales
Chao Wei, Zhizhou Zhang, Dongxu Cheng, Zhe Sun, Menghui Zhu and Lin Li
Citation: Wei C, Zhang Z Z, Cheng D X, Sun Z, Zhu M H et al. An overview of laser-based multiple metallic material additive manufacturing: from macro- to micro-scales. Int. J. Extrem. Manuf. 3, 012003(2021).

Laser metal deposition of refractory high-entropy alloys for high-throughput synthesis and structure-property characterization
Henrik Dobbelstein, Easo P George, Evgeny L Gurevich, Aleksander Kostka, Andreas Ostendorf and Guillaume Laplanche
Citation: Dobbelstein H, George E P, Gurevich E L, Kostka A, Ostendorf A et al. Laser metal deposition of refractory high-entropy alloys for high-throughput synthesis and structure-property characterization. Int. J. Extrem. Manuf. 3, 015201(2021).
Investigation of melt-growth alumina/aluminum titanate composite ceramics prepared by directed energy deposition

Yunfei Huang, Dongjiang Wu, Dake Zhao, Fangyong Niu* and Guangyi Ma

Key Laboratory for Precision and Non-traditional Machining Technology of Ministry of Education, Dalian University of Technology, Dalian, Liaoning Province 116024, People’s Republic of China

E-mail: niufangyong@dlut.edu.cn

Received 12 November 2020, revised 2 January 2021
Accepted for publication 12 April 2021
Published 3 May 2021

Abstract

$\text{Al}_2\text{O}_3/\text{Al}_6\text{Ti}_2\text{O}_{13}$ composite ceramics with low thermal expansion properties are promising for the rapid preparation of large-scale and complex components by directed energy deposition-laser based (DED-LB) technology. However, the wider application of DED-LB technology is limited due to the inadequate understanding of process conditions. The shaping quality, microstructure, and mechanical properties of $\text{Al}_2\text{O}_3/\text{Al}_6\text{Ti}_2\text{O}_{13}$ (6 mol% TiO$_2$) composite ceramics were systematically investigated as a function of energy input in an extensive process window. On this basis, the formation mechanism of solidification defects and the evolution process of microstructure were revealed, and the optimized process parameters were determined. Results show that high energy input improves the fluidity of the molten pool and promotes the uniform distribution and full growth of constituent phases, thus, facilitating the elimination of solidification defects, such as pores and strip gaps. In addition, the microstructure size is strongly dependent on the energy input, increasing when the energy input increases. Moreover, the morphology of the $\alpha$-$\text{Al}_2\text{O}_3$ phase gradually transforms from cellular into cellular dendrite with increasing energy input due to changing solidification conditions. Under the comprehensive influence of solidification defects and microstructure size, the fracture toughness and flexural strength of $\text{Al}_2\text{O}_3/\text{Al}_6\text{Ti}_2\text{O}_{13}$ composite ceramics present a parabolic law behavior as the energy input increases. Optimal shaping quality and excellent mechanical properties are achieved at an energy input range of 0.36–0.54 W$\cdot$min$^2$ g$^{-1}$ mm$^{-1}$. Within this process window, the average microhardness, fracture toughness, and flexural strength of $\text{Al}_2\text{O}_3/\text{Al}_6\text{Ti}_2\text{O}_{13}$ composite ceramics are up to 1640 Hv, 3.87 MPa m$^{1/2}$, and 227 MPa, respectively. This study provides practical guidance for determining the process parameters of DED-LB of melt growth $\text{Al}_2\text{O}_3/\text{Al}_6\text{Ti}_2\text{O}_{13}$ composite ceramics.

Keywords: additive manufacturing, $\text{Al}_2\text{O}_3$, composite ceramics, microstructure, mechanical properties

* Author to whom any correspondence should be addressed.

Original content from this work may be used under the terms of the Creative Commons Attribution 3.0 licence. Any further distribution of this work must maintain attribution to the author(s) and the title of the work, journal citation and DOI.
1. Introduction

In order to improve thermal efficiency and reduce pollutant emissions such as NOx for aeroengine and gas turbine systems, an urgent demand has been imposed for developing a new generation of ultra-high-temperature structural materials that can remain stable in aggressive environments at elevated temperatures [1–3]. More recently, Al2O3-based melt growth ceramics (MGCs) have attracted extensive attention for their superior high-temperature performance, inherent thermochemical stability, and outstanding oxidation resistance at temperatures close to their melting point under oxidizing environments [2–6]. Waku et al [2] found that Al2O3/GdAlO3 and Al2O3/Y3Al5O12 MGCs had no marked microstructure coarsening and mass gain after heat treatment in an air environment at 1700 °C for 500 h. The flexural strength of Al2O3/GdAlO3 [4] and Al2O3/Er3Al5O12/ZrO2 [5] MGCs was maintained at approximately 700 MPa from room temperature to 1600 °C. The value of Al2O3/Y3Al5O12/ZrO2 [6] reached 860 MPa. Additionally, the density of Al2O3-based ceramics (<5 g cm⁻³) [7] is much lower than that of nickel-based and cobalt-based superalloys (>8 g cm⁻³) [8–10], which is beneficial to improve the thrust-to-weight ratio of aeroengines. Naturally, Al2O3-based MGCs possess fascinating application potential in the aerospace industry.

The main preparation methods of Al2O3-based MGCs, including the Bridgman method [4–6], edge-defined film-fed growth [11], and the laser floating zone method [12, 13], are difficult to meet comprehensive requirements of high density, high toughness, and complex shape. These methods usually have long preparation periods, consume high amounts of energy, and can only obtain simple block structures similar to crucible shape. Therefore, it is imperative to develop effective near-net shaping techniques for high-efficiency and high-quality preparation of Al2O3-based MGCs. The development of laser additive manufacturing (LAM), such as selective laser melting [14, 15] and directed energy deposition-laser based (DED-LB) technology [16, 17], provides new possibilities for the rapid preparation of high-performance ceramics with complex shapes. During the LAM process, a molten pool is first formed by irradiating ceramic powders with a high-energy laser beam. Subsequently, the microstructure with a clean phase interface and strong interfacial bonding directly nucleates and grows from the ceramic melt. The unique interfacial bonding characteristic of Al2O3-based MGCs imparts excellent ultra-high-temperature performance close to their melting point [12, 18]. Moreover, the melt solidification process of LAM technology gives Al2O3-based MGCs more compact, unique microstructure and better mechanical properties [18, 19]. Furthermore, the bottom-up and layer-by-layer deposition methods rid LAM of mold restrictions, implying the feasibility of rapidly preparing ceramics with arbitrary shapes. However, the LAM process generates extremely high thermal stress due to its high cooling rate (10⁶ K s⁻¹ [16]) and temperature gradient (10⁴ K cm⁻¹ [20]). The original solidification defects with high stress concentration easily expand and form macro cracks under the action of thermal stress, leading to cracking or even scrapping of Al2O3-based MGCs.

In order to alleviate the cracking problem of Al2O3-based MGCs, aluminum titanate (Al1Ti5O12, AT) was doped during LAM shaping Al2O3 ceramics to reduce thermal stress in our previous work [21]. Fortunately, the doping of AT (0.5 × 10⁻⁶ °C⁻¹) with low thermal expansion coefficient (TEC) effectively reduced the TEC of Al2O3 ceramics, which was conducive to the reduction of thermal stress [21]. The local residual stress and microcracks generated by the thermal expansion mismatch between Al2O3 and AT are beneficial to improve crack propagation and thermal shock resistance in Al2O3-based MGCs [22–24]. Therefore, Al2O3/Al1Ti5O12 (A/AT) was selected as the target material for LAM shaping large-scale and crack-free ceramics. Additionally, the cracking of Al2O3-based MGCs is related to the original solidification defects, which are mainly regulated by the solidification behavior of molten pool. As a highly nonlinear additive manufacturing technology, the solidification behavior including temperature gradient and solidification rate during the LAM shaping process depends on the process conditions [25–27]. The optimization of important process parameters, such as scanning speed [25, 26] and laser power [27], was expected to significantly reduce solidification defects and obtain ideal microstructure and mechanical properties. However, there are few current studies on the preparation of A/AT MGCs by LAM. Furthermore, the influence mechanism of energy input on the microstructure evolution and solidification process of A/AT MGCs prepared by LAM has not been systematically studied. It is difficult to achieve the advantage of LAM technology for tailoring microstructure and obtaining high-performance, crack-free ceramics by adjusting the solidification behavior of molten pool.

For the current research needs, dense A/AT MGCs were directly produced in a single step by DED-LB technology with Al2O3 and TiO2 as the raw materials, providing a new option for the rapid preparation of A/AT MGCs. The relationship between energy input and shaping quality, microstructure characteristics, and mechanical properties of the solidified ceramics was further investigated on the basis of [21]. Furthermore, the formation mechanism of solidification defects and the microstructure evolution process were analyzed, providing necessary theoretical support for the preparation of high-performance A/AT MGCs by DED-LB.

2. Material and methods

The DED-LB system used in the experiment is depicted in figure 1. It mainly consists of an Nd:YAG continuous laser (GSI Lumonics, JK1002), a three-cylinder powder feeder (Beijing Aeronautical Manufacturing Engineering Research Institute, DPSF-3D), a CNC machine (RESUM, LP7002), and a circulating cooling water system. During the DED-LB process, high-purity argon (99.99%) with a gas flow rate of about 0.4 l min⁻¹ was selected as the powder feeding and shielding gas. Commercially pure (>99%) Al2O3 (BESTRY, BAK-0700) and TiO2 (Sunspraying, SY-514)
spherical powders, with a diameter of 40–90 µm, were weighted in the proportion of Al₂O₃:TiO₂ = 94 mol:6 mol [28]. Prior to DED-LB processing, the starting powders were dried in an oven at 120 °C for 4 h to remove moisture so as to ensure their fluidity. Subsequently, the dried powders were evenly mixed by a mechanical mixing method and placed in the powder feeder.

Commercial Al₂O₃ ceramic plate (XiYuan, XY-95), with a size of 150 mm × 100 mm × 25 mm³, was selected as the shaping substrate due to its high physical compatibility with the deposited material. At the beginning of shaping, a laser beam was transmitted through an optical fiber to either the substrate or the most recently deposited layer. Under the irradiation of laser beam, the substrate or deposited layer was rapidly melted, and an initial molten pool was formed. The mixed ceramic powders, which were continuously and steadily supplied during deposition process, were captured for the growth of the molten pool. With the relative circumferential cladding movement (Φ 4 mm) between the nozzle and substrate, the molten pool continuously underwent forming-growing-solidification until the solidified ceramics reached the design height. In the deposition process, the nozzle immediately raised a layer increment (ΔZ) after depositing a layer of material to ensure that the spot was in a stable focusing state of about 2 mm. The comparison parameter $S = P/VQ$ [29] is suitable for energy input evaluation in the DED-LB process. In this study, five groups of laser power, $P$, were set to correspond to different $S$ values, as shown in table 1. The other process parameters were scanning speed, $V = 300$ mm min⁻¹, layer increment, $ΔZ = 0.4$ mm, and powder feeding rate, $Q = 1.86$ g min⁻¹.

The transverse and longitudinal sections of the solidified ceramics were treated with the conventional metallographic technique. Phase analyses of the solidified ceramics were detected by x-ray diffraction (XRD, panalytical B.V., empyrean) and energy dispersive spectrometer (EDS). The Co Kα (30 kV, 50 mA) was selected for the test with a step size of 0.02° and a scanning range of 20–90°. The rapidly solidified microstructure characteristics were analyzed by scanning electron microscopy (SEM, Zeiss, Supra55) equipped with EDS. The microhardness of the solidified ceramics was performed using a Vickers hardness tester (HUAYIN, DHVS-1000A) with an indentation load of 9.8 N and a holding time of 15 s. At least ten valid indentations were made for each measurement on the polished transverse and longitudinal sections. The fracture toughness was assed using the indentation technique. The crack lengths were observed by SEM and measured by Image Pro Plus. The flexural strength of the cylindrical solidified ceramics was measured in a three-point fixture (INSTRON, INSTRON 2344) with a 30 mm loading span and a crossbeam speed of 0.5 mm min⁻¹. Five samples were selected for each measurement and the flexural strength was calculated using equation (1) [30]:

$$σ_f = \frac{8FL}{πd^3}$$  \hspace{1cm} (1)

where $σ_f$ is the flexural strength, $F$ is the maximum load, $L$ is the length of the loading span, and $d$ is the diameter of tested solidified ceramics.

![Diagram of DED-LB system.](image)

**Table 1. Laser power and energy input.**

| $P$(W) | 150 | 200 | 250 | 300 | 350 |
|-------|-----|-----|-----|-----|-----|
| $S/(W\cdot min^-2 \cdot g^-1 \cdot mm^-1)$ | 0.27 | 0.36 | 0.45 | 0.54 | 0.63 |
3. Results and discussion

3.1. Shaping quality of DED-LB A/AT MGCs

Figure 2 presents the typical macro morphology of DED-LB A/AT cylindrical MGCs solidified at different energy inputs. Due to the technological characteristics of layer-by-layer deposition, the surface of the A/AT MGCs exhibit typical interlayer bonding characteristics. No crack defects were observed on the surface of the A/AT MGCs, indicating that the doping of AT with low TEC suppresses cracks effectively. The increased energy input nearly doubled the diameter, d, of the cylindrical A/AT MGCs (table 2). Additionally, the surface finish of the A/AT MGCs improved significantly with increasing energy input, while the shape precision, in this case, the cylindricity, was worse.

When the energy input is 0.27 W min⁻² g⁻¹ mm⁻¹, only a small stable molten pool with relatively low temperature was maintained. The molten pool’s width was smaller than the diameter of the ceramic powder flow focus. During the DED-LB process, the powders near the molten pool are inevitably captured and absorbed. However, the laser beam radiated to the molten pool fails to completely melt the ceramic powders adhering to the edge. Therefore, there are a large number of unmelted powder particles attached to the surface of the A/AT MGCs, which significantly affects the surface finish. Moreover, the low temperature molten pool is accompanied by high viscosity, large surface tension, poor fluidity, and poor wettability. Consequently, the ceramic melt does not spread well on the deposited layer, resulting in cylindrical A/AT MGCs with diameters of only 3.57 mm. After the energy input was increased to 0.36–0.45 W min⁻² g⁻¹ mm⁻¹, the increase in temperature reduced the viscosity and surface tension of molten pool, which improved its fluidity and wettability. As a result, the width of the molten pool increased and more ceramic powders were captured, giving the solidified ceramics a larger diameter. However, a few unmelted powder particles remained on the surface of the A/AT MGCs despite the increased energy input. With a further increase in energy input, the spreading ability of the melt improved and the width of molten pool increased. Naturally, the diameter of cylindrical A/AT MGCs increases further until it reaches 6.82 mm when the energy input is 0.63 W min⁻² g⁻¹ mm⁻¹. In addition, the improved surface finish indicates that the ceramic powders adhering to the molten pool edge were fully melted under such a high energy input. Unfortunately, the improvement in fluidity was not conducive to shaping the melt, so A/AT MGCs that solidify under high energy input conditions (0.54–0.63 W min⁻² g⁻¹ mm⁻¹) have poor cylindricity.

3.2. Solidification defects of DED-LB A/AT MGCs

Pores are one of the major solidification defects in most DED-LB MGCs. Pores are divided into gas cavities and shrinkage cavities, both of which are potential crack sources. The existence of pores easily induces the nucleation and propagation of macro cracks and seriously weaken the mechanical properties of MGCs [31]. Although argon, as shielding gas, helps reduce porosity [32], the high viscosity and gas solubility of ceramic melts make it impossible for the molten pool to completely avoid gas absorption [33]. A portion of gas fails to escape in time due to the characteristics of rapid solidification, which is responsible for the pore defects in DED-LB MGCs. Three samples were selected to calculate the porosity. Figure 3 illustrates the distribution of pores in A/AT MGCs, the variation in porosity, and the corresponding standard deviation with energy input.

Energy input in the DED-LB process significantly influences pore characteristics. When the energy input ranged between 0.27 and 0.36 W min⁻² g⁻¹ mm⁻¹, the porosity

![Figure 2](image-url)
of A/AT MGCs exceeded 3.6%. Pores with diameters larger than 500 µm were observed in the solidified ceramics. The observed pores included regular gas cavities with smooth inner walls and irregular shrinkage cavities with rough inner walls. The shrinkage cavities were distributed in the center of the rod-shaped structure, and a large number of small gas cavities with diameters less than 100 µm diffused towards the edge, as shown in figures 3(a) and (b). The porosity dropped dramatically to 0.44% at an energy input of 0.45 W min$^{-2}$ g$^{-1}$ mm$^{-1}$. The large pore defects disappeared, the diameter of small gas cavities decreased to less than 50 µm, and the small gas cavities displayed distinct edge distribution characteristics, as shown in figure 3(c). No obvious pore defects were detected after further increasing the energy input (0.54–0.63 W min$^{-2}$ g$^{-1}$ mm$^{-1}$). Only a few small gas cavities with a diameter of about 30 µm were observed at the edge of the rod-shaped structure, and the porosity was stable at about 0.05%.

In order to clearly explain the distribution characteristics of the pores and the variation of porosity, it is necessary to clarify the formation mechanism of pores during DED-LB deposition. Figure 4 is a simplified schematic diagram of the gas cavity formation process. During the DED-LB deposition process, the molten pool absorbs and dissolves the gas hidden in the ceramic powders. Since the specific free energy is higher between solid/gas than between solid/liquid and liquid/gas, gas tends to dissolve in liquids \([34]\). With the nucleation and growth of the solid phase, the gas in the molten pool is squeezed to the liquid phase. Once the concentration reaches a certain level, the gas nucleates and forms bubbles near the solid/liquid interface \([35]\). As shown in figure 4, the bubbles grow continuously through diffusion and coalescence. Until reaching a critical size, $R_{critical}$, the buoyancy and surface tension of bubbles are sufficient to overcome the viscosity resistance of ceramic melt, and the bubbles gradually separate from the solid/liquid interface. In order to reduce the interfacial free
energy, the bubbles migrate from low temperature regions with high surface tension to high temperature liquid regions with low surface tension [36]. In DED-LB, the highest molten pool temperature appears on the top surface [37]. Consequently, the bubbles larger than \( R_{\text{critical}} \) continuously move from the solid/liquid interface toward the top of the molten pool. When the molten pool material is determined, the bubble movement velocity, \( v_c \), depends on its size, \( R \), and the molten pool viscosity, \( \eta \); and the relationship between them is expressed in equation (2) [33]:

\[
v_c = \frac{2gR^2(\rho_m - \rho_b)}{9\eta}
\]

where \( g \) is the gravitational acceleration, \( \rho_m \) is the molten pool density, and \( \rho_b \) is the bubble density. Equation (2) indicates that \( v_c \) is proportional to the square of the bubble size. Naturally, large bubbles formed by diffusion or coalescence are more likely to escape from the molten pool in time. In contrast, some small bubbles, whose movement velocity is lower than the solidification rate of molten pool or even fail to move due to the viscosity resistance, are entrapped in the melt and form gas cavities, as shown in figure 4.

Shrinkage cavities are widely believed to be caused by the untimely filling of the liquid phase during DED-LB rapid solidification [33]. A simplified schematic diagram of the formation process is illustrated in figure 5. In the solidification process of molten pool, the primary phase precipitates first, and the surrounding residual liquid phase adheres to the solid phase for solidification and contraction. In the absence of timely supplement from the liquid phase, the voids generated by solidification and contraction gradually expand into shrinkage cavities as the solidification progresses, as shown in figure 5. Shrinkage cavities usually develop in the center of the rod-shaped structure, as shown in figures 3(a) and (b), which is closely related to the solidification sequence in the molten pool. Due to direct contact with the surrounding environment, the temperature gradient and cooling rate are highest near the edge of molten pool. Conversely, the central region of the molten pool is composed of melt with a similar temperature and has the lowest temperature gradient. Similar conclusions have been obtained in other reports [37]. During the DED-LB shaping process, the melt at the molten pool edge solidifies first and the central region solidifies last. At the initial stage of solidification, the melt at the molten pool edge solidifies rapidly in an environment with sufficient liquid phase and forms a closed region. When the liquid phase has no capacity to fill the entire molten pool before the closed region forms, the voids in the final solidification region, i.e. the central region, cannot be bridged to form shrinkage cavities.

When the energy input ranged between 0.27 W\(^{\ast}\)min\(^{-2}\)g\(^{-1}\)mm\(^{-1}\) and 0.36 W\(^{\ast}\)min\(^{-2}\)g\(^{-1}\)mm\(^{-1}\), the molten pool had small volume, low temperature, and high viscosity, which impeded the bubble movement velocity. Furthermore, because of the short duration of the small volume molten pool, most of the small bubbles moved too slowly to escape before the melt solidification. Under such harsh conditions, even some large bubbles (>500 \( \mu \)m) became trapped in the escape result, resulting in a large number of gas cavities diffused toward the edge of the rod-shaped structure. Shrinkage cavities also formed in the DED-LB A/AT MGCs under low energy inputs (0.27–0.36 W\(^{\ast}\)min\(^{-2}\)g\(^{-1}\)mm\(^{-1}\)). The high viscosity hindered the full flow of melt, and thus the liquid phase failed to replenish in time during the melt solidification in the central region of molten pool. Therefore, the gas cavities and shrinkage cavities coexisted in the solidified ceramics shown in figures 3(a) and (b). In contrast, when the energy input is increased to 0.45 W\(^{\ast}\)min\(^{-2}\)g\(^{-1}\)mm\(^{-1}\), the molten pool has increased volume, improved temperature, reduced viscosity, and extended retention time. The full flow of the melt is conducive to the elimination of shrinkage cavities and escape of gas cavities, which corresponded to a sharp decrease in porosity. In particular, the DED-LB A/AT MGCs were almost considered pore-free after the energy input exceeded 0.54 W\(^{\ast}\)min\(^{-2}\)g\(^{-1}\)mm\(^{-1}\). Even smaller bubbles (30–50 \( \mu \)m) escaped from the molten pool. Only the bubbles smaller than 30 \( \mu \)m with relatively low movement velocity were trapped at the edge of molten pool during the escape process. Correspondingly, the gas cavities were generated and characterized by small size and edge distribution.

3.3. Microstructure evolution of DED-LB A/AT MGCs

3.3.1. Phase composition. The elemental distribution of A/AT MGCs prepared under different energy inputs is displayed in figure 6. The microstructure of all DED-LB A/AT MGCs is composed of a dark discrete phase and a bright matrix phase. The dark phase is an \( \alpha \)-Al\(_2\)O\(_3\) phase, which accounts for the main volume fraction and is rich in Al and O. The bright phase with a small volume proportion is the Ti element enrichment region, corresponding to the AT phase. In
addition, the distribution of elements becomes more uniform with the increase of energy input. On one hand, the molten pool has a longer retention time under high energy input conditions, which means that the diffusion time of solute elements is prolonged. On the other hand, the atomic diffusion force is mainly controlled by the molten pool temperature, $T$, when the material composition is constant, as expressed in the equation (3):

$$D = D_0 \exp\left(-\frac{\Delta E}{kT}\right)$$

where $D$ is the diffusion coefficient, $D_0$ is the diffusion coefficient at 293 K, and $\Delta E$ is the diffusion activation energy. The molten pool has a higher temperature under high energy input conditions, meaning that the solute elements have a faster diffusion rate. Therefore, the solute elements diffuse more fully and distribute more uniformly with the increase in energy input.

The XRD patterns of A/AT MGCs prepared under different energy inputs are presented in figure 7. The sharp and narrow characteristic diffraction peaks indicate that DED-LB A/AT MGCs have excellent crystallinity and crystal integrity. Figure 7(a) illustrates that energy input has little effect on the phase composition of the solidified ceramics, and A/AT MGCs are mainly composed of $\alpha$-$\text{Al}_2\text{O}_3$ phase and AT phase, which is consistent with the EDS results. Interestingly, the characteristic diffraction peaks of the $\alpha$-$\text{Al}_2\text{O}_3$ phase show a phenomenon of moving towards large diffraction angle as the energy input increases, as shown in figure 7(b). As can be seen from figure 6, some Ti$^{4+}$ atoms (0.068 nm), whose atomic radius is larger than an Al$^{3+}$ atom (0.05 nm) [38], dissolve in the $\alpha$-$\text{Al}_2\text{O}_3$ phase, increasing the lattice parameters of the $\alpha$-$\text{Al}_2\text{O}_3$ phase. As mentioned above, since the diffusion
capacity of solute elements increases with increasing energy input, the number of Ti^{4+} atoms dissolved in the α-Al_{2}O_{3} phase decreases. In other words, the lattice parameters of the α-Al_{2}O_{3} phase gradually decrease with the increase in energy input, and the crystal plane spacing also decreases. According to Bragg’s law, a small crystal plane spacing corresponds to a large diffraction angle, which explains the fact that the characteristic diffraction peaks of the α-Al_{2}O_{3} phase move to right.

Figure 8. Effects of energy input on the microstructure of DED-LB A/AT MGCS: (a) 0.27 W^*min^2 g\^{-1} mm\^{-1}, (b) 0.36 W^*min^2 g\^{-1} mm\^{-1}, (c) 0.45 W^*min^2 g\^{-1} mm\^{-1}, (d) 0.54 W^*min^2 g\^{-1} mm\^{-1}, and (e) 0.63 W^*min^2 g\^{-1} mm\^{-1}, respectively (1 stands for longitudinal section, and 2 stands for cross section).
3.3.2. Microstructure evolution. The microstructure of DED-LB A/AT MGCs is closely related to the energy input, as shown in figure 8. The microstructure of the A/AT MGCs prepared under different energy inputs is micron scale and composed of a discretely-distributed dark primary $\alpha$-Al$_2$O$_3$ phase and a network-distributed bright AT matrix phase. When the energy input was less than 0.36 W$^\ast$min$^{-2}$ g$^{-1}$ mm$^{-1}$, the microstructure of the A/AT MGCs presented isotropic growth (figures 8(a1)–(b2)). Several interphase bonding defects, called strip gaps, were distributed between the cellular $\alpha$-Al$_2$O$_3$ phase and the film-like AT matrix phase with typical metallurgical bonding characteristics (figures 8(a1)–(b2)). Moreover, the AT phase with network distribution exhibited discontinuous interruptions in some regions, as shown in the high magnification SEM in figure 8(b1). After the energy input exceeded 0.45 W$^\ast$min$^{-2}$ g$^{-1}$ mm$^{-1}$, the microstructure presented obvious anisotropy. Both phases grew preferentially along the deposition direction, and the aspect ratio $L/W$ of the $\alpha$-Al$_2$O$_3$ phase remained about 1–2. The strip gaps decreased sharply, and the discontinuity of the AT phase disappeared after increasing the energy input (figures 8(c1)–(e2)). With the energy input ranging from 0.45 W$^\ast$min$^{-2}$ g$^{-1}$ mm$^{-1}$ to 0.63 W$^\ast$min$^{-2}$ g$^{-1}$ mm$^{-1}$, more and more $\alpha$-Al$_2$O$_3$ phases developed secondary dendrites and transformed from cellular to cellular dendrite morphology. As shown in figures 8(e1)–(e2), all $\alpha$-Al$_2$O$_3$ phases developed secondary dendrite arms until the energy input reached 0.63 W$^\ast$min$^{-2}$ g$^{-1}$ mm$^{-1}$.

The technological characteristics of rapid melting-solidification endow DED-LB MGCs’ microstructure with typical non-equilibrium solidification characteristics; therefore, the solid/liquid interface is prone to instability and disturbance during solidification. However, the extremely high temperature gradient restricts the composition supercooling, so that the A/AT MGCs’ microstructure generally appears as cellular structures. According to the Al$_2$O$_3$-TiO$_2$ binary phase diagram [39], the $\alpha$-Al$_2$O$_3$ phase precipitates first during DED-LB solidification process, while the Ti element is squeezed into the residual liquid phase in the molten pool. Under the influence of composition supercooling, the planar interface becomes unstable and extends into the liquid phase in the form of cellular bulges, as shown in the simplified schematic diagram (figure 9). After the primary $\alpha$-Al$_2$O$_3$ phase bulges, the solutes discharged from the solid phase tend to drain away from the top of cellular bulges with good diffusion conditions, leading to significant solute concentration and composition supercooling reduction on both sides. In contrast, the top of the $\alpha$-Al$_2$O$_3$ phase bulge, located in the supercooled region, has a higher growth rate and forms a stable convex shape. Therefore, the primary $\alpha$-Al$_2$O$_3$ phase should theoretically grow into a directional cellular structure parallel to the direction of the heat flow. As the solidification progresses, Ti elements in the residual liquid phase gradually enrich and induce a peritectic reaction ($3\alpha$-Al$_2$O$_3$ + 2TiO$_2$→Al$_6$Ti$_2$O$_13$) to form the AT phase. Therefore, the AT phase is pushed to the phase boundary by the growing $\alpha$-Al$_2$O$_3$ phase, and finally presents a network distribution.

The energy input has a marked influence on the solidification behavior of the molten pool, so the growth and distribution characteristics of constituent phases are sensitive to changes in energy input. As mentioned above, the growth direction of the crystal is usually inversely parallel to the heat flow direction. The main heat dissipation mode during DED-LB solidification is bottom-to-top thermal conduction. Therefore, the microstructure should show clear directional growth along the deposition direction. However, the molten pool with an energy input lower than 0.36 W$^\ast$min$^{-2}$ g$^{-1}$ mm$^{-1}$ had poor fluidity and short duration, resulting in the incomplete development of a primary $\alpha$-Al$_2$O$_3$ phase. Therefore, the cellular $\alpha$-Al$_2$O$_3$ phases in figures 8(a1)–(b2) are isotropic rather than directional growth. When the energy input...
exceeded 0.45 W·min$^2$·g$^{-1}$·mm$^{-1}$, the aspect ratio of the primary $\alpha$-Al$_2$O$_3$ phase increased and exhibited an anisotropic growth characteristic (figures 8(c1)–(e2)). On the one hand, the increase in energy input improved the fluidity and retention time of molten pool, which provided favorable conditions for the full development of primary $\alpha$-Al$_2$O$_3$ phase. On the other hand, there was a serious heat accumulation phenomenon due to the remelting of the previous deposition layer during the DED-LB process. The heat accumulation intensified with increasing energy input, leading to a decrease in the temperature gradient between adjacent deposited layers. Under the condition of a certain solidification rate, the decrease in temperature gradient intensified the composition supercooling degree at the front of solid/liquid interface [40]. The intensified composition supercooling induced cellular bulges that extended further into the liquid phase, which promoted the directional growth of the primary $\alpha$-Al$_2$O$_3$ phase along the deposition direction. In addition, the transformation of the $\alpha$-Al$_2$O$_3$ phase morphology with energy input was also highly correlated with composition supercooling. The high transverse solute diffusion rate caused by large composition supercooling stimulated the generation of secondary dendrite arms, resulting in the transformation of the cellular $\alpha$-Al$_2$O$_3$ phase into a cellular dendrite morphology with a hexagonal substructure. As a result, the $\alpha$-Al$_2$O$_3$ phase experienced an evolution of cellular (figures 8(a1)–(b2))-cellular coexisting with cellular dendrite (figures 8(c1)–(d2))-cellular dendrite (figures 8(e1)–(e2)) when the energy input increased from 0.27 W·min$^2$·g$^{-1}$·mm$^{-1}$ to 0.63 W·min$^2$·g$^{-1}$·mm$^{-1}$. Moreover, the secondary dendrite arms of the $\alpha$-Al$_2$O$_3$ phase continued developing, which was attributed to the prolonged retention time and improved fluidity of the molten pool under high energy input conditions.

The energy input also has a decisive influence on the distribution continuity of the AT matrix phase and the bonding condition between the $\alpha$-Al$_2$O$_3$ and AT phases. According to equation (3), the diffusion capacity of solute elements mainly depends on the molten pool temperature. Under low energy inputs (0.27–0.36 W·min$^2$·g$^{-1}$·mm$^{-1}$), the molten pool temperature was low and the diffusion of Ti element was limited. The inadequate diffusion of Ti element in the residual liquid phase leads to the inhomogeneous peritectic reaction, thus ‘discontinuous’ appears in local regions of the AT phase. Additionally, the viscosity of the melt in the low temperature molten pool was large, which was not conducive to the improvement of molten pool fluidity. In the DED-LB process, poor fluidity means that the residual liquid phase between adjacent deposited layers. Naturally, the microstructure size of energy input conditions was measured using the circle interception method to evaluate the influence of energy input on the microstructure size. The measurement results are plotted in figure 10.

With the increase in energy input, the size of the primary $\alpha$-Al$_2$O$_3$ phase increased significantly. The $\alpha$-Al$_2$O$_3$ phase size increased sharply from 20.3 $\mu$m at 0.27 W·min$^2$·g$^{-1}$·mm$^{-1}$ to 30.79 $\mu$m at 0.63 W·min$^2$·g$^{-1}$·mm$^{-1}$, corresponding to an increase of 50.67%. According to the solidification theory, the smaller the product of the temperature gradient, $G$, and the solidification rate, $V_s$, the larger the microstructure size. As described in section 3.3.2, the intensified heat accumulation with increased energy input reduces the temperature gradient between adjacent deposited layers. Naturally, the microstructures of A/AT MGCs that solidify under high energy input are coarser. Moreover, the prolonged duration of the molten pool under high energy input conditions allows the $\alpha$-Al$_2$O$_3$ phase to grow fully, which increases the phase size.

3.3.3. Microstructure size. The microstructure size, which significantly affects the crack propagation and the original defect size, is an important factor for determining the mechanical properties of DED-LB MGCs. Obviously, the volume of the primary $\alpha$-Al$_2$O$_3$ phase is absolutely dominant, which is the main factor controlling the performance of A/AT MGCs. Consequently, the size of the $\alpha$-Al$_2$O$_3$ phase was measured using the circle interception method to evaluate the influence of energy input on the microstructure size. The measurement results are plotted in figure 10.

With the increase in energy input, the size of the primary $\alpha$-Al$_2$O$_3$ phase increased significantly. The $\alpha$-Al$_2$O$_3$ phase size increased sharply from 20.3 $\mu$m at 0.27 W·min$^2$·g$^{-1}$·mm$^{-1}$ to 30.79 $\mu$m at 0.63 W·min$^2$·g$^{-1}$·mm$^{-1}$, corresponding to an increase of 50.67%. According to the solidification theory, the smaller the product of the temperature gradient, $G$, and the solidification rate, $V_s$, the larger the microstructure size. As described in section 3.3.2, the intensified heat accumulation with increased energy input reduces the temperature gradient between adjacent deposited layers. Naturally, the microstructures of A/AT MGCs that solidify under high energy input are coarser. Moreover, the prolonged duration of the molten pool under high energy input conditions allows the $\alpha$-Al$_2$O$_3$ phase to grow fully, which increases the phase size.

3.4. Mechanical properties of DED-LB A/AT MGCs

3.4.1. Microhardness and fracture toughness. The SEM image of the typical Vickers indentation is displayed in figure 11(a). Since the microhardness is mainly related to the physical properties of material itself, the microhardness of A/AT MGCs is almost independent of the energy input when determining the constituent phases. The microhardness of both sections ranging from 1400 Hv to 1600 Hv (figure 11(d)), much higher than that of sintered A/AT composite ceramics [41]. Especially, the microhardness of cross- and longitudinal-sections were similar when the energy input was less than 0.36 W·min$^2$·g$^{-1}$·mm$^{-1}$, while the
Figure 11. Microhardness and fracture toughness of A/AT MGCs: (a) typical indentation, (b) and (c) crack propagation path and inhibition mechanism, and (d) variation trend of microhardness and fracture toughness with increasing energy input (L stands for longitudinal section, and T stands for cross section).

Microhardness of longitudinal-section was slightly higher than that of cross-section when the energy input exceeded 0.45 W min$^{-2}$ g$^{-1}$ mm$^{-1}$. As shown in figures 8(c1)–(e2), when the energy input was between 0.45 W min$^{-2}$ g$^{-1}$ mm$^{-1}$ and 0.63 W min$^{-2}$ g$^{-1}$ mm$^{-1}$, the microstructure grew directionally along the deposition direction. The deformation failure and crack propagation direction caused by indentation on the longitudinal section were perpendicular to the growth direction of the microstructure, so the sizes of the indentation and crack were relatively small and the microhardness was relatively high. In contrast, when the energy input was between 0.27 W min$^{-2}$ g$^{-1}$ mm$^{-1}$ and 0.36 W min$^{-2}$ g$^{-1}$ mm$^{-1}$ (figures 8(a1)–(b2)), the microstructure shows isotropic growth characteristics, so the microhardness of cross- and longitudinal-sections was essentially the same.

Figure 11(a) reveals that the radial cracks in the four corners of the Vickers indentation were mainly transgranular fractures that terminated in the AT matrix (marked by white circles). The reason is that the AT matrix, with low TEC, was subjected to compressive stress during solidification, which had a healing effect on the crack tips, as described in figure 11(b). Furthermore, there are microcracks, induced by intercrystalline stress, developed in the AT phase. Similar findings have been reported by Cleveland et al [42] and Bueno et al [43]. Microcracks are conducive to promoting crack branching and deflection, thus effectively reducing the driving force of crack propagation and improving the fracture toughness, as shown in figures 11(b) and (c). Figure 11(c) shows the crack bridge, branching, and deflection phenomena along the crack propagation path, which are beneficial to the consumption of propagation energy and crack suppression.

The crack and indentation dimensions, measured by Image Pro Plus, satisfy the relationship of $0.25 \leq (c-a)/a \leq 2.5$ (where $a$ is the indent diagonal and $c$ is the radial crack size), indicating that the indentation crack is a Palmqvist crack. Hence, the fracture toughness, $K_C$, was calculated by the Niihara formula [44], and the result is shown in figure 11(d). With the increase in energy input, the fracture toughness of both sections presented a parabolic trend and reached a maximum value of 3.87 MPa m$^{1/2}$ and 3.27 MPa m$^{1/2}$ at $S = 0.54$ W min$^{-2}$ g$^{-1}$ mm$^{-1}$, respectively. Similar to microhardness, the microstructure growth characteristic depends on the energy input and leads to differences in fracture toughness between cross- and longitudinal-sections. The fracture toughness of cross- and longitudinal-sections were similar under low energy inputs (0.27–0.36 W min$^{-2}$ g$^{-1}$ mm$^{-1}$), but the fracture toughness of longitudinal-section was higher under high energy inputs (0.45–0.63 W min$^{-2}$ g$^{-1}$ mm$^{-1}$). The specific analysis was mentioned above.

The solidification defects and microstructure size are the main factors affecting the fracture toughness of A/AT MGCs. Figure 3 shows that the porosity of the A/AT MGCs reached 6.54% at an energy input of 0.27 W min$^{-2}$ g$^{-1}$ mm$^{-1}$. As
the flexural strength, \( \sigma \), of A/AT MGCs is plotted as a function of energy input was between 0.36 W and 0.63 W. The fracture toughness of the A/AT MGCs was higher when the energy input ranged between 0.36 W and 0.63 W. When the energy input was between 0.36 W and 0.63 W, the porosity remained nearly unchanged when the energy input reached 0.63 W. The porosity decreased dramatically and the strip gap defects almost disappeared. In contrast, the increment of the microstructure size was small, so the flexural strength significantly increased to more than 200 MPa. The porosity remained nearly unchanged when the energy input reached 0.63 W. The porosity decreased dramatically and the strip gap defects almost disappeared. In contrast, the increment of the microstructure size was small, so the flexural strength significantly increased to more than 150 MPa.

4. Conclusions

Crack-free melt-growth Al\(_2\)O\(_3\)/Al\(_3\)Ti\(_2\)O\(_3\) composite ceramics were prepared by DED-LB technology. The effects of energy input on the shaping quality, microstructure, and mechanical properties of A/AT MGCs were systematically studied in a large process window. On this basis, the solidification defects formation mechanism and microstructure evolution process were revealed, and the process window for obtaining A/AT MGCs with excellent comprehensive properties was determined. The specific conclusions are as follows:

(a) A/AT MGCs had no macroscopic crack defects, indicating that doping AT with low TEC has crack suppression effects. With the increase in energy input, the improved fluidity significantly improved the surface finish of A/AT MGCs, while the shape precision gradually decreased. When the energy input was between 0.36 W and 0.63 W, the shaping quality of A/AT MGCs, including dimensional precision, shape precision, and surface finish, was maintained at a high level.

(b) The characteristic diffraction peaks of the \( \alpha \)-Al\(_2\)O\(_3\) phase move to the right with increasing energy input due to the decrease of lattice parameters. However, all of the A/AT MGCs were mainly composed of an \( \alpha \)-Al\(_2\)O\(_3\) phase and an AT phase. This means that the energy input had an almost negligible effect on the phase composition. Conversely, the energy input significantly influenced the microstructure of the A/AT MGCs. The fluidity of the molten pool increased with increasing energy input, and the discontinuity of the film-like AT phase with network distribution was

\[
\sigma_f = \sigma_0 \exp (-\alpha p)
\]  

where \( \sigma_0 \) is the flexural strength at porosity \( p = 0 \), and \( \alpha \) is a constant between 4 and 7. Zhou et al. pointed out that the relationship between the strength and microstructure size of ceramics is similar to that of metals, which conforms to the Hall–Petch relationship:

\[
\sigma_f = \sigma_c + C d_0^{-1/2}
\]

where \( \sigma_c \) is the strength of an infinite single crystal, \( d_0 \) is the microstructure size, and \( C \) is a constant. Studies on polycrystalline TiO\(_2\) and Al\(_2\)O\(_3\) ceramics prove that the flexural strength is remarkably improved when the microstructure size decreases.

Although the phase size was small when the energy input was 0.27 W, the microstructure size, and 0.54 W, the porosity decreased dramatically and the strip gap defects almost disappeared. In contrast, the increment of the microstructure size was small, so the flexural strength significantly increased to more than 200 MPa. The porosity remained nearly unchanged when the energy input reached 0.63 W. The porosity decreased dramatically and the strip gap defects almost disappeared. In contrast, the increment of the microstructure size was small, so the flexural strength significantly increased to more than 150 MPa.
eliminated. The dispersed primary $\alpha$-Al$_2$O$_3$ phase transformed from the cellular morphology of isotropic growth to the cellular dendrite morphology of directional growth along the deposition direction, which was mainly attributed to the decrease of the temperature gradient caused by the increase of energy input.

(c) The microhardness of A/AT MGCs was independent of energy input, while the fracture toughness and flexural strength were highly sensitive to energy input. On the one hand, the improved fluidity and prolonged duration of the molten pool reduced solidification defects, such as pores and strip gaps, which improved the mechanical properties. On the other hand, the resulting coarsening of the $\alpha$-Al$_2$O$_3$ phase had a negative effect on the mechanical properties. Since the microstructure was relatively fine and the solidification defects almost disappeared, A/AT MGCs exhibited excellent comprehensive properties when the energy input was between 0.36 and 0.54 W mm$^{-2}$ g$^{-1}$ mm$^{-1}$.

This study revealed the relationship between energy input, an important process condition, and the microstructure and properties of DED-LB MGCs, and provided guidance for the selection and optimization of process parameters in the preparation of DED-LB A/AT MGCs.

Acknowledgments

This work was funded by the National Natural Science Foundation of China (Nos. 51805070, 51790172), and the Fundamental Research Funds for the Central Universities (No. DUT19RC(3)060).

ORCID iD

Fangyong Niu https://orcid.org/0000-0002-9403-2761

References

[1] Mazerolles L, Perriere L, Lartigue-Korinek S, Piquet N and Parlier M 2008 Microstructures, crystallography of interfaces, and creep behavior of melt-growth composites J. Eur. Ceram. Soc. 28 2301–8

[2] Nakagawa N, Ohtsubo H, Mitani A, Shimizu K and Waku Y 2005 High temperature strength and thermal stability for melt growth composite J. Eur. Ceram. Soc. 25 1251–7

[3] Hirano K 2005 Application of eutectic composites to gas turbine system and fundamental fracture properties up to 1700°C J. Eur. Ceram. Soc. 25 1191–9

[4] Waku Y, Nakagawa N, Nakamoto T, Ohtsubo H, Shimizu K and Kohtoku Y 1997 A ducile ceramic eutectic composite with high strength at 1.873 K Nature 389 49–52

[5] Waku Y, Sakata S, Mitani A, Shimizu K, Ohtsuka A and Hasebe M 2005 Microstructure and high-temperature strength of Al$_2$O$_3$/Er$_5$Al$_{12}$O$_{37}$ZrO$_2$ ternary melt growth composite J. Mater. Sci. 40 711–7

[6] Waku Y, Sakata S, Mitani A, Shimizu K and Hasebe M 2002 Temperature dependence of flexural strength and microstructure of Al$_2$O$_3$/Y$_2$O$_3$/Al$_2$O$_3$ZrO$_2$ ternary melt growth composites J. Mater. Sci. 37 2975–82

[7] Song C Y, Wang S H, Liu J C and Zhai S Y 2018 Effect of solidification process on microstructure and properties of Al$_2$O$_3$/Er$_5$Al$_{12}$O$_{37}$ZrO$_2$ eutectic ceramic Ceram. Int. 44 17407–14

[8] Liu Y H, Ning Y Q, Yang X M, Yao Z K and Guo H Z 2016 Effect of temperature and strain rate on the workability of FGH4096 superalloy in hot deformation Mater. Des. 95 669–76

[9] Liu P, Huang H Y, Antonov S, Wen C, Xue D Z, Chen H W, Li H W, Feng Q, Omori T and Su Y J 2020 Machine learning assisted design of γ'-strengthened Co-base superalloys with multi-performance optimization npj Comput. Mater. 6 62

[10] Menou E, Rame J, Desgranges C, Ramstein G and Tancret F 2019 Computational design of a single crystal nickel-based superalloy with improved specific creep endurance at high temperature Comput. Mater. Sci. 170 109194

[11] Carroz L and Duffar T 2018 Tuning the sapphire EFG process to the growth of Al$_2$O$_3$/YAG/ZrO$_2$:Y eutectic J. Cryst. Growth 489 5–10

[12] Huamán-Mamani F A, Jiménez-Melendo M, Mesa M C and Oliete P B 2012 Microstructure and high-temperature mechanical behavior of melt-growth Al$_2$O$_3$/Er$_5$Al$_{12}$O$_{37}$ZrO$_2$ ternary eutectic composites J. Alloys Compd. 536 5527–31

[13] Mesa M C, Serrano-Zabaleta S, Oliete P B and Larrea A 2014 Microstructural stability and orientation relationships of directionally solidified Al$_2$O$_3$/Er$_5$Al$_{12}$ZrO$_2$ eutectic ceramics up to 1600°C J. Eur. Ceram. Soc. 34 2071–80

[14] Liu H F, Su H J, Shen Z L, Wang E Y, Zhao D, Guo M, Zhang J, Liu L and Fu H Z 2018 Direct formation of Al$_2$O$_3$/GdAlO$_3$/ZrO$_2$ ternary eutectic ceramics by selective laser melting: microstructure evolutions J. Eur. Ceram. Soc. 38 5144–52

[15] Zhang C, Zhu J K, Zheng H, Li H, Liu S and Cheng G J 2020 A review on microstructures and properties of high entropy alloys manufactured by selective laser melting Int. J. Extreme Manuf. 2 032003

[16] Balia V K, Bose S and Bandypadhyay A 2008 Processing of bulk alumina ceramics using laser engineered net shaping Int. J. Appl. Ceram. Technol. 5 234–42

[17] Niu F Y, Wu D J, Zhou S Y and Ma G Y 2014 Power prediction for laser engineered net shaping of Al$_2$O$_3$ ceramic parts J. Eur. Ceram. Soc. 34 3811–7

[18] Mesa M C, Oliete P B, Pastor J Y, Martín A and LLorca J 2014 Mechanical properties up to 1900 K of Al$_2$O$_3$/Er$_5$Al$_{12}$O$_{37}$ZrO$_2$ eutectic ceramics grown by the laser floating zone method J. Eur. Ceram. Soc. 34 2081–7

[19] Ester F J, Larrea A and Merino R I 2011 Processing and microstructural study of surface laser remelted Al$_2$O$_3$-YSZ-YAG eutectic plates J. Eur. Ceram. Soc. 31 1257–68

[20] Yan S, Wu D J, Niu F Y, Ma G Y and Kang R K 2017 Al$_2$O$_3$/ZrO$_2$ eutectic ceramic via ultrasonic-assisted laser engineered net shaping Ceram. Int. 43 15905–10

[21] Niu F Y, Wu D J, Huang Y F, Yan S, Ma G Y, Li C J and Ding J 2019 Direct additive manufacturing of large-sized crack-free alumina/aluminum titanate composite ceramics by directed laser deposition Rapid Prototyp. J. 25 1370–8

[22] Ohya Y, Yamamoto S, Ban T, Tanaka M and Kitaoka S 2017 Thermal expansion and mechanical properties of self-reinforced aluminum titanate ceramics with elongated grains J. Eur. Ceram. Soc. 37 1673–80

[23] Padture N P, Bennison S J and Chan H M 1993 Flaw-tolerance and crack-resistance properties of melt-grown alumina-aluminum titanate composites with tailored microstructures J. Am. Ceram. Soc. 76 2312–20
Yan S, Huang Y F, Zhao D K, Niu F Y, Ma G Y and Wu D J 2008 Directional solidification of $\text{Al}_2\text{O}_3$ ceramics JOM 69 557–62

Huang Y F, Wu D J, Zhao D K, Niu F Y, Zhang H, Yan S and Ma G Y 2020 Process optimization of melt growth alumina/aluminum titanate composites directed energy deposition: effects of scanning speed Addit. Manuf. 35 101210

Wu D J, Zhang Y L, Niu F Y, Ma G Y, Sun B and Kang R K 2014 Influence of laser power on the forming quality of $\text{Al}_2\text{O}_3$–$\text{ZrO}_2$ eutectic ceramic Rare Met. Mater. Eng. 43 2814–8

Wu D J, Huang Y F, Niu F Y, Ma G Y, Yan S, Li C J and Ding J 2019 Effects of $\text{TiO}_2$ doping on microstructure and properties of directed laser deposition alumina/aluminum titanate composites Virtual Phys. Prototyp. 14 371–81

Niu F Y, Wu D J, Lu F, Liu G, Ma G Y and Jia Z Y 2018 Microstructure and macro properties of $\text{Al}_2\text{O}_3$ ceramics prepared by laser engineered net shaping Ceram. Int. 44 14303–10

Traxel K D, Malhi D, Starkey K and Bandyopadhyay A 2020 Model-driven directed-energy-deposition process workflow incorporating powder flowrate as key parameter Manuf. Lett. 25 88–92

Sola D, Ester F J, Oliete P B and Peña J I 2011 Study of the stability of the molten zone and the stresses induced during the growth of $\text{Al}_2\text{O}_3$–$\text{Y}_2\text{O}_3$–$\text{Al}_2\text{O}_3$ eutectic composite by the laser floating zone technique J. Eur. Ceram. Soc. 31 1211–8

Oliete P B and Peña J I 2007 Study of the gas inclusions in $\text{Al}_2\text{O}_3$–$\text{Y}_2\text{O}_3$–$\text{Al}_2\text{O}_3$ and $\text{Al}_2\text{O}_3$–$\text{Y}_2\text{O}_3$–$\text{ZrO}_2$ eutectic fibers grown by laser floating zone J. Cryst. Growth 304 514–9

Yan S, Huang Y F, Zhao D K, Niu F Y, Ma G Y and Wu D J 2019 3D printing of nano-scale $\text{Al}_2\text{O}_3$–$\text{ZrO}_2$ eutectic ceramic: principle analysis and process optimization of pores Addit. Manuf. 28 120–6

Saito M 1985 Gas-bubble formation of ruby single crystals by floating zone method with an infrared radiation convergence type heater J. Cryst. Growth 71 664–72

Wilcox W R and Kuo V H S 1973 Gas bubble nucleation during crystallization J. Cryst. Growth 19 221–8

Triantafyllidis D, Li L and Stott F H 2003 Mechanisms of porosity formation along the solid/liquid interface during laser melting of ceramics Appl. Surf. Sci. 208–209 458–62

Fan Z Q, Zhao Y T, Tan Q Y, Mo N, Zhang M X, Lu M Y and Huang H 2019 Nanostructured $\text{Al}_2\text{O}_3$–$\text{YAG}$–$\text{ZrO}_2$ ternary eutectic components prepared by laser engineered net shaping Acta Mater. 170 24–37

Naghizadeh R, Rezaie H R and Golestani-Fard F 2009 The influence of composition, cooling rate and atmosphere on the synthesis and thermal stability of aluminum titanate Mater. Sci. Eng. B 157 20–25

Berger M H and Sayir A 2008 Directional solidification of $\text{Al}_2\text{O}_3$–$\text{Al}_2\text{TiO}_3$ system J. Eur. Ceram. Soc. 28 2411–9

Fu L S, Fu X S, Chen G Q, Han W B and Zhou W L 2017 Tailoring the morphology in $\text{Y}_2\text{O}_3$ doped melt-grown $\text{Al}_2\text{O}_3$–$\text{ZrO}_2$ eutectic ceramic Scr. Mater. 129 20–4

Meybodi S M, Bafrooei H B, Ebadzadeh T and Tazike M 2013 Microstructure and mechanical properties of $\text{Al}_2\text{O}_3$–20 wt% $\text{Al}_2\text{TiO}_3$ composite prepared from alumina and titania nanopowders Ceram. Int. 39 977–82

Cleveland J J and Bradt R C 1978 Grain size/microcracking relations for pseudobrookite oxides J. Am. Ceram. Soc. 61 478–81

Bueno S, Berger M H, Moreno R and Baudín C 2008 Fracture behaviour of microcrack-free alumina–aluminum titanate ceramics with second phase nanoparticles at alumina grain boundaries J. Eur. Ceram. Soc. 28 1961–71

Niinara K 1983 A fracture mechanics analysis of indentation-induced Palmqvist crack in ceramics J. Mater. Sci. Lett. 2 221–3

Coble R L and Kingery W D 1956 Effect of porosity on physical properties of sintered alumina J. Am. Ceram. Soc. 39 377–85

Zhou Y 2004 Ceramic Materials 2nd edn (Beijing: Science Press) pp 343–5

Mesa M C, Oliete P B, Orera V M, Pastor J Y, Martín A and Lloreta J 2011 Microstructure and mechanical properties of $\text{Al}_2\text{O}_3$–$\text{Er}_2\text{O}_3$–$\text{Al}_2\text{O}_3$ eutectic rods grown by the laser-heated floating zone method J. Eur. Ceram. Soc. 31 1241–50