Effect of Process Parameters on Selective Laser Melting Al₂O₃-Al Cermet Material

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Abstract
The cermet composite material is one of the researches focuses in the field of materials, for it can combine the toughness of metal and the hardness of ceramics. In this work, Al₂O₃-Al cermet composite with a mass ratio of 1:1 was fabricated by selective laser melting process. The effect of process parameters on the relative density and Al₂O₃ loss rate, as well as the Al₂O₃ loss mechanism, was investigated in detail. The results show that Al₂O₃ undergoes melt recrystallization and is significantly aggregated. The aggregated Al₂O₃ exhibits a network distribution in the metal matrix. The process parameters have a great influence on the relative density and the Al₂O₃ loss rate. As the scanning speed decreases, the relative density and the Al₂O₃ loss rate are changed with a contrary tendency. The loss mechanism is that the aluminum acts as a reducing agent, causing the Al₂O₃ becoming a gaseous substance Al₂O during selective laser melting process.

Introduction
Al₂O₃ ceramics have excellent wear resistance and corrosion resistance, which are rich in resources and low in price[1,2]. Therefore, the development and application of Al₂O₃ ceramic materials have a great economic effect. However, the brittleness of ceramics seriously restricts the development of their application[3]. Recently, the development of cermet material has made up for the lack of plasticity of Al₂O₃ ceramics. The cermet is composed of metal and ceramic, and the latter has a volume fraction of 15 to 85%[3].

Aluminum is one of the most widely used metals with high strength to weight ratio and low densities. It is also commonly used in the fabrication of cermet[4]. Al₂O₃-Al cermet materials have both excellent properties, such as high specific strength, high specific modulus, good wear resistance, etc[5,6]. They are promising in industry. Conventional methods for fabricating Al₂O₃-Al cermet materials generally include stir casting[7,8], hot isostatic pressing[9,10], and sintering[11,12]. However, these methods limit the application of Al₂O₃-Al cermet due to their inability to form part with complex structure. It is urgent to develop a new method for fabricating cermet part with complex structure.

Selective laser melting (SLM) is now widely used in the aerospace industry because of its ability to form complex shaped parts[13–15]. Studies have shown that this method can also be applied to the manufacture of cermet materials[16,17]. This paper investigates the effect of process parameters on the selective laser melting of Al₂O₃-Al cermet with a mass fraction of 50wt%.

Materials and experiment procedures
Materials and SLM process
The polygonal Al₂O₃ powder used in this experiment has an average particle size of 26.6 μm. The aluminium alloy powder is the AlSi10Mg with an average particle size of 33.1μm. Ball milling was performed in a tumbling ball mill to obtain uniform Al₂O₃-AlSi10Mg composite powder, according to the weight ratio of
It reveals that the morphology and dispersion state of the mixed Al$_2$O$_3$-AlSi$_{10}$Mg powders do not change markedly under the condition of mechanical blending.

**Fig.1.** SEM image of the mixed Al$_2$O$_3$-AlSi$_{10}$Mg composite powders after ball milling

The experiments were carried out on a self-developed SLM-100 machine that equipped with an IPG YLR-200 fiber laser ($\lambda=1.07\mu$m) with a maximum output power of 200W, an automatic powder layering appliance and an inert argon gas protection system. The SLM machine was described in detail in our previous publication[18]. The specimens were to be prepared on an AA 2024 aluminum alloy substrate with dimensions of 140mm×140mm×35mm. The process was carried out under argon atmosphere with the concentration of O$_2$ controlled below 200ppm.

The sample with a size of 5mm×5mm×5mm was preliminarily fabricated using several parameters sets in order to study the effect of processing parameter. The used detailed parameters are shown in Table 1.

### Table 1

<table>
<thead>
<tr>
<th>Manufacturing parameters</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Laser power $P$, W</td>
<td>200</td>
</tr>
<tr>
<td>Scanning speed $v$, m/s</td>
<td>150,200,250,300 and 350</td>
</tr>
<tr>
<td>Layer thickness $t$, $\mu$m</td>
<td>20</td>
</tr>
<tr>
<td>Hatching space $h$, mm</td>
<td>0.05, 0.10</td>
</tr>
</tbody>
</table>

**Characterization**

The relative density and Al$_2$O$_3$ loss rate of the samples were evaluated by an image process algorithm (IPA) based on C# language. This method was described in detail in our previous study[17]. The Al$_2$O$_3$ loss rate ($S$) is determined by Eq. (1):

$$S = \frac{M_0 - M_1}{M_0} \times 100\%$$

(1)

where $M_0$ is the mass fraction of Al$_2$O$_3$ in the starting mixed powder, $M_1$ is the mass fraction of Al$_2$O$_3$ in the SLMed sample. The $M_1$ can be calculated as Eq. (2):

$$M_1 = \frac{V_1 \times \rho_{Al_{2}O_{3}}}{V_1 \times \rho_{Al_{2}O_{3}} + (1 - V_1) \times \rho_{Al}}$$

(2)

where $\rho_{Al_{2}O_{3}}$ and $\rho_{Al}$ are the theoretical densities of Al$_2$O$_3$ and Al, respectively. $V_1$ is the area fraction of Al$_2$O$_3$, which can be determined by IPA[17]. The laser energy density is defined as Eq. (3).

$$E_d = \frac{P}{\rho \cdot v \cdot h \cdot \delta}$$

(3)

where $E_d$ is the laser energy density in J/mm$^3$, $v$ is the scanning speed (mm/s), $h$ is the hatching space, $\delta$ is the layer thickness.

The metallographic samples were prepared according to the standard procedures and then etched by a solvent composed of 95 ml water, 2.5 ml HNO$_3$, 1.5 ml HCl and 1.0 ml HF (every 100 ml solution). The
microstructure was characterized using the scanning electron microscopy (SEM, FEI Nova NanoSEM 450) equipped with an energy dispersive spectrometry (EDS, Oxford X-Max 50) microprobe system and optical microscopy (OM, Nikon EPIPHOT). Phase identification was performed by X-ray diffraction (XRD) using a PANalytical X’pert PRO with Cu Kα radiation. On all averaged values, a confidence interval of 95%, based on the variance $\sigma_{n-1}$, is used to indicate the spread.

**Results and discussions**

**Microstructure**

Fig. 2 shows the SEM image of the vertical cross of SLMed sample fabricated under the speed of 350mm/s and hatching space of 0.10mm. The white substance is Al₂O₃. It is apparent that the Al₂O₃ undergoes melting and recrystallization. The molten Al₂O₃ aggregates in the molten pool and exhibits a network distribution. The red arrow identifies the unfused pore near the Al₂O₃ in Fig. 2a. As an additive, Al₂O₃ affects the flow of AlSi10Mg in the molten pool, especially the molten Al₂O₃, which easily hinders the flow of the molten pool. Thereby, unfused pores are formed. As shown in Fig. 2b, the aggregated Al₂O₃ ceramics have microcracks. It can be seen that the ceramic still exhibits strong brittleness. Of course, this is also related to the SLM being a fast cooling rate and high-temperature gradient. Al₂O₃ is prone to form micro-cracks during the process.

Fig. 3 is a photomicrograph of SLMed sample under different process parameters. The process parameters have a great influence on the distribution state of Al₂O₃. When the hatching space is 0.05 mm and scanning speed is 150mm/s, Al₂O₃ exhibits local aggregation, as shown by the white area in Fig. 3a-1. As the scanning speed increases, the Al₂O₃ distribution is more uniform and the content of Al₂O₃ in the matrix increases. When the hatching space is 0.10mm, the Al₂O₃ exhibits a network distribution, especially when the scanning speed is greater than 250 mm/s.

Fig. 4 is the XRD diffraction pattern of the typical SLM formed Al₂O₃-AlSi10Mg composite. The XRD results show that the diffraction peak of Al₂O₃ decreases with the increase of the energy density. This confirms the occurrence of Al₂O₃ loss. As the energy density increases, i.e. the hatching space or scanning speed decreases, the Al₂O₃ loss increases significantly, as shown in Fig. 3a-1 and Fig. 3b-5. A uniform distribution of Al₂O₃-AlSi10Mg composites is obtained when scanning speed is in the range of 250 mm/s to 350 mm/s and the hatching space is 0.10mm.

![Fig.2. SEM image of the vertical cross of SLMed sample fabricated under the scanning speed of 350mm/s and hatching space of 0.10mm](image-url)
**Fig. 3.** Polished cross-sections of SLM-fabricated Al$_2$O$_3$-AlSi10Mg sample under varying process parameters

**Fig. 4.** The XRD diffraction pattern of typical SLM formed Al$_2$O$_3$-AlSi10Mg composite

**Relative density and Al$_2$O$_3$ loss**

Fig. 5 shows the relative density of SLMed samples at different scanning speeds. As the scanning speed increases, the relative density slowly increases. When the hatching space is 0.10mm and the scanning speed is 250mm/s, the highest relative density can be achieved to 94%. When the scanning speed is greater than 250mm/s, higher than 93% relative density also can be achieved. This is because at low scanning speed, the energy density is high and a large number of pores appear. The pores are formed at low scanning speeds from gases trapped within the molten pool or evolved from the powder during solidification[19]. The distribution of Al$_2$O$_3$ is also more uniform as the scanning speed increases as shown in Fig. 3, which reduces the possibility of defects. Noteworthy that as the scanning speed continues to increase, the relative density may decrease.
Fig. 5. The relative density under varying scanning speed and hatching space

Fig. 6 shows the variation of Al₂O₃ loss rate with the scanning speed at different hatching space. Apparently, the relative density and the Al₂O₃ loss rate show a contrary tendency. As the scanning speed and hatching space increase, the loss rate of Al₂O₃ gradually decreases. When the hatching space is 0.10 mm and the scanning speed is higher than 250 mm/s, the Al₂O₃ loss rate is less than 7.6%. It has been confirmed that Al₂O₃ and aluminum undergo redox reactions under high temperature conditions. Al₂O₃ is easily reduced by aluminum to form Al₂O gas and overflows the molten pool[17].

Fig. 6. The Al₂O₃ loss rate under varying scanning speed and hatching space

Conclusions

The microstructure of SLMed Al₂O₃-AlSi10Mg cermet material with a mass ratio of 1:1 were investigated. The Al₂O₃ ceramic in Al₂O₃-AlSi10Mg undergoes melting recrystallization under laser irradiation. When the hatching space is 0.05 mm, the Al₂O₃ tends to locally aggregate. As the scanning speed or hatching space increases, the Al₂O₃ gradually disperses and exhibits a network distribution. The effect of process parameters on the relative density and Al₂O₃ loss rate was also studied. The relative density and the Al₂O₃ loss show with a contrary tendency. As the hatching spacing or scanning speed increases, the density increases and the Al₂O₃ loss decreases. The Al₂O₃ loss is caused by the reduction of the aluminum alloy at high temperature to form Al₂O gas.
Future works

As that process parameters affect the loss and distribution of Al$_2$O$_3$. Future work will focus on how to avoid loss and agglomeration of Al$_2$O$_3$. And the optimized process parameters will be obtained after doing more experiments and analysis.

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References

