Chemical composition analysis for laser solid forming of titanium alloys from blended elemental powders

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Laser solid forming (LSF) from blended elemental powders is an advanced technique to investigate new alloy systems and to create innovative materials. Accurate composition control is critical for the applications of this technique. In this letter, the composition analysis is performed on LSF titanium alloys from blended Ti, Al, and V powders. It is found that the composition of as-deposited sample can be controlled by keeping the identity of the divergence angle of each elemental powder stream. Based on the consistency condition for divergence angles of different elemental powder streams, the matching relation among the Ti, Al, and V powder characteristics (particle size and density) can be obtained, which ensures the consistency in composition between the laser deposits and the blended elemental powders under different laser processing parameters.

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Laser solid forming (LSF) is a solid-freeform-fabrication method, which can be used to manufacture near net-shape metallic components directly from computer-aided design (CAD) model[1]. During LSF, the powder is fed into a molten pool created by a sharply-focused laser beam, and a part is built in point-by-point and layer-by-layer fashion. Recently, more attention has been focused on LSF titanium and titanium alloys. Blackwell *et al.* examined the mechanical properties of LSF Ti-6246 high-strength titanium alloy obtained by Nd:YAG and CO_2 lasers[2]. Qian *et al.* established a finite element model to predict the thermal histories of LSF Ti-6Al-4V thin wall samples to understand the microstructural evolution of LSF Ti-6Al-4V[3]. Bontha *et al.* investigated the effect of process variables on the grain morphology of LSF Ti-6Al-4V[4]. Jia *et al.* developed a finite element model to simulate the temperature and stress field of LSF Ti-6Al-4V hollow blade[5]. Han *et al.* fabricated the titanium coping and titanium base by LSF and so on[6−8]. The previous studies have used the pre-alloyed powders with the required compositions as the deposited materials. However, since the powders are injected into the molten pool synchronously during LSF, it allows the flexibility to deposit blended elemental powders and create an alloy *in situ* in LSF, which could potentially reduce the processing costs to a large extent. LSF from blended elemental powders is also a powerful tool for synthesis of novel materials. In addition, by controlling the mixing process of the powders from several powder feeders, graded compositions within the same sample can be obtained more flexibly. Thus, a number of recent efforts have been conducted on LSF titanium alloys and functionally graded materials from blended elemental powders[9−15].

However, LSF from blended elemental powders suffers from instability and non-repeatability in achieving the accurate as-deposited composition compared with LSF from pre-alloyed powders. Accurate composition control is critical for the applications of this technique. Collins investigated the effect of powder type on composition of as-deposited Ti-Al and Ti-Al-Mo alloys using five types of Al powders[16]. It was indicated that a variable including the powder size and material density might influence the deviation between the target and actual compositions. He *et al.* observed the behavior of the Nb, Ti, and Al mixing-powder stream concentration from a coaxial nozzle in LSF process by a charge-coupled device (CCD) camera[17]. However, few works have been done to study the mechanism of the deviation in composition, and there is still the lack of effective method to realize the exact control in composition in LSF from blended elemental powders. In this letter, LSF experiments are carried out using blended Ti, Al, and V powders. The composition analysis is performed on as-deposited samples and the delivery processes of the elemental powders are monitored by a high speed camera. The mechanism of the deviation between the target and actual compositions of as-deposited sample is investigated and an effective composition control method is proposed.

The titanium alloy samples were fabricated by using a LSF system consisting of a 5-kW continuous wave (CW) CO_2 laser, a four-axis numerical control working table, a powder feeder, and a coaxial nozzle (with four symmetrical powder nozzles). The experiments were carried out inside a glove box filled with argon gas. A schematic of the experimental setup is presented in Fig. 1. Thin wall samples with the size of $50\times12\times3$ (mm) were fabricated. The processing parameters used in the experiments are shown in Table 1. Commercially pure Ti, Al, and V powders were used as the deposited materials. The powders were dried in a vacuum oven for 24 h and then mixed in a ball grinder for 2 h. The equivalent diameters of the powder particles were measured by a LS-230 laser particle size analyzer, as listed in Table 2. The composition of as-deposited sample was characterized by an inductively coupled plasma atomic emission spectrometer (ICP-AES).

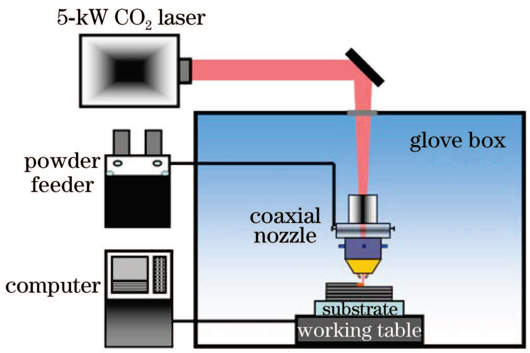

Fig. 1. Schematic of the LSF system.

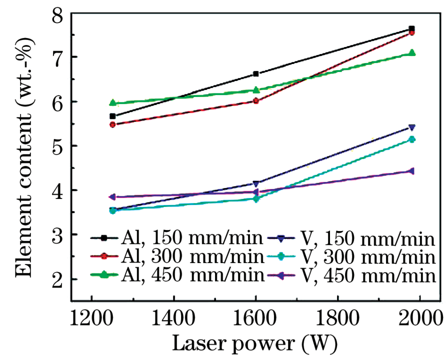

Fig. 2. Influence of laser power and scanning velocity on the Al and V content.

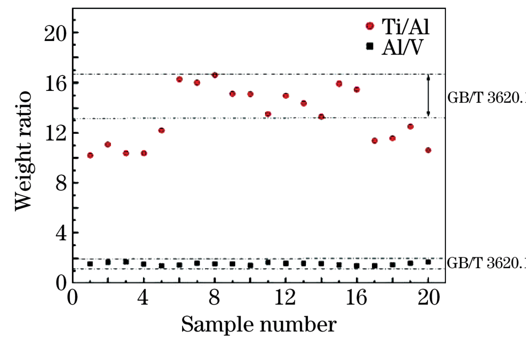

Fig. 3. Weight ratios of Ti to Al and Al to V of as-deposited samples.

Table 1. Processing Parameters of the LSF Route

Laser Power (W)	1250 – 2000
Scanning Velocity (mm/min)	150 – 600
Spot Diameter (mm)	3.0
Nozzle Diameter (mm)	2.5
Powder Feeding Rate (g/min)	4.5
Carrier Gas Flow (L/h)	180

Table 2. Deposited Materials

Powder Material	Shape	Density (kg/m^3)	Equivalent Diameter (μm)
Ti	Spherical	4510	131.40
Al	Spherical	2696	83.55
V	Irregular	6110	58.51

Table 3. Chemical Compositions of the Blended Elemental Powders and As-Deposited Samples

Blended Elemental Powder (wt.-%)	As-Deposited Sample		
	Ti (wt.-%)	Al (wt.-%)	V (wt.-%)
90Ti+6Al+4V	86.19 – 90.98	5.48 – 8.33	3.54 – 5.43

The chemical compositions of the blended elemental powders and as-deposited samples are shown in Table 3. Figure 2 presents the influence of laser power and scanning velocity on the composition of as-deposited sample. It is found that the chemical composition of as-deposited sample varies with the laser processing parameters. Both the Al content and V content of as-deposited sample increase with the laser power at a certain scanning velocity. At a high scanning velocity of 450 mm/min, the weight concentrations of Al and V increase slightly with the laser power. While at low scanning velocities of 150 and 300 mm/min, the weight concentrations of Al and V increase more rapidly with the increase of laser power. Interestingly, the variations of weight concentrations of Al and V present a similar trend. Figure 3 illustrates the weight ratios of Ti to Al and Al to V of all as-deposited samples. It is found that the weight ratio of Ti to Al changes remarkably with the processing parameters, while that of Al to V is approximately constant ($\approx 6/4$) under different laser processing parameters.

In order to understand the distribution of the elemental powder particles in the mixing-powder stream, the delivery processes of Ti, Al, and V powders were monitored by a PCO.1200hs high speed camera with the exposure time of 400 ms. The divergence angle ϕ of the powder stream was measured using an image analysis software (Analysis Five), as shown in Fig. 4. As can be seen, the divergence angle of Ti stream is much larger (12.16°), while that of Al stream (5.09°) is almost the same as that of V stream (5.23°). So the delivery process of the mixing-powder stream can be considered as that shown in Fig. 5. Since only the powder particles blown into the molten pool will be melted, and the size of the molten pool will change with the laser processing parameters, it is easy to understand why the weight ratios

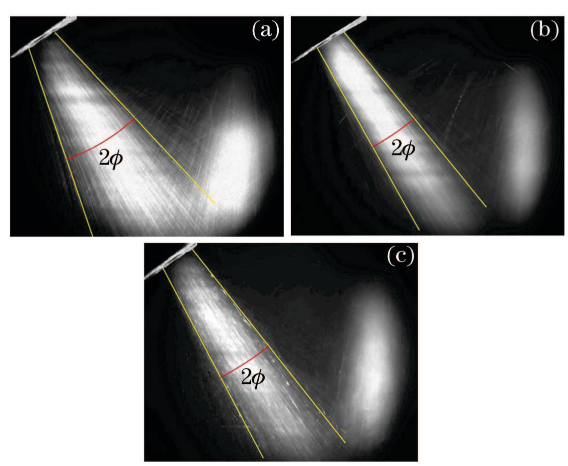

Fig. 4. *In-situ* observation results of the powder delivery processes. (a) Ti, (b) Al, (c) V.

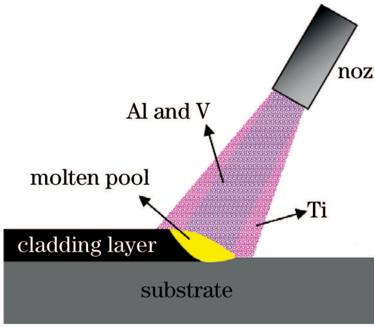

Fig. 5. Schematic of the delivery process of the mixing-powder stream.

of Ti to Al and Ti to V of as-deposited samples change remarkably while that of Al to V keeps constant under different processing parameters. It can be deduced that the spatial distribution of the elemental powder particles in the mixing-powder stream, combined with the size and shape of the molten pool, has a large influence on the composition of as-deposited sample.

Thus, the composition of as-deposited sample can be controlled by two methods. One is to control the size and shape of the molten pool by optimizing the laser processing parameters with the powder stream concentration field obtained. However, a number of parameters govern the LSF process. They are sensitive to the environmental variations, and they are also interdependent. It is difficult and inflexible to realize composition control by optimizing the laser processing parameters. The other one is to choose the elemental powders with matched characteristics as deposited materials. If the divergence angles of the elemental powder streams are identical during the powder delivery process, the composition of as-deposited sample will be the same as that of blended elemental powders no matter what the processing parameters are, i.e., the composition of as-deposited sample can be controlled by keeping the identity of the divergence angle of each elemental powder stream, which is mainly influenced by the powder characteristics, including particle size, shape, and density.

The second method was employed in this letter. A mathematical model, including the variables of powder particle size and density, was established to describe the powder delivery process inside and outside the nozzle. Considering the restriction on the length of the letter, the mathematical model will be described in detail in another paper. By using the model, the consistency condition for divergence angles of different elemental powder streams can be obtained as

$$v_{\text{pe}}(A) = v_{\text{pe}}(B) = v_{\text{pe}}(C) = \cdots = v_{\text{pe}}(X), \quad (1)$$

where $v_{\text{pe}}(X)$ represents the exit velocity of the elemental powder X.

Based on the consistency condition in Eq. (1), the matching relation among the elemental powder characteristics can be obtained.

To verify the usefulness of the model in composition control for LSF from blended elemental powders, the exit velocities of the Ti, Al, and V powders used in the experiments are calculated by the model, which are 1.28, 1.77, and 1.71 m/s, respectively. It can be seen that the exit velocities of Al and V powders are basically equivalent, while that of Ti powder is much smaller, which agrees well the experimental results.

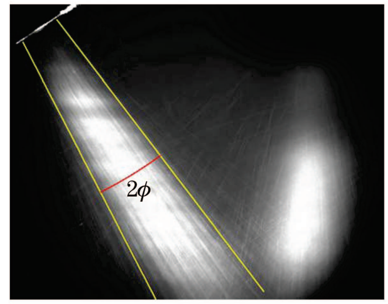

Fig. 6. *In-situ* observation of the Ti powder (with the equivalent diameter of 65 μm) delivery process.

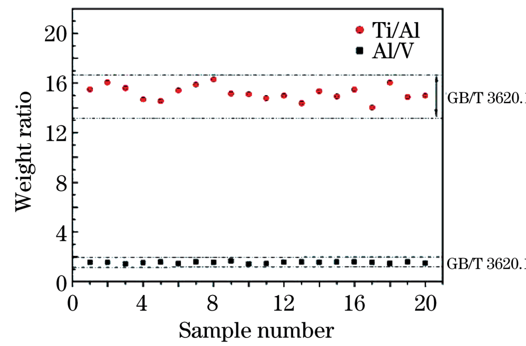

Fig. 7. Weight ratios of Ti to Al and Al to V of as-deposited samples obtained in repeated experiments.

Submitting the exit velocity of 1.74 m/s into the model, and the equivalent diameter of the Ti powder is calculated to be 65 μm so that the divergence angle of the Ti powder stream is consistent with that of Al and V powder streams. We chose the Ti powder with the equivalent diameter of 65 μm as the deposited material and repeated the LSF experiments using the blended Ti, Al, and V powders. Figure 6 shows the delivery process of the Ti powder with the equivalent diameter of 65 μm. It is found that the divergence angle of this Ti powder stream is about 5.82°, which is basically identical to that of Al and V powder streams. Figure 7 presents the weight ratios of Ti to Al and Al to V of all as-deposited samples. It is found that the Ti-6Al-4V alloy meeting the requirements specified in GB/T 3620.1 is obtained under different laser processing parameters. The results clearly demonstrate that, based on the consistency condition for divergence angles of different elemental powder streams, the matching relation among the elemental powder characteristics can be obtained, and the composition of as-deposited sample can be controlled exactly.

In conclusion, LSF from blended elemental powders is a promising laser material processing technique, which offers a variety of interesting cost-effective possibilities for the near net-shape manufacturing of novel alloys and functionally graded materials. Based on the chemical composition analysis performed on LSF Ti-6Al-4V from blended Ti, Al, and V powders, an effective composition control method is proposed for LSF from blended elemental powders, and it is verified by the experimental results.

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