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Experimental Measurement of Vacuum Evaporation of Aluminum in Ti-Al, V-Al, Ti6Al4V Alloys by Electron Beam

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Abstract: Titanium alloys have been widely used in aerospace engineering due to their excellent mechanical properties, especially their strength-to-weight ratio. In addition, Ti6Al4V (TC4) alloy is the most widely used among α+β alloys. The main three elements of TC4 alloy are titanium (Ti), aluminum (Al) and vanadium (V). Since the boiling point of aluminum is much lower than the melting point of the other two elements, the consistency of TC4 alloy during smelting, additive manufacturing and surface treatment is difficult to control. Therefore, in order to study the difficult problem of composition control in TC4 alloy production, we measured the vacuum evaporation of Al, Ti and V in Ti-Al, V-Al and TC4 alloys, and tracked the changes of molten pool temperature, heating time and weight. According to the results, the Al started to evaporate near 1300 ± 10 °C in vacuum and totally evaporated after 225 s heating to 1484 °C at 10^{-2} Pa. However, V and Ti barely evaporated below 2000 °C. The Al in Ti-Al alloy started to evaporate at 1753 ± 10 °C and lost 20.6 wt.% aluminum during 500 s at 1750~1957 °C. The Al in V-Al alloy started to evaporate at 1913 ± 10 °C and lost 26.4 wt.% aluminum during 543s at 1893~2050 °C. The Al in TC4 alloy started to evaporate at 1879 ± 10 °C and lost 79.6 wt. % aluminum after 113 s at 1879~1989 °C. The results indicate that smelting TC4 alloy with Ti-Al and V-Al alloys by EBM below 1900 °C improves the consistency and performance. Additionally, the lowest loss of Al occurred in the additive manufacturing of TC4 alloy below 1900 °C.

Keywords: Ti6Al4V; vacuum evaporation; Ti-Al alloy; V-Al alloy; electron beam

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1. Introduction

Electron beam melting (EBM) is a unique method for the metallurgy [1–7] and purification of refractory metals [8–13]. The vacuum heating principle of electron beam as a heat source is that the high-speed focused electron beam contains a large amount of kinetic energy. When the electron beam impinges on the surface of a material, the kinetic energy is transformed into heat energy, and the material is rapidly heated. The EBM process is complex and there are many variates of the system, such as vacuum degree, beam power, beam size, scan model and scan speed [1,4,6,13]. Different heating patterns may have their own unique variates, which makes the EBM method difficult to accurately control, compare and standardize. Recently, it has been frequently mentioned as a kind of metal powder bed fusion additive manufacturing technology to fabricate three-dimensional workpieces and products directly from computer models, developing a great diversity of applications including aerospace, military [14–29] and medical fields [30–32]. In these fields, it is necessary to improve the quality of products through the precise control of appropriate process parameters, which is inseparable from the research of vacuum evaporation.

Ti-6Al-4V (TC4) is a kind of α+β titanium alloy famous for its excellent properties combining both the high strength of an α alloy and the good ductility of a β alloy. The main elements in TC4 alloy are titanium, aluminum and vanadium [4,9,15–17]. Both titanium and vanadium are refractory metals with high melting and boiling points, with both having melting points above 1600 °C and boiling points above 3287 °C. The melting point of aluminum is 660 °C and the boiling point is 2327 °C. At the same temperature, the vapor pressures of aluminum, titanium and vanadium are very different. Therefore, after EBM treatment or other similar high-temperature (above 1600 °C) treatments, due to the huge gap between the vapor pressure of aluminum and the other two elements, evaporation loss will occur, resulting in changes in the composition of the alloy [4,15,16,19]. The performance changes, and even the alloy phase composition will be changed. Consequently, studying the vacuum evaporation loss of aluminum in TC4 during the EBM process is of great significance for the preparation of titanium alloys and the additive manufacturing of TC4. Most of the loss of TC4 in vacuum must be attributable to the evaporation of aluminum. This research includes a great deal of related work, including measuring the evaporation of aluminum in titanium, vanadium, aluminum and their alloys. It also provides data support for the standardization of electron beam melting technology.

2. Materials and Methods

In this study, in order to evaluate the evaporation process of Al in Ti6Al4V alloy under vacuum EBM heating, we considered the evaporation of all the main elements in the alloy. All the raw materials used in this study are listed in Table 1, including Ti, V, Al elementary elements and some common binary alloys such as Ti-Al and V-Al alloys. All raw materials were purchased online with quality assurance.

Table 1. The raw materials in this study.

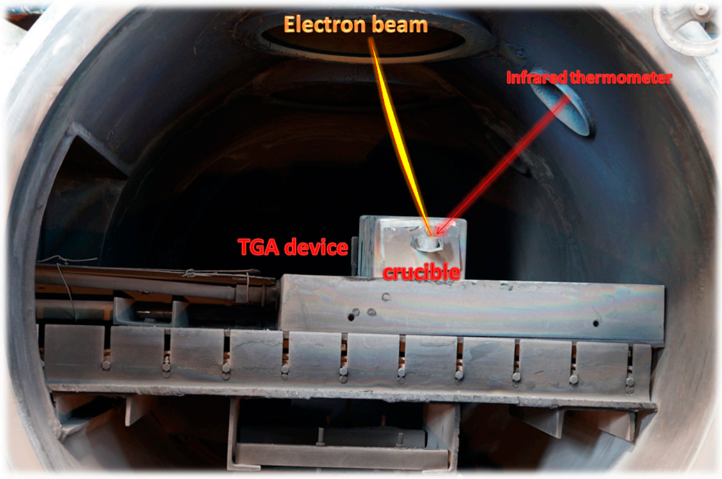
Material	Constituents (wt.)	Shape
Ti	99.9%	small pieces
V	99.9%	small pieces
Al	99.9%	chunk
Ti-Al (64:36)	Ti: 64%, Al: 36%	small pieces
V-Al (65:35)	V: 65%, Al: 35%	small pieces
TC4	Ti: 89%, Al: 5.8%, V: 3.9%	chunk

The EBM process was carried out in a huge vacuum electron beam melting furnace. Table 2 lists the common process parameters of the EBM process.

Table 2. Parameters of the EBM process.

Parameter	Value
Scan pattern	point focused
Point size	3~4 mm
Beam power	3~20 kW
Vapor pressure	(1.5~3) × 10^{-2} Pa
Cooling water temperature	20 °C

The experimental process followed in this study was as follows. First, weigh the crucible and raw materials, and turn on the TGA measuring instrument. Then, the crucible containing the alloys is placed in a fixed position on the TGA device and vacuumed. When the vacuum of the system reaches 10^{-2} Pa, the material is heated with a focused electron beam, and the heating time, molten pool temperature and weight change are collected on the computer in real time. Finally, the EBM process is stopped and cooled to room temperature, and the crucible is taken out of the furnace. For different alloys, in order to ensure the heating requirements of the test process, the electron beam power is adjusted appropriately. The crucible used in the experiment is made of high-temperature-resistant graphite. The inside of the furnace is shown in Figure 1, showing the relative positions of the electron beam and the TGA device.



Inside of the electron beam melting furnace

Figure 1. The actual setup and orientation of the TGA.

In this study, the main method of intensive evaporation during the measurement process is the thermogravimetric analysis (TGA) method. However, due to the very high temperature and vacuum pressure in the EBM furnace, the existing TGA measurement equipment was not suitable for the measurement of the electron beam intensive evaporation. Therefore, we designed and manufactured a new type of TGA device designed for the EBM process. The new device consists of two systems, a temperature measurement system and a quality measurement system. The temperature measurement is done by a non-contact infrared thermometer (Raytek Marathon MR1) placed outside the furnace through the observation window. The thermometer is connected to a computer so that it can continuously measure the temperature of the molten pool, sending a signal to the computer every second to record temperature and time data. In addition, the quality measurement system is uniquely adapted to harsh conditions. It is actually a large balance. The balance arm of the balance is made of molybdenum metal. The crucible is installed on the side of the long arm, and the pressure sensor is installed on the side of the short arm. Molybdenum was chosen for the balance arm because molybdenum is a refractory metal with a high melting point and can withstand high temperatures. The pressure sensor measures gravity data and transmits it to the same computer every second. Most importantly, during the entire EBM process, temperature, time and gravity data can be measured once per second and recorded by the computer at the same time. In order to ensure the accuracy of each measurement, the equipment must be calibrated before each experiment. By recording the temperature and weight changes over time during the test, the entire test process can be recorded effectively and in detail.

The heating rate of the traditional TGA is set constant because the time has a relationship with the temperature. However, it is impossible to set the heating rate constant in the electron beam heating process because of the high energy density of the electron beam. Therefore, the time of the electron beam evaporation experimentation must be recorded synchronously to show the heating rate in the curve of temperature as a function of time, as well as the cooling rate. The TGA device and the crucible have no cooling water, so the heating time is limited. The heating rate is related to the electron beam power, as listed in Table 3.

Table 3. Electron beam power and average heating and cooling rates in the experiment.

Material	Electron Beam Power	Heating Rate (°C/s)	Cooling Rate (°C/s)
Al substance	4.2 kW	53	-
Ti substance	5.7 kW (<160 s) 9.3 kW (≥160 s)	74	-
V substance	8.1 kW (<320 s) 7.8 kW (≥320 s)	94	11
Ti-Al alloy	4.2 kW (<500 s) 6.9 kW (≥500 s)	40	15
V-Al alloy	3.6 kW (<400 s) 6.3 kW (≥400 s)	22	11
TC4 alloy	4.2 kW (<140 s) 7.2 kW (≥140 s)	65	4

3. Results

The TGA data of all metals and alloys for the electron beam evaporation experiments consist of time, temperature and mass percent. Temperature as a function of time is presented as a red curve, and a green curve shows the mass percent as a function of time in Figures 2–7.

In the figures the *x*-axis is the time of the process in seconds. The left *y*-axis is the mass percent (%) of the raw material during the EBM process. It is calculated by Equation (1).

$$\rho = \frac{m_1}{m_0} \times 100\% \tag{1}$$

where m_0 is the initial mass of the raw material; m_1 is the mass of remaining heated material and ρ is the mass percent of the material left in the crucible. The right *y*-axis is the temperature of the molten pool during the EBM process. Although the accuracy of the mass measurement device is high enough to reach 2‰, mechanical vibration from the environment can still interfere with the measurement, which requires some future work to reduce the interference and make the data obtained more accurate.

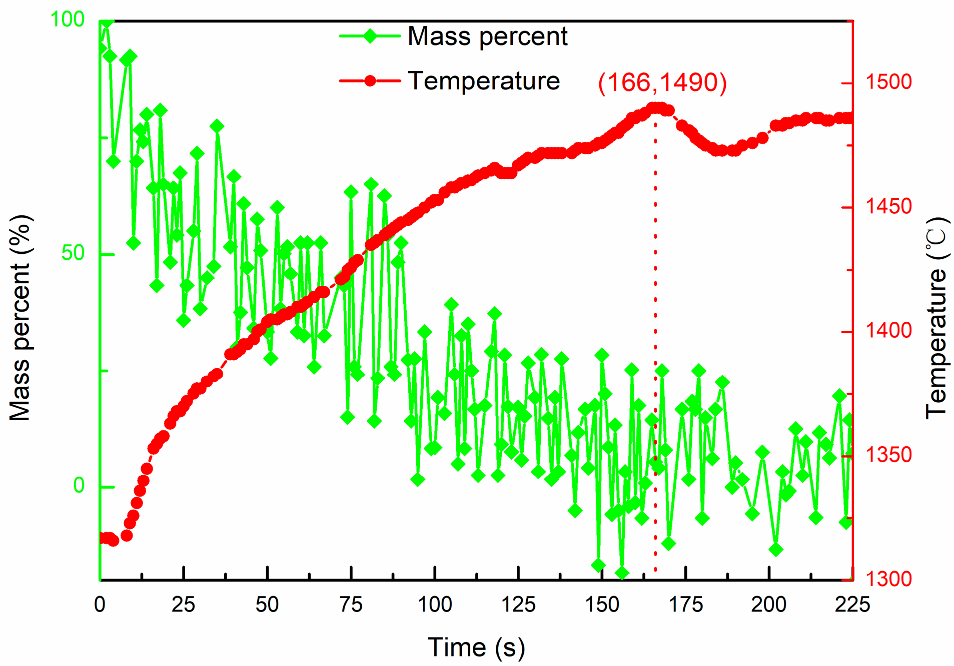


Figure 2. The vacuum evaporation of Al by EBM.

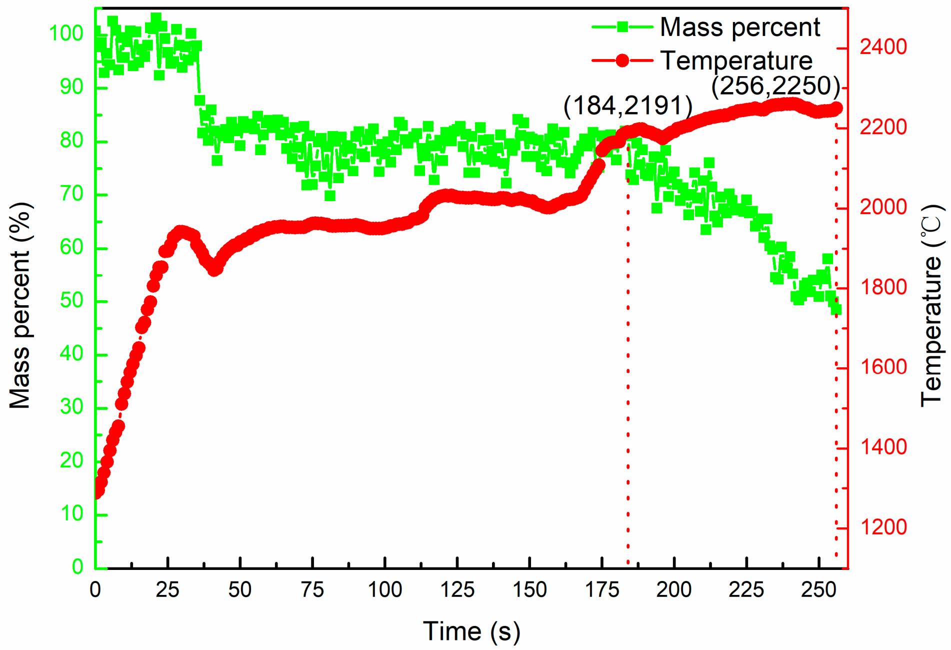


Figure 3. The vacuum evaporation of Ti by EBM.

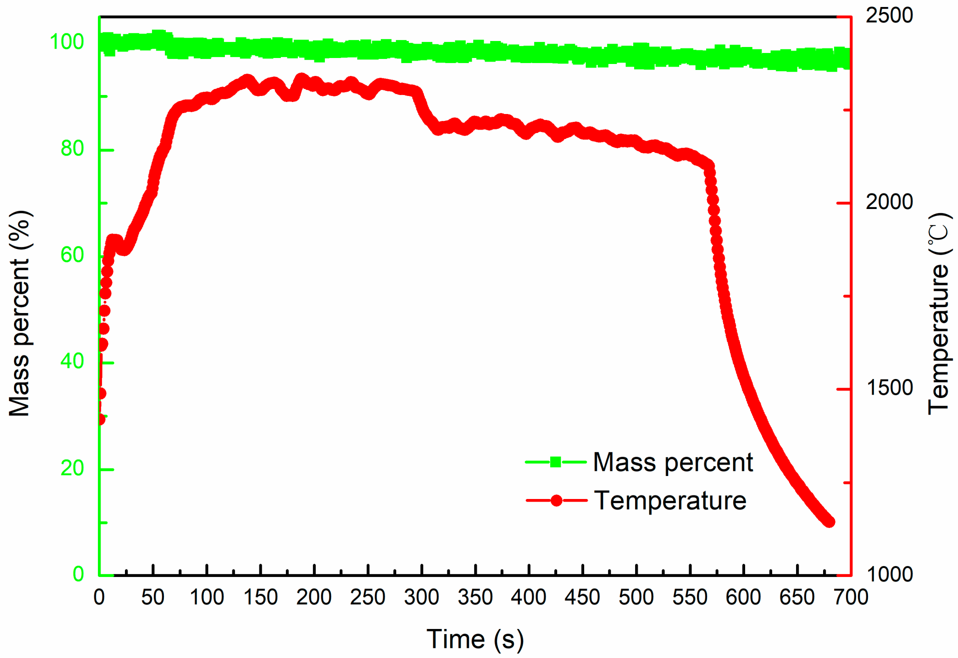


Figure 4. The vacuum evaporation of V by EBM.

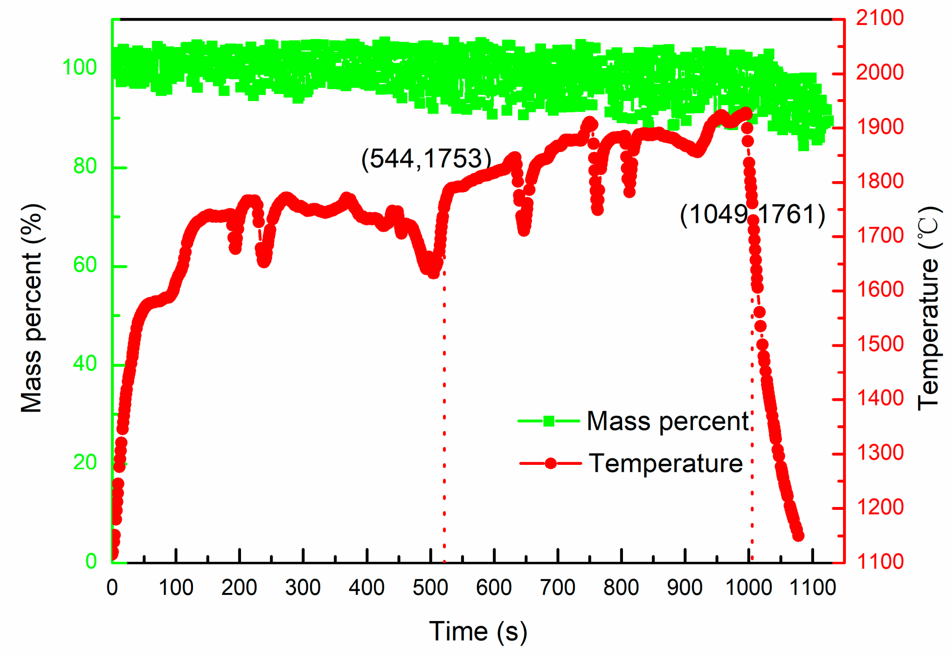


Figure 5. The vacuum evaporation of Al in Ti-Al alloy by EBM.

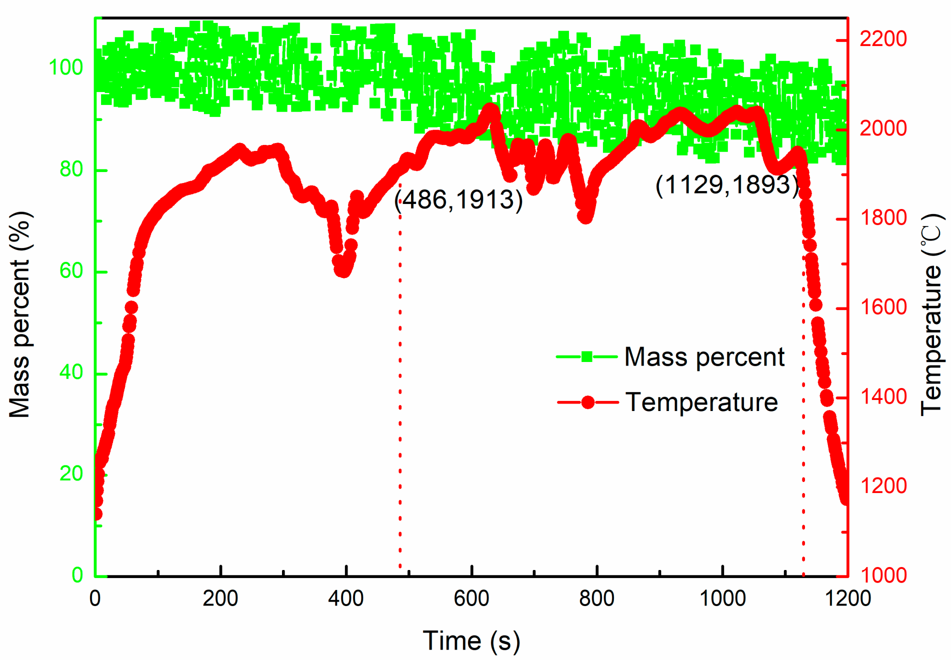


Figure 6. The vacuum evaporation of Al in V-Al alloy by EBM.

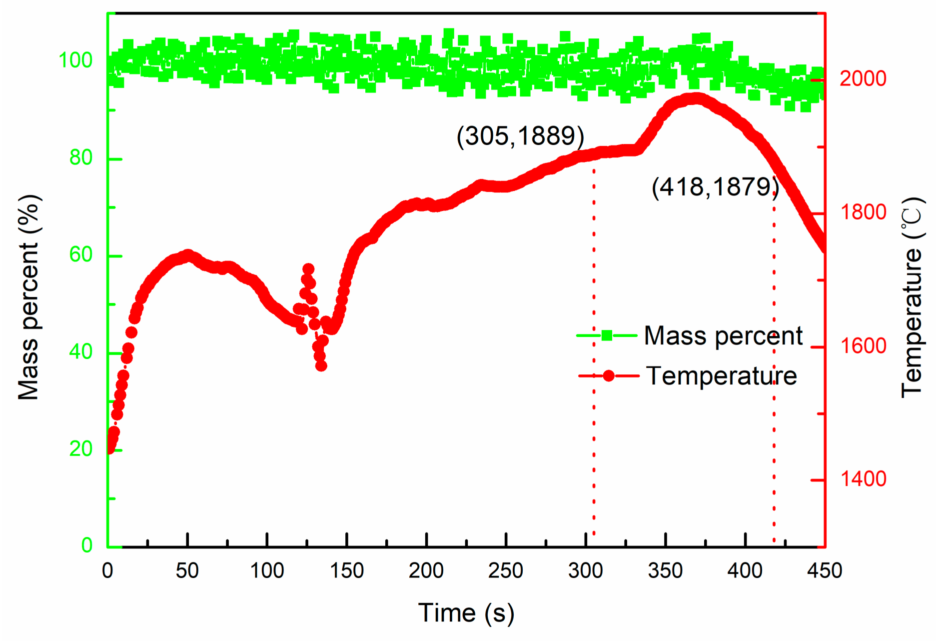


Figure 7. The vacuum evaporation of Al in TC4 alloy by EBM.

The vacuum evaporation of aluminum is shown in Figure 2. The melting point of aluminum is lower than the range offline, so the heating process before 1300 °C could not be recorded. However, the mass percent data could be recorded. As can be seen from the green curve, Al evaporated intensively beyond 1300 °C and almost 100% evaporated at 166 s. The dip in temperature after 166 s corresponds to the almost 100% weight loss of the Al substance. As all the Al was evaporated, this temperature is not of the molten pool of Al but of the surface of the crucible, with lower-temperature heating by the electron beam.

The vacuum evaporation of titanium is shown in Figure 3. Obvious evaporation occurred at around 184 s, and the temperature was above 2190 °C. The decrease in weight before 184 s was caused by melt flow resulting in the movement of the center of gravity. This was an accidental error and can be eliminated.

The vacuum evaporation of vanadium is shown in Figure 4. Vanadium is a refractory metal with high melting and boiling points. The highest temperature during the TGA experiment was 2317 °C, but vanadium still did not evaporate. The total mass loss of vanadium was less than 2.1% during 675 s of heating. Therefore, it can be said that titanium and vanadium show little evaporation below 2000 °C. The mass loss in the alloys below 2000 °C was caused mostly by aluminum evaporation.

The vacuum evaporation of Ti-Al alloy is shown in Figure 5. Obvious evaporation occurred at around 544 s, and the temperature was 1753 °C. The evaporation lasted for nearly 500 s, resulting in a total of 20.6% loss of aluminum in alloy. The highest temperature was 1957 °C.

The vacuum evaporation of V-Al alloy is shown in Figure 6. Similarly, obvious evaporation occurred at 486 s, 1913 °C and ended at 1129 s, 1893 °C, lasting for nearly 543 s, losing 26.4% of the aluminum in the alloy. The highest temperature was 2050 °C.

The vacuum evaporation of TC4 alloy is shown in Figure 7. Obvious evaporation occurred at 305 s, 1889 °C and ended at about 418 s, 1879 °C, lasting for nearly 113 s, losing 79.6% of the aluminum. The highest temperature was 1989 °C.

In the process of the experiment, pure aluminum at 660 °C melted into liquid. At 1350 °C the saturated vapor pressure calculated by the saturated vapor pressure formula is nearly 3.12 Pa, which is far greater than the limit vacuum degree of the equipment, so the crucible of pure aluminum would continue to rapidly evaporate until all evaporation was completed. However, the titanium alloy would melt into liquid at 1600 °C and above; at the same time, the concentration of aluminum in the alloy is low, and under the action of the base metal, the probability of aluminum atom overflow interface is low, so it would begin to significantly evaporate at 1800 °C and above.

4. Discussion

The experimental results of this study are novel because of the new measurements. The evaporation data of Ti and V in TC4 alloy agrees well with the results by Powell et al. [33] and the calculation by Ivanchenko et al. [34], in consideration of the difference of heating pattern, temperature range and cooling condition. However, the evaporation of Al in the TC4 alloy is not consistent with the results of these studies. One of the reasons is that the Langmuir equation used in both articles, calculating the evaporation rates from the temperature, fit the experimental measurements well in the weak evaporation period, but not the intensive evaporation at the higher temperature and fast heating rate caused by the electron beam in this study. This study focuses on intensive evaporation more than weak evaporation.

The characteristics of the EBM heating process can be clearly seen by consulting the red curves in the figures. It can be divided into three periods. The first period is the initial heating period, where there is no evaporation and the thermal radiation is negligible. The molten pool is still small, and the temperature is low, but it heats up most quickly in this period. The second period is the middle heating section. The characteristic of this part is that the temperature of the molten pool is high, and the heat is rapidly transmitted to the outside of the molten pool. Evaporation occurs only in the pool with higher temperature, and thermal radiation also increases slowly. Due to the heat absorption of melting and evaporation, the molten pool expands, making the temperature rise increasingly slowly. The last part is the heat balance period. The main characteristic of this part is that the temperature is stable, and the size of the molten pool does not change. At this point, the heat generated by the electron beam is equal to the heat lost from the pool. There are three causes of loss: heat radiation, evaporation and heat conduction. Because the temperature difference is small, the heat conduction in this part is relatively small and negligible. Thus, when thermal radiation can be calculated, the average rate of evaporation at the same temperature can be estimated.

The electron beam heating process is a very complex process, and there is still a lot of work to be done in its research; the TGA device in this study is only a small step. TC4's EBM process is a typical example. Based on the data of this study, the process of synthesizing TC4 alloy with (Ti-Al) alloy and (V-Al) alloy as raw materials by EBM under vacuum and accurately controlling the maximum temperature below 1900 °C can make the composition of the synthesized TC4 alloy more accurate, so as to improve the alloy properties. At the same time, the data of this study also have important reference value for the improvement of the processing parameters and product quality of electron beam additive manufacturing, and for the refining process of high-purity metal using EBM.

5. Conclusions

1. The Al in Ti-Al alloy started to evaporate at 1753 ± 10 °C, and after heating for 500 s in the temperature range of 1750~1957 °C, the evaporative loss of aluminum was 20.6 wt.%.
2. The Al in V-Al alloy started to evaporate at 1913 ± 10 °C, and the evaporation loss of aluminum was 26.4 wt.% after heating for 543 s in the temperature range of 1893~2050 °C.
3. The Al in TC4 alloy started to evaporate at 1889 ± 10 °C, and 79.6 wt.% aluminum was lost after heating for 113 s in the temperature range of 1879~1989 °C.

The TC4 alloy was fused with Ti-Al and V-Al alloys by EBM method at 1900 °C; the alloy composition was more accurate and the properties were better. In addition, at 1900 °C, the TC4 alloy melted by the additive manufacturing process had the lowest aluminum loss.

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Conflicts of Interest: The authors declare no conflict of interest.

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