

Research Article Study of Controlled Aluminum-Vanadium Alloy Prepared by Vacuum Resistance Furnace

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Received 7 May 2021; Accepted 22 June 2021; Published 9 July 2021

Academic Editor: Yaohui Xu

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In this paper, V_2O_3 is used as the raw material, and an Al-V alloy is synthesized by vacuum resistance furnace heating. The results show that with the extension of the heating time, the separation effect of the alloy and the slag and the uniformity of the alloy are significantly improved. The slag is fully separated from the alloy, and the composition of the alloy is more uniformly distributed when the heating time is controlled at 1 hour. In addition, the composition of the alloy is affected by increasing content of Al. In particular, the obtained alloy composition meets commercial standards when the mass ratio of Al is 31-36 wt%. The effect of different mass ratios of CaO on the quality of slag series and alloys was also investigated. The results show that the composition distribution of the slag system can be effectively changed with increasing the mass ratio of CaO, and the uniformity of the alloy composition is also optimized. When the mass ratio of CaO is 27 wt%, the alloy and slag are well separated, and the composition distribution in the obtained alloy is more uniform. In addition, the content of impurities in the alloy can be effectively improved under vacuum. Therefore, it is suggested that a vacuum resistance furnace should be applied in the industrial production of high-quality Al-V alloy.

1. Introduction

The properties of titanium alloys can be effectively optimized when a certain proportion of vanadium is added to the titanium alloys, especially the ductility, corrosion resistance, and high temperature resistance of the alloy that are significantly improved [1-3]. Titanium alloys with metallic vanadium are widely used in major areas such as aerospace, nuclear energy materials, and weaponry [4-8]. Generally, vanadium is added to the titanium alloy in the form of aluminum-vanadium (Al-V) alloy. Al-V alloy is the main raw material of vanadium-containing master alloy in the preparation of vanadium-containing titanium alloy, and the high-end titanium alloys have very strict requirements for their composition; therefore, most Al-V master alloys cannot be used in large-scale production of high-end vanadiumcontaining titanium alloys, and only a small amount of Al-V alloys can meet the demand standards [9-11]. This is

mainly due to the strict control of the production process of Al-V alloy. It is necessary to ensure the uniformity of the composition and low impurity content, especially for impurity elements such as oxygen, carbon, and nitrogen, which increase the grain boundary hardening and brittle phases of the alloy and affect the properties of the alloy.

The traditional production methods of Al-V alloy are the self-propagating method and two-step method [12–15]. In the self-propagating method, V_2O_5 and Al are used as raw materials, CaF_2 or CaO is selected as a slag-forming agent, and Mg or KClO₃ is used as an igniter to obtain V-Al alloy with different V contents. This method has a poor controllability; in addition, it has higher requirements on the material of the furnace lining, and the improvement of product quality and the reduction of production cost are severely restricted. The two-step method is an improvement based on the self-propagating method. Firstly, V_2O_5 and Al are used as raw materials for reduction reaction, and an Al-V

alloy with a V content of more than 85% is obtained. Subsequently, the alloy and a certain amount of Al are smelted under vacuum conditions to finally obtain the desired Al-V alloy. This method can obtain high-quality Al-V alloy, but the production process is long, the operation process is complicated, and the requirements for various parameters are high, which restricts large-scale production.

Because Al is used to reduce V₂O₅, heat is quickly released in the early stage of the reaction, resulting in insufficient heat in the later stage of the reaction and rapid condensation of the alloy, which affects the quality of the alloy and the yield of V. At present, many researchers have begun to pay attention to how to supplement the heat of the entire reaction to ensure that the reaction proceeds fully and the alloy composition is uniform. Liu et al. [16] used the microwave heating method to supplement heat, and the nonmetallic inclusion particles in the final alloy product have been significantly reduced. Li et al. [17-20] tried to prepare Al-V alloy with electrode-assisted heating, and the electrodes was used to ignite the mixed raw material particles and then use the electrodes for heating in the later stage of the reaction. This not only makes the reduction reaction complete, but also prolongs the solidification time of the Al-V alloy and makes the alloy composition more uniform. The high-standard Al-V alloy samples can be obtained by this method, but the contact time between the electrode and the slag and alloy must be strictly controlled when using the electrode for heating; otherwise, the carbon content in the alloy will be increased significantly.

When V_2O_5 is used as a raw material, it has a large reaction heat effect and a large heat release rate. Compared with V_2O_5 , V_2O_3 has a lower thermal effect, and the reaction has stronger controllability. This paper studies a new preparation method of Al-V alloy, which can realize the controllability of the reaction process. V_2O_3 and the Al are used as raw materials, CaO is used as raw materials, and resistance furnaces are used as auxiliary heating equipment. During the heating process, the raw materials are continuously and slowly heated through the resistance furnace, and the reaction between the V_2O_3 and the Al proceeds slowly, which achieves the controllability of the entire Al-V alloy preparation process. Finally, high-purity Al-V alloy is obtained by optimizing the ratio of raw materials and heating time.

2. Experimental Details

2.1. Materials. Vanadiumtrioxide (V_2O_3) particles were used in our experiment, which was manufactured by Chengsteel HBIS. And the commercial grade aluminum granules and CaO powder were also used in the experiment. All the form and purity of raw materials are shown in Table 1.

2.2. Experimental Procedures. The raw materials of V_2O_3 , Al particles, and CaO are mixed in a certain proportion and then put into the crucible. The crucible and raw materials are put into vacuum resistance furnace for heating, and the experimental process and device are shown in Figure 1. A resistance furnace was used for high heating temperature experiments. The hot zone with a high consistency of temperature range was determined before the experiment

TABLE 1: The raw materials in the experiments.

Material	Form	Purity (wt %)
		$V_2O_3 > 95$
V_2O_3	Powder (technical grade)	Fe < 0.05
		Si < 0.08
		Al > 99.85
Al	Granules (technical grade)	Fe < 0.1
		Si < 0.08
		CaO > 98
CaO	Powder (technical grade)	Fe < 0.1
		Si < 0.4



FIGURE 1: Schematic diagram of the experiments.



FIGURE 2: Phase diagram of the slag system of CaO and Al₂O₃.



The composition of the slag under the heating time is 5 min (wt %)

No.	Ca	О	Al	V
1	8.8	44.7	46.3	0.2
2	13.6	43.5	42.6	0.4
3	1.8	0.0	84.5	13.7
4	0.0	0.0	99.5	0.5



The composition of the alloy under the heating time is 5 min (wt %)				
No.	Ca	0	Al	V
1	0.2	0.0	60.4	39.4
2	1.0	0.0	63.7	35.3
3	28.3	41.4	33.7	0.3



The composition of the slag
under the heating time is 10 min (wt %)

No.	Ca	0	Al	V
1	25.6	40.4	33.9	0.0
2	15.6	43.0	41.3	0.0

The composition of the alloy under the heating time is 10 min (wt %)					
No.	Ca	0	Al	V	
1	0.0	0.0	31.0	68.9	
2	0.0	0.0	44.4	55.4	

(d)

FIGURE 3: Microstructure and composition of sample: (a) is the slag of heating 5 min, (b) is the alloy of heating 5 min, (c) is the slag of heating 5 min, and (d) is the alloy of heating 5 min.

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The composition of the slag under the heating time is 5 min (wt %)				
No.	Ca	0	Al	V
1	25.5	40.4	34.0	0.0
2	32.2	38.7	29.1	0.0
3	15.6	43.0	41.4	0.0

(a)



The composition of the alloy under the heating time is 30 min (wt %)				
No.	Ca	0	Al	V
1	0.0	0.0	60.1	39.9
2	0.0	0.0	49.0	51.0

(b)



The composition of the slag
under the heating time is 60 min (wt %)

No.	Ca	0	Al	V
1	20.1	26.9	33.0	0.0
_2	15.6	43.0	41.3	0.0

(c)



No.	Ca	0	Al	V
1	0.0	0.0	60.0	39.4
2	0.0	0.0	50.1	49.3

FIGURE 4: Microstructure and composition of sample: (a) is the slag of heating 30 min, (b) is the alloy of heating 30 min, (c) is the slag of heating 60 min, and (d) is the alloy of heating 60 min.

(d)

TABLE 2: Composition of raw materials used in the experiments (wt%).

No.	CaO	V ₂ O ₃	Al
1	24	41.0	35.0
2	24	41.5	34.5
3	24	42.0	34.0

TABLE 3: Composition of sample present in the laboratory	samples
(wt %).	

Sample		Compositions			
	Point	CaO	Al ₂ O ₃	Al	V
Slag	1	21.7	78.3	0.0	0.0
	2	35.6	64.4	0.0	0.0
	3	0.0	0.0	43.6	56.4
Alloy	4	0.0	0.0	43.5	56.5
	5	0.0	0.0	43.1	56.9



FIGURE 5: Microstructure of slag and alloy of No. 1.



FIGURE 6: Microstructure of slag and alloy of sample No. 2.

started. And a type-B thermocouple was used to monitor the temperature during the reaction. The furnace was filled with argon (purity > 99%) gas throughout the experiment to prevent the oxidation of the raw materials and crucible at high temperature. The crucible was placed in the heating zone, and the temperature was raised to 1600° C.

2.3. Characterization of Samples. The slag and alloy samples obtained were mounted in epoxy resins, followed by polishing and carbon-coating. For EPMA, an accelerating voltage of 15 kV and probe current of 15 nA were adopted. The composition standard of slag and alloy in the sample were obtained from Charles M. Taylor Co. (Stanford, California,

TABLE 4: Compositions of	in the laboratory	7 samples ((wt%).
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	Doint	Compositions (%)				
Sample	Politi	CaO	Al_2O_3	Al	V	
Slag	1	35.8	64.2	0.0	0.0	
	2	21.9	78.1	0.0	0.0	
Alloy	3	0.0	0.0	42.2	57.8	
	4	0.0	0.0	41.9	58.1	

USA). The alloy samples prepared from different experimental conditions were analyzed by an inductively coupled plasma atomic emission spectrometer (ICP-AES), using a PS-6 Plasma Spectrovac, Baird (USA).



FIGURE 7: Microstructure of the slag and alloy of sample No. 3.

TABLE 5: Compositions of samples present in the laboratory samples(wt %).

Delat	Compositions (wt%)				
Point	CaO	Al_2O_3	Al	V	
1	20.6	79.4	0.0	0.0	
2	33.5	66.5	0.0	0.0	
3	0.0	0.0	41.4	58.6	
4	0.0	0.0	41.2	58.8	
5	0.0	0.0	41.0	59.0	
	Point 1 2 3 4 5	Point CaO 1 20.6 2 33.5 3 0.0 4 0.0 5 0.0	$\begin{array}{c c} \mbox{Point} & \begin{tabular}{c c c c c } CaO & Al_2O_3 \\ \hline CaO & Al_2O_3 \\ \hline 1 & 20.6 & 79.4 \\ 2 & 33.5 & 66.5 \\ \hline 3 & 0.0 & 0.0 \\ \hline 4 & 0.0 & 0.0 \\ \hline 5 & 0.0 & 0.0 \\ \end{array}$	$\begin{array}{c c c c c c c } Point & Compositions (wt%)\\ \hline CaO & Al_2O_3 & Al \\ \hline 1 & 20.6 & 79.4 & 0.0 \\ 2 & 33.5 & 66.5 & 0.0 \\ 3 & 0.0 & 0.0 & 41.4 \\ 4 & 0.0 & 0.0 & 41.2 \\ 5 & 0.0 & 0.0 & 41.0 \\ \end{array}$	

TABLE 6: Compositions of raw materials in the tests (wt %).

No.	CaO	m _{V2O3:Al}
1	21.0	1.2:1
2	23.0	1.2:1
3	27.0	1.2:1
4	30.0	1.2:1

3. Results and Discussion

3.1. Theoretical Analysis. The reduction reaction between Al and V_2O_3 is as follows.

$$V_2O_3 + 2AI \longrightarrow 2V + Al_2O_3,$$

$$\Delta H298 \text{ K} = -456.901 \frac{\text{KJ}}{\text{mol}}.$$
(1)

During the reaction, a large amount of heat is released, and a large amount of alumina slag is produced, which has an important influence on the quality of the alloy. The main function of the slag is to prevent the alloy from being oxidized, while the impurities in the alloy are absorbed into the slag. The influence of these effects mainly depends on the fluidity of slag; so, it is necessary to improve the fluidity of slag. In this study, the phase diagram was used to analyze and study the slag system with different proportions of CaO, and the composition change of the slag is shown in Figure 2. It can be seen from the figure that with the change of CaO addition, the melting point of the slag system is significantly reduced. The liquid phase composition is appeared in the slag when the temperature is lower than 1500°C, and the mass ratio of CaO is increased to 38%. However, the melting point of the slag is raised again when the mass ratio of CaO is increased to 65%. Therefore, in order to obtain a lower melting point slag system, the mass ratio of CaO in the slag should be controlled at 38-65%.

3.2. Effect of Heating Time on the Slag and Alloy. The influence of different heating times on the reaction process was investigated, and the heating time is set to 5, 10, 30, and 60 min. The morphology and composition of the product obtained for the heating times of 5 min and 10 min are shown in Figure 3. Figure 3(a) displays the microstructure of the slag phase for the heating time of 5 min, which shows that the slag system contains a certain amount of V, with the V content at point 3 of 13.7 wt%. At the same time, a large amount of CaO and Al_2O_3 are found in the alloy, as shown in Figure 3(b). This indicates that the alloy and slag obtained in the reaction process cannot be effectively separated, eventually giving rise to the above described phenomenon and preventing the fabrication of high-quality alloy samples. The products obtained using the heating time of 10 min are shown in Figures 3(c)and 3(d). The slag and alloy are well separated as shown by the analysis of the phase composition of the alloy and slag.

The microstructures of the products obtained when the heating time is further extended to 30 min and 60 min are shown in Figure 4. It is observed that the vanadium oxide and vanadium are not found in the slag system when the heating time is 30 min, as shown in Figure 4(a). Correspondingly, the CaO and Al_2O_3 impurities are not detected in the alloy as shown in Figure 4(b). In particular, when the heating time is controlled at 60 min, the obtained slag and alloy sample has the best composition uniformity, as shown in Figures 4(c) and 4(d). This shows that the supplementary heat not only helps to separate the slag and the alloy but also has a significant effect on the improvement of the alloy quality. This



FIGURE 8: Microstructure of slag and alloy of sample No. 1.

C	Delat	Composition (wt%)				
Sample	Point	CaO	Al_2O_3	Al	V	
Slag	1	36.1	63.9	0.0	0.0	
	2	22.1	77.8	0.0	0.0	
	3	0.0	0.0	42.0	57.7	
Alloy	1	0.0	0.0	98.6	1.3	
	2	0.0	0.0	85.3	14.2	
	3	0.0	0.0	61.4	53.8	

TABLE 7: Compositions of samples present in the laboratory samples(wt %).

phenomenon has similar consistency with some reported effects of microwave heating and electrode heating [21–24].

3.3. The Effect of Composition of Different Raw Materials on Alloy. V_2O_3 is used as the raw material, and Al is used as the reducing agent. Firstly, the effect of different ratios of V_2O_3 and Al on the alloy product was studied. The mass ratios of different raw materials are shown in Table 2. During the reaction process, the time for the raw materials is heated by a resistance furnace at 1600°C for 1 hour. The microstructures of the product obtained when the mass ratio of V_2O_3 and Al in the raw material are 44 wt% and 31 wt%, respectively, are shown in Figure 4. It is found that the slag is composed of two main material structures as shown in Figures 4(a) and 4(b). A small amount of

alloy phases are found in the slag phase as shown in Figure 4(b). The region where the content of CaO is 35.6 wt% is the liquid slag phase that is more conducive to the separation of alloy and slag. And the area with a CaO content of 21.7 wt% is the solid phase during heating as shown in Table 3. In addition, the main difference between the two phases is the amount of CaO contained, and the distribution of the two phases is not uniform. Figures 4(c) and 4(d) display the microstructures of the alloy products, which show that the slag and oxide are not coated in the alloy. This indicates that the reduction reaction is completed. However, the V content varies greatly at different positions in the alloy, as shown in Table 3, indicating that the poor uniformity of the alloy.

When the Al content in the raw material is further increased to 36 wt%, the microstructure of the resulting slag and alloy is shown in Figure 5. More heat is released, and the slag system area of the liquid phase is increased during the reaction process as shown in Figure 5(a). The CaO content in the liquid slag phase shown in Table 1 is 35.8 wt%, enhancing the fluidity of the slag and improving the uniformity of the alloy as shown in Figure 6(b). The content of V in the alloy is 57-58%, as shown in Table 4.

The microstructure of the slag and alloy obtained when the amount of Al added in the raw material is further expanded to 38 wt% is shown in Figure 7. It was found that the slag system is mainly a liquid slag system, as shown in Figure 7(a), and Table 5 shows that the CaO content in this material is reduced to 33.5 wt%.



FIGURE 9: Microstructure of slag and alloy of sample No. 2.

TABLE 8: Compositions of samples present in the laboratory samples (wt %).

Commlo	Deline		on (wt%)		
Sample	Point	CaO	Al_2O_3	Al	V
	1	35.7	64.3	0.0	0.0
Slag	2	21.7	78.3	0.0	0.0
	3	47.6	52.4	0.0	0.0
	4	0.0	0.0	43.2	56.8
Alloy	1	0.0	0.0	41.5	58.5
	2	0.0	0.0	41.2	58.8

Additionally, as the Al content in the alloy increases, three different material structures appeared in the alloy, and Al and V contents are shown in Table 3. These structures lead to the poor uniformity of the alloy; in addition, the V content of the alloy is further reduced due to the increase in the Al content. The content of V in some areas of the alloy is 35 wt% and 33 wt%, respectively, which is lower than the required standard for an Al-V alloy.

Since the addition of CaO has a significant effect on the properties of the slag system, the effect of adding different proportions of CaO on the alloy was investigated. The different CaO addition ratios in the raw materials are shown in Table 6.

Figure 8 shows the microstructure of the product obtained when the addition of CaO in the raw material is

21%. It found that there are three main phase structures in the contact position between the slag and the alloy, as shown in Figure 8(a), and these three phases are $CaO \times Al_2O_3$, $CaO \times 2Al_2O_3$, and Al-V alloy, as shown in Table 1. It indicates that the slag and alloy are not effectively separated, and resulting in the alloy be presented in the slag. However, the alloy is not detected in the upper part of the slag, and there are two substances of CaO and Al₂O₃, as shown in Figure 8(b). The microstructure of the synthesized alloy is shown in Figures 8(c) and 8(d), and it is observed that there are three structures in the alloy and with poor distribution uniformity. The V contents of these three regions are 1.3, 14.2, and 53.8 wt%, respectively, as shown in Table 7. These phenomena indicate that the fluidity of the slag is weak, which affects the separation of alloy and slag and the uniformity of the alloy.

In order to improve the flow effect of the slag, the addition amount of CaO in the raw material is increased to 23 wt%, and the product structure is shown in Figure 9. There are still two material composition structures on the upper surface of the slag layer, and their composition is shown in Table 8. However, in the slag layer in contact with the alloy, it was found that the alloy content was reduced, but the composition and structure of the slag changed, as shown in Figure 9(b). In addition, Figures 9(c) and 9(d) shows the structure of the alloy. It is observed that the uniformity of the distribution of the two constituent substances in the alloy has been improved, and the compositions of these two regions are presented in Table 8.



FIGURE 10: Microstructure of slag and alloy of sample No. 3.

TABLE 9: Compositions of samples present in the laboratory samples(wt%).

Sample	Doint				
	Politi	CaO	Al_2O_3	Al	V
Slag	1	35.8	64.1	0.0	0.0
	2	21.8	78.1	0.0	0.0
Alloy	1	0.0	0.0	41.1	58.9
	2	0.0	0.0	41.6	58.4

When the proportion of calcium oxide in the raw material is further increased to 27 wt%, the microstructure of the obtained slag and alloy is shown in Figure 10. The structure of the slag system is shown in Figures 10(a) and 10(b), and the main components of the slag phase are shown in Table 9. Correspondingly, the microstructure of the alloy sample is shown in Figures 10(c) and 10(d). Table 9 shows the distribution of the V content in the main phases of the alloy, and the V content is 58.9 wt% and 58.4 wt%, respectively. This V content is mainly determined by the two phase structures present in the alloy, namely, AlV_3 and Al_8V_5 , as shown in Figure 11.

The microstructure of the resulting slag and alloy is shown in Figure 12 for the proportion of CaO in the raw material that is increased to 30 wt%. At this time, the phase composition in the slag and alloy has not changed as shown in Figure 12(a). However, in Figure 12(b), the uniformity of the alloy is further improved, and the V content in different



FIGURE 11: X-ray diffraction of the alloy.

regions of the alloy is 58.3 wt% and 60.0 wt%, respectively, as shown in Table 10. It reveals that the effect of slag on the alloy is more significant with the increase of CaO addition. And then on the alloy surface, the oxidation phenomena and the impurities (O, N, H) are not detected when the alloy is synthesized. It illustrates these impurities are effectively prevented from entering the alloy under vacuum.



FIGURE 12: Microstructure of slag and alloy of sample No. 4.

TABLE 10: Compositions of samples present in the laboratory samples (wt %).

Commlo	Doint	Composition (wt%)				
Sample	Politi	CaO	Al_2O_3	Al	V	
Slag	1	35.8	64.2	0.0	0.0	
	2	21.9	78.1	0.0	0.0	
Alloy	1	0.0	0.0	41.7	58.3	
	2	0.0	0.0	40.0	60.0	

4. Conclusions

Laboratory experiments were carried out using V_2O_3 , CaO, and Al as raw materials. Slag and alloy samples were detected by EPMA to identify the content of elements and analyze their compositions, and at the same time, different heating times have also been studied. The results showed that with the extension of the heating time, the separation effect of the alloy and the slag and the uniformity of the alloy are significantly improved. The slag is fully separated from the alloy, and the composition of the alloy is more uniformly distributed when the heating time is controlled at 1 hour by vacuum resistance furnace. The synthesized alloy is not oxidized when the raw material is heated under vacuum.

In the study of the optimization of the raw material ratio, it was found that the increase in the Al content affects the composition of the alloy. The composition of the obtained alloy composition meets commercial standards when the amount of Al added is controlled to be 31-36 wt%. Then, the effect of the different proportions of CaO on the quality of slag series and alloys has also been studied. The results show that the composition of the slag system can be effectively changed by increasing the proportion of CaO, so that the flow effect of the liquid slag during the heating process is improved, and the uniformity of the alloy composition is also optimized. When the proportion of CaO is 27 wt%, the alloy and slag are well separated, and the composition distribution in the obtained alloy is more uniform. To achieve more efficient Al-V alloy production and reduce its environmental impact, it is suggested that resistance furnace should be applied in the industrial production of high-quality Al-V alloy.

Data Availability

All the data are contained in the manuscript.

Conflicts of Interest

The authors declare no conflicts of interest.

Acknowledgments

The authors are grateful for the financial support by the National key Research and Development Program (No. 2016YFC0400403), Sustainable Development Demonstration Zone Special Project of Chengde (No. 202008F003), and Construction of Vanadium and Titanium Industry Technology Innovation Research Institute (No. 202008F027).

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