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Removing Ti_5Si_3 phase in Ti alloy via desilication of upgraded titania slag using low-temperature alkali leaching

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Abstract: Silicon in two types of upgraded titania slag (UGS) containing different components was removed using a low-temperature alkali solution and melt leaching. The effects of temperature, time, and NaOH solution concentration on desilication rate were investigated. Desilication in melt can lead to the loss of titanium in upgraded titania slag. After agitating at a speed of 600 r/min and 80 °C for 300 min, the silicon content in domestic slag decreased from 0.96 wt.% to 0.29 wt.%, and the leaching rate was 69.8% after leaching in 4 mol/L sodium hydroxide solution. Under the same conditions, the silicon content in imported slag decreased from 1.11 wt.% to 0.12 wt.%, and the desilication rate was up to 89.2%. The alloy powders obtained from the reduction of upgraded titania slag with magnesium were sintered to produce titanium alloy, and the alloy was characterized. It was determined that the Ti_5Si_3 phase in the titanium alloy product was successfully eliminated. In addition, the two UGS materials and the chemical principle of desiliconization were analyzed using a thermodynamic software.

Key words: upgraded titania slag; alkali leaching; desilication; Ti_5Si_3 phase

1 Introduction

As a titanium-rich material with more than 85 wt.% titanium dioxide and a small amount of valuable alloying elements, including Fe, Mn, Al, and Si, upgraded titania slag (UGS) has been used for the preparation of titanium dioxide and titanium chloride [1−4]. The mainstream methods used to prepare titanium dioxide from UGS include the sulfuric acid method [5−7] and the chlorination method [8−10]. The most significant disadvantage of titanium dioxide production through the sulfuric acid method is the generation of by-products that include $FeSO_4$ and dilute H_2SO_4 waste liquids. These two varieties of by-products not only result in severe environmental pollution but also incur high recovery costs, which restricts the further development of the titanium dioxide industry. Materials and products involved in the production of titanium dioxide via chlorination are the most toxic and corrosive. Moreover, it is difficult to treat the generated waste [11−14].

Recently, our team has proposed the preparation method of titanium alloys from UGS, which can significantly improve the time efficiency of the existing process flow [15,16]. Titanium alloy was prepared by sintering the alloy powder obtained through the reduction of UGS using a one-step process involving magnesium powder. However, the prepared titanium alloy contained a certain amount of Si (>1 wt.%), which can lead to the formation of the Ti_5Si_3 phase with titanium, thereby reducing the plasticity of titanium alloy to a

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significant extent [17−19]. The addition of silicon in titanium alloys enhances the tensile strength and creep properties via silicide precipitation, and the silicon content should be as low as 0.1−0.2 wt.% to prevent the coarsening of titanium silicide [20−22].

To prevent the negative impact of Ti_5Si_3 on the plasticity of the titanium alloy while reducing the content of Si in the alloy, desilication of two kinds of UGS (of which one was produced by a domestic titanium factory and the other by Rio Tinto) by means of alkali leaching was performed in this study. In addition to investigating the difference between the removal of Si in NaOH melt and solution, a study was conducted regarding the effects of the concentration of NaOH solution, leaching temperature, time, and stirring speed on the outcome of Si removal. By comparing these two types of raw materials, a study was conducted on the differences in desilication via the alkali leaching of these two types of UGS. This study provides not only a technical reference for the desilication of high-titanium slag but also a new technique for the preparation of low-silicon titanium alloys via thermal reduction.

2 Experimental

2.1 Experimental methods

The process flow used in this study is illustrated in Fig. 1.

As shown in Fig. 1, the prepared solution was firstly poured into a 250 mL three-neck flask before being placed in a constant-temperature oil bath formulated using a hot plate equipped with a temperature control system (accuracy ±2 °C). Subsequently, a condenser with circulating ice water was employed to minimize the potential evaporation of the solutions from the reactor during the experiment. Next, the UGS was ground to less than 38 μm and then added to a flask when the expected temperature was reached. Afterward, the agitator was activated, and the slurry was stirred at specified intervals. Following the reaction, the slurry was swiftly removed, and cold water was added for cooling, followed by filtration and drying. The slurry was washed with deionized water for 30 min and dried. Finally, the silicon content in the residue was analyzed using ICP-OES, and the leaching rate of silicon was calculated using the following equation:

$$X=(1-x_r/x_s)\times100\% \quad (1)$$

where X represents the leaching rate of Si; x_r indicates its mass fraction in the residue, and x_s denotes the mass fraction in the slag.

The materials and products used in the study were characterized through the X-ray fluorescence (XRF), ICP-OES, and mineral liberation analyzer (MLA). The micromorphology of the sintered alloy was analyzed using scanning electron microscopy (SEM) along with energy-dispersive spectroscopy (EDS).

2.2 Experimental materials

Two varieties of UGS were used in this study: one produced by a titanium smelting enterprise in China and the other produced by Rio Tinto. Electronic-grade NaOH was purchased from Aladdin. Anhydrous $MgCl_2$ powder was used as flux. Mg metal powder with particle sizes between 75 and 150 μm, sourced from Sinopharm Chemical Reagent Co., Ltd., was used as the reducing agent. Figures 2 and 3 show the composition of these two types of UGS as determined using the MLA. As shown in Fig. 2, the rutile phase in the domestic high-titanium slag contains elements such as Al,

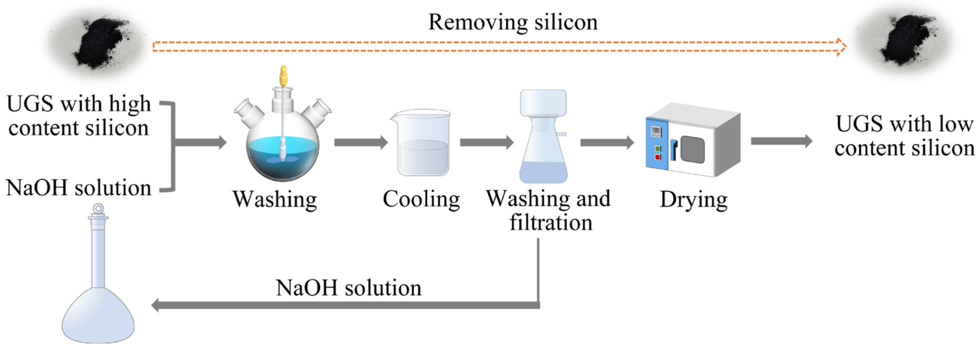

Fig. 1 Flowchart of desilication of UGS

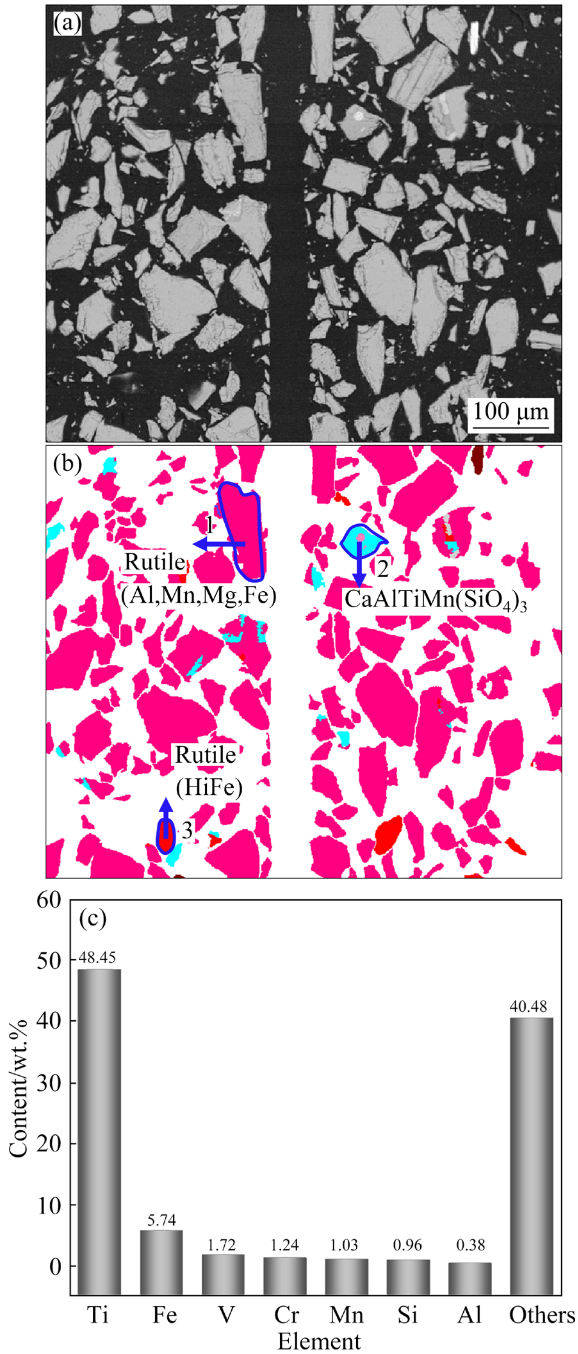

Fig. 2 Chemical composition and phase of UGS from China: (a) Backscattered image; (b) Mineral distribution image; (c) Elemental composition

Mn, and Fe, with Si present primarily in the form of complex silicate. The Ti content in the slag was 48.45 wt.%, and the Fe content was 5.74 wt.%. The slag produced by Rio Tinto was relatively pure in composition, as shown in Fig. 3. Si primarily existed in the form of quartz. The Ti content in the slag was 54.89 wt.%, and the Fe content was 2.11 wt.%; other elements were mostly oxygen and trace metal elements.

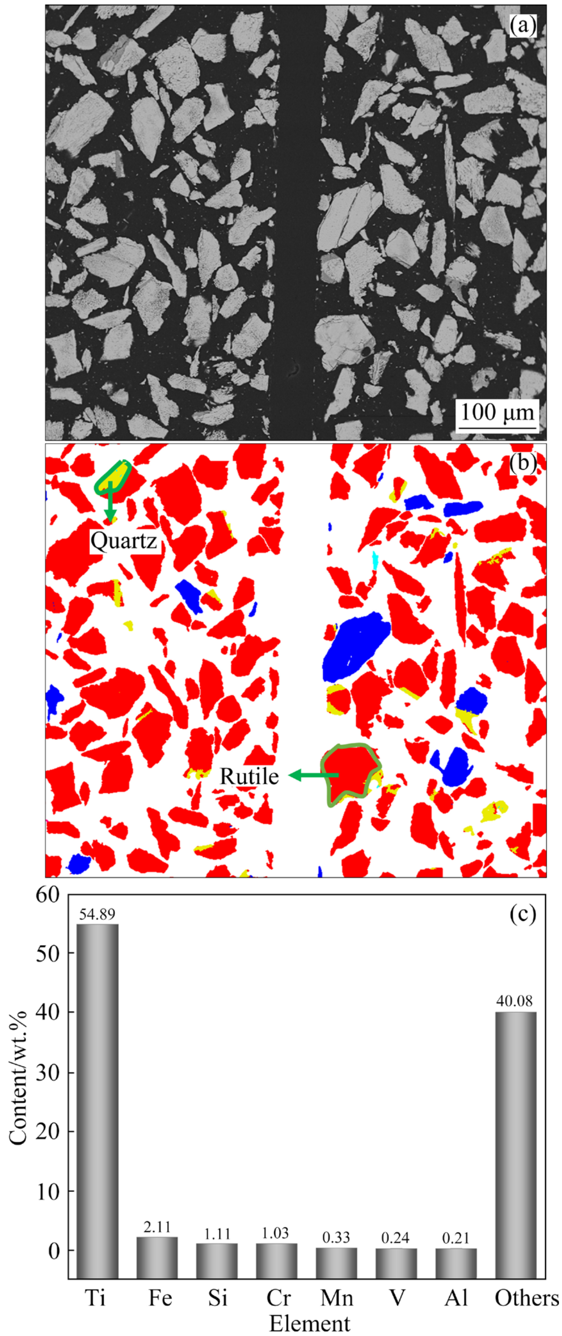

Fig. 3 Chemical composition and phase of UGS produced by Rio Tinto: (a) Backscattered image; (b) Mineral distribution image: (c) Elemental composition

3 Results and discussion

3.1 Chemical principle of desilication

Most of Si in UGS exists in the form of free SiO_2, although some is in the form of complex silicate compounds. Under certain conditions, free SiO_2 can react with NaOH solution, whereas the reaction between complex silicates and NaOH is more complex. Therefore, the Factsage and HSC chemical thermodynamic softwares were used to briefly explain the principle of chemical reaction. Figures 4(a−c) show the potential (φ)−pH diagrams of various reaction systems at 80 °C. Figure 4(a) shows the equilibrium composition of the Na−Si−H_2O system, which indicates that SiO_2 reacts with NaOH to form water-soluble $NaSiO_3$ in a 4 mol/L NaOH solution. Figures 4(b) and (c) show the φ−pH diagrams of Si−Na−Ca−H_2O and Si−Na−Mg−H_2O, respectively. These diagrams also indicate that complex silicates do not react with the NaOH solution to form soluble substances. The equilibrium composition results (Fig. 4(d)) calculated using the HSC thermodynamic software suggest that TiO_2 can also react with pure NaOH melt to form titanate, resulting in the loss of TiO_2. Therefore, the removal of Si using NaOH solution and NaOH melt is discussed.

3.2 Desilication of domestic UGS

3.2.1 Experimental factors affecting desilication in NaOH solution

A single-factor experiment was performed to investigate the effects of agitation speed, temperature, holding time, and NaOH solution concentration on the desilication of the UGS produced in China, and the results are shown in Fig. 5.

As shown in Fig. 5, when the volume was 250 mL, the mass of titanium slag was 15 g, and the NaOH solution concentration was 4 mol/L, the Si content in the slag first decreased and then increased slightly as the temperature increased from 40 to 100 °C, which may be attributed to the high temperature and the failure of fine crystals attached to the surface of the UGS to be washed thoroughly after the reaction between the NaOH solution and UGS. At 80 °C, the Si content was 0.29 wt.%, and the removal rate of Si was 69.8%. As the leaching temperature continued to increase, the removal rate of Si showed a slight decline. In addition, as the leaching temperature increased, it became more difficult to wash the UGS in the solution. Therefore, 80 °C was considered the optimal temperature. Figure 5(b) shows the effect of agitation speed on

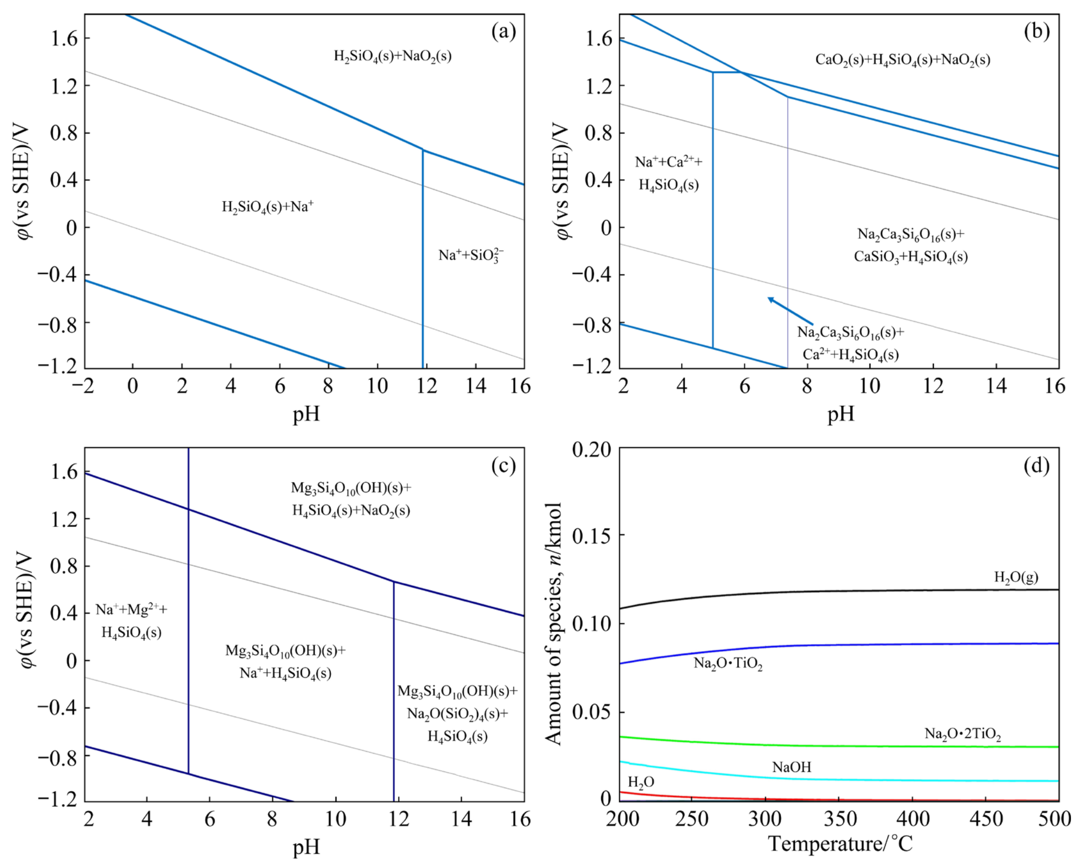

Fig. 4 φ−pH diagrams of Si−Na−H_2O (a), Si−Na−Ca−H_2O (b), and Si−Na−Mg−H_2O (c) systems at 80 °C calculated using Factsage software, and equilibrium diagram of TiO_2−NaOH system calculated using HSC software (d): (a) 0.5<Na/(Si+Na) molar ratio<0.667; (b) Na/(Si+Na+Ca) molar ratio=0.04, Ca/(Si+Na+Ca) molar ratio=0.15; (c) Na/(Si+Na+Mg) molar ratio=0.04, Mg/(Si+Na+Mg) molar ratio=0.150

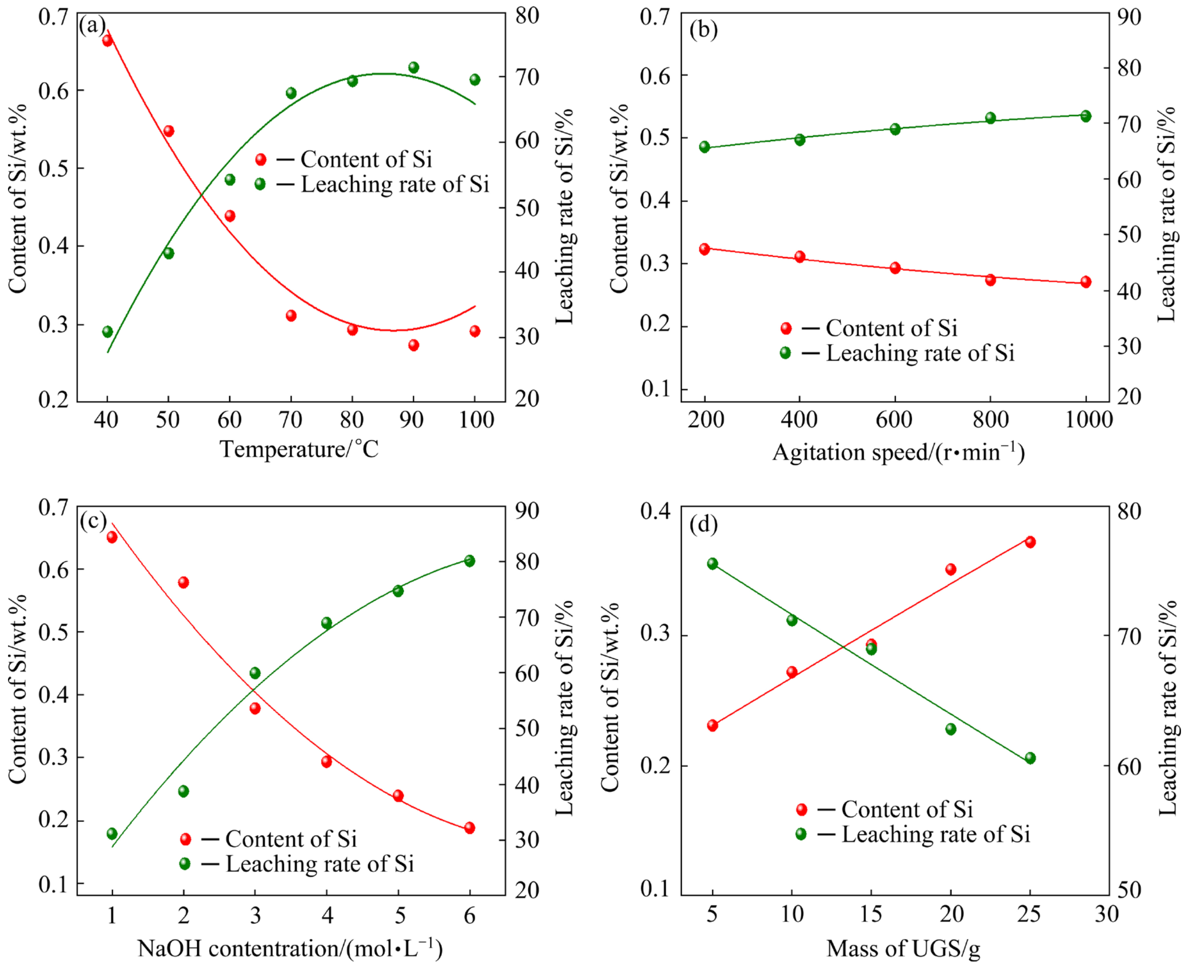

Fig. 5 Effects of temperature (a), agitation speed (b), NaOH concentration (c), and raw material mass (d) on content and leaching rate of Si from domestic UGS

the leaching rate of Si. As the speed increased from 200 to 1000 r/min, the leaching ratio also increased.

An agitation speed of 600 r/min and a solution volume of 250 mL were set as the experimental conditions, and the effect of NaOH concentration on the leaching rate of Si was investigated, the results are shown in Fig. 5(c). Clearly, the Si content in the UGS decreased rapidly with increasing NaOH concentration. When the NaOH concentration was 4 mol/L, the Si content was 0.29 wt.%. As the NaOH concentration increased beyond 4 mol/L, the leaching rate of Si began to decreased slightly. Figure 5(d) shows the leaching rate of Si obtained when the slag of varying quality is added. As suggested by the results, the leaching rate decreased at a slow pace with an increase in the slag quality.

These results suggest that temperature has the most significant impact on the outcome of desilication. Therefore, the changes in the silicon leaching rate with temperature were explored at various leaching time; a NaOH concentration of 4 mol/L and a volume of 250 mL, and an agitation speed of 600 r/min were utilized, and an the results are shown in Fig. 6.

From Fig. 6, the Si content in the residue was less than 0.3 wt.% when the temperature was at least 80 °C and the duration exceeded 300 min, and the leaching rate was higher than 70%. Therefore, to improve the leaching rate, it is necessary to restrict the duration to above 300 min and the temperature to above 80 °C.

3.2.2 Evolution of elemental composition after leaching in NaOH solution

The desilication reaction in NaOH solution was performed to ensure the removal of Si content, and it is also expected to maintain the consistency of the Ti content. Table 1 lists the contents of the elements present in the slag after being held for 300 min at various leaching temperatures.

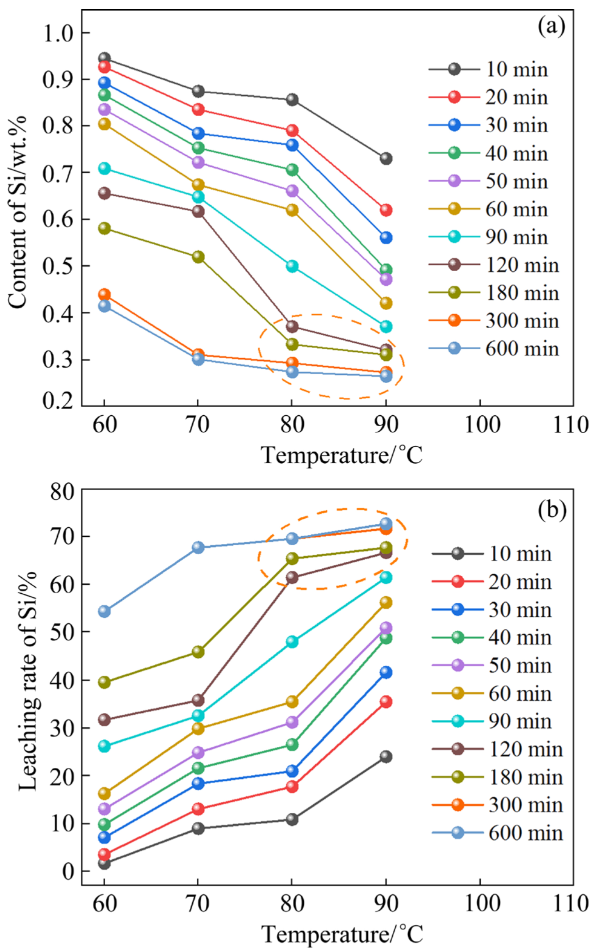

Fig. 6 Effects of temperature on desilication of UGS from China: (a) Content of Si; (b) Leaching rate of Si

Table 1 Elemental contents of UGS after being held in NaOH solution for 300 min at various leaching temperatures

T/°C	Elemental content/wt.%				
	Ti	Fe	Mn	Al	Si
60	45.46	8.31	2.07	0.91	0.51
70	45.87	8.16	2.06	0.97	0.47
80	46.61	8.05	2.07	0.87	0.29
90	45.86	7.92	2.03	0.78	0.31

From Table 1, not only does the desilication in NaOH solution contribute to the leaching of Si, but also ensures the preservation of Ti. Compared with the raw material, the Ti content in the leaching residue showed a slight decrease, from 48.45 wt.% to approximately 46 wt.%.

3.2.3 Desilication in molten NaOH

The desilication of UGS in NaOH melt was also investigated. Specifically, 40 g of solid NaOH was weighed and mixed with 15 g of UGS before being placed in a stainless-steel crucible. The stainless-steel crucible was then placed in a well furnace, and the furnace was heated to 350, 400, and 450 °C, respectively. After holding for 3 h, the temperature in the furnace was reduced to 30 °C. The leached slag was then removed, washed, and dried. Table 2 lists the elemental contents of the leached slag.

Table 2 Elemental contents of UGS after being held in NaOH melt for 180 min at various leaching temperatures

T/°C	Elemental content/wt.%					
	Ti	Fe	Mn	Al	Si	Na
350	41.31	8.12	1.86	0.12	0.31	10.57
400	39.96	8.15	1.78	0.05	0.27	12.67
450	40.50	8.25	1.74	0.04	0.29	11.63
500	39.99	8.03	1.86	0.03	0.35	12.76

According to Table 2, the leaching rate of Si did not significantly change during the desilication of domestic UGS with NaOH melt. The Ti content also showed a decreasing trend during leaching, which is attributable to the partial reaction of TiO_2 with the NaOH melt.

3.3 Desilication of UGS produced by Rio Tinto

To compare the leaching rate of Si in UGS produced both domestically and abroad, an investigation of the Si leaching rate of UGS produced by Rio Tinto was conducted. With the agitation speed of 600 r/min, the effects of NaOH concentration, UGS quality, temperature, and time on the desilication of UGS produced by Rio Tinto were investigated, and the results are shown in Fig. 7.

According to Fig. 7, temperature has the most significant effect on the leaching of Si in the UGS produced by Rio Tinto. Because the SiO_2 phase in the imported UGS exists primarily in the form of relatively pure quartz, the desilication effect is more significant than that in the domestic UGS. With heating at 80 °C for 5 h, it was possible to obtain a Si content as low as 0.12 wt.% when 15 g of UGS was added. The Si leaching rate was 89.2%, which was higher than that of domestic UGS.

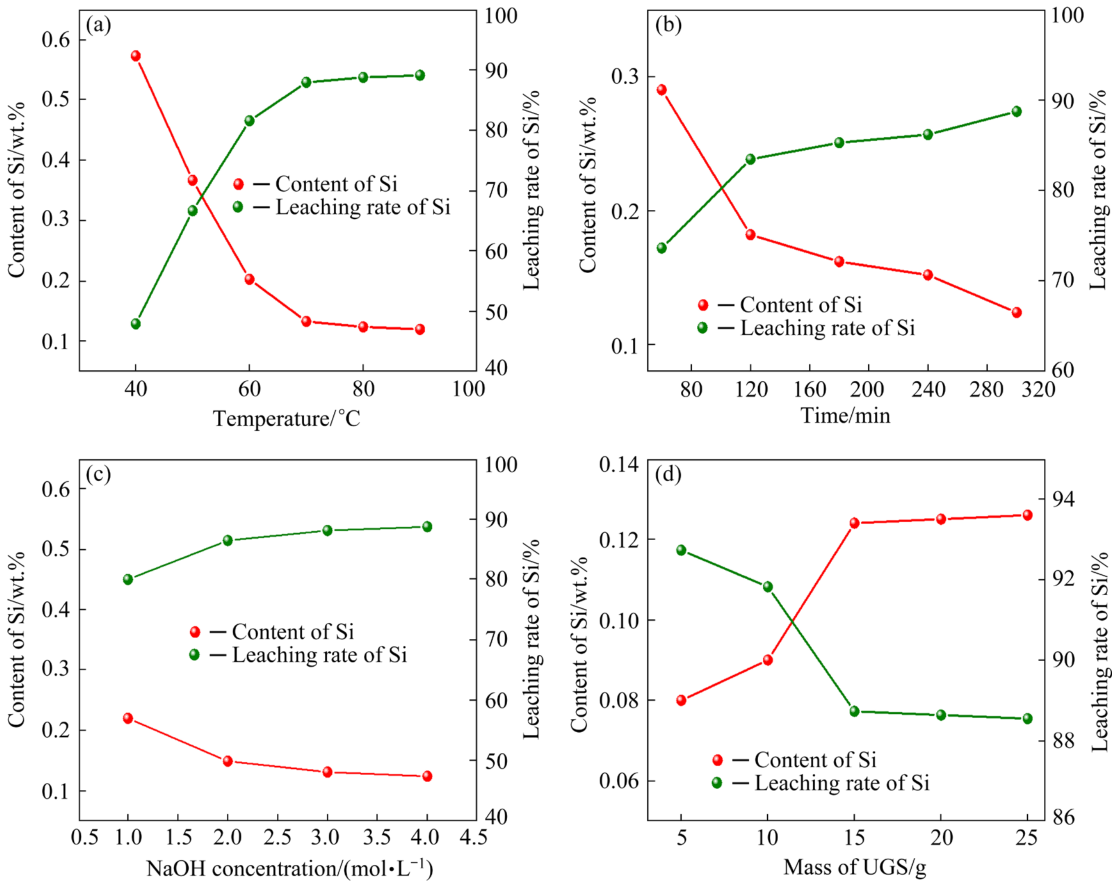

Fig. 7 Effects of temperature (a), time (b), NaOH concentration (c), and mass of UGS (d) on content and leaching rate of Si for UGS produced by Rio Tinto

3.4 Effect of Si content in UGS on microstructure of alloy

Ti alloy powder was prepared via the Mg reduction of UGS before and after desilication. Subsequently, the alloy powder was subjected to cold isostatic pressing and sintering, and the morphology of the sintered alloy products was observed before and after desilication. The results of which are shown in Figs. 8−11. Figures 8 and 9 present cross-sectional views of the alloy prepared before and after the desilication of the domestic UGS, respectively.

An analysis of the section morphology exhibited by the Ti alloy derived from domestic UGS was conducted to reveal the elemental distribution in the alloy. The Si in the alloy was primarily combined with Fe, as shown in Fig. 8. After desilication, as shown in Fig. 9, the content of Si in the Ti alloy prepared via Mg reduction decreased abruptly, with a small amount of remaining Si combining with Fe.

The Fe content of the Ti alloy prepared by reducing UGS produced by Rio Tinto was lower than that of the Ti alloy prepared by reducing domestic UGS. Prior to desilication, three phases were present in the alloy. As shown in Fig. 10, the presence of Si leads to the formation of the Ti_5Si_3 phase. After desilication, only two phases were observed in the alloy, and the Ti_5Si_3 phase was difficult to detect, as shown in Fig. 11.

The Si contents of the alloy are listed in Table 3. The content of Si in the alloy obtained by the reduction of imported UGS was 0.18 wt.%, suggesting that the formation of the Ti_5Si_3 phase was prevented.

Table 3 Contents of Si in raw materials and product detected using ICP-OES (wt.%)

Type	UGS (domestic)	UGS (imported)
UGS (before desilication)	0.96	1.11
UGS (desilication)	0.29	0.12
Alloy (before desilication)	1.11	1.51
Alloy (desilication)	0.37	0.18

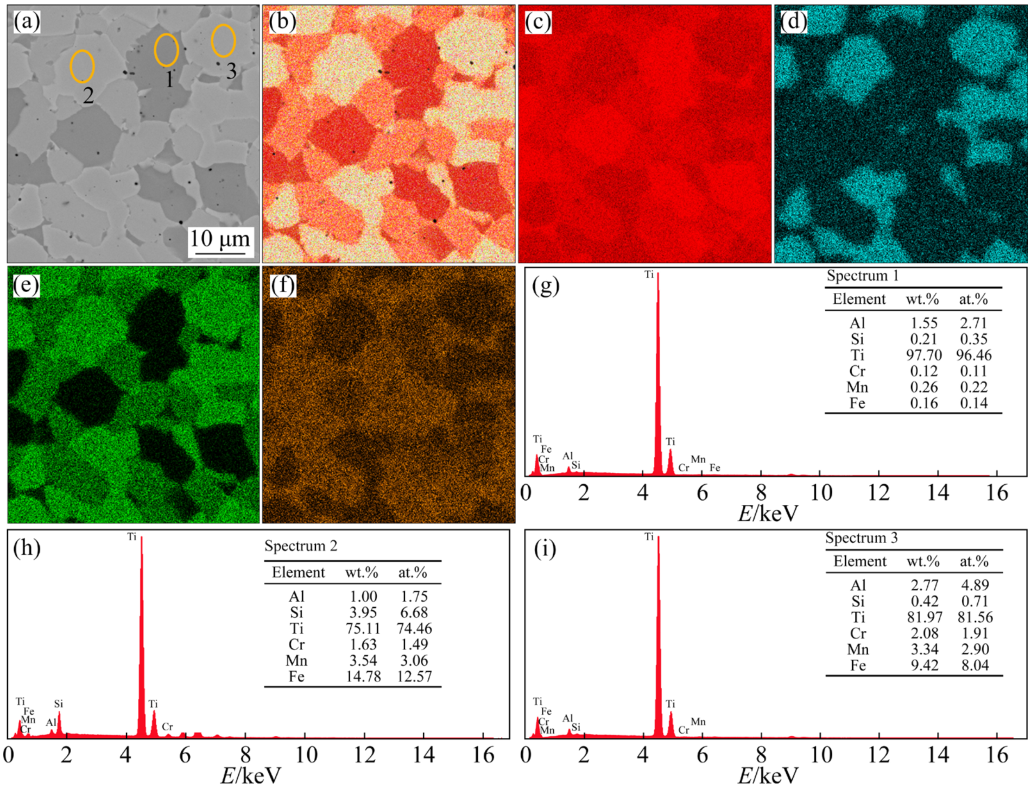

Fig. 8 SEM image and EDS mapping results for Ti alloy prepared via reduction of domestic UGS before desilication: (a) Backscattered image; (b) Combination of EDS map; (c–f): EDS mapping patterns of Ti, Si, Fe, and Al, respectively; (g) EDS result at Point 1; (h) EDS result at Point 2; (i) EDS result at Point 3

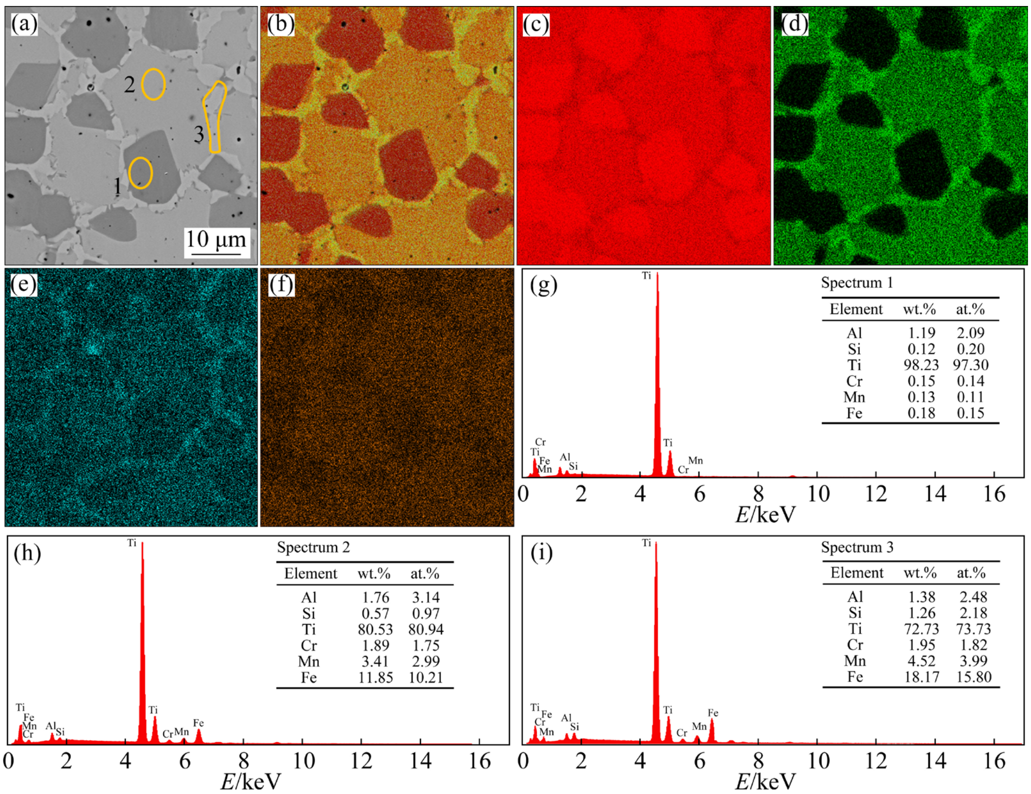

Fig. 9 SEM image and EDS mapping results for Ti alloy prepared via reduction of domestic UGS after desilication: (a) Backscattered image; (b) Combination of EDS map; (c–f): EDS mapping patterns of Ti, Fe, Si, and Al, respectively; (g) EDS result at Point 1; (h) EDS result at Point 2; (i) EDS result at Point 3

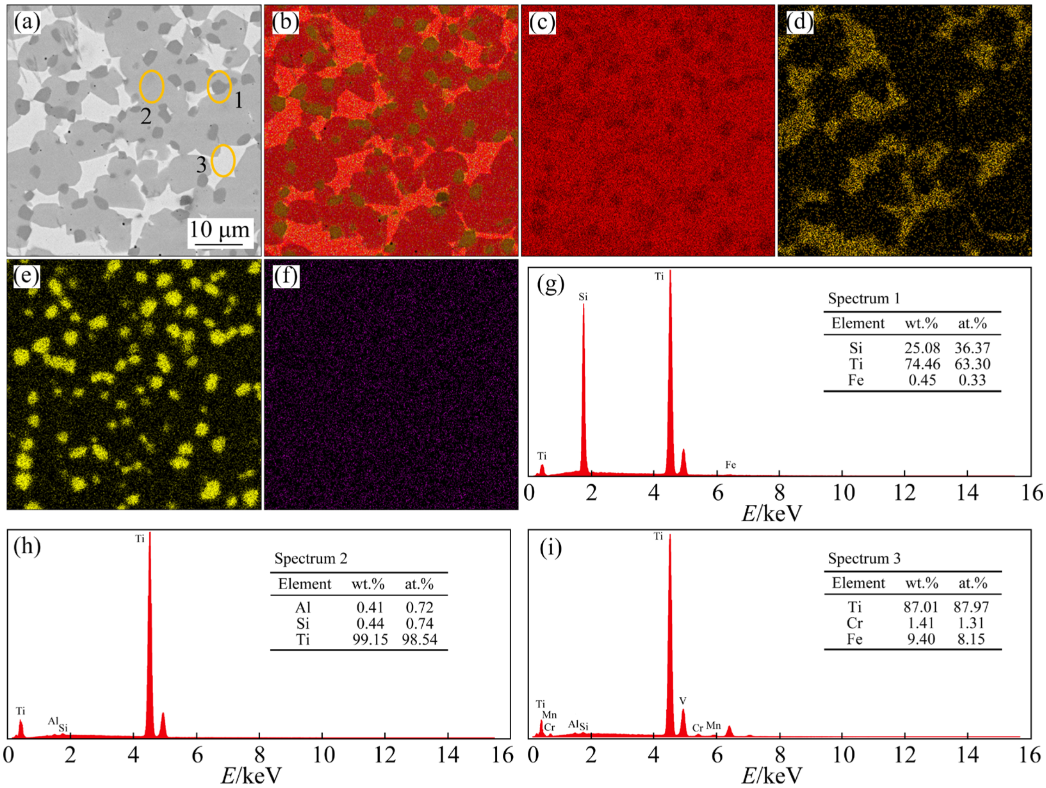

Fig. 10 SEM image and EDS mapping results for Ti alloy prepared via reduction of UGS produced by Rio Tinto before desilication: (a) Backscattered image; (b) Combination of EDS map; (c–f): EDS mapping patterns Ti, Fe, Si, and Cr, respectively; (g) EDS result at Point 1; (h) EDS result at Point 2; (i) EDS result at Point 3

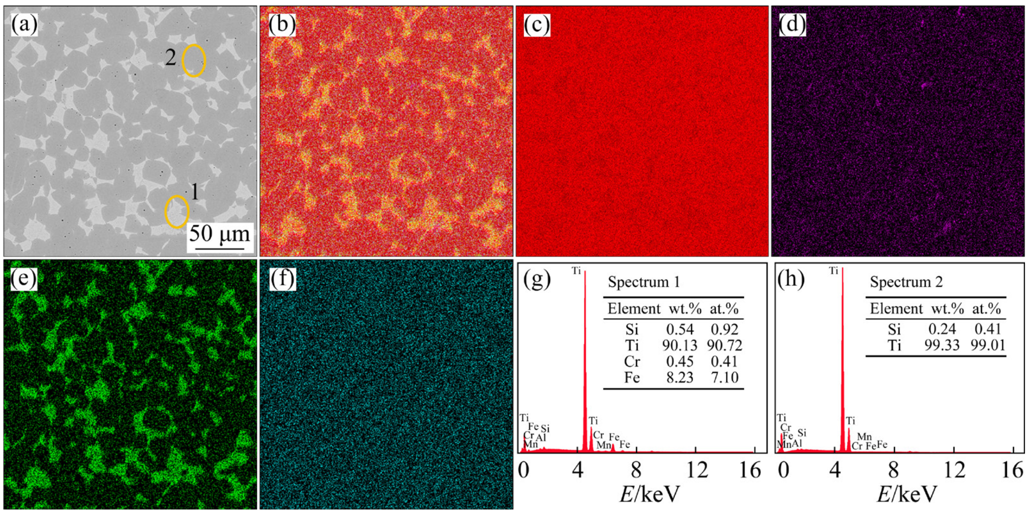

Fig. 11 SEM image and EDS mapping results for Ti alloy prepared via reduction of UGS produced by Rio Tinto after desilication: (a) Backscattered image; (b) Combination of EDS maps; (c–f): EDS mapping patterns of Ti, Si, Fe, and Cr, respectively; (g) EDS result at Point 1; (h) EDS result at Point 2

4 Conclusions

(1) After agitating at a speed of 600 r/min and 80 °C for 300 min, the Si content in the domestic leached slag was 0.29 wt.%, and the leaching rate was 69.8%. The Si content of the alloy decreased from 1.11 wt.% to 0.37 wt.%. Under the same conditions, the Si content in the imported slag was as low as 0.12 wt.%, and the highest desilication rate was 89.2%. The Si content of the alloy decreased from 1.51 wt.% to 0.18 wt.%.

(2) Desilication can be achieved using both an aqueous NaOH solution and melt. Desilication using an aqueous solution is effective in ensuring the stability of the Ti content, while desilication using melt can lead to the partial loss of Ti.

(3) Silicon in domestic UGS is composed of complex silicates, and the leaching rate is significantly lower than that in UGS produced by Rio Tinto. In addition, due to the high Fe content in domestic UGS, Si mainly forms Fe−Si phase. By removing Si, not only can Ti_5Si_3 phase be eliminated, but also Fe−Si phase be removed.

(4) The desilication of UGS contributes to the removal of the Ti_5Si_3 phase during the preparation of the Ti alloy through the Mg thermal reduction of UGS, which may be beneficial to improving the properties of the prepared alloy.

Acknowledgments

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低温碱浸高钛渣脱硅去除钛合金中的 Ti_5Si_3 相

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摘 要：利用低温碱溶液及熔体浸出对两种不同的高钛渣进行脱硅，研究温度、时间及氢氧化钠溶液浓度对脱硅率的影响。熔体除硅可导致高钛渣中的钛损失。用 4 mol/L 的氢氧化钠溶液浸出高钛渣，在温度为 80 °C、搅拌速率为 600 r/min 及保温时间为 300 min 的条件下，国内高钛渣中硅含量由 0.96%(质量分数)降低至 0.29%(质量分数)，脱硅率为 69.8%。在相同条件下，进口的高钛渣中硅含量由 1.11%(质量分数)降低至 0.12%(质量分数)，脱硅率为 89.2%。对采用镁还原高钛渣得到的合金粉末进行烧结，制备钛合金，并对合金进行表征。结果表明，钛合金中的 Ti_5Si_3 相已成功去除，此外，对两种高钛渣原料进行对比分析，并利用热力学软件对脱硅原理进行分析。

关键词：高钛渣；碱浸；脱硅；Ti_5Si_3 相

(Edited by Wei-ping CHEN)