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Research Article

Effects of mixing technique, molar ratio, and filtration methods on the production of titanium power from secondary titanium slag through magnesiothermic reduction

Nam Ho Phung Khac, Duong Duc La, Hoai Phuong Nguyen Thi*

ABSTRACT

The application of titanium has been widening and the demand for titanium has also been increasing in recent years as it is a lightweight, extremely strong, and highly corrosion resistant metal. The most popular production process for titanium metal in the world is the Kroll process. However, this method is complicated, chlorine waste generated during the chlorination process, which causes serious environmental problems., Furthermore, its product cost is high mainly dueto the multistep, time-consuming, and high-temperature batch-type process. During the last decade a variety of investigations have been conducted to find alternative routes to the Kroll process for production of titanium metal. Various reductants and feed materials have been investigated for the production of titanium metal. In this study, magnesiothermic reduction has been considered as effective pathway to fabricate titanium powder from secondary titanium slag. The feed material (Secondary titanium slag from Binh Dinh Mineral Joint Stock Company), reductant (Mg), and $MgCl_2.6H_2O$ were mixed and reduced in an argon atmosphere at 900 ^{o}C in 4 h. To obtain good yield and quality of the titanium product, many factors such as: mixing method of raw materials, the molar ratio of Mg/TiO_2, and filtering and separating the products (water washing and acid washing) should be optimized. In this study, these influencing factors were extensively investigated and optimized. The prepared titanium powder was characterized using X-ray diffraction, energy-dispersive X-ray spectroscopy, and scanning electron microscopy. The research results showed that with the molar ratio of Mg/TiO_2 of 2.6, interleaved or even disposition of raw materials were optimal conditions to convert secondary titanium slag to titanium powder. The titanium product after pyrolysis washed and filtered with water followed by the acid washing, was a high-purity porous with a titanium content of up to 94.75%wt.

Key words: EDX, Magnesiothermic reduction, Secondary titanium slag, SEM, XRD

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INTRODUCTION

Titanium is the 9th most abundant element and makes up 0.6 % of the elements in the earth's crust, occurring mainly in the form of ilmenite (40-80% TiO_2), rutile (~95% TiO_2), anatate (> 95%), leucoxene (> 65% TiO_2)[1]. According to the U.S. Geological Survey (USGS) "Mineral Commodity Summaries 2023" (U.S. Geological Survey, 2023), World resources of anatase, ilmenite, and rutile total more than 2 billion tons, mainly distributed in Australia (reserves accounting for 27.29%), followed by China (27.14%), India (13.2%), Brazil (6.14 %), Norway (5.29 %), South Africa (5.21%), etc[2]. Vietnam is also a country, having potential reserves of titanium placers, located mainly along the coast of the Middle spreading across provinces from Thanh Hoa to Ba Ria - Vung Tau. One of the technologies being used in the mining and deep processing of titanium ores today is titanium slag production. The by-product of this process is secondary titanium slag with a low titanium dioxide content that cannot be exported, but is still higher level than in ilmenite and leucoxene ores (example, secondary titanium slag at Binh Dinh Mineral Joint Stock Company have TiO_2 content up to 85%).

Titanium and its alloys have special properties, such as low thermal expansion coefficient, good performance tribological properties[3], high strength/density ratio, high corrosion resistance, biocompatibility[4], high electrical conductivity, good ductility, excellent fracture resistance, cryogenic properties, shape memory behavior, and hydrogen affinity (for hydrogen storage)[5]... Thus, they have been applied to a broader range of applications. They are widely used for building aircraft turbine disks, blades, and airframe structural components in the aerospace industry[6]. More recently, porous titanium bulks have been extensively investigated as potential replacements for human bone[7]. Titanium and its alloys are widely

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used for medical and dental implant devices - artificial joints, bone fixators, spinal fixators, dental implant, etc[8].

Titanium sponge is commercially produced by the Kroll process, which is based on the magnesiothermic reduction of titanium tetrachloride ($TiCl_4$) in a sealed vessel filled with argon gas at 800-900 °C[9]. Overall the Kroll process can produce high quality titanium; however, its production cost is high mainly because of the multistep, high-temperature, and time-consuming batch-type process. Moreover, the Kroll process is complex, its product is contaminated with iron due to the reaction vessel, and the process causes serious environmental problems due to chloride wastes, generated in the chlorination process[10]. Given the above-mentioned background, many researchers in the world have endeavored to find alternatives to the Kroll process with new titanium production processes such as the Fray Farthing Chen (FFC) Cambridge process[11], Ono and Suzuki (OS) process[12], preform reduction process (PRP) [13], and hydrogen-assisted magnesiothermic reduction (HAMR) process[13,14]. These processes all use Calcium (Ca) and magnesium (Mg) are used as reducing agents, TiO_2 as raw material, which is safer to handle and easier to transport than $TiCl_4$, which is a highly toxic chemical. Besides, there were also some research works on the magnesiothermic reduction of titanium dioxide with the addition of magnesium dichloride ($MgCl_2$), sodium chloride (NaCl), magnesium chloride hexahydrate ($MgCl_2$. H_2O), calcium hydroxide ($Ca(OH)_2$) in hydrogen, nitrogen, or argon atmospheres to obtain titanium hydrogen, titanium nitride, or titanium[15–18].

Melting and boiling point of magnesium is less than that of calcium, so it is used as a reducing agent that could save energy in the reduction process in comparison to calcium[19]. Additionally, in terms of cost, magnesium is more affordable compared to calcium. Moreover, magnesium exhibits lower reactivity than calcium, resulting in less stringent requirements for labor safety, storage, and handling. Due to these benefits, magnesium is a favored reductant among researchers for the reduction of titanium dioxide.

In this study, method of magnesiothermic reduction has been used to fabricate titanium powder from secondary titanium slag (by-product of titanium slag production process at Binh Dinh Mineral Joint Stock Company) as a raw material in $MgCl_2.6H_2O$ media in argon atmosphere, based on the following consideration: mixing method of raw materials, the molar ratio of Mg/TiO_2, and filtering and separating the products (water washing and acid washing).

MATERIAL AND METHODS

Materials

Metallic magnesium (Mg) (purchase from Xilong, a particle size of around 6 mm, 99% purity); magnesium chloride hexahydrate ($MgCl_2.6H_2O$) (purchase from Xilong, with a purity of 98%). Secondary titanium slag with a particle mainly in the arrange of 0.08-0.35mm, (accounting for 96% of the weight) from Binh Dinh Mineral Joint Stock Company, its chemical Composition as shown in Table 1. Analytical-grade hydrochloric acid (37 wt%) (purchase from Xilong); pH paper (manufacturer: Merck); Distilled water, (origin: Vietnam). Analytical balance PA 213 OHAUS; kiln KJ-T1200R, particle size sieve set, porcelain bowl, mortar and pestle, crucible, measuring tube, beaker, and pipette.

Table 1: Chemical Composition of the secondary titanium slag

Compounds	Concentration (%)
TiO_2	86.60
Fe_2O_3	3.30
MnO	3.11
SiO_2	2.80
Al_2O_3	2.08
V_2O_5	0.56
CaO	0.56
MgO	0.45
K_2O	0.20
SO_3	0.13

Experiment

Fabricate titanium powder from secondary titanium slag

Titanium was fabricated by magnesiothermic reduction at 900 ^oC for 4 hours in argon atmosphere. The primary material was secondary titanium slag which was ground by a ball mill, then sifted to obtain a particle size under 0.08 mm. The secondary titanium slag powder and magnesium chloride hexahydrate salt were dried at 100 °C for 2 hours before starting the heating procedures. The materials loaded in a porcelain crucible, and fed into a rotary tube furnace (KJ-T1200R) for reduction. The cycle of heating, charging/discharging of argon and vacuum is carried out according to the diagram in Figure 1.

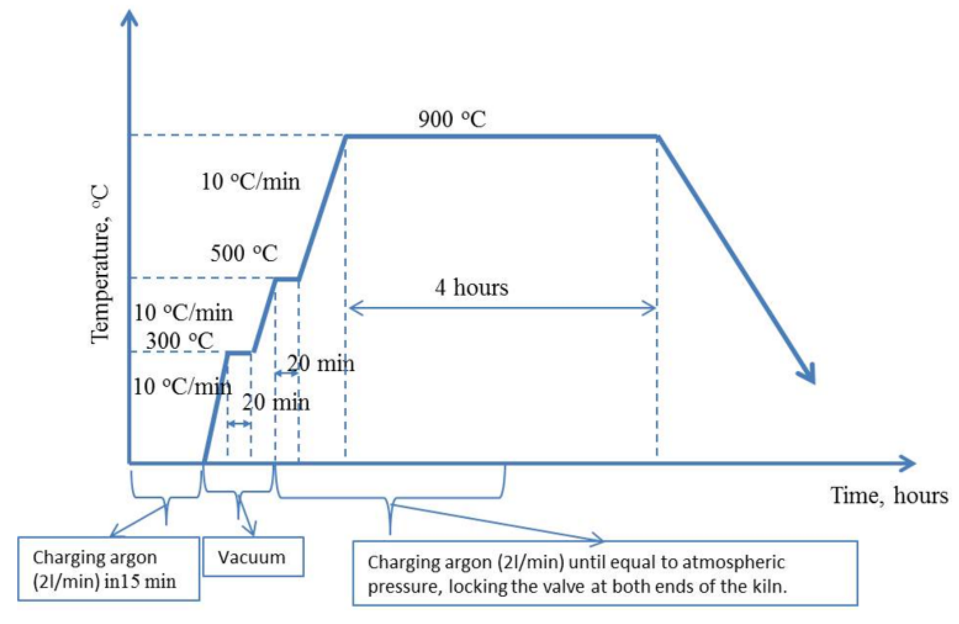

Figure 1: The cycle of heating, charging/discharging of argon and vacuum in the magnesiothermic reduction

The leaching after reduction was carried out as follows: after cooling to room temperature, the reacted mass was immersed in distilled water and stirred at room temperature. Then, it was filtered and washed several times with distilled water. Then excess acetic acid HCl 5% was added and stirred at room temperature for about 4 hours, then the remaining solid was filtered out and washed by distilled water until neutrality was achieved. The final product powder was oven-dried at 100 °C prior to further characterization.

Material Characterization

The chemical composition was determined by energy dispersive X-ray (EDX) spectroscopy and the morphology was determined by scanning electron microscopy (SEM) on the Hitachi S-4800 instrument. The material phase composition was characterized by X-ray diffraction (XRD) technique on X'Pert instrument using CuKα X-ray source with λ = 1.5406 Å, 45 kV, 40 mA, scan step 0.1 ^{o}/s, scanning angle from 5^o - 90^o. The ASTM D1895A (method of density determination) was employed to determine the bulk density of the material.

Effect of the mixing method

Experiments are carried out with 3 mixing methods for materials: 1) Secondary titanium slag, Magnesium, and magnesium chloride hexahydrate are mixed evenly (even disposition), then they charged in a porcelain crucible; 2) Magnesium and magnesium chloride hexahydrate are mixed evenly, charged in at the bottom of porcelain crucible, then secondary titanium slag was overlapped (individual disposition); 3) Magnesium, and magnesium chloride hexahydrate are mixed evenly, then set-up interleaved layers between this mixture and secondary titanium slag in a porcelain crucible (interleaved disposition). A detailed description of the mixing method is shown in the Figure 2.

Each experiment was carried out with 30 g of secondary titanium slag and 20.543 g of magnesium; Use magnesium chloride salt in sufficient quantity to completely dissolve the generated MgO (1 mol $MgCl_2$ dissolved completely to form 2 mol MgO) [19,20].

Effect of the molar ratio of Mg/TiO_2

Various Mg/TiO_2 molar ratios of 2/1, 2.6/1, and 3.2/1 were employed to investigate the effect of the Mg/TiO_2 molar ratio on the formation of titanium powder. The crystal phase of the products was characterized by powder X-ray diffraction (XRD).

Effect of filtering and separating methods (water washing, acid washing)

The reduced products of magnesiothermic reduction process were leached with distilled water and hydrochloric acid as mentioned above. Obtained products of the leaching (product after reducing, product

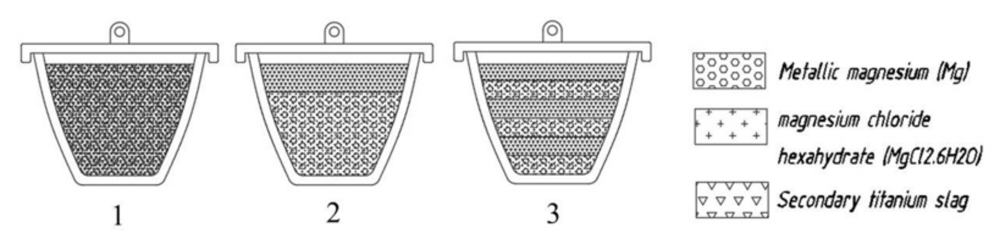

Figure 2: Mixing methods for materials: 1) Seven disposition, 2) individual disposition), and 3) Interleaved disposition

after water washing, and procduct after acid washing) were characterized by powder X-ray diffraction and energy-dispersive X-ray spectroscopy (EDX).

RESULTS AND DISCUSSIONS

Effect of mixing methods on the formation of titanium powder

The crystal phase of the products corresponding to the three methods of mixing materials was determined by X-ray diffraction. The results are as shown in Figure 3. Based on the XRD patterns provided, it is evident that when the magnesium and secondary titanium slag are not in close contact (individual disposition), the resulting product does not exhibit a titanium phase. Instead, several other phases such as $MgTiO_3$, $MgTi_2O_5$, and TiO_2 are observed. This lack of contact between magnesium and the secondary titanium slag likely leads to a significantly low reduction efficiency. On the other hand, in the cases of even disposition and interleaved disposition, there is substantial contact between magnesium and the secondary titanium slag, resulting in a high reduction efficiency. The products obtained in these two cases demonstrate a similar phase composition, primarily comprising a clearly discernible titanium phase with prominent peak patterns. Only a minor presence of Ti_5Si_3 impurities with low intensity is observed. So, methods of even disposition and interleaved disposition have equivalent effectiveness for formation of titanium powder. However, methods of even disposition are facilely implemented in the large scale, thus this mixing method is chosen for next experiments.

Effect of Mg/TiO_2 molar ratios on the formation of titanium powder

Figure 4 illustrates the XRD results obtained from the products synthesized using various Mg/TiO_2 molar ratios. By comparing the three XRD patterns corresponding to different molar ratios, it was observed that the product obtained with an Mg/TiO_2 molar ratio of 2.6 exhibits the highest and most distinct characteristic titanium peaks. This finding indicates that the optimal reconstitution of titanium is achieved when employing a molar ratio of Mg/TiO_2 equal to 2.6.

In magnesiothermic reduction, the theoretical stoichiometry suggests that 2 moles of magnesium should react with 1 mole of TiO_2. Nonetheless, magnesium particles frequently undergo oxidation, forming an oxide layer on their surface. Consequently, not all the material employed in the experiment consists of pure metallic magnesium. Additionally, some of the vaporized magnesium escapes and does not engage in the reduction reaction. Consequently, the actual molar ratio of Mg/TiO_2 is lower than the theoretical ratio of Mg/TiO_2. As a result, an excess amount of magnesium is often used to ensure the maximum efficiency of the reduction process. However, when the magnesium quantity is further increased, the excess magnesium no longer contributes to the reduction reaction, and titanium undergoes re-oxidation. Consequently, the titanium content decreases.

Effect of Mg/TiO_2 molar ratios on the formation of titanium powder filtering and separating methods (water washing and acid washing)

Table 2 and Figure 5 display the results of EDX measurements conducted on the composition of the products at different stages: after reduction, after water washing, and after acid washing. The product after reducing contains mainly the O, Mg, and Cl elements with high content (40.97% O, 24.59% Mg, and 26.62% Cl). Titanium content is low (5.77% Ti). X-ray diffraction spectrum (Figure 6) shows the presence of many phases: MgO phase with high peak, followed by $MgCl_2(H_2O)_6$ phase, and titanium phase with low peak intensity. For the sample after water washing, from the EDX measurement results, it

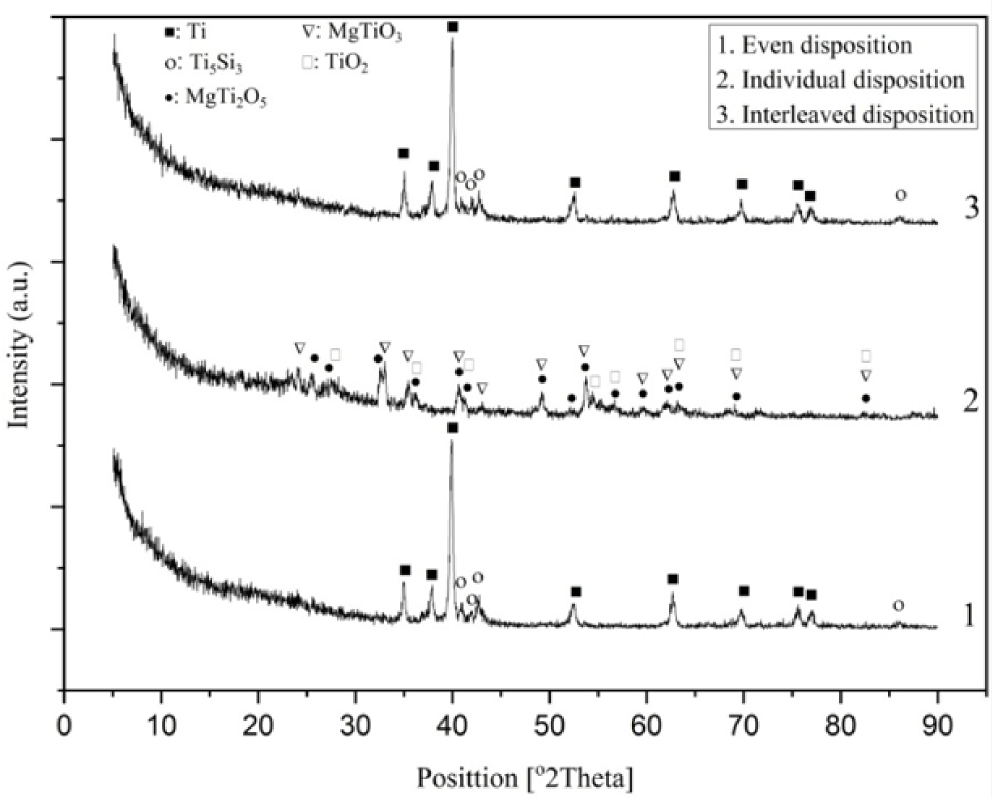

Figure 3: XRD profiles of the product corresponding to 3 methods of mixing materials: 1- Even disposition, 2- Individual disposition, 3- Interleaved disposition

was found that the chlorine content in the sample decreased significantly. However, the content of Mg and O were still high with weight percentages of 33.08% Mg, and 55.41% O, respectively, the titanium content increased slightly compared to the unwashed sample, but still at a low level (8.66% Ti). The results of X-ray diffraction measurement (Figure 5) showed that there was no longer $MgCl_2(H_2O)_6$ phase, because it was dissolved in water and filtered. This result is consistent with the EDX measurement result (chlorine content decreased significantly after water washing). The peak set of MgO phase is still high, and the peaks of the titanium phase with intensity also increases slightly (relatively compared with the peak of the MgO phase). In addition, $Mg(OH)_2$ phase appears, Ti_5Si_3 phase with low spectral line intensity.

The EDX measurements conducted on the samples after water and acid washing revealed a significant titanium content, reaching as high as 94.75%. The remaining impurities (such as Mg, Fe, Si, and Al) were found to be present in very small quantities. The X-ray diffraction spectrum also predominantly displayed the presence of the titanium phase, with only minimal impurities detected, indicated by low peak intensities, such as the Ti_5Si_3 phase. Consequently, after undergoing acid washing to dissolve $Mg(OH)_2$ and MgO, followed by filtration, the resulting titanium product exhibited high purity.

The morphology of titanium powder fabricated from secondary titanium slag by magnesiothermic reduction method with Mg/TiO_2 molar ratio of 2.6, with even disposition of raw materials is shown in Figure 7. The SEM images showed that the obtained product had irregular shape. We can see that the fabricated titanium powder has a relatively porous structure, some of which tend to agglomerate together possibly due to the temperature in reducing process is high, leading to melting of titanium powder and agglomeration of particles. The titanium slag is mixed with the Mg powder, when reacting at high temperature, the Mg vapor will reduce the titanium slag, resulting in the formation of metallic titanium with a porous structure.

The densities of the primary secondary titanium slag sample and the titanium powder product were evaluated according to ASTM D1895A, the results being

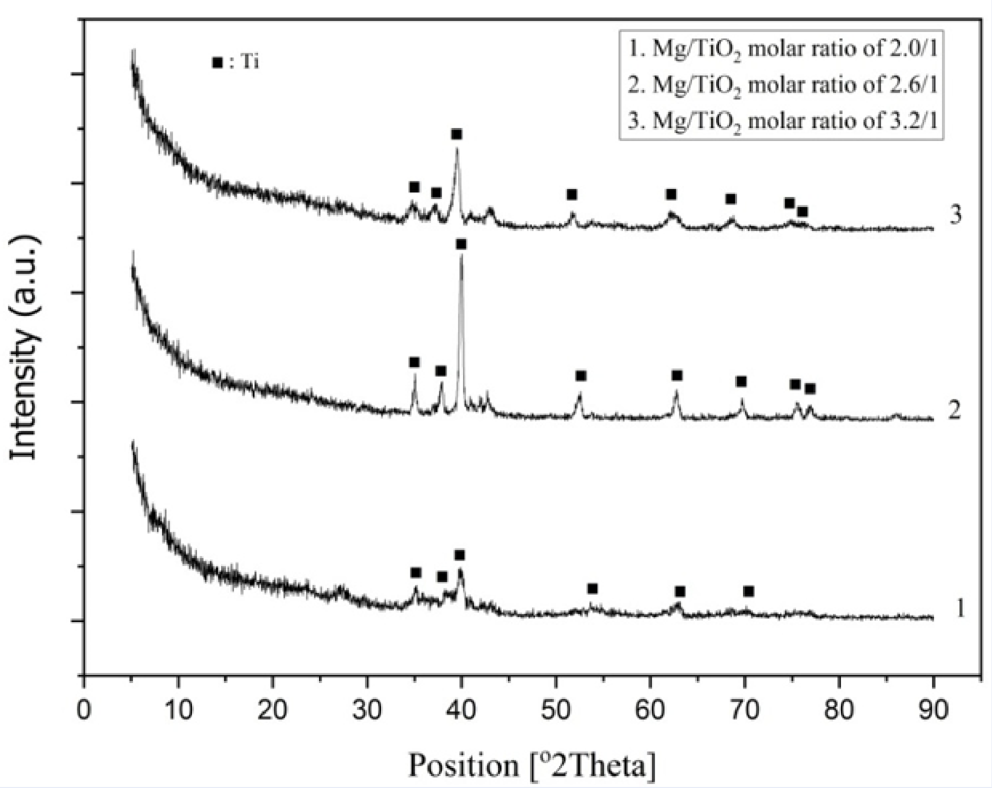

Figure 4: XRD patterns of the products various Mg/TiO_2 molar ratios of 2/1, 2.6/1, and 3.2/1

Table 2: Composition % by mass of elements in products of each leaching stage

Element	wt. %		
	product after reducing	product after water washing	product after acid washing
O	40.97	55.41	-
Mg	24.59	33.08	1
Al	-	0.41	2.46
Si	-	0.73	0.64
Cl	26.62	1.11	-
K	0.60	-	-
Ti	5.77	8.66	94.75
Fe	1.44	0.6	1.15

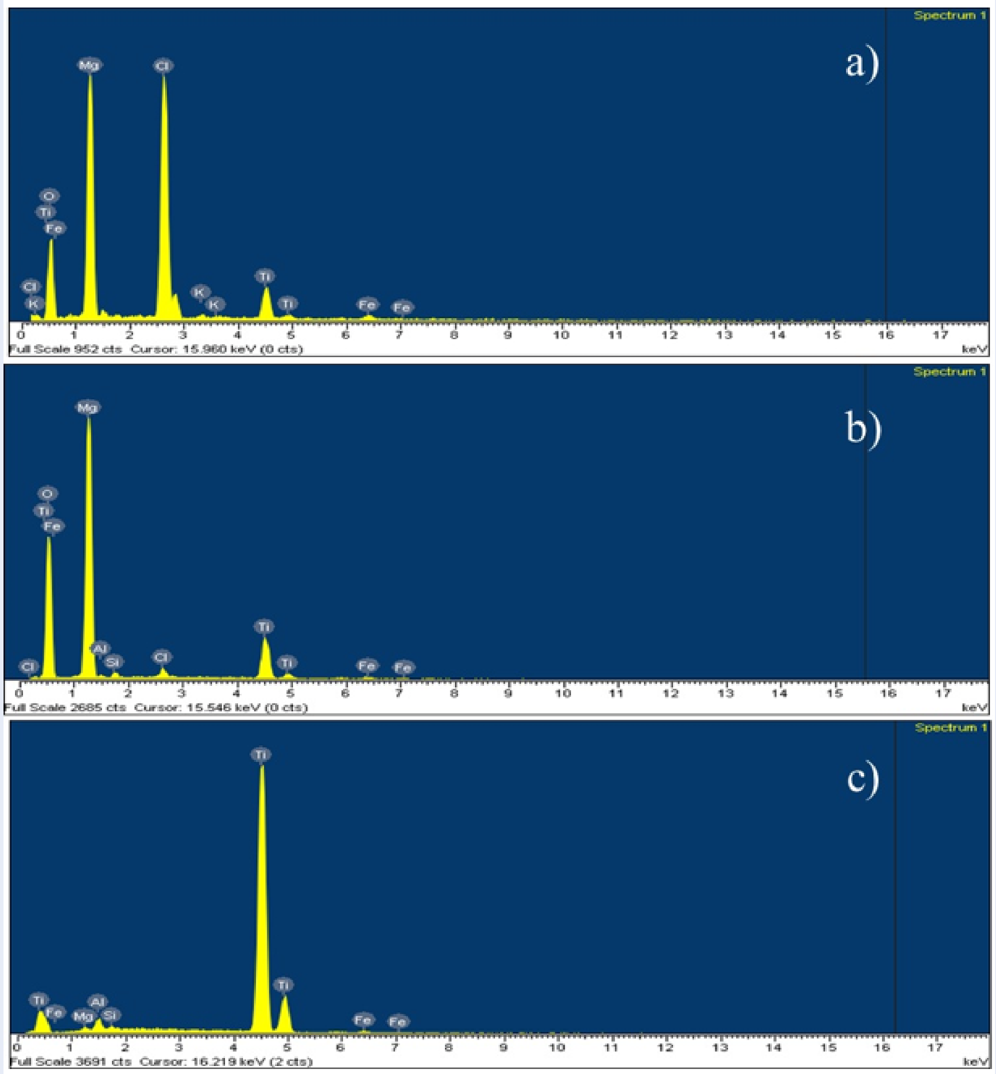

Figure 5: EDX spectrum of sample after reducing (a), sample after washing (b) and sample after acid washing (c).

2.352 and 1.299 g/cm^3, respectively. This result shows that after heat reduction by magnesium, the density of obtained product has reduced by half compared to that of the input material. This result is also consistent with the porous structure of titanium obtained from SEM.

CONCLUSION

In summary, the study has identified the optimal molar ratio of the reducing agent to reconstituted material for titanium conversion (Mg/TiO$_2$ ratio of 2.6). The raw materials can be mixed either alternately or evenly. The resulting product is subjected to water and acid washing, followed by filtration, to achieve high purity. The obtained product exhibits high purity, with a titanium content of up to 94.75%. The composition includes alloying elements beneficial to the intended application, such as Mg and Fe, while Si and Al are present in small quantities without harmful impurities. The fabricated titanium powder possesses a porous structure with grain sizes ranging from 100 to 500 nm. The study's findings establish optimal conditions for the magnesiothermic reduction process of secondary titanium slag to produce titanium powder, which can be implemented for industrial-scale fabrication of titanium from secondary titanium slag.

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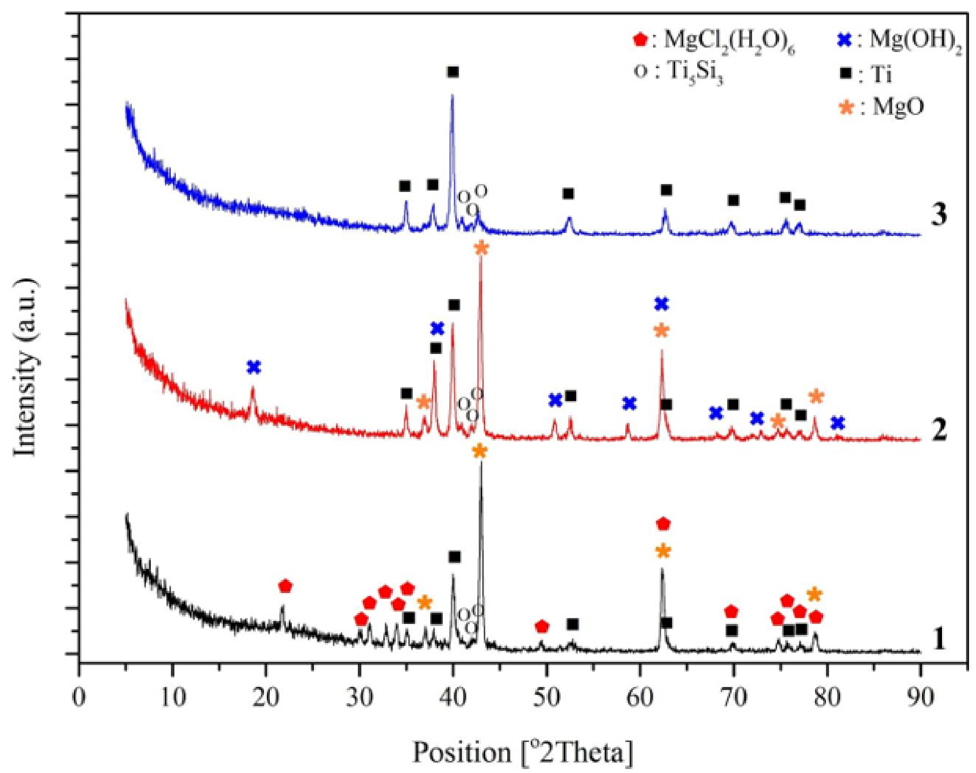

Figure 6: XRD profiles of the sample after reducing (1), sample after water washing (2) and sample after acid washing (3).

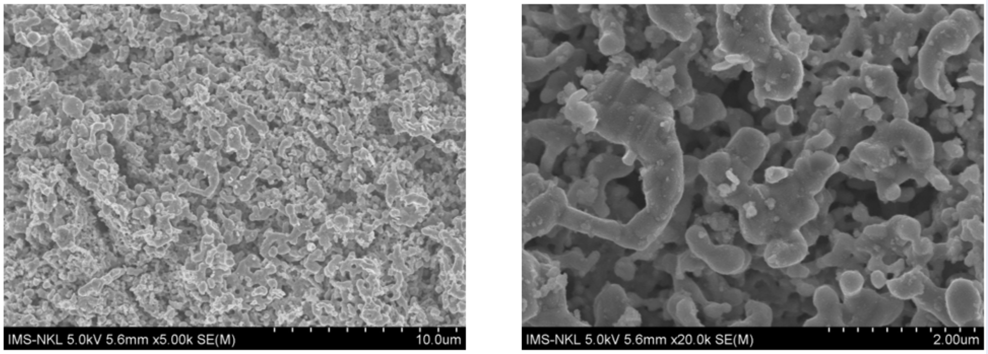

Figure 7: Morphology of titanium powder fabricated from secondary titanium slag by magnesiothermic reduction method with Mg/TiO2 molar ratio of 2.6, with even disposition of raw materials

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LIST OF ABBREVIATIONS

EDX: Energy-Dispersive X-ray spectroscopy
FFC: Fray Farthing Chen
OS: Ono and Suzuki
PRP: Preform Reduction Process
HAMR: Hydrogen-Assisted Magnesiothermic Reduction
SEM: Scanning Electron Microscopy
XRD: X-ray Diffraction

COMPETING OF INTERESTS

The author(s) declare that they have no competing interests.

AUTHORS' CONTRIBUTION

Nam Ho Phung Khac: Investigation, Writing - original draft, Methodology, Formal analysis, Data curation.

Duong Duc La: resources, Formal analysis, validation.

Hoai Phuong Nguyen Thi: Methodology, writing-reviewing scientific contents and editing.

All authors have read and agreed to the published version of the manuscript.

REFERENCES

1. Thambiliyagodage C, Wijesekera R, Bakker MG. Leaching of ilmenite to produce titanium based materials: a review. Discover Materials. 2021;1(1):20;Available from: https://doi.org/10.1007/s43939-021-00020-0.
2. U.S. Geological Survey. Mineral commodity summaries 2023. U.S. Geological Survey; 2023. 210 p;Available from: https://doi.org/10.3133/mcs2023.
3. Yang Y, Zhang C, Dai Y, Luo J. Tribological properties of titanium alloys under lubrication of SEE oil and aqueous solutions. Tribology International. 2017;109:40-47;Available from: https://doi.org/10.1016/j.triboint.2016.11.040.
4. Prasad S, Ehrensberger M, Gibson Mpietlal. Biomaterial properties of titanium in dentistry. J Oral Biosci. 2015;57:192-199;Available from: https://doi.org/10.1016/j.job.2015.08.001.
5. Hurless BE, Froes FH. Lowering the cost of titanium. AMPTIAC Q. 2022;6:3-9;.
6. Leyens C, Peters M. Titanium and Titanium Alloys: Fundamentals and Applications. John Wiley & Sons; 1980;.
7. Schwartz Z, Olivares-Navarrete R, Wieland M, Cochran DL, Boyan BD. Mechanisms regulating increased production of osteoprotegerin by osteoblasts cultured on microstructured titanium surfaces. Biomaterials. 2009;30:3390-3396;Available from: https://doi.org/10.1016/j.biomaterials.2009.03.047.
8. Hanawa T. Titanium-Tissue Interface Reaction and Its Control With Surface Treatment. Front Bioeng Biotechnol. 2019;7:170;Available from: https://doi.org/10.3389/fbioe.2019.00170.
9. Feng Q, Lv M, Mao L, Duan B, Yang Y, Chen G, et al. Research Progress of Titanium Sponge Production: A Review. 2023;13(2):408;Available from: https://doi.org/10.3390/met13020408.
10. Zheng H, Ito H, Okabe TH. Production of Titanium Powder by the Calciothermic Reduction of Titanium Concentrates or Ore Using the Preform Reduction Process. Materials Transactions. 2007;48(8);Available from: https://doi.org/10.2320/matertrans.MER2007115.
11. Chen GZ, Fray DJ, Farthing TW. Direct electrochemical reduction of titanium dioxide to titanium in molten calcium chloride. Nature. 2000;407:361-363;Available from: https://doi.org/10.1038/35030069.
12. Suzuki RO, Ono K, Teranuma K. Calciothermic reduction of titanium oxide and in-situ electrolysis in molten CaCl2. Metall Mater Trans B. 2003;34(3):287-295;Available from: https://doi.org/10.1007/s11663-003-0074-1.
13. Okabe TH, Oda T, Mitsuda Y. Titanium powder production by preform reduction process (PRP). J Alloys Compd. 2004;364(1-2):156-163;Available from: https://doi.org/10.1016/S0925-8388(03)00610-8.
14. Xia Y, Fang ZZ, Zhang Y, Lefler H, Zhang T, Sun P, et al. Hydrogen assisted magnesiothermic reduction (HAMR) of commercial TiO2 to produce titanium powder with controlled morphology and particle size. Mater Trans. 2017;58(3):355-360;Available from: https://doi.org/10.2320/matertrans.MK201628.
15. Zhang Y, Fang ZZ, Xia Y, Sun P, Van Devener B, Free M, et al. Hydrogen assisted magnesiothermic reduction of TiO2. Chem Eng J. 2017;308:299-310;Available from: http://dx.doi.org/10.1016/j.cej.2016.09.066.
16. Nersisyan H, Won H, Won C, et al. Direct magnesiothermic reduction of titanium dioxide to titanium powder through combustion synthesis. Chem Eng J. 2014;235:67-74;Available from: https://doi.org/10.1016/j.cej.2013.08.104.
17. Kan X, Ding J, Zhu H, Deng C, Yu C. Low temperature synthesis of nanoscale titanium nitride via molten-saltmediated magnesiothermic reduction. Powder Technol. 2017;315:81-86;Available from: https://doi.org/10.1016/j.powtec.2017.03.042.
18. Nersisyan HH, Won HI, Won CW, Kim JB, Park SM, Kim JH. Combustion synthesis of porous titanium microspheres. Mater Chem Phys. 2013;141:283-288;Available from: http://dx.doi.org/10.1016/j.matchemphys.2013.05.012.
19. Zelikman AN, Krein OE, Samsonov GV. Metallurgy of rare metals. Second edition. Izdatel'stvo Metallurgiya; Moskva; 1964;.
20. Li Q, Zhu X, Zhang Y, Fang ZZ, Zheng S, Sun P, et al. An investigation of the reduction of TiO2 by Mg in H2 atmosphere. Chem Eng Sci. 2019;195:484-493;Available from: https://doi.org/10.1016/j.ces.2018.09.047.

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Bài nghiên cứu

Ảnh hưởng của kỹ thuật phối trộn, tỷ lệ mol và phương pháp lọc đến quá trình chế tạo titan từ xỉ titan thứ cấp bằng phương pháp nhiệt magiê

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TÓM TẮT

Ứng dụng của titan ngày càng mở rộng và nhu cầu về titan cũng tăng lên trong những năm gần đây vì đây là kim loại nhẹ, cực kỳ bền và có khả năng chống ăn mòn cao. Quy trình sản xuất Ti kim loại phổ biến nhất trên thế giới là quy trình Kroll. Tuy nhiên, phương pháp này phức tạp, chất thải clo sinh ra trong quá trình clo hóa cũng là yếu tố gây ra các vấn đề nghiêm trọng về môi trường. Hơn nữa, giá thành sản phẩm cao chủ yếu do quy trình sản xuất theo mẻ nhiều bước, tốn thời gian và nhiệt độ cao. Trong thập kỷ qua, nhiều nghiên cứu đã được tiến hành để tìm ra các quy trình thay thế cho quy trình Kroll để sản xuất kim loại titan. Nhiều chất khử và nguyên liệu đầu vào khác nhau đã được nghiên cứu để sản xuất kim loại titan. Trong nghiên cứu này, phương pháp khử nhiệt magiê đã được xem là một cách hiệu quả để chế tạo bột titan từ xỉ titan thứ cấp. Nguyên liệu đầu vào (xỉ titan thứ cấp của Công ty Cổ phần Khoáng sản Bình Định), chất khử (Mg) và $MgCl_2.6H_2O$ được phối trộn và khử trong môi trường khí argon ở 900 ^oC trong vòng 4 giờ. Để thu được sản phẩm titan với hiệu suất cao và chất lượng tốt, cần tối ưu hóa nhiều yếu tố như: phương pháp phối trộn nguyên liệu, tỷ lệ mol Mg/TiO_2, quá trình lọc tách sản phẩm (rửa nước và rửa axit). Trong nghiên cứu này, các yếu tố ảnh hưởng này đã được nghiên cứu chi tiết và tối ưu hóa. Bột titan tạo ra đã được đánh giá đặc trưng bằng phương pháp nhiễu xạ tia X, phổ tán xạ năng lượng tia X và kính hiển vi điện tử quét. Kết quả nghiên cứu cho thấy, với tỷ lệ mol Mg/TiO_2 là 2,6, việc bố trí phối trộn liệu xen kẽ hoặc trộn đều là điều kiện tối ưu để chuyển hóa xỉ titan thứ cấp thành bột titan. Sản phẩm titan sau khi nhiệt phân được rửa và lọc bằng nước, sau đó rửa bằng axit, là dạng bột xốp có độ tinh khiết cao với hàm lượng titan lên tới 94,75 %.

Từ khoá: EDX, Khử nhiệt magiê, Xỉ titan thứ cấp, SEM, XRD

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