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REVIEW

Research Status and Progress on Preparation and Purification of Niobium Metal by Pyrometallurgy

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Abstract: As a strategic critical metal, niobium is widely used in the fields of aerospace, nuclear industry, and superconductors owing to its superior properties. With the requirement of sustainable development, the energy consumption in niobium melting processes has attracted increasing attention. Green, low-carbon, and energy-saving practices have become the new development direction. In addition, microelectronics technology requires high-purity niobium as a sputtering target material. Although niobium with a purity of up to 5N has been achieved, a low-cost high-purity technique is still challenging. This review summarized a variety of pyrometallurgical methods for the preparation and purification of crude niobium. As a traditional method for producing crude niobium, the key challenge of thermal reduction is how to reduce energy consumption. As a technique with industrial propects, molten salt electrolysis has been developed into a variety of methods, but the efficiency needs to be further improved. In addition, some new purification techniques are constantly emerging, such as fully-automated melting technique assisted by the digital twin and artificial intelligence. In the future, a variety of technical means will be combined to purify niobium metal. This review also briefly introduced the current status of niobium recovery and further explored the full lifecycle of niobium based on the concept of urban mine, to provide direction for achieving niobium recycling.

Key words: niobium; reduction; purification; pyrometallurgy; recycling

1 Introduction

As a refractory metal, niobium possesses a variety of physical and chemical properties, such as a low coefficient of thermal expansion, heat resistance, corrosion resistance, and a low thermal neutron absorption cross-section[1–2]. Niobium has become an indispensable critical metal in the development of modern high-tech industry[3–5]. It is mainly used to produce ferroniobium and functional materials such as superconducting alloys, nuclear fuel cladding materials, and heat-resistant alloys in the fields of superconductivity[6–8], nuclear industry[9–10], and aerospace[11–12]. Meanwhile, it is worth noting that high-purity niobium is critically needed in metal sputtering targets and nanotechnology[13–14].

In recent years, global demand for niobium has increased significantly[15]. Natural niobium deposits are predominantly concentrated in Brazil, with pyrochlore-type ores representing the primary economically viable source. Following beneficiation, the ore proceeds to the metallurgical stage. Niobium metallurgy encompasses three key processes: separation and purification of niobium compounds, synthesis of intermediate niobium compounds, and the production and purification of metallic niobium. Metallic niobium production primarily involves the pyrometallurgical reduction of compounds like Nb_2O_5 or $NbCl_5$. Subsequent purification further refines the metal to enhance its purity. Although established techniques exist for both production and purification, each method faces inherent restrictions. Consequently, selecting appropriate techniques tailored to specific niobium product requirements remains essential[16–17]. Furthermore, the imperative for energy conservation and emission reduction necessitates developing more environmentally sustainable processes. This represents a

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critical future direction, alongside deeper investigation into emerging technologies. Notably, urban mines constitute a significant potential source of secondary niobium resources. Implementing efficient recycling strategies could substantially reduce energy consumption across the niobium value chain[18].

This review comprehensively summarized recent pyrometallurgical methods for preparing and purifying crude niobium. The current status of secondary niobium recovery was introduced. This work aims to provide references for developing new high-purity niobium production techniques and identify future research directions.

2 Thermal Reduction Method

Crude niobium is primarily produced via reduction of niobium oxide (Nb_2O_5) or niobium chloride ($NbCl_5$). Fig. 1 illustrates the conventional process for producing high-purity niobium from Nb_2O_5. Reducing agents encompass metallic elements (e.g., Ca, Mg, Al, and Na) and non-metallic elements (C and H_2), with aluminum as the predominant reductant. When $NbCl_5$ serves as the precursor, alloy powders can be synthesized through liquid metal reduction analogous to the Kroll process[19]. The resultant crude niobium undergoes subsequent refinement and purification through techniques including electron beam melting (EBM), zone melting, vacuum arc melting (VAM), and electrolysis refining.

2.1 Metal thermal reduction

Metal thermal reduction of oxides represents a fundamental reaction methodology. Fig. 2 shows the oxygen-potential diagram for major elements calculated using FactSage software (FactPS and FToxid databases). Thermodynamic analysis reveals that the standard Gibbs free energy for oxygen reactions with Al, Mg, and Ca is lower than that with Nb across 0–2000 °C, confirming their viability as reductants for Nb_2O_5. Carbon can also reduce Nb_2O_5 at elevated temperatures through CO formation. While sodium demonstrates reduction capability at lower temperatures, its extreme reactivity with oxygen restricts the practical application for Nb_2O_5 reduction. Sodium reduction is preferentially employed for K_2NbF_7 processing. Hydrogen reduction of Nb_2O_5 to metallic niobium remains thermodynamically unfavorable even at elevated temperatures.

2.1.1 Aluminothermic reduction

Aluminothermic reduction is the dominant industrial method for producing niobium. Characterized by a strongly exothermic nature, the reduction reaction becomes self-sustaining once initiated[20]. The core chemical reaction is: $3Nb_2O_5+10Al \rightarrow 6Nb+5Al_2O_3$. In this process, Nb_2O_5 is initially reduced by aluminum to lower-valent oxides (such as NbO_2 or NbO), which are then further reduced to metallic niobium. The procedure involves blending niobium oxide with aluminum powder, placing the mixture into a crucible, and topping it with additional aluminum powder. The crucible is then heated to 1000 °C to ignite the reaction. The reduction products consist of a niobium-aluminum alloy melt and an Al_2O_3 slag. Subsequently, the alloy melt is separated from the slag and undergoes EBM to remove aluminum. The recovery rate of niobium metal from this reduction process exceeds 95%.

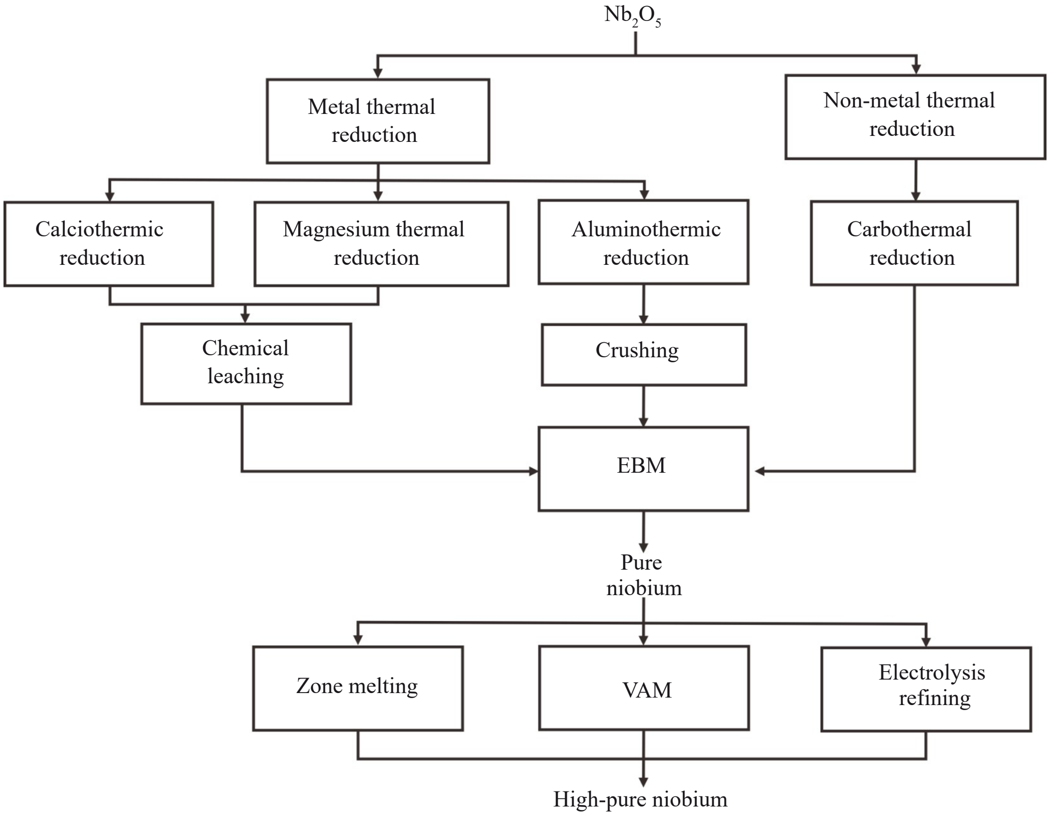

Fig.1 Schematic diagram of conventional methods for producing high-purity niobium from Nb_2O_5

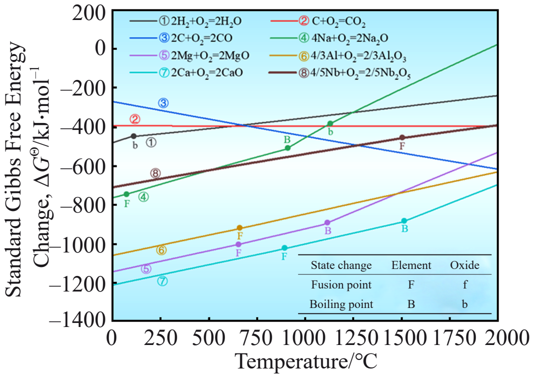

Fig.2 Oxygen-potential diagram

The primary factors influencing the thermal response of the aluminothermic reaction are the inherent exothermicity, additives, aluminum particle size, aluminum content, and process parameters[21]. When the intrinsic heat generation is insufficient, thermal enhancers and fluxes must be added. Commonly used thermal enhancers are strong oxidizers, like nitrates, chlorates, and peroxides, which react vigorously with aluminum, releasing substantial heat to compensate for the deficit in the primary reaction. For the aluminothermic reduction process, CaO is the most common flux due to its effectiveness and low cost. Besides lowering the melting point of slag, its addition also reduces the reaction initiation temperature.

Compared to other preparation methods, niobium produced via the aluminothermic reduction process typically contains aluminum impurities, which are readily removed through EBM under high vacuum and high temperature[22]. Additionally, the aluminothermic reduction method offers advantages in terms of lower environmental impact and straightforward scalability for industrial production, making it one of the most effective processes for producing metallic niobium. Consequently, the mainstream approach for high-purity niobium extraction combines aluminothermic reduction with EBM. However, the growing demands of target materials, 3D printing, and powder metallurgy require increasingly stringent powder characteristics, particularly regarding particle size and purity. Niobium produced by aluminothermic reduction tends to exhibit inadequate purity and inconsistent particle size. Furthermore, the subsequent refining process is highly energy-intensive. This has created an urgent need for alternative methods, positioning molten salt electrolysis as a promising new development trend.

2.1.2 Sodiothermic reduction

The sodiothermic reduction method typically employs K_2NbF_7 as raw material, using sodium as the reducing agent for thermal reduction[23], as shown in Fig. 3. This process provides consistent niobium yield, enables precise control over product morphology, and produces high-purity niobium. Consequently, niobium powder synthesized via this method is well-suited for capacitor manufacturing. However, the equipment faces severe corrosion risks, while operational hazards exist due to the use of strong acids during purification.

2.1.3 Magnesium thermal reduction

The reduction of Nb_2O_5 with magnesium produces fine powder with a high specific surface area, though its highly exothermic nature poses a significant challenge. To address reaction control difficulties, methods such as periodic Nb_2O_5 addition[24–25] or controlled magnesium vapor dosing[26] are typically employed. Further challenges arise from the tendency to form magnesium niobate and difficulties in separating the metal powder from magnesium oxide, which may partially sinter during the process. Separation is usually achieved through chemical leaching. Through the two-step reduction process, involving magnesium vapor reaction followed by chemical treatment, the oxygen content can be reduced to 0.65%[27].

2.1.4 Calciothermic reduction

As a reactive metal, calcium serves as an effective reducing agent for converting metal oxides to their metallic state[28]. Achieving the separation of solidified niobium from calcia slag requires elevated temperatures to melt the slag. This is accomplished by direct reactor heating or exothermic additions (e. g., I_2 or S) that react with calcium to generate supplemental heat without metal contamination.

In 2002, Ono and Suzuki[29] pioneered the Ono-Suzuki (OS) process, wherein CaO dissociation in molten $CaCl_2$ via electrolysis produces calcium for calciothermic reduction of metal oxides. This methodology is equally applicable to niobium production. Fig.4 contrasts two distinct calciothermic reduction methods.

Building on OS process, Okabe et al[30] developed the preform reduction process (PRP) in 2004. PRP method involves pre-forming metal oxide compacts followed by reduction with calcium vapor, which is a technique also effective for producing uniform niobium powder. Although PRP enables precise control over product purity and particle size, it demands higher-purity reducing agents.

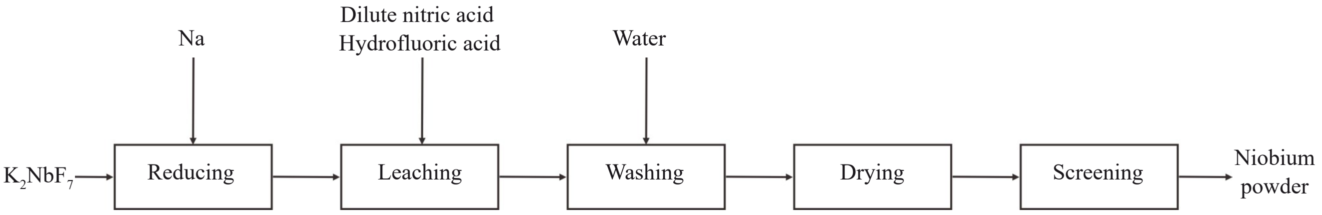

Fig.3 Schematic diagram of sodiothermic reduction process[23]

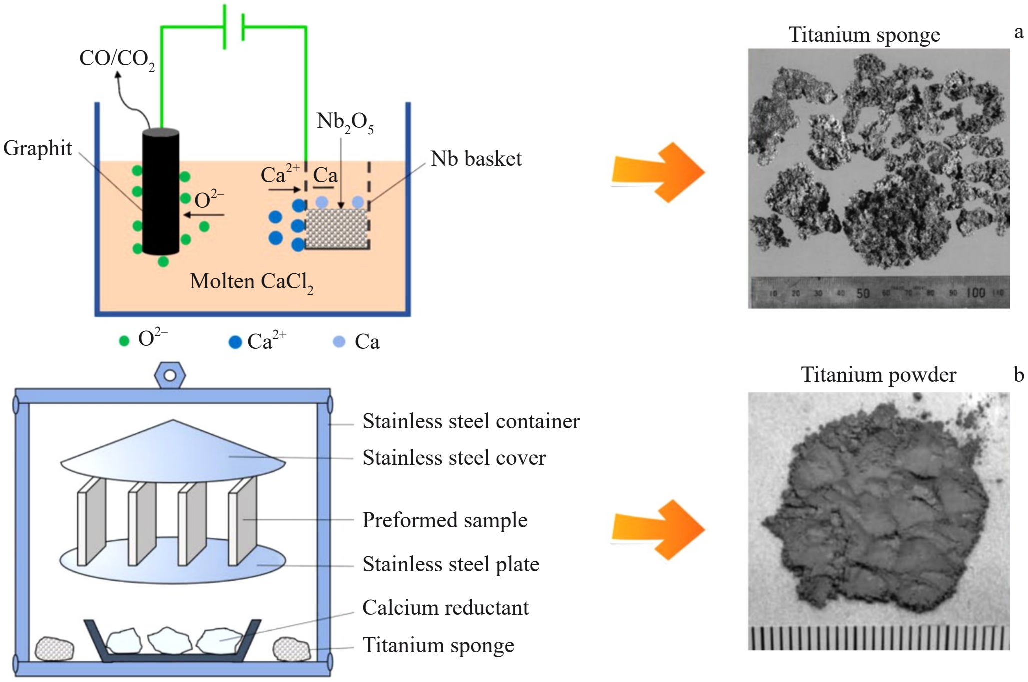

Fig.4 Schematic diagrams of two processes of calciothermic reduction[29–30]: (a) OS; (b) PRP

2.2 Non-metal thermal reduction

Non-metallic reduction methods enable the conversion of niobium oxides and chlorides using carbon or hydrogen. Due to its low cost, easy availability, and facile separation of CO byproduct from the metallic product, the carbon reduction method dominates industrial production before the aluminum thermal reduction method. Alternatively, hydrogen reduction of $NbCl_5$ facilitates the preparation of metallic niobium coatings and ultrafine powders. This approach integrates with chlorination processes for treating niobium concentrates or complex low-grade raw materials, offering a rapid and economically viable processing method.

2.2.1 Carbothermal reduction

The carbon reduction method for niobium production involves high-temperature vacuum reactions between carbon and Nb_2O_5. This reduction process is highly complex, as studies confirm niobium metal forms not directly from Nb_2O_5, but through intermediate phases, including lower oxides (NbO_2 and NbO), solid solutions (NbC_xO_y), and carbides (NbC)[31–32].

Niobium production via carbothermal reduction employs either single-stage or two-stage processing methods. The single-stage method, conducted primarily in vacuum carbon tube furnaces, directly reduces Nb_2O_5 with carbon to yield niobium powder. The two-stage approach involves: (1) reacting carbon black with Nb_2O_5 to form niobium carbide (NbC); (2) reduction of residual Nb_2O_5 with the synthesized NbC to produce metallic niobium, with Nb_2O_5 typically exceeding stoichiometric requirements by 1% – 5%. This method can increase the output of niobium and shorten the reduction cycle.

As the oldest and most economical reducing agent, carbothermal reduction dominates industrial niobium production before aluminothermic reduction processes. However, during reduction, niobium vapor reacts with evolving carbon monoxide, contaminating condensation products with oxides and carbon. Furthermore, the strong carbon affinity of niobium promotes carbide formation, compromising product purity. Advances in vacuum technique enable high-temperature processing under extreme vacuum, significantly enhancing purity in modern carbothermal reduction.

2.2.2 Hydrogen reduction

Chemical vapor deposition is commonly employed to produce ultrafine niobium powder through hydrogen reduction. Zhu et al[33] used hydrogen reduction of gaseous chlorides at 950 ℃ to synthesize ultrafine niobium and tantalum powders. The resulting powder exhibits uniform particle size distributions with average diameters of 30–40 nm.

2.2.3 Nitride process

The nitride process has been developed for niobium metal production[34]. This method reacts niobium oxide with ammonia or nitrogen to form niobium nitride, which subsequently undergoes thermal decomposition under vacuum at 2103–2373 K to yield metallic niobium. A key advantage of this process is its continuous operation capability. Once formed, the nitride decomposes spontaneously into metal without process interruption. The resulting metal typically achieves purity of 99.7% – 99.8% with a total recovery rate of 96%.

3 Electroreduction Method

To reduce niobium production costs, researchers have conducted extensive studies. Compared to thermal reduction methods, the electroreduction method offers advantages, including enhanced process control and lower environmental impact. Consequently, it is regarded as a promising alternative, with significant research progress already achieved[35]. However, challenges remain due to complex electrode reactions involving metal ions and inadequate electrolytic efficiency, necessitating deeper investigation into reaction mechanisms and process optimization[36–38].

3.1 Conventional molten salt electrolysis

In conventional molten salt electrolysis for niobium production, K_2NbF_7 or $NbCl_5$ typically serve as raw materials, while alkali or alkaline earth metal chlorides function as the electrolyte[39]. Cathodic reaction mechanisms vary significantly with electrode materials and molten salt systems. Nb^{5+} reduction frequently requires multiple electron transfer steps before metallic niobium deposition occurs. Crucially, fluoride ions stabilize high-valence niobium species and streamline reduction pathways[40]. Table 1 summarizes the electrochemical reduction steps of niobium ions across various molten salt systems.

3.2 FFC Cambridge process

The molten salt electro-deoxidation (FFC Cambridge process), pioneered by Chen et al[58], was initially developed for oxygen removal from titanium. This technique has evolved into a direct electrolysis method for producing pure metals from solid metal oxides[59]. In niobium production, the process employs molten $CaCl_2$ to directly electrolyze Nb_2O_5. The deoxidation pathway follows a stepwise reduction sequence: $Nb_2O_5\rightarrow NbO_2\rightarrow NbO\rightarrow Nb$, occurring progressively from the cathode surface to its core[60]. Crucially, oxide reduction is driven by electron transfer, enabling direct electrochemical reduction within the melt[61]. When electrolyte oxide ion activity is elevated, dissolved NbO forms negatively charged complexes[62]. Incorporating graphite powder, CaO, or $CaCO_3$ into the Nb_2O_5 cathode significantly enhances electrode porosity and accelerates reaction kinetics[48].

3.3 EMR process

Electronically mediated reaction (EMR) was first proposed by Okabe et al[63]. In their setup, Nb_2O_5 and Ca-Al-Ni alloy

Table 1 Electrochemical reduction steps for niobium ions in various molten salt systems

Molten salt system	Reduction step	Ref.
$NaCl\text{-}KCl\text{-}NbCl_5$	$Nb^{5+}\rightarrow Nb^{4+}\rightarrow Nb$	[41]
	$Nb^{5+}\rightarrow Nb^{4+}\rightarrow Nb^{3+}\rightarrow Nb$	[40]
	$Nb^{5+}\rightarrow Nb^{3+}\rightarrow Nb^{+}\rightarrow Nb$	[42]
$NaCl\text{-}KCl\text{-}NaF\text{-}NbCl_5$	$Nb^{5+}\rightarrow Nb^{4+}\rightarrow Nb$	[40]
	$Nb^{5+}\rightarrow Nb^{3+}\rightarrow Nb$	[43]
	(mole ratio: F^-/Nb^{5+}=1, 2, 3)	
	$Nb^{5+}\rightarrow Nb^{4+}\rightarrow Nb$	
	(mole ratio: F^-/Nb^{5+}=5, 10)	
$NaCl\text{-}KCl\text{-}MgCl_2\text{-}DyCl_2$	$Nb^{n+}\rightarrow Nb$	[44]
$NaCl\text{-}AlCl_3\text{-}NbCl_5$	$Nb^{5+}\rightarrow Nb^{4+}\rightarrow Nb^{2.67+}\rightarrow Nb^{2.33+}$	[45]
	$Nb^{5+}\rightarrow Nb^{2+}\rightarrow Nb^{+}\rightarrow Nb$	[43]
$LiCl\text{-}KCl\text{-}NbCl_5$	$Nb^{5+}\rightarrow Nb^{4+}\rightarrow Nb^{3+}\rightarrow Nb$	[46]
	$Nb^{5+}\rightarrow Nb^{3+}\rightarrow Nb^{2+}\rightarrow Nb$	[47]
$NaCl\text{-}AlCl_3\text{-}NaF\text{-}NbCl_5$	$Nb^{5+}\rightarrow Nb^{3+}\rightarrow Nb$	[43]
	(mole ratio: F^-/Nb^{5+}=1, 3, 5, 10)	
	$Nb^{5+}\rightarrow Nb^{4+}\rightarrow Nb^{2+}\rightarrow Nb$	
	(mole ratio: F^-/Nb^{5+}=2)	
$NaCl\text{-}CaCl_2\text{-}Nb_2O_5$	$Nb^{5+}\rightarrow Nb^{4+}\rightarrow Nb^{2+}\rightarrow Nb$	[48]
$CaCl_2\text{-}Nb_2O_5$	$Nb^{5+}\rightarrow Nb^{4+}\rightarrow Nb$	[49]
$NaCl\text{-}KCl\text{-}K_2NbF_7$	$Nb^{5+}\rightarrow Nb^{4+}\rightarrow Nb$	[50]
	$Nb^{5+}\rightarrow Nb^{4+}\rightarrow Nb$	[51]
	$Nb^{5+}\rightarrow Nb^{4+}\rightarrow Nb$	[52]
$NaCl\text{-}KCl\text{-}NaF\text{-}K_2NbF_7$	$Nb^{5+}\rightarrow Nb^{4+}\rightarrow Nb$	[38]
$CaCl_2\text{-}Nb_2O_5$	$Nb^{5+}\rightarrow Nb^{4+}\rightarrow Nb$	[53]
$LiF\text{-}NaF\text{-}K_2NbF_7$	$Nb^{5+}\rightarrow Nb^{4+}\rightarrow Nb$	[54]
$LiF\text{-}KF\text{-}K_2NbF_7$	$Nb^{5+}\rightarrow Nb^{4+}\rightarrow Nb$	[55]
$LiF\text{-}NaF\text{-}KF\text{-}K_2NbF_7$	$Nb^{5+}\rightarrow Nb^{4+}\rightarrow Nb$	[36]
	$Nb^{5+}\rightarrow Nb^{4+}\rightarrow Nb$	[56]
$LiCl\text{-}KCl\text{-}KF\text{-}Na_2O\text{-}K_2NbF_7$	$Nb^{5+}\rightarrow Nb^{4+}\rightarrow Nb^{3+}$	[57]
$KCl\text{-}KF\text{-}K_2NbF_7$	$Nb^{5+}\rightarrow Nb^{4+}\rightarrow Nb$	[39]

(nominal composition: 63.7wt% Ca, 24.3wt% Al, 12.0wt% Ni) were placed in electronically isolated compartments within molten $CaCl_2$ at 1173 K. A measurable current was detected in the external circuit connecting the Nb_2O_5 and the calcium alloy. Notably, niobium powder with low aluminum and nickel content was obtained despite the use of the liquid Ca-Al-Ni alloy as the reductant. This contrasts with the conventional concept of calciothermic reduction, which relies on reaction and mass transport involving medium (Fig. 5a). Crucially, EMR process eliminates the need for direct physical contact between the feed material (Nb_2O_5) and the reductant (Ca) (Fig. 5b). EMR concept thus provides deeper insight into the role of molten salt during calciothermic reduction of niobium. This mechanism holds promise for developing processes to produce high-purity niobium using less pure reductant alloys[64].

3.4 SOM method

The solid oxygen permeable membrane (SOM) method uses a solid oxygen-ion-conducting membrane to selectively control the ionic species participating in the reaction[65–66], thereby enabling the electrolytic production of metals. In this process, graphite serves as the cathode, while the anode consists of a cermet whose molten salt-facing surface is coated with a zirconia-based solid oxygen permeable membrane. As this membrane permits only oxygen ions (O^{2-}) to migrate to the anode, O^{2-} is the sole anion involved in the anodic reaction. He et al[53] employed SOM method to prepare metallic niobium using Nb_2O_5 powder as the raw material and identified the optimal electrolytic conditions. During the electro-deoxygenation process, the porosity of the cathode was found to influence the reaction rate. Electrochemical analysis revealed that the reduction of Nb_2O_5 proceeds stepwise: Nb(V)→Nb(IV)→Nb. Notably, the deoxygenation rate is faster during the initial stage of the SOM electrolytic reduction. Consequently, SOM method exhibits a higher overall reaction rate than FFC Cambridge process.

3.5 USTB process

Jiao and Zhu[67] proposed a method for preparing pure titanium using TiC_xO_y soluble anode electrolysis, known as USTB process. The principle of this method is that the soluble anode dissolves under the action of an electric field, and metal ions migrate to the anode and are reduced to pure metal. Meanwhile, carbon and oxygen in the anode combine to form CO. This method has been successfully applied to metallic vanadium[68] and zirconium[69]. Using this method, the research team prepared single-phase NbC_xO_y solid solution by carbothermal reduction of Nb_2O_5 and demonstrated that it is an excellent soluble anode[70]. The reduction process of niobium ions was clarified through electrochemical testing, and metallic niobium was successfully obtained at the cathode through electrolysis[71]. The flow chart of the process is shown in Fig.6.

Table 2 provides a detailed comparison of pyrometallurgical methods for preparing metallic niobium. Compared with the aluminothermic and carbothermal reduction processes, the other methods show significant potential for enhancing product purity and reducing energy consumption. However, these alternative techniques remain under development.

4 Niobium Purification Method

4.1 EBM

EBM is the most common method for producing high-purity niobium[72]. The purification principle relies on the removal of impurities via evaporation and degassing under the combined effects of high temperature and high vacuum, while the crude niobium is in a molten state[73]. The efficacy of purification by EBM depends critically on process parameters such as melting time, melting rate, vacuum level, and power input. Furthermore, the temperature distribution within the melt, as well as the size and shape of the metal, also significantly influence the purification level[74–76].

Niobium readily reacts with various gases, and its interactions with H, N, O, and C are critical during EBM. Under high vacuum and high temperature, hydrogen and nitrogen are removed by diffusing to the melt surface until degassing and gettering achieve equilibrium[77]. This method efficiently achieves dehydrogenation and denitrification[78]. In the Nb-O system, the equilibrium oxygen partial pressure is extremely low, which falls below the residual pressure in an ultra-high vacuum system, preventing oxygen removal. However, since most refractory metals form volatile oxides[79], oxygen is removed through the formation of niobium oxides[80], primarily NbO and NbO_2[81]. Carbon impurities in niobium can only be eliminated through CO formation. At

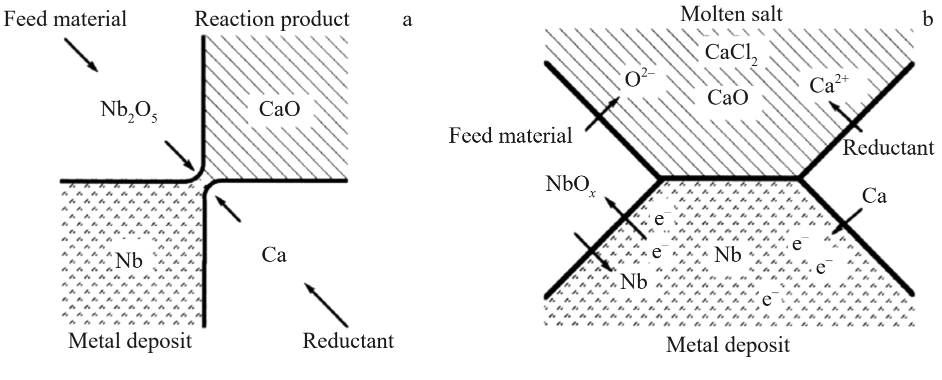

Fig.5 Schematic diagrams of conventional process (a) and EMR process (b)[63]

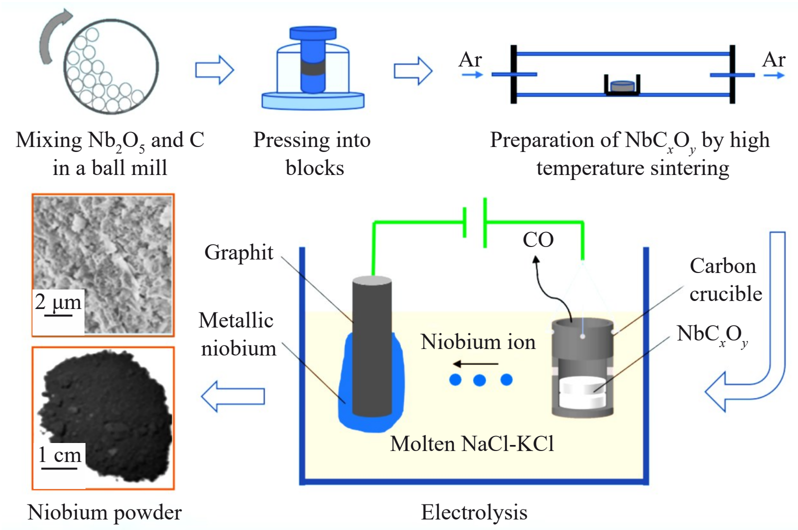

Fig.6 Flow chart of USTB process

Table 2 Comparison of various pyrometallurgy methods for preparing metallic niobium

Method	Status	Advantage	Disadvantage	Purity of niobium	Energy consumption level
Aluminothermic reduction	Mainstream industrial technique	Simple process and high output	Low product purity	Low	High (including subsequent processing)
Sodiothermic reduction	Semifinished industrialization	Controllable morphology	High requirements for equipment	Medium	Medium
Magnesium thermal reduction	Semifinished industrialization	Small particle size	Difficult to separate the product	Medium	Medium
Calciothermic reduction	Semifinished industrialization	High product purity	High cost	High	Medium
Carbothermal reduction	Industrialization	Low cost and easy to operate	Easy to generate niobium carbide	Low	High
Hydrogen reduction	Industrialization	High purity and small particle size	Flammable and explosive hydrogen	High	Low
Nitride process	R&D phase	High flexibility in feedstock	Complex process and high energy consumption	Medium	High
Conventional electrolysis method	R&D phase	Mature and easy to implement	Complex reaction and low electrolysis efficiency	Medium	Medium
FFC Cambridge process	R&D phase	Simple and low pollution	Low deoxidation efficiency	Medium	Medium
EMR process	R&D phase	High purity	Low electrolysis efficiency and high cost	High	Medium
SOM process	R&D phase	High reaction rate	Complex device	High	Medium
USTB process	R&D phase	Low raw material requirements and low pollution	Long process flow	Medium	Medium

Note: R&D phase means research and development phase.

higher oxygen concentrations, carbon is removed as CO while oxygen is concurrently removed as NbO. Conversely, at low oxygen levels, only volatile NbO forms. Consequently, excess oxygen is frequently employed to remove carbon via CO formation[82]. EBM is highly ineffective in removing refractory metals like tungsten and tantalum from crude niobium, necessitating a combination with other purification techniques. Multiple EBM passes are known to yield exceptionally pure niobium[83–84].

EBM offers both high temperature and high vacuum environment, enabling remarkable purification effects. However, the associated equipment is costly and energy-intensive. Current development efforts focus on enhancing energy efficiency and material yield of EBM. Key measures include stabilizing beam power, precisely targeting the melt zone, and implementing programmed control over the melting process[85]. For instance, Germany's ALD vacuum uses fully automated EBM, leveraging digital twin technique to cut melting energy consumption by 15% and elevate metal purity to 99.99%. Similarly, Ningxia Orient Tantalum Industry Co., Ltd (China) employs machine vision on its zone melting line to monitor the melt pool morphology. This system automatically modulates heating power, reducing manual intervention by 90%.

4.2 VAM

VAM uses an arc as its heat source and shares a similar impurity removal mechanism with EBM[86]. While the equipment costs of VAM are lower than those of EBM, the process requires preparatory electrode fabrication, leading to a longer overall procedure.

4.3 Plasma refining

Metallic niobium can be purified via Ar-H_2 plasma arc melting[87]. Hydrogen addition creates a reducing atmosphere, facilitating the removal of impurities like oxygen, carbon, and nitrogen[88]; however, excessive hydrogen can cause hydrogen embrittlement. Hydrogen possesses higher thermal conductivity than argon, and its addition significantly enhances heat transfer efficiency, promoting both metal melting and impurity volatilization. This process achieves impurity removal rates exceeding 80%[89]. Compared to EBM, this method presents challenges, including more complex equipment, poorer plasma stability, and higher residual hydrogen content in the niobium metal. Future enhancements involve adopting radiofrequency or microwave plasma sources to improve stability, integrating artificial intelligence (AI) for real-time plasma parameter regulation, and optimizing gas recycling technique to reduce energy consumption.

4.4 Chemical vapor transport

Chemical vapor transport (CVT) involves niobium reacting with halogens such as I_2 at elevated temperatures to form volatile compounds like niobium pentaiodide (NbI_5). These compounds subsequently decompose at lower temperatures to deposit high-purity niobium[72]. Alternatively, niobium can react with Cl_2 to form niobium pentachloride ($NbCl_5$), which is then reduced by hydrogen to yield metallic niobium[90]. This method achieves exceptionally high purity levels (⩾6N, 99.9999%), with extremely low yield.

4.5 Zone melting

Zone melting is a purification method that exploits the difference in impurity solubility between the solid and molten states of a metal to redistribute soluble impurities. The metal to be purified is placed on a sample table, and a small localized region is heated to form a molten zone. As the heating zone moves, the molten zone correspondingly moves. Due to the phenomenon where the melting point of an impure mixture is lower than that of the pure substance, impurities gradually accumulate within the molten zone. Consequently, as the heating zone advances, impurities are progressively driven toward the end of the metal sample and ultimately removed[91]. This process produces no gaseous pollutants. The resulting product exhibits high purity with uniform structure and composition[92]. For refractory metals, electron beams remain a common heating source. Furthermore, niobium purity can be enhanced using floating zone melting combined with magnetic stirring[93], and this technique can also be employed to prepare niobium single crystals[94].

4.6 Molten salt electrorefining

Molten salt electrorefining uses potential differences of elements at electrodes to purify metals[95]. In this process, crude niobium forms the anode while corrosion-resistant and highly conductive metal rods serve as the cathode. In crude niobium, refractory metals such as tantalum, tungsten, and molybdenum can be removed through the molten salt electrorefining method. Particularly, the use of molten fluoride is effective in removing metallic tantalum from crude niobium. However, metals that are more electrochemically active than niobium dissolve and co-deposit on the cathode. To further reduce C, N, and O content, this method is typically combined with EBM. Crude niobium with an initial mass fraction of 99.8% can be purified to >99.99% through this technique[96].

4.7 Solid-state electrotransport

Solid-state electrotransport purification operates under ultra-high vacuum or inert atmospheres, where direct currents of hundreds of amperes are passed through a metal rod at elevated temperatures. Under these combined thermal and electric field effects, impurity ions within the metal lattice migrate directionally to achieve purification[97]. As impurity ions exhibit higher mobility than metal ions, they accumulate at the cathode while high-purity niobium concentrates at the anode. This process reduces key impurities (carbon, oxygen, hydrogen, and nitrogen) to concentration approaching detection limits[98]. The technique is particularly effective for high-melting-point metals with low vapor pressures and high impurity mobility, notably refractory metals (e.g., W, Mo, Ta, and Nb). Kirchheim et al[99] demonstrated oxygen and nitrogen electrotransport in niobium and tantalum. However, the method requires prolonged processing time (typically hundreds of hours) and is energy-intensive, restricting its scalability for industrial production.

As summarized in Table 3, the advantages, disadvantages, target impurities, and removal efficiencies of various niobium refining methods are compared. Currently, EBM and VAM represent the industrially mature techniques. Plasma refining remains at the pilot stage, while CVT, zone melting, molten salt electrorefining, and solid-state electrotransport are currently confined to small-scale laboratory sample preparation.

Table 3 Comparison of various refining methods of metal niobium

Method	Advantage	Disadvantage	Target impurity	Removal efficiency
EBM	It has an extremely strong ability to remove impurity elements and the output is large.	The equipment is expensive and has high energy consumption, which may lead to loss of raw materials.	C, O, N, Fe, Ni, Al	High
VAM	The cost is relatively low and it has excellent degassing capabilities.	The removal effect of impurities is not as good as that of EBM.	O, N, H	High
Plasma refining	It has high energy density and is operable in inert or reactive atmospheres.	The equipment is complex and the technique is not yet fully mature.	C, O, N	High
CVT	The product has an extremely high purity and is precisely controllable.	The reaction reagents (e.g., I_2) may be introduced as impurities, and the equipment requirements are high.	Ta, W, Mo	Low
Zone melting	It has no contamination from the crucible and can produce single crystals.	The process is slow, the efficiency is extremely low, and the output is small.	W, Mo, Ta, Ti, Zr	Low
Molten salt electrorefining	It has low energy consumption and low requirements for raw materials.	The efficiency is low, and it may introduce molten salt ions, thereby causing pollution.	Fe, Ni, Cr, Ti, Al	Medium
Solid-state electrotransport	The removal effect of the gap elements is very significant.	The process is extremely slow and can only handle small samples.	C, O, N	Low

5 Current Situation of Secondary Niobium Recovery

Metal recycling is widely recognized as an impactful sustainability strategy. The viability of niobium recovery is primarily determined by waste source composition. During niobium and alloy production, processing waste (e. g., chips, shavings, and defective ingots) exhibits simple composition and high purity, enabling straightforward recovery through direct remelting for production reuse. In end products, although approximately 90% of niobium serves as steel additives in ferro-niobium (FeNb) form, its concentration in steel remains low (0.01%–0.1%) and recovery hinges on steel recycling efficiency. Decommissioned superconducting materials (e. g., NbTi and Nb_3Sn) constitute high-value recycling streams due to their elevated niobium content and exceptional purity. Conversely, high-temperature alloy waste contains only 1.5%–5.0% niobium dispersed as solid solutions or strengthening phases, rendering recovery technologically challenging with suboptimal yields. In electronic waste, niobium primarily exists as trace by-products that lose functional integrity, leading to significant losses through dispersion into low-value products. Pyrometallurgy currently dominates bulk niobium waste processing. Metallic waste requires initial oxidation followed by aluminothermic reduction and electron beam refining. Complex and low-grade niobium sources are predominantly treated via hydrometallurgical routes.

Urban mines constitute a critical secondary source for niobium recovery. Globally, average end-of-life functional recovery rates and recycled content exceed 50% owing to the inherent chemical stability of niobium in steel matrices[100]. The consumed energy during niobium recycling represents approximately 7% of the total carbon emissions across the niobium supply chain, underscoring its significant potential for energy conservation and emission reduction[101–102]. Future advancements will focus on green, refined, and intelligent recovery pathways: minimizing pyrometallurgical energy consumption via low-temperature processes, developing bio-hydrometallurgical or physical separation alternatives, enhancing separation efficiency for chemically similar elements (e. g., Nb/Ta), and implementing AI-optimized process control systems.

6 Future Prospects

Currently, no single niobium preparation or purification technique can simultaneously achieve ultra-high purity and low energy consumption. Consequently, comprehensive utilization of complementary methodologies through scenario-specific multi-technique integration is essential. While thermal reduction typically yields products with purity restrictions and elevated energy consumption, its mass/heat transfer processes can be optimized via computational simulation. Employing hydrogen or molten salt as auxiliary reductants alongside by-product recycling offers significant improvements. Electrolytic methods face inherent efficiency constraints due to the transition-metal characteristics of niobium—its propensity for disproportionation reactions across multiple valence states. Research focusing on niobium ion coordination environments in molten salts and disproportionation inhibition represents a critical pathway toward enhanced current efficiency.

AI integration during purification can further elevate efficiency and product quality: (1) digital twin technique enables full-process simulation to reduce experimental costs; (2) adaptive electron beam power regulation minimizes energy waste; (3) AI-driven impurity distribution prediction optimizes beam scanning trajectories and enables autonomous parameter adjustment. From a lifecycle perspective, restructuring energy inputs and refining recycling protocols can substantially reduce cumulative energy demand of niobium. Strategic implementation includes deploying renewable energy (e. g., solar and microwave) for heat production to displace fossil fuels and applying high-energy ball milling for waste activation to boost recovery rates. Collectively, these advancements will transition niobium pyrometallurgy from traditional process refinement toward emerging technological paradigms, amplifying its strategic importance in advanced materials manufacturing.

7 Conclusions

1) The current status and progress on the preparation and purification of metallic niobium by pyrometallurgy were reviewed, and the advantages and disadvantages of different methods were compared. At present, the focus of the thermal reduction method is on reducing energy consumption and improving product purity, including calorific value simulation and hydrogen plasma/molten salt-assisted reduction. Meanwhile, the electrolytic method aims to inhibit disproportionation reactions to improve efficiency. Comprehensive utilization of complementary methodologies through scenario-specific multi-technique integration is essential.

2) The difficulty in metal purification lies in the trace removal of similar elements, which requires the coupling of multiple physical fields to enhance the separation process. Meanwhile, poor process stability remains a challenge. The introduction of AI can further improve product quality, for example, through digital twins, impurity distribution prediction, and adaptive parameter adjustment.

3) From a lifecycle perspective, urban mines are gradually becoming an important source of niobium. Improving the recovery rate of niobium helps reduce the overall energy demand of the niobium industry chain. Replacing fossil fuels with clean energy sources such as solar and wind power for thermal reduction can effectively lower carbon emissions.

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金属铌火法制备和提纯的研究现状与进展

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摘　要：铌作为一种战略性关键金属，因其卓越的性能而被广泛应用于航空航天、核工业、超导体等领域。随着可持续发展的要求，铌冶炼过程中的能耗问题日益突出。绿色、低碳和节能已成为未来的新发展方向。此外，微电子行业需要高纯铌作为溅射靶材。尽管铌的纯度最高已达到5N，但低成本的高纯化技术仍颇具挑战性。本文总结了通过火法冶金制备和提纯粗铌的各种方法。作为生产粗铌的传统方法，热还原法的关键问题在于如何降低能耗。作为有工业前景的技术，熔盐电解已发展出多种方法，但效率仍有待提高。此外，一些新兴技术不断涌现，例如借助数字孪生和人工智能的全自动熔炼技术。未来，需要结合多种技术手段来提纯金属铌。本文还简要介绍了铌回收的现状，并基于城市矿山的概念进一步探讨了铌的全生命周期，以期为实现铌的回收利用提供方向。

关键词：铌；还原；提纯；火法冶金；回收

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