

## Overview of *in-situ* oxygen production technologies for lunar resources

Youpeng Xu, Sheng Pang, Liangwei Cong, Guoyu Qian, Dong Wang, Laishi Li, Yusheng Wu, and Zhi Wang

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
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## Overview of *in-situ* oxygen production technologies for lunar resources

Youpeng Xu<sup>1,2</sup>, Sheng Pang<sup>2,3,4</sup>,, Liangwei Cong<sup>1,2</sup>, Guoyu Qian<sup>2,3,4</sup>,, Dong Wang<sup>2,3,4</sup>, Laishi Li<sup>1</sup>,  
Yusheng Wu<sup>1</sup>,, and Zhi Wang<sup>2,3,4</sup>,

1) College of Materials Science and Engineering, Shenyang University of Technology, Shenyang 110870, China

2) CAS Key Laboratory of Green Process and Engineering, National Engineering Research Center for Green Recycling of Strategic Metal Resources, Institute of Process Engineering, Chinese Academy of Sciences, Beijing 100190, China

3) Innovation Academy for Green Manufacture, Chinese Academy of Sciences, Beijing 100190, China

4) School of Chemical Engineering, University of Chinese Academy of Sciences, Beijing 100049, China

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**Abstract:** The rich resources and unique environment of the Moon make it an ideal location for human expansion and the utilization of extraterrestrial resources. Oxygen, crucial for supporting human life on the Moon, can be extracted from lunar regolith, which is highly rich in oxygen and contains polymetallic oxides. This oxygen and metal extraction can be achieved using existing metallurgical techniques. Furthermore, the ample reserves of water ice on the Moon offer another means for oxygen production. This paper offers a detailed overview of the leading technologies for achieving oxygen production on the Moon, drawing from an analysis of lunar resources and environmental conditions. It delves into the principles, processes, advantages, and drawbacks of water-ice electrolysis, two-step oxygen production from lunar regolith, and one-step oxygen production from lunar regolith. The two-step methods involve hydrogen reduction, carbothermal reduction, and hydrometallurgy, while the one-step methods encompass fluorination/chlorination, high-temperature decomposition, molten salt electrolysis, and molten regolith electrolysis (MOE). Following a thorough comparison of raw materials, equipment, technology, and economic viability, MOE is identified as the most promising approach for future *in-situ* oxygen production on the Moon. Considering the corrosion characteristics of molten lunar regolith at high temperatures, along with the Moon's low-gravity environment, the development of inexpensive and stable inert anodes and electrolysis devices that can easily collect oxygen is critical for promoting MOE technology on the Moon. This review significantly contributes to our understanding of *in-situ* oxygen production technologies on the Moon and supports upcoming lunar exploration initiatives.

**Keywords:** lunar resources; *in-situ* oxygen production; space metallurgy; molten lunar regolith electrolysis

### 1. Introduction

In recent years, the rapid development of human technologies has refocused global attention on space exploration [1]. The Moon, Earth's satellite, is widely regarded as an ideal location for training and as a base for developing and utilizing extraterrestrial resources [2]. From the Apollo program to the Chang'e program, the Moon has been proven to possess abundant resources [3]. For instance, lunar basalt contains over 15 trillion tons of ilmenite that could be exploited [4]. The lunar soil is estimated to have between 100 and 500 million tons of helium-3, a potential fusion fuel that could satisfy human energy demands for more than 10000 years [5–6]. In addition, the water-ice resources in the permanently shadowed areas of the Moon are extremely abundant. According to the three-stage development concept of lunar exploration, which includes landing, exploring, and establishing bases, manned lunar landings are anticipated by 2030, with the construction of lunar scientific research bases to follow. There-

fore, the effective *in-situ* utilization of lunar resources is crucial.

Oxygen is crucial for sustaining human life on the Moon, and liquid oxygen serves as an excellent propellant for space missions. However, transporting materials from Earth to the Moon is currently estimated to cost between \$20000 and \$30000 per pound [7]. Consequently, the current supply-based oxygen delivery method is ill-suited to meet the future demands of constructing large-scale bases and launching spacecraft. Yet, the Moon, abundant in resources, contains approximately 45% oxygen in its soil in the form of metal oxides alongside substantial reserves of water ice [8–9]. Employing suitable technologies for oxygen production on the Moon would significantly bolster the large-scale development and utilization of its resources. Achieving technological breakthroughs in lunar *in-situ* oxygen production will play a vital role in enabling future lunar bases to reduce reliance on Earth for oxygen supply, thereby enhancing human sustainability and resilience on the lunar surface.

 Corresponding authors: Zhi Wang E-mail: [zwang@ipe.ac.cn](mailto:zwang@ipe.ac.cn); Sheng Pang E-mail: [spang@ipe.ac.cn](mailto:spang@ipe.ac.cn);

Guoyu Qian E-mail: [gyqian@ipe.ac.cn](mailto:gyqian@ipe.ac.cn); Yusheng Wu E-mail: [wuyus@sut.edu.cn](mailto:wuyus@sut.edu.cn)

Various oxygen production technologies using water and metal oxides as raw materials have been developed on Earth [10]. However, the Moon's environmental conditions, such as low gravity, high vacuum levels, prolonged daylight periods, and intense radiation, significantly differ from those on Earth. It is therefore necessary to comprehensively evaluate the advantages and disadvantages of various oxygen production technologies, which will help in selecting the most suitable *in-situ* oxygen production technology for future lunar applications [11]. This review provides a brief overview of the resources available on the Moon and its environmental characteristics. It systematically introduces the mainstream technologies that are expected to enable *in-situ* oxygen production on the Moon. The discussion primarily focuses on the principles and processes of different oxygen production methods, such as water-ice electrolysis, two-step oxygen production from lunar regolith, and one-step oxygen production from lunar regolith. After a comprehensive assessment of the raw material availability and the equipment, technology, and economic feasibility, this review concludes that the electrolysis of molten lunar regolith presents the most promising method for future *in-situ* oxygen production on the Moon.

## 2. Current status of lunar exploration

### 2.1. Overview of lunar resources

In the context of *in-situ* resource utilization (ISRU) for lunar exploration, the presence of water ice and surface regolith on the Moon holds particular significance for O<sub>2</sub> extraction and metal smelting [12]. Minerals found on the Moon, such as ilmenite, apatite, olivine, feldspar, and pyroxene, contain numerous oxides [13]. Therefore, through a series of technologies, it is possible to either substitute the bound oxygen within these minerals or to directly release it. Additionally, the availability of water resources on the Moon represents an alternative pathway for oxygen extraction.

#### 2.1.1. Water ice

From the 1960s to the present day, research has extensively focused on the water resources located within the cold, permanently shadowed impact craters at the lunar poles [14]. Early reports indicated the presence of water in the form of water ice in these permanently shadowed regions, with estimated quantities ranging from 66 to 200 billion tons [15–17]. Deutsch *et al.* [16] discovered that significant water-ice deposits are not confined to large impact craters but are also present in micro cold traps, rough patches, and intercrater terrains. Numerous scientists have investigated the abundance and distribution of water and other volatiles on the Moon [18–21]. In 2015, Crawford estimated the total mass of surface water in the uppermost layer of the permanently shadowed regolith to be around 29 billion tons [19]. In 2017, Fisher *et al.* [20] used various techniques to confirm the presence of surface water ice in the lunar polar regions. In 2018, Li *et al.* [21] confirmed the presence of valuable water resources on the lunar surface, suggesting that the ice content dispersed within the regolith could be as high as 30wt%

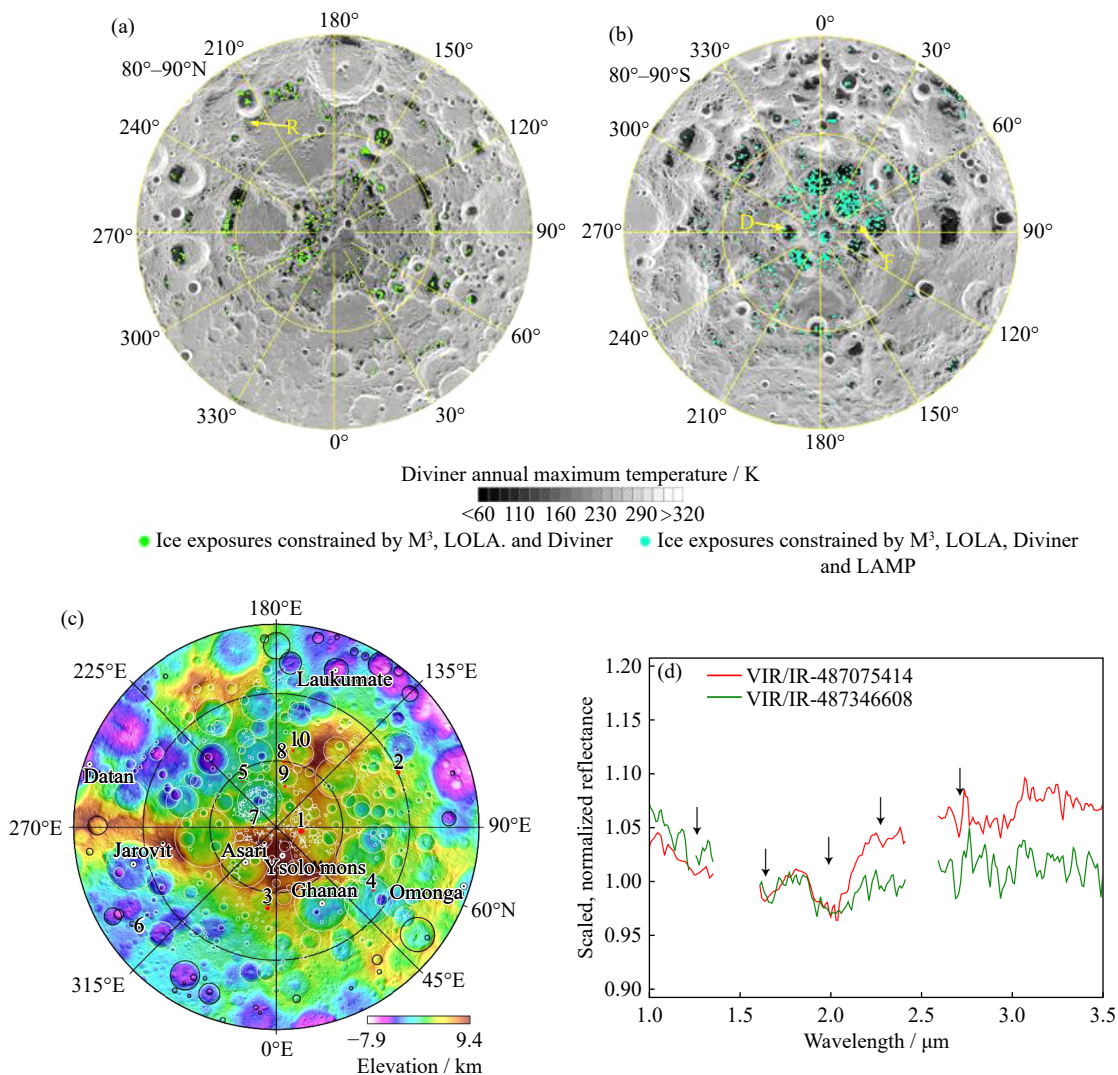
(Fig. 1(a) and (b)). Platz *et al.* [17] discovered bright deposits at the bottom of ten impact craters, with spectroscopic analysis identifying one of these bright deposits as water ice. This finding further substantiates the preservation of water ice in the permanently shadowed regions on airless celestial bodies (Fig. 1(c) and (d)).

#### 2.1.2. Lunar regolith

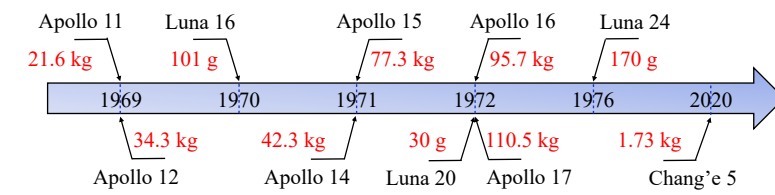
The lunar regolith is a significant source of oxygen, containing up to 44wt% in the form of stable metal oxides. This ubiquity across the lunar surface means that oxygen can be extracted virtually anywhere on the Moon. Prior to the 21st century, human understanding of lunar geological evolution stemmed largely from samples returned from the Apollo and Luna missions between 1969 and 1976, during which astronauts from the United States and the Soviet Union successfully landed on the Moon nine times and brought back a total of 382 kg of lunar samples. However, after the last manned lunar sampling mission, Apollo 24, lunar exploration activities by both nations ceased for a period. It was not until recent years that China resumed lunar exploration under the “Chang’e Project.” On December 17, 2020, the Chang’e-5 returner successfully brought back 1.73 kg of lunar samples to Earth, concluding China’s three-phase lunar exploration project of “orbiting, landing, and returning,” as depicted in Fig. 2 [22].

The lunar regolith represents the primary mineral resource on the lunar surface. Extensive research has confirmed that it is rich in oxygen and polymetallic oxides. It primarily contains metal oxides such as SiO<sub>2</sub>, TiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, FeO, Fe<sub>2</sub>O<sub>3</sub>, MgO, and CaO, as listed in Table 1 [23–26]. Owing to the limited availability of actual lunar regolith, experimental research often relies on simulated samples such as JSC-1 from NASA Johnson Space Center [22], MLS-1 from the University of Minnesota [23], CAS-1 from the Institute of Geochemistry, Chinese Academy of Sciences [25], and NEU-1 from Northeastern University [26]. The regolith contains complex oxides, including feldspar ((Ca,Na)(Al,Si)<sub>4</sub>O<sub>8</sub>), pyroxene ((Ca,Mg,Fe)<sub>2</sub>Si<sub>2</sub>O<sub>6</sub>), olivine ((Mg,Fe)<sub>2</sub>SiO<sub>4</sub>), and ilmenite (FeTiO<sub>3</sub>). Actual lunar regolith samples include Apollo 17 soil 70051 [23] and those returned by Chang’e-5 [27]. Utilizing mineral resources on the lunar surface to produce oxygen for human life activities and extract metals for base construction is crucial. Current methods for extracting oxygen and metals from lunar regolith include hydrogen reduction, carbon thermal reduction, metal thermal reduction, hydrometallurgy, high-temperature decomposition, and electrolytic reduction. Given the unique environmental characteristics of the lunar surface, developing an efficient method for direct metallurgical extraction of metals and oxygen from lunar regolith, without the need for ore selection, is imperative.

The lunar regolith collected by Chang’e-5 represents the first lunar return sample obtained in 44 years, leading to groundbreaking discoveries in several areas. Guo *et al.* [28] were the first to discover chalcantite (Cu<sub>1.8</sub>S) in lunar regolith (Fig. 3(a)), a finding that underscored the vital role of



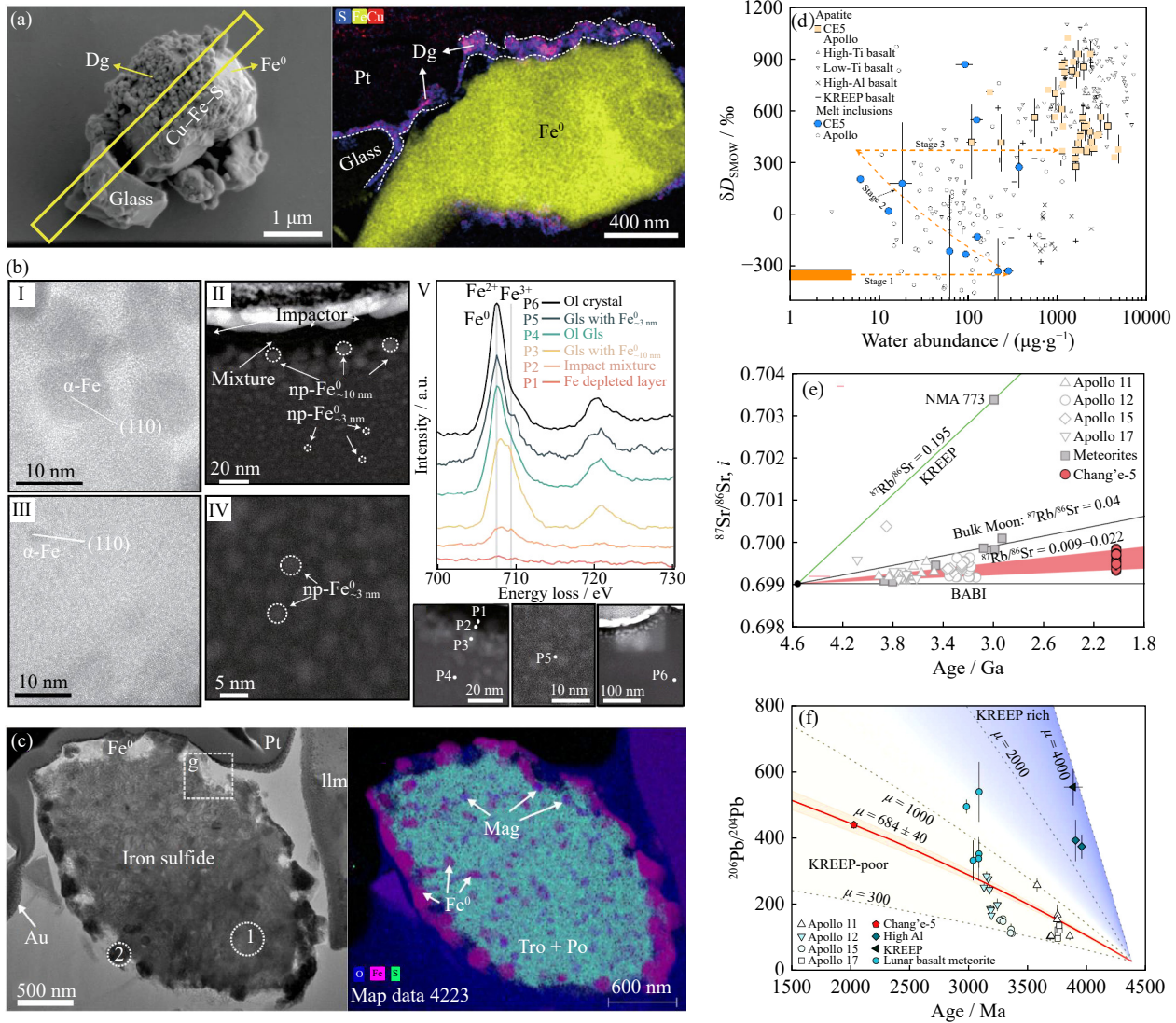
**Fig. 1.** Distribution of water-ice-bearing pixels (green and cyan dots) overlain on the Diviner annual maximum temperature for the northern (a) and southern (b) polar regions. Reprinted from [21]. (c) Craters hosting permanent shadows and bright deposits and (d) water-ice signatures within Crater 2 in visible mapping spectrometer (VIR) spectra [17]. Reprinted by permission from Springer Nature: *Nat. Astron.*, Surface water-ice deposits in the northern shadowed regions of Ceres, T. Platz, A. Nathues, N. Schorghofer, et al., Copyright 2017.



**Fig. 2.** Timeline diagram of humans bringing lunar regolith back to Earth [22].

**Table 1.** Chemical compositions of lunar regolith and some simulated lunar regolith samples

Sample	SiO <sub>2</sub>	TiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	FeO	Fe <sub>2</sub> O <sub>3</sub>	MgO	CaO	Na <sub>2</sub> O	K <sub>2</sub> O	Cr <sub>2</sub> O <sub>3</sub>	MnO	P <sub>2</sub> O <sub>5</sub>	Total	wt%
Appllo17 [23]	42.2	5.09	15.7	12.4	—	10.3	11.5	0.24	0.07	—	0.15	—	97.7	
CE-5 [24]	42.1	5.7	11.6	22.2	—	5.8	10.9	0.6	0.1	0.2	0.3	0.2	99.7	
JSC-1 [23]	47.71	1.59	15.02	7.35	3.44	9.01	10.42	2.7	0.82	0.04	0.18	0.66	98.9	
MLS-1 [23]	43.86	6.32	13.68	13.4	2.6	6.68	10.13	2.12	0.28	—	0.2	0.2	99.5	
CAS-1 [25]	49.24	1.91	15.8	11.47	—	8.72	7.25	3.08	1.03	—	0.14	0.3	98.9	
NEU-1 [26]	44.92	2.87	17.23	13.09	—	4.37	9.44	3.79	3.01	0.04	0.34	0.54	99.6	



**Fig. 3.** (a) The blue copper mineral chalcanthite, formed by evaporative deposition in the lunar regolith of the Chang'e-5 mission [28]. Reprinted from *Sci. Bull.*, 68, Z. Guo, C. Li, Y. Li, *et al.*, Vapor-deposited digenite in Chang'e-5 lunar soil, 723-729, Copyright 2023, with permission from Elsevier. (b) Results of crystal spacing (I–IV) and electron energy loss spectra (V) of different grain sizes of np-Fe<sup>0</sup> in the olivine fusion layer at the bottom of the micro-impact crater [29]. Adapted by permission from Springer Nature: *Nat. Astron.*, Impact-driven disproportionation origin of nanophase iron particles in Chang'e-5 lunar soil sample, C. Li, Z. Guo, Y. Li, *et al.*, Copyright 2022. (c) Spherical troilite grains containing magnetite in the lunar regolith from the Chang'e-5 mission. Reprinted from [30]. (d) Water content and hydrogen isotopic composition of apatite and melt inclusions in Chang'e 5 basalt samples. Reprinted from [31]. (e) Nd isotopic analyses for Chang'e-5 basalt. Reprinted from [32]. (f) Age and source-area  $\mu$ -value distributions of returned lunar samples and selected lunar meteorites. Reprinted from [33].

meteorite impacts in facilitating gas–solid deposition processes on the Moon. This discovery sheds light on the migration of gas-phase components and their profound effects on lunar surface materials. Li *et al.* [29] initially confirmed the presence of metallic iron in the lunar regolith, originating from the disproportionation reactions in iron olivine, enriching our understanding of the origin of metallic iron on the lunar surface. They examined the crystal lattice spacing within different-sized np-Fe<sup>0</sup> in the olivine melt layer at the bottom of micro-impact craters, complemented by electron energy loss spectroscopy analysis and Gibbs free energy calculations for the disproportionation and decomposition reactions of FeO (Fig. 3(b)). Guo *et al.* [30] also identified native magnetite in lunar regolith for the first time (Fig. 3(c)), point-

ing out that sulfides belong to weathering-sensitive minerals. This discovery offers new evidence for lunar magnetic carrier minerals and potentially leads to the development of new criteria for assessing lunar regolith maturity. Hu *et al.* [31] found a clear negative correlation between water content and hydrogen isotopic composition in most melt inclusions, indicating magma degassing processes and a trend of decreasing mantle water content over time (Fig. 3(d)). Their findings challenge the theory that prolonged abnormal lunar volcanic activity on the Moon was driven by high mantle water content reducing the melting point. Tian *et al.* [32] found significant differences in Sr and Nd isotopes in Chang'e-5 lunar basalts compared to those in KREEP (Fig. 3(e)), with less than 0.5% contribution from KREEP components. This in-

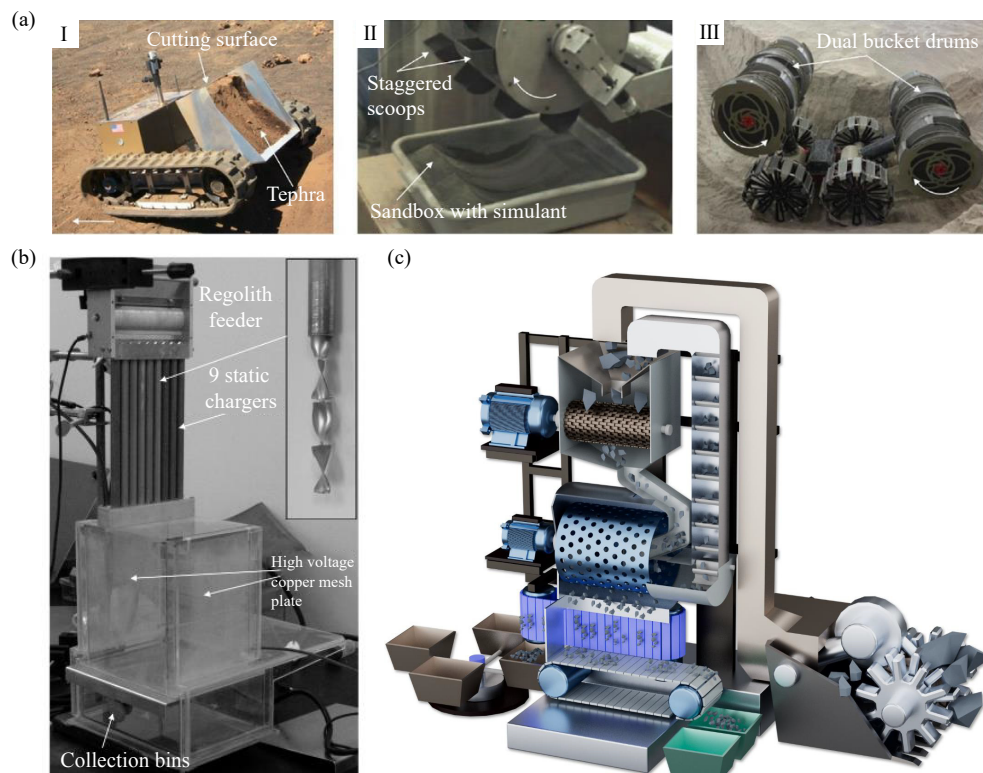
dicates that the Moon's long-term lunar activity is not maintained by KREEP abundance in the mantle. Through their research, Li *et al.* [33] found that the age of Chang'e-5 lunar basalts provides a crucial anchor point at 2 billion years on the geochronological curve (Fig. 3(f)), greatly improving the accuracy of the "crater dating method" and providing an important methodological basis for precise dating of future lunar samples.

In addition, current lunar scientific exploration and research results indicate that the Moon may harbor abundant rare resources such as helium-3 ( $^3\text{He}$ ). The lunar surface is exposed to solar wind and other forms of particle bombardment. In hexagonal crystal structures like ilmenite ( $\text{FeTiO}_3$ ), these conditions are conducive to capturing and retaining  $^3\text{He}$ , leading to its high enrichment and preservation. Therefore, research into the extraction technology of ilmenite is crucial for future utilization of lunar resources. Successfully extracting and enriching ilmenite from lunar regolith would provide the necessary materials for *in-situ* oxygen production and metal extraction on the lunar surface, thereby enhancing human capabilities for lunar residency and long-term space survival. This achievement could pave the way for new interstellar exploration ventures, support innovative developments in China's manned spaceflight program, and serve

strategic goals for the peaceful utilization of space.

The premise for the collection and utilization of ilmenite in lunar regolith is its collection. Experimental research on lunar regolith collection has been conducted extensively both domestically and internationally. For instance, the Robotics Institute at Carnegie Mellon University in the United States has developed a discrete excavation machine characterized by disconnecting contact with the soil between cuts to clear the cutting surface and dump collected lunar regolith. For continuous regolith collection, researchers have proposed the use of a continuous excavation machine. Additionally, a continuous excavation machine has been proposed, including a variant with a reverse rotating drum and cutting surface indicator. The three lunar regolith collection devices are illustrated in Fig. 4(a) [34].

For lunar regolith samples containing ilmenite, further analysis and evaluation of the content and selectivity of ilmenite are necessary [35]. Ilmenite enrichment in lunar regolith is a crucial aspect of space resource utilization. Based on this premise, national aeronautics and space administration (NASA) [36] has introduced a technology combining frictional charging with a free-fall parallel plate separator for mineral enrichment of ilmenite-containing lunar regolith. Fig. 4(b) depicts a free-fall frictional charging separator,



**Fig. 4.** (a) Examples for the three different main excavator system classes: (I) discrete complete system with indication of cutting surface and excavation direction, (II) continuous partial system with indication of cutting surface and direction of bucket rotation, and (III) continuous complete system with indication of counter rotating bucket drums and cutting surfaces [34]. Reprinted from *Planet. Space Sci.* 180, G.H. Just, K. Smith, K.H. Joy, and M.J. Roy, Parametric review of existing regolith excavation techniques for lunar *in situ* Resource Utilisation (ISRU) and recommendations for future excavation experiments, art. No. 104746, Copyright 2020, with permission from Elsevier. (b) Electrostatic beneficiation apparatus as designed for the RGF. Reprinted from Ref. [36], with permission from American Society of Civil Engineers. (c) The integrated prototype for lunar regolith crushing, sieving, and magnetic enrichment of ilmenite.

which has successfully enriched ilmenite from lunar soil simulant. The technology is notable for its simplicity and the use of lightweight and low-density components, and has been tested across a broad range of environmental conditions. Our team is also developing an integrated prototype that encompasses the crushing, screening, and magnetic separation enrichment of ilmenite from lunar regolith. This prototype will be based on principles of miniaturization, lightweight design, and precision, employing an intelligent precision machining and forming system for its production. We plan to conduct integrated construction and performance verification tests on the ore selection and enrichment prototype, and these tests will evaluate the compatibility and integration level among the various systems through practical testing, aiming to strengthen the seamless integration among key components, scenarios, and objects. We will also conduct full-process simulation tests for ore selection and enrichment of ilmenite-containing lunar regolith in a simulated lunar surface environment, testing the long-term stability of the prototype, especially its key core components) and, based on the outcomes, optimize and improve the prototype. Fig. 4(c) illustrates the conceptual design of the prototype for the ore selection and enrichment of ilmenite-containing lunar regolith.

## 2.2. Lunar environment

The lunar surface is abundant in regolith and solar energy, presenting the opportunity to utilize solar for decomposing regolith for oxygen production. Achieving this breakthrough could provide crucial support for future lunar bases, making them independent from Earth's oxygen supply and enhancing human sustainability for long-term lunar residency. However, oxygen production from lunar resources involves complex chemical reactions and multiphase separations. The rates and efficiencies of these processes are highly constrained by external environmental factors such as pressure and gravity. Characterized by ultra-high vacuum, low gravity, and significant temperature variations, the lunar environment poses challenges for simulating these unique and harsh conditions over extended periods on Earth and in near-lunar space stations. Therefore, developing reliable oxygen production technology and equipment necessitates research conducted directly on the lunar surface. According to data from the Chang'e-4 mission, lunar surface radiation levels reach 60 microsieverts per hour, twice the level of radiation level experienced on the International Space Station and 5–10 times higher than that of long-haul flights, and 200 times the radiation level on Earth's surface [37]. In the first and second lunar days following the Chang'e-4 landing, the Lunar Lander Neutrons and Dosimetry instrument measured the time evolution of the Moon's radiation environment. Furthermore, data from spacecraft missions such as Apollo 15 and Apollo 16 indicate that the lunar magnetic field intensity is generally low, only a few nanoteslas, varying significantly across different regions [38]. Additionally, the lunar surface is covered by a large number of fine particles ranging from tens of nanometers to hundreds of micrometers in diameter,

known as lunar dust [39]. The Lunar Dust Experiment charted the distribution of lunar dust [40]. Owing to their small size and highly irregular structure, lunar dust particles easily adhere to the surfaces and interiors of lunar exploration equipment, leading to mechanical jamming and component wear, which can significantly affect the long-term operation of lunar exploration equipment.

## 3. In-situ oxygen production methods for lunar resources

So far, there are eight main methods for *in-situ* oxygen production from lunar resources, as illustrated in Fig. 5.

### 3.1. Electrolysis of water ice

Since water electrolysis can directly produce oxygen), the presence of water ice on the Moon provides important insights for further ISRU research and development. The principal process of oxygen production through electrolysis, utilized in the American space station [41], is illustrated in Fig. 6(a). Currently, various technologies for extracting oxygen from lunar water ice are being explored, including solid polymer electrolytes, flowing alkaline electrolytes, static water supply fixed electrolytes, and solid oxide electrolysis cells.

The solid polymer electrolysis technology system employs a perfluoro sulfonic acid ion exchange membrane as the sole electrolyte for water electrolysis, eliminating the need for alkaline or acidic solutions. The Nafion membrane, a type of perfluoro sulfonic acid thin film, demonstrates excellent ionic conductivity when hydrated. Its operation principle involves the voltage-driven water decomposition into hydrogen and oxygen. The General Electric Company of the United States successfully developed a fuel cell electrolyzer utilizing the Nafion membrane as the electrolyte, which was first applied on the "Gemini" spacecraft [42–44], as depicted

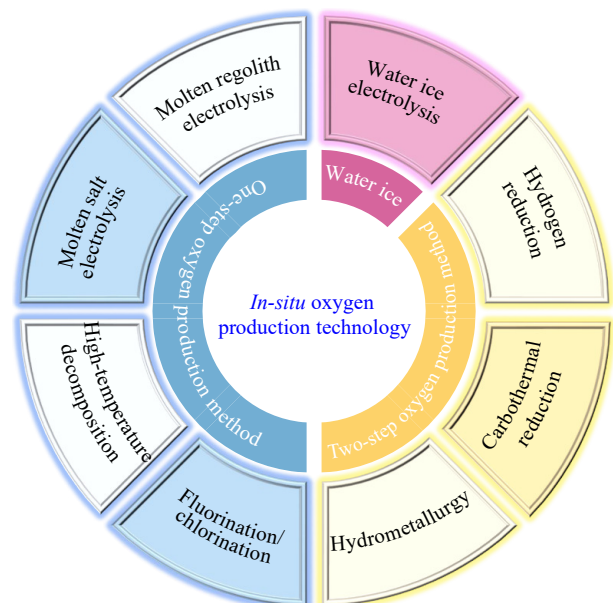


Fig. 5. Main methods of oxygen production.

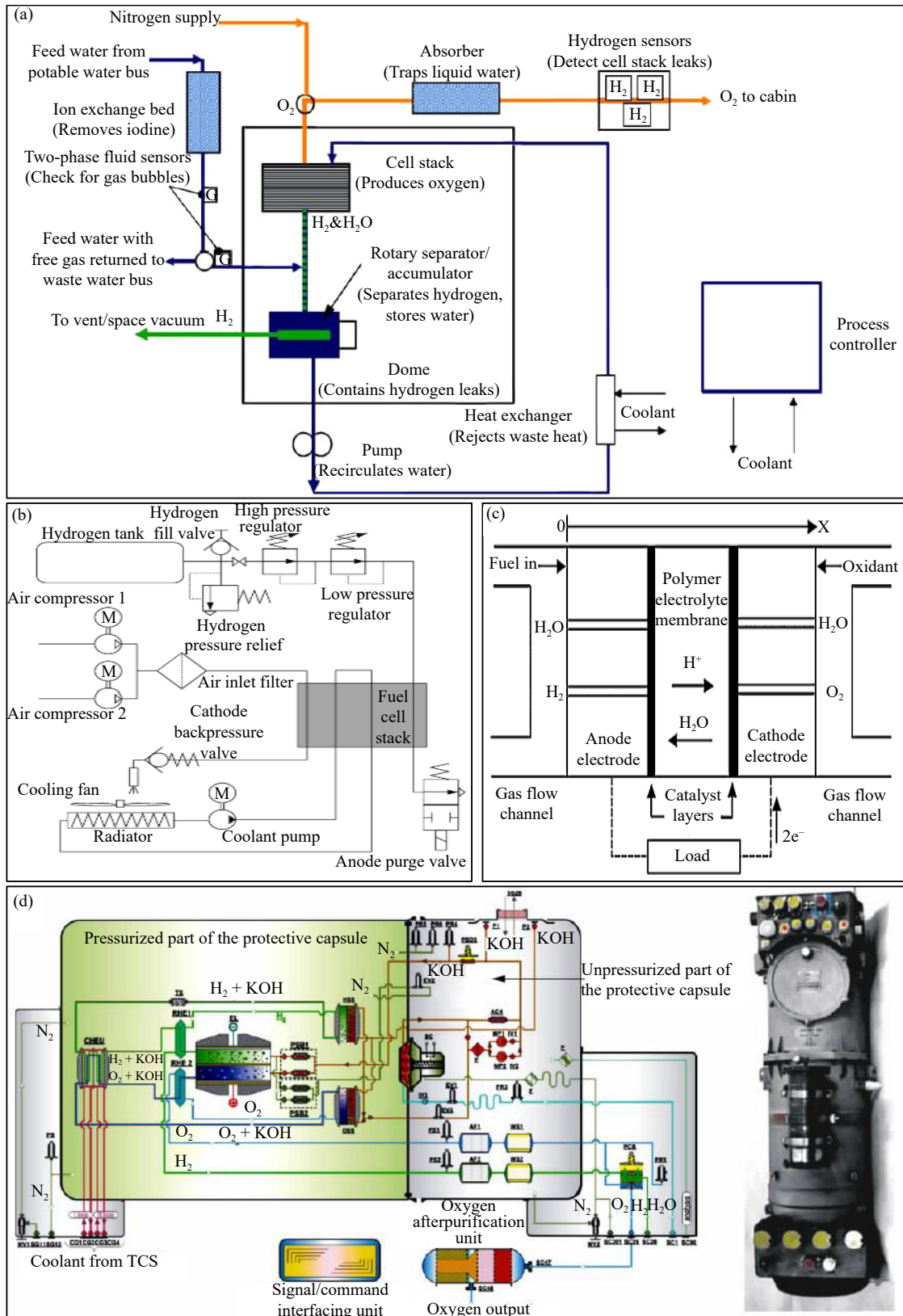


Fig. 6. (a) Process of electrolytic oxygen production in the American space station. Reprinted from Ref. [41], with permission from SAE International. (b) Fuel cell powerplant diagram and system specifications [42]. Reprinted from *J. Power Sources*, 171, T.H. Bradley, B.A. Moffitt, D.N. Mavris, and D.E. Parekh, Development and experimental characterization of a fuel cell powered aircraft, 793-801, Copyright 2007, with permission from Elsevier. (c) Schematic diagram of a proton exchange membrane fuel cell (PEMFC) [43]. Reprinted from *J. Power Sources*, 196, C.H. Lee and J.T. Yang, Modeling of the Ballard-Mark-V proton exchange membrane fuel cell with power converters for applications in autonomous underwater vehicles, 3810-3823, Copyright 2011, with permission from Elsevier. (d) Electrolysis-based oxygen generation system on the Russian "Mir" space station. Reprinted from [45], with permission from SAE International.

in the fuel cell power unit diagram and the proton exchange membrane fuel cell schematic in Fig. 6(b) and (c) [42–43], respectively. A significant advantage of this system is that it solely uses water, eliminating the use of corrosive liquids, thereby enhancing safety and simplifying the electrode structure for optimizing catalytic activity. However, the unique properties of the electrolyte lead to lower electrolysis efficiency compared to alkaline electrolytes, resulting in higher water vapor content in the product gas and poor dehydration tolerance of the membrane material.

The flowing alkaline electrolyte oxygen production technology utilizes a highly conductive KOH solution as the electrolyte. This process, under voltage, breaks down water molecules into hydrogen and oxygen. Russia applied this approach to develop an oxygen production system used on the “Mir” space station (Fig. 6(d)) [45]. A primary benefit of this technology is its efficient management of heat and by-products. The electrolyte facilitates both temperature control within the cell and the swift removal of gases generated from the reaction zone. Furthermore, the electrolyte can be fully replenished to the electrolyte electrode, allowing for high current densities and versatile operational capabilities. However, the corrosive nature of the flowing alkaline electrolyte poses a challenge to equipment longevity, necessitating a purifier to ensure the purity of the produced oxygen.

The static water supply fixed electrolyte technology, developed by Russia, aims to reduce the system’s water and gas separation processes, thereby reducing equipment and operational load. This approach uses a porous electrode electrolysis cell with cathode feeding, where the electrolyte is embedded within the electrode material for enhanced stability, forming a fixed alkaline electrolyte (FAE) [46]. Potassium hydroxide is favored as the electrolyte for its ability to reduce the vapor pressure and decrease the water content in the output gas. The advantages of this technology include its lightweight and compact oxygen production system, simple operation, long service life, near-perfect Faraday efficiency, and relatively low power consumption. However, the electrolysis cell’s intricate structure, comprising oxygen, hydrogen, water supply, and cooling chambers, introduces pressure control and potential electrolyte loss.

Solid oxide electrolysis cells (SOEs) function at high temperatures above 500°C, where water vapor is converted into chemical energy through electrical and thermal inputs. In recent years, some ceramic proton conductors have garnered widespread attention for their application in steam electrolysis. These materials exhibit good ionic conductivity and efficiency at 500–700°C and demonstrate excellent chemical compatibility with commonly used hydrogen-evolving electrodes, such as nickel, showing promising prospects in oxygen production through electrolysis [47]. The advantage of SOE electrolysis cells lies in the solid form of the electrolyte, enabling operation under pressure differentials. However, the drawbacks include the susceptibility to electrode breakage, safety hazards, and the requirement for high-level sealing.

In addition, Ethridge and Kaukler developed a low-tem-

perature apparatus to test the feasibility of extracting water from the regolith using microwaves [48]. In a vacuum, they evaporated 85% of the water from the regolith permafrost (0.5wt%–2wt%) and captured 99% of the water using a liquid nitrogen trap to demonstrate the feasibility of *in-situ* microwave water extraction without prior excavation.

Another challenge in water electrolysis for oxygen production lies in the separation and purification of newly formed gases, as various impurities such as hydrogen sulfide, CO, organic compounds, and NH<sub>3</sub> may appear during the electrolysis of the ice-regolith layer. To address this, Schlüter *et al.* [49] proposed a volatile ice extraction process with low-temperature distillation and purification steps.

The extraction and melting of water ice are highly energy-intensive processes. This challenge is compounded by the fact that lunar polar craters are perpetually shaded and therefore cannot directly utilize solar energy. To overcome this, the ice-rich lunar soil must either be transported to sunlit areas of the lunar surface where temperatures are higher or energy must be delivered to the shaded locations where the ice is located. While water is a crucial source of oxygen, the extraction of water ice does not yield useful metallic by-products. This limitation, combined with the current technological constraints on water-ice extraction, makes the sole use of water-ice electrolysis for oxygen production on the Moon impractical in the short term. Consequently, the integration of water electrolysis technology with other techniques is emerging as a promising approach for developing *in-situ* oxygen production capabilities on the Moon.

### 3.2. Two-step oxygen production method using lunar regolith

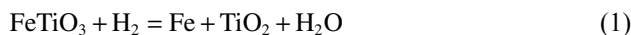
The lunar regolith’s two-step oxygen production method involves initially converting lunar soil into water through a reaction, thereby obtaining water resources. Subsequently, this water is utilized in an electrolysis reaction to decompose it into oxygen and hydrogen.

#### 3.2.1. Hydrogen reduction

The hydrogen reduction process emerged in the 1960s as one of the first proposed ISRU techniques for lunar oxygen production. This method involves reacting solid lunar regolith with a stream of hydrogen gas at temperatures between 800 and 1000°C, resulting in water production. The water is then separated from the hydrogen gas stream using a condensation water trap. However, this method requires a significant amount of energy to cool the gas down to 100°C and then reheat the hydrogen gas to the process temperature. Subsequently, the water produced undergoes isolation, purification, and electrolysis to generate H<sub>2</sub> and O<sub>2</sub>. The produced oxygen can be stored for use, while the hydrogen can be recycled back into the system.

Materials utilized in the study of H<sub>2</sub> reduction of lunar soil include ilmenite, volcanic glass, simulated lunar soil, high-titanium soil, and iron-rich volcanic ash, among others [7,50–53]. The H<sub>2</sub> reduction reaction of ilmenite follows a shrinking core model, where factors such as the gas flow rate,

reaction temperature and pressure, and reactor type significantly influence the reaction. At temperatures ranging from 900 to 1200°C, ilmenite undergoes reduction, breaking down into its metallic components and oxygen, using H<sub>2</sub> as the reducing agent. This method can also reduce oxides with weaker chemical bonds, such as FeO, TiO<sub>2</sub>, and SiO<sub>2</sub>, as illustrated by Eqs. (1) and (2):



In 1990, Gibson *et al.* [54] utilized lunar basalt 70035 as a raw material and conducted an oxidation-reduction reaction with hydrogen at temperatures ranging from 900 to 1050°C, resulting in an oxygen mass comprising 4.6% of the initial sample mass. In 1996, McKay and Allen [51] performed hydrogen reduction experiments on 16 lunar soil samples and 3 types of volcanic glass-rich lunar soil, achieving oxygen yields of 4.67% of the sample mass. That same year, they also synthesized findings from various experiments involving hydrogen reduction using Apollo samples and confirmed the relationship between oxygen yield and iron oxide content [55]. In 2000, Yoshida *et al.* [56], using a 40-g lunar soil simulant FJS-1 sample, confirmed that a reaction temperature of 1000°C and smaller particles with larger surface areas enhanced water production. They identified mass trans-

fer within the particles as the primary factor controlling the water production rate. In 2010, Lu *et al.* [57] conducted a thermodynamic analysis of the hydrogen reduction of lunar regolith and concluded that despite highly complex side reactions, iron oxide reduction is the only reaction capable of significantly producing oxygen and metal.

The advantages of this method are manifold. First, the required reaction temperature is relatively low (around 900°C), reducing energy requirements for the hydrogen reduction reaction. Second, hydrogen exhibits strong reducibility and does not produce other by-products during the reaction process, simplifying the reaction and contributing to equipment maintenance [58]. Additionally, H<sub>2</sub> can be recycled on-site. Third, the regolith remains granular throughout the process, posing low operational risks. Furthermore, both fluidized bed technology and water electrolysis technology are relatively mature. Moreover, the raw materials, including hydrogen derived from solar wind ions (primarily H and He), are abundant on the lunar surface. The concentration of H in representative lunar soil is approximately 50 µg/g, equivalent to a concentration of 100–200 g/m<sup>3</sup> in soil assuming a density of 1.75 g/cm<sup>3</sup>, partially compensating for the loss of hydrogen. Owing to its conceptual simplicity, this process has seen further development in NASA's exploration technology and development programs [59]. The hydrogen reduction reactor is illustrated in Fig. 7.

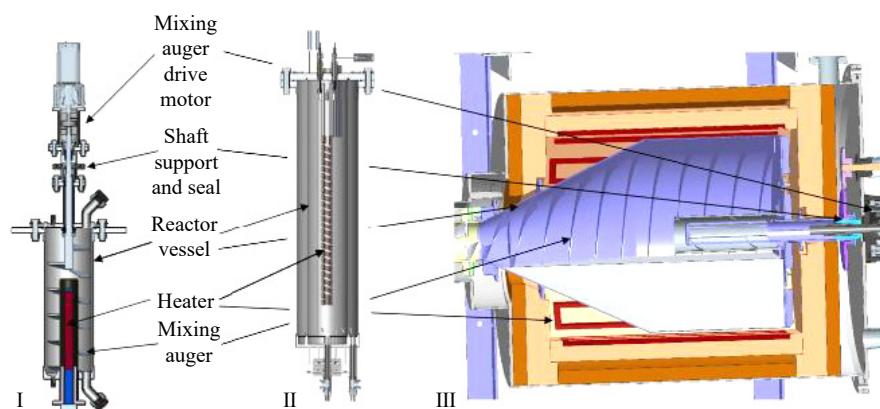
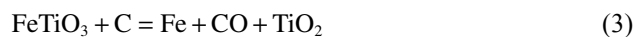


Fig. 7. Hydrogen reduction reactors: (I) ROxygen Gen I reactor, (II) ROxygen Gen II reactor, and (III) PILOT rotating reactor. Reprinted from Ref. [59], with permission of American Society of Civil Engineers.

The drawback of the hydrogen reduction method for producing hydrogen from lunar soil, despite its relative maturity, lies in the indirect acquisition of oxygen. Fe<sub>2</sub>O<sub>3</sub> in ilmenite is easily reduced by H<sub>2</sub> at temperatures above 700°C, resulting in oxygen production. However, reducing iron oxide in olivine and pyroxene with H<sub>2</sub> to produce metallic Fe is more challenging, leading to relatively lower yields. Ore dressing can further improve this process by increasing the ilmenite content in the raw materials to 70wt% [60]. If the ilmenite in the raw material has a sufficiently high titanium content, it can be directly reduced to produce metal and oxygen without the need for ore dressing [61–62]. Gas sealing and circulation in the reduction equipment also represent significant issues that need to be addressed [63].

### 3.2.2. Carbon/carbon thermal chlorination/carbon monoxide/methane reduction

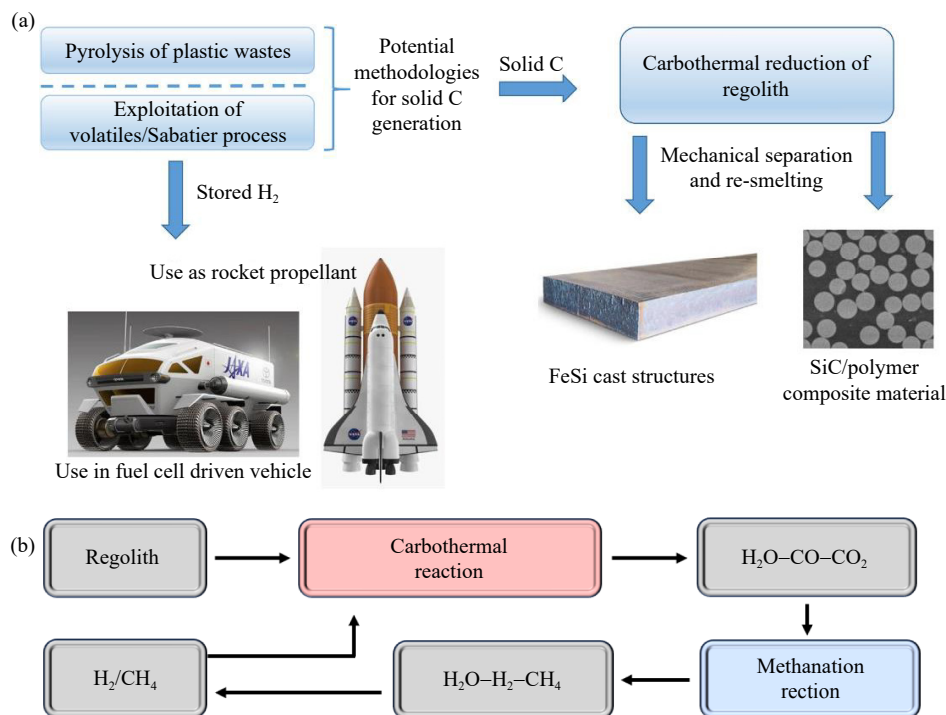
The principle of the carbon thermal reduction method involves utilizing a reducing agent to reduce oxides to metals at high temperatures. The reducing agent can be pure carbon, C-Cl<sub>2</sub>, or CO. Cutler and Krag [64] successfully produced oxygen through multistep reactions using carbon as a reducing agent during the carbon thermal reduction of ilmenite. The carbon reduction reactions in ilmenite to produce iron and oxygen are shown in Eqs. (3)–(6):





Sen *et al.* [65] achieved mass fractions of 91.9% for Fe and 4.59% for Si in the products obtained after reacting lunar soil simulant JSC-1 mixed with graphite at 1500°C for 1 h. Similarly, Samouhos *et al.* [66] utilized graphite powder as a reducing agent for the carbon thermal reduction of lunar soil simulant BP-1, successfully producing a silicon–iron alloy (Fe–Si) with a Si content of 23.78wt%, an Al content of 1.80wt%, a Ti content of 0.94wt%, and the by-product silicon carbide. During the experimental process, it was observed that an excess of carbon had a negligible impact on the reduction process at 1300°C; however, temperatures exceeding

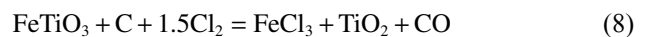
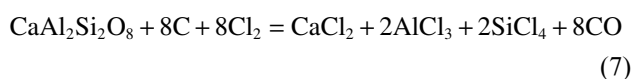
1300°C contributed to increasing the amount of SiC produced. Nevertheless, this led to two adverse outcomes: the enrichment of silicon in the silicon–iron alloy and diffusion issues between the alloy and the graphite crucible. The carbon thermal reduction process, its products, and the method for *in-situ* solid carbon production on the Moon are illustrated in Fig. 8(a). Granular silicon–iron alloy is a suitable raw material that can be used as feedstock for 3D-printing processes, as well as for metal casting processes, structural building components, and mechanical construction. Additionally, SiC can be utilized in the construction of composite polypropylene/SiC for space radiation shielding.



**Fig. 8.** (a) Comprehensive approach for the carbothermal reduction, its products, and the lunar *in-situ* production of solid carbon [66]. Reprinted from *Planet. Space Sci.*, 212, M. Samouhos, P. Tsakiridis, M. Iskander, M. Taxiarchou, and K. Betsis, *In-situ* resource utilization: Ferrosilicon and SiC production from BP-1 lunar regolith simulant via carbothermal reduction, art. No. 105414, Copyright 2022, with permission from Elsevier. (b) Process flow for methane carbothermal reduction [72]. Adapted from *Planet. Space Sci.*, 225, J. Prinetto, A. Colagrossi, A. Dottori, Ivan Troisi, and Michèle Roberta Lavagna, *Terrestrial demonstrator for a low-temperature carbothermal reduction process on lunar regolith simulant: Design and AIV activities*, art. No. 105618, Copyright 2023, with permission from Elsevier.

While this method demonstrates the potential for oxygen production in terrestrial simulations, its application in lunar environments mainly faces significant challenges, primarily involving the transportation and acquisition of carbon-based reducing agents on the lunar surface [67]. Furthermore, this method can only reduce Fe<sub>2</sub>O<sub>3</sub> leaving higher SiO<sub>2</sub> content, and the incomplete reduction of silicon in carbon thermal reduction process leads to inefficiency [68].

Carbon chlorination involves treating lunar soil simulants (calcium feldspar and ilmenite) with carbon and chlorine gas in a fluidized bed reactor, as depicted in Eqs. (7) and (8):



Under normal circumstances, Cl<sub>2</sub> struggles to react with the calcium feldspar in silicate minerals, necessitating the addition of carbon to facilitate the reaction. Multiple steps are required after carbon chlorination to obtain O<sub>2</sub>, and the recycling of C and Cl<sub>2</sub> is also extremely challenging, rendering this reaction practically unfeasible on the lunar surface [69].

Zhao and Shadman [70] investigated the kinetics and mechanisms involved in carbon monoxide (CO) reduction of ilmenite. The reaction for CO reduction of ilmenite, producing metallic iron and oxygen, is represented by Eqs. (9) and (10):

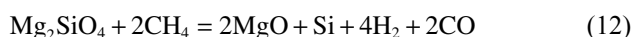




The CO reduction of FeTiO<sub>3</sub> yields Fe, TiO<sub>2</sub>, and CO<sub>2</sub>. Subsequently, CO<sub>2</sub> undergoes decomposition to produce CO and O<sub>2</sub>, thus achieving CO recycling. However, this method can result in CO leakages, necessitating ground transportation for replenishment.

Lu and Reddy [71] combined thermodynamic simulations and experimental results to analyze the carbon thermal reduction of lunar soil across a temperature range of 100–1500°C. Below 1100°C, only iron oxide can be significantly reduced, as depicted in the methane carbon thermal reduction process in Fig. 8(b) [72]. Therefore, a weathered layer rich in iron is advantageous for the solid weathered layer carbon thermal reduction, potentially requiring ore beneficiation to increase the iron content. Williams *et al.* [50] investigated ore beneficiation processes to increase the content of ilmenite (FeTiO<sub>3</sub>). Exceeding 1200°C, the weathered layers are partially or completely molten, and compounds such as TiC, FeSi, and Fe<sub>3</sub>Si can form, thereby releasing some of the oxygen bound in SiO<sub>2</sub> and TiO<sub>2</sub>. When the temperature of carbon thermal reduction exceed 1625°C, not only iron oxide but also silicon dioxide can be reduced, achieving an oxygen production rate of 28 kg per 100 kg of weathered layer [73]. However, metallic silicon is not formed at this stage; instead, SiC and gaseous SiO are produced, with metallic silicon only forming at temperatures above 2000°C [74–75]. Since SiO<sub>2</sub> is the most abundant oxide in lunar weathered layers, comprising 40wt%–50wt% of lunar soil mass, the production of molten lunar soil through carbon thermal reduction does not rely on the landing site, allowing the use of any weathered layer without the need for ore beneficiation.

The reactions representing the use of CH<sub>4</sub> to reduce ilmenite (FeTiO<sub>3</sub>) [76] and olivine (Mg<sub>2</sub>SiO<sub>4</sub>) to produce O<sub>2</sub> are represented by Eqs. (11) and (12):



In addition to obtaining Fe and Si, the reduction products include CO and H<sub>2</sub>. The CO hydrogenation reaction facilitates the recycling of the reducing agent CH<sub>4</sub> and the production of H<sub>2</sub>O, which can then be electrolyzed to yield H<sub>2</sub> and the desired product, O<sub>2</sub>. During the reduction process, the oxides present in the molten silicates may be reduced by CH<sub>4</sub>, H<sub>2</sub>, or CO, rendering the reduction process rather complex.

Balasubramaniam *et al.* [77] developed a model that elucidates the chemical transformation process by which the lunar regolith generates CO in a carbon thermal reactor, ultimately producing oxygen. Initially, methane is thermally decomposed upon contact with the hot, molten regolith surface. The resulting solid carbon deposits onto the melt and becomes uniformly mixed with it. Subsequent reactions occur between the carbon and the metal oxides in the melt, yielding CO, which then escapes the melt as gas. Finally, CO is processed to generate oxygen. ORBITEC, an industry partner of the RESOLVE project, is developing a small-scale reactor [78] that employs the carbon thermal reduction process to ex-

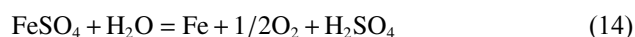
tract oxygen from lunar regolith. This process involves using a carbon source such as methane gas to reduce titanium iron ore and silicates present in lunar regolith at high temperatures (approximately 1625°C).

The advantages of this method include its fast reaction rate, mature experimental equipment development, and the ability to recycle hydrogen and CH<sub>4</sub>. Additionally, the process allows for direct use of molten regolith in carbon thermal reduction, eliminating the need for ore beneficiation and ensuring substantial oxygen production. However, the method has its drawbacks. First, the reaction of the molten regolith requires high temperatures, resulting in significant energy consumption. Second, the high temperatures and the corrosive nature of the molten salt can severely corrode the reactor lining, thereby posing relatively high risks. Furthermore, although the regolith contains carbon introduced by solar winds, the quantity is insufficient to offset the consumption of carbon-based reducing agents and still need to be resupply from Earth.

### 3.2.3. Hydrometallurgy

Hydrometallurgy involves the chemical extraction of metals and their compounds from ore materials using aqueous solutions of acidic (H<sub>2</sub>SO<sub>4</sub>, HF) or alkaline (NaOH) mediums.

Sullivan *et al.* [79] first proposed the use of sulfuric acid to process raw materials rich in titanium iron ore, wherein the raw materials react with sulfuric acid to produce ferrous sulfate, titanium dioxide, and water. Subsequently, the ferrous sulfate dissolves in water and undergoes electrolysis to generate iron and oxygen while also recovering sulfuric acid. The chemical reactions involved in the H<sub>2</sub>SO<sub>4</sub> leaching process are illustrated by Eqs. (13) and (14):



Leaching lunar soil with hydrofluoric acid and NaOH entails several complex chemical separation processes, which pose significant challenges for implementation on the lunar surface, making it relatively under-researched. Hydrometallurgical techniques can dissolve lunar soil into a solution, facilitating the extraction of individual compounds through leaching, followed by metal extraction. The drawback of this method lies in its highly complex reaction processes. For raw materials with low titanium iron ore content, the oxygen yield is low, and sulfuric acid can cause severe corrosion to the equipment used for the process. Additionally, the necessity of replenishing raw materials for leaching from Earth compromises the feasibility of this method.

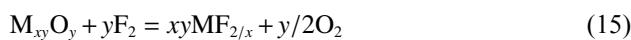
### 3.3. One-step oxygen production method using lunar regolith

The one-step oxygen production directly converts lunar regolith into oxygen through one process. This approach simplifies the oxygen production process by eliminating the need to first extract water and then perform electrolysis. Through direct reaction, oxygen can be efficiently extracted from lun-

ar regolith, saving time and resources. The development and application of this one-step oxygen production from lunar regolith hold promise for providing a sustainable oxygen supply for future space exploration and lunar base construction.

### 3.3.1. Fluorination/chlorination process

Fluorine gas ( $F_2$ ) is the most reactive element and a potent oxidizing agent, surpassing oxygen in reactivity. Therefore, in the reaction of minerals or oxides with fluorine gas, all oxygen atoms are replaced by fluorine atoms, and the oxygen is released in the form of  $O_2$ . The primary products of this fluorination reaction include fluoride salts, as well as gaseous fluorides such as  $SiF_4$ . The reaction of metal oxides  $M_{xy}O_y$  (M stands for metal) with fluorine gas is illustrated by Eq. (15) [80]:



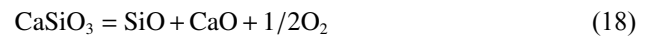
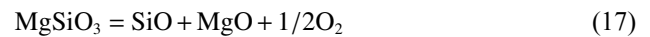
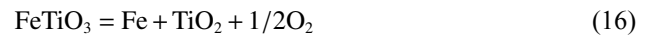
Burt [81–82] and Landis [83] conducted fluorination experiments using plagioclase ( $CaAl_2Si_2O_8$ ) and ilmenite ( $FeTiO_3$ ) as reactants and combined with the electrolysis method to demonstrate the feasibility and cyclic capability of this approach. The advantage of the fluorination process lies in the direct use of fluorine gas to displace oxygen from oxides without involving unnecessary steps, allowing for processing raw materials without the need for enrichment and beneficiation. Through fluorination, elements within these minerals can be separated and obtained as silicon and various metals while simultaneously generating oxygen.

However, the shortcomings of this process are evident. The entire fluorination and fluorine recycling process requires 6–8 different reactors, complicating the operational aspect, and each reactor must be resilient to fluorine corrosion [83]. Furthermore,  $F_2$  cannot be recycled, necessitating the transportation of fluorine from Earth in the form of stable fluoride salts, such as  $KF$ ,  $NaF$ , or  $CaF_2$ . Additionally, the electrolysis process requires anode materials resistant to high-temperature fluoride, while fluorine loss must be compensated by terrestrial supply owing to partial reactions of metal fluorides with corresponding metals or metal oxides [84]. Moreover,  $F_2$  is highly toxic, and the fluorination process generates a significant amount of intermediate products, which are detrimental to the extraction and utilization of oxygen and other desired products. In addition to the fluorination process, cold plasma  $Cl_2$  can reduce titanium ore to metal chlorides and oxygen. Subsequently, metal chlorides are processed through molten salt electrolysis to obtain metal and  $Cl_2$ , thereby achieving  $Cl_2$  recycling. Factors such as temperature, chlorine gas partial pressure, and cold plasma density all affect the rate of the cold plasma decomposition reaction process.

### 3.3.2. High-temperature decomposition method

The high-temperature decomposition method can be divided into vacuum thermal decomposition and thermal plasma decomposition based on the reaction temperature. When using the vacuum thermal decomposition method to process lunar regolith simulants, a high temperature of 2000–3000°C is required, which is achieved through solar

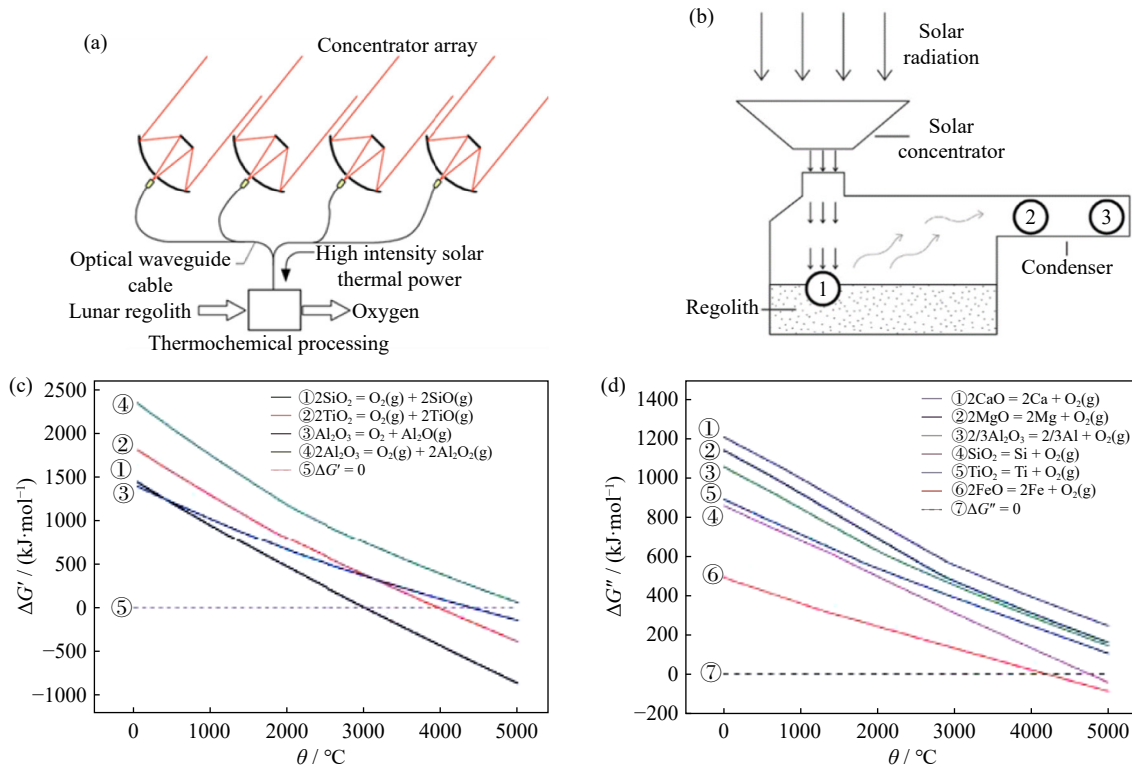
energy focusing. The high vacuum environment of the Moon happens to meet the conditions required for the extraction of metals and oxygen using the vacuum thermal decomposition method. At high temperatures, oxides undergo stepwise decomposition, as depicted in Eqs. (16)–(18):



Senior [85] utilized this method to process lunar regolith simulants for the extraction of metals and oxygen. They investigated the thermodynamic behavior of major oxides in the gas-phase pyrolysis weathering layer. By rapidly condensing the gas-phase products, they managed to separate oxygen and low-valent metastable metal oxides, along with a minimal amount of metal. Owing to the incomplete thermal decomposition, only a minimal amount of metal was obtained. Shaw *et al.* [86] proposed a theoretical approach for the selective thermal decomposition of oxides in lunar regolith to produce sodium and potassium metals under lunar environmental conditions using concentrated solar energy (The theoretical model of a solar concentrator is visualized in Fig. 9(a) [59]). They also successfully simulated the process route for producing sodium, potassium, and FeO from lunar regolith using the FactSage software package, and they considered vacuum thermal dissociation as one of the most promising pathways for metal reduction [87]. The basic concept of the selective thermal decomposition reactor is illustrated in Fig. 9(b). Shi *et al.* [88] calculated the Gibbs free energy change for oxide decomposition in lunar regolith into low-valent oxides and complete decomposition into metals and oxygen (Fig. 9(c) and (d)). Thermodynamic calculations indicated that the temperature required for  $SiO_2$ ,  $TiO_2$ , and  $Al_2O_3$  decomposition into oxygen and low-valent oxides at a standard atmospheric pressure is very high. Among  $SiO_2$ ,  $TiO_2$ ,  $Al_2O_3$ ,  $FeO$ ,  $CaO$ , and  $MgO$ ,  $FeO$  requires the lowest temperature for complete decomposition into metals and oxygen.

The advantage of this method lies in its relatively simple process and direct utilization of solar energy. However, it suffers from high energy consumption and low decomposition efficiency. Additionally, this method requires that oxide materials be finely ground and sieved, as only small particle materials can undergo thermal decomposition. However, the difficulty in achieving fine grinding of materials on the lunar surface makes it extremely challenging to employ this technique for metal and oxygen preparation.

The low decomposition efficiency of lunar regolith in vacuum thermal decomposition is attributed to the low reaction temperature. Increasing the temperature and using thermal plasma decomposition could enhance the efficiency of metal and oxygen production from lunar regolith. Thermal plasma decomposition operates at temperatures ranging from 6700 to 9700°C, leveraging the fact that metals can be more easily extracted than oxygen in an electrostatic or magnetic field. This leads to the instantaneous ionization of oxides and the



**Fig. 9.** (a) Solar concentrator. Reprinted from Ref. [59], with permission from American Society of Civil Engineers. (b) Basic reactor concept [86]. Reprinted from *Planet. Space Sci.*, 204, M.G. Shaw, G.A. Brooks, M.A. Rhamdhani, A.R. Duffy, and M.I. Pownceby, Thermodynamic modelling of ultra-high vacuum thermal decomposition for lunar resource processing, art. No. 105272, Copyright 2021, with permission from Elsevier. (c) Gibbs free energy changes in some oxides decomposed into suboxides and oxygen and (d) Gibbs free energy changes in some oxides completely decomposed into metals and oxygen. Reprinted from [88] and with permission.

separation of metals and oxygen. When the temperature rises from 7700 to 9700 $^{\circ}\text{C}$ , the conversion of lunar regolith into oxygen increases by 10%, resulting in a significant improvement in oxygen yield. At 7700 $^{\circ}\text{C}$ , over 90% of the metal undergoes ionization, while only 1% of oxygen is ionized, facilitating the separation of oxygen and metals [89]. Ionized metals can be captured by electrostatic or electric field trapping, while non-ionized  $\text{O}_2$  is collected from the other end.

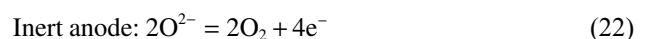
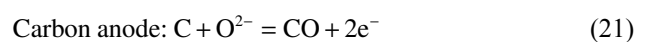
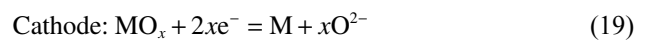
An advantage of this method is that it eliminates the need for screening lunar regolith. Theoretically, this process only necessitates abundant resources on the Moon, such as sunlight, vacuum, and lunar weathering layers. Moreover, the oxygen production efficiency is relatively high, with a theoretical oxygen production rate of over 24%. However, a significant drawback of this method is the excessively high operating temperatures, resulting in high energy consumption. Producing one ton of oxygen requires an energy input of 34500 kWh [90]. Furthermore, high temperatures impose extremely stringent requirements on equipment, as conventional container materials struggle to withstand temperatures exceeding 7000 $^{\circ}\text{C}$ . Additionally, the reduction products are generated in the gaseous phase, making them highly susceptible to reverse reactions. Rapid cooling is necessary to prevent these reverse reactions, and oxygen must be continuously removed from the process to maintain the vacuum.

### 3.3.3. Molten salt electrolysis

The molten  $\text{CaCl}_2$  is considered an important electrolyte

for novel electrochemical metallurgical processes owing to its high oxygen ion solubility. Several new processes based on the electrolysis of oxide molten salts have been proposed, with the Fray–Farthing–Chen (FFC)–Cambridge process and the One–Suzuki (OS) process being typical examples [91–94].

In the FFC–Cambridge process, lunar regolith pellets, sintered as cathodes, undergo ionization of their oxygen atoms, which are transported to the graphite anode to be oxidized, forming  $\text{CO}$  or  $\text{CO}_2$ , while  $\text{O}_2$  is formed on the inert anode (Fig. 10(a) [95]). The cathode gradually undergoes reduction, ultimately yielding pure metal or alloys, as depicted in Eqs. (19)–(22):



Lomax *et al.* [95], using simulated lunar regolith as raw material, employed the FFC process to electrolyze it for 50 h, extracting 96% of the oxygen from the weathered layer and obtaining a metal product consisting of three separated alloys: Al–Fe, Fe–Si, and Ca–Si–Al. Schwandt *et al.* [96] electrolyzed  $\text{FeTiO}_3$  in a  $\text{CaCl}_2$ – $\text{CaO}$  molten salt at 900 $^{\circ}\text{C}$  using an inert anode, resulting in the production of Fe–Ti alloy and

oxygen. In the OS process, a porous metal filled with granular oxides serves as the cathode. On the cathode surface,  $\text{Ca}^{2+}$  is reduced to Ca metal, which subsequently reacts with the metal oxide within the cathode to produce the corresponding metal and CaO. CaO exists in the form of  $\text{Ca}^{2+}$  and  $\text{O}^{2-}$  ions in  $\text{CaCl}_2$ , with the latter being transported to the anode and oxidized, ultimately releasing oxygen. To ensure good conductivity of both calcium and oxygen ions, CaO is added to  $\text{CaCl}_2$  in the application of both the FFC and OS processes [97].

In addition to the FFC and OS processes, lunar regolith can be dissolved in molten salt media (such as fluoride molten salts) for electrolysis, with oxygen evolving at the anode and the deposition of metal or alloys at the cathode. Kvande and Haupin [98] electrolyzed lunar regolith dissolved in cryolite molten salt, obtaining an Al–Si alloy at the cathode and  $\text{O}_2$  at the inert anode, with continuous enrichment of CaO in the electrolyte. The CaO-enriched molten salt was further electrolyzed in LiF– $\text{CaF}_2$  molten salt, ultimately yielding metallic calcium. The electrolyte in molten salt electrolysis should possess high ionic conductivity and electrical conductivity, as well as a low melting point, viscosity, and vapor pressure. It should also not react with the electrodes or the metals produced [99].

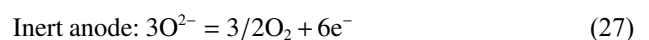
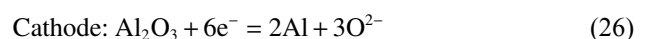
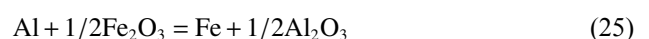
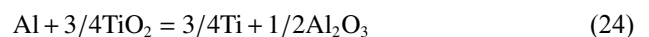
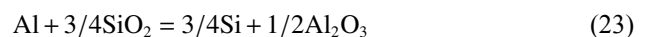
The primary advantage of molten salt electrolysis lies in the substantial reduction in electrolysis temperature and energy consumption, as well as decreased requirements for electrolytic cells and electrode materials [100]. Furthermore, it allows for the direct extraction of oxygen and metals on the electrode surface, utilizing raw materials such as lunar regolith without needing extensive ore selection. However, the drawbacks of molten salt electrolysis include the high vapor pressure of the electrolyte at operating temperatures, necessitating the recovery or replenishment of evaporated salts from Earth. Additionally, in the FFC method, the pressing of oxides into shapes prior to electrolysis results in poor electrode conductivity, and the selection of inert anodes poses a major challenge for molten salt electrolysis.

Research on inert anodes for molten salt electrolysis is ongoing, with numerous materials being tested. One such material is doped tin oxide ( $\text{SnO}_2$ ). While ordinary tin oxide is an insulating and brittle material, doping it with approximately 2wt% antimony oxide ( $\text{Sb}_2\text{O}_3$ ) and 1wt% copper oxide ( $\text{CuO}$ ) improves its conductivity and mechanical strength [101]. In a melt composed of calcium chloride and dissolved calcium oxide, doped tin oxide successfully evolved oxygen as the anode [101–102], with the mass changes and optical images of the graphite and  $\text{SnO}_2$ -based anodes after electrolysis depicted in Fig. 10(b) and (c). However, owing to the erosion of the molten salt, the doped tin oxide was completely corroded within a few hours, and its conductivity gradually decreased owing to the slow formation of calcium stannate ( $\text{CaSnO}_3$ ) insulating surface layers. Another promising material is the solid solution of calcium titanate and ruthenium oxide ( $\text{CaTi}_{1-x}\text{Ru}_x\text{O}_3$ ) [103–104]. Depending on the proportion of ruthenium and titanium, the conductivity of

this material varies from metallic to semiconducting, with the resistivity changes in composite particles composed of different  $\text{CaRuO}_3$  components depicted in Fig. 10(d). This material has sustained over 100 h of electrolysis in a calcium chloride melt. Jiao and his co-workers [105] developed a  $\text{TiO}_2$ - $\text{RuO}_2$  inert anode, processing simulated lunar regolith to prepare the cathode. Using this inert anode instead of a graphite anode, they successfully electrolyzed the metal oxides in the simulated lunar regolith to produce oxygen and metal alloys, as depicted in Fig. 10(e). The photographs and scanning electron microscopy images of the anode before and after 24 h of electrolysis are shown in Fig. 10(f), with almost no observed changes in the anode shape, indicating its stability despite the continuous evolution of oxygen on the anode surface.

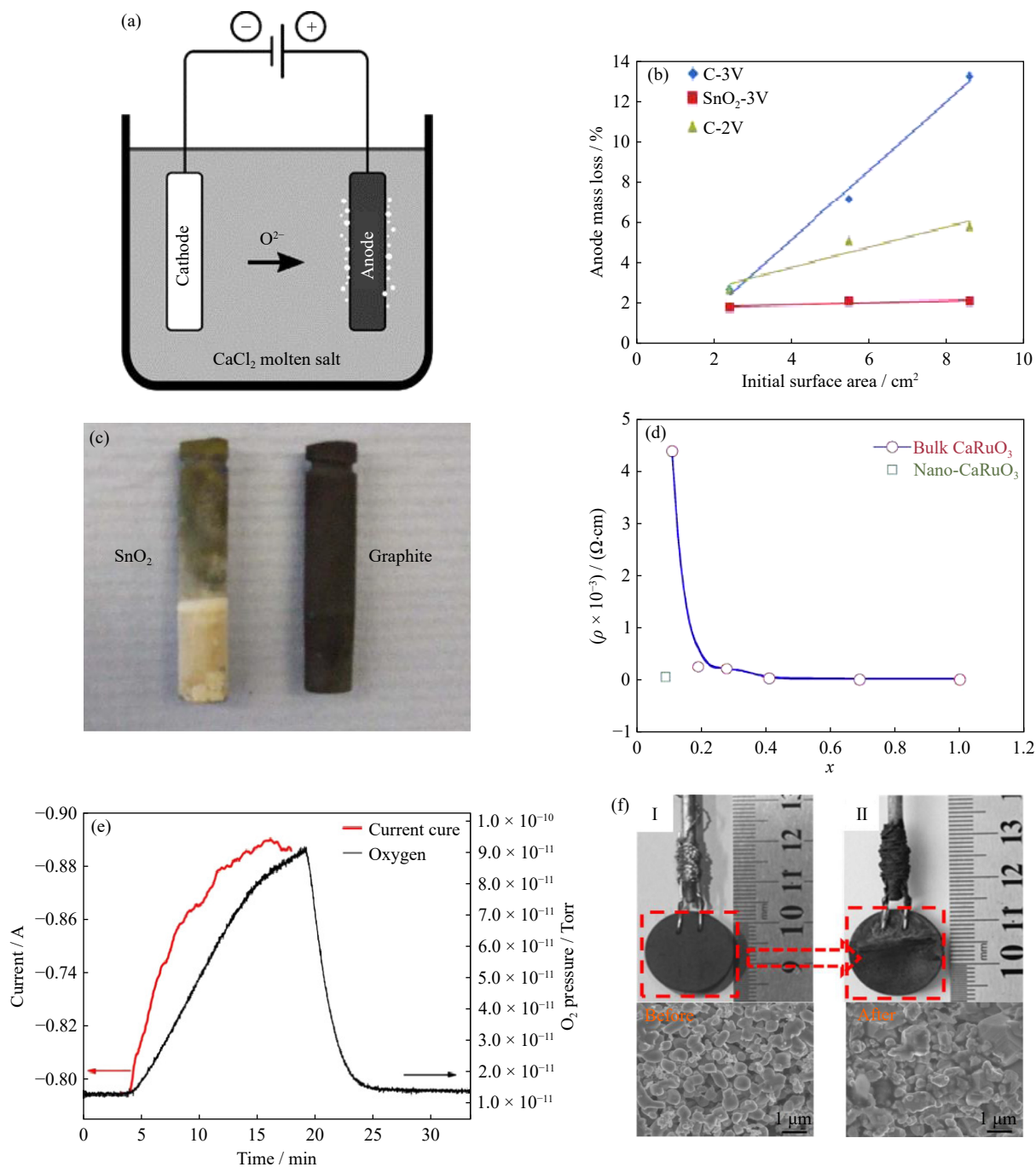
### 3.3.4. Molten salt electrolysis–metal thermal reduction combined method

The metal thermal reduction method is a process for preparing metals by using active metals to reduce less active metal compounds. The combined method of molten salt electrolysis and metal thermal reduction involves the integration of two metallurgical methods. Initially, lunar regolith simulant is dissolved in a cryolite molten salt system, and aluminum is used as a reducing agent [106] to thermally reduce the lunar regolith simulant to an Al–Si–Ti–Fe alloy, and aluminum oxide-enriched cryolite molten salt. Subsequently, inert anode electrolysis enriches the aluminum oxide in the cryolite molten salt, with oxygen evolving at the anode and metallic aluminum obtained at the cathode, thus achieving the recyclable use of aluminum, as depicted in Eqs. (23)–(27):



The metal thermal reduction method was first used to reduce lunar regolith simulant by Semkow and Sarmell [107], who used Li and Na to reduce  $\text{FeO}$ ,  $\text{TiO}_2$ , and  $\text{SiO}_2$  in the lunar regolith simulant to Fe, Ti, and Si while also obtaining  $\text{Li}_2\text{O}$  and  $\text{Na}_2\text{O}$ . However,  $\text{Al}_2\text{O}_3$ , CaO, and  $\text{MgO}$  were not reduced. Subsequently,  $\text{Li}_2\text{O}$  was added to a LiF–LiCl molten salt, and inert anode electrolysis was performed, resulting in the evolution of oxygen at the anode and the production of metallic Li at the cathode, thus achieving the recyclable use of lithium. The main challenge of this method lies in separating  $\text{Li}_2\text{O}$  from the unreduced mixed oxides.

In a cryolite molten salt medium, Shi *et al.* [108] used aluminum thermal reduction to prepare an aluminum–titanium–iron alloy from lunar regolith simulant ilmenite, investigating the effects of the amount of reducing agent and reduction time on the alloy product. Subsequently, inert anode electrolysis with an Fe–Ni base was conducted, resulting



**Fig. 10.** (a) Basic concept of the FFC-Cambridge process [95]. Reprinted from *Planet. Space Sci.*, 180, B.A. Lomax, M. Conti, N. Khan, N.S. Bennett, A.Y. Ganin, and M.D. Symes, Proving the viability of an electrochemical process for the simultaneous extraction of oxygen and production of metal alloys from lunar regolith, art. No. 104748, Copyright 2020, with permission from Elsevier. (b) Mass change of the graphite and  $\text{SnO}_2$ -based anodes after FFC-Cambridge electrolysis [100]. Reprinted by permission from Springer Nature: *Int. J. Miner. Metall. Mater.*, Applications of molten salt and progress of molten salt electrolysis in secondary metal resource recovery, X.L. Xi, M. Feng, L.W. Zhang, and Z.R. Nie, Copyright 2020. (c) Anodes after use [102]. Reprinted by permission from Springer Nature: *Metall. Mater. Trans. B*, Reduction of tantalum pentoxide using graphite and tin-oxide-based anodes via the FFC-Cambridge process, R. Barnett, K.T. Kilby, and D.J. Fray, Copyright 2009. (d) The change in electrical resistivity for composite pellets consisting of varying amounts of  $\text{CaRuO}_3$  [103]. Reprinted from *Mater. Res. Bull.*, 44, S.Q. Jiao, K.N.P. Kumar, K.T. Kilby, and D.J. Fray, Preparation and electrical properties of  $x\text{CaRuO}_3/(1-x)\text{CaTiO}_3$  perovskite composites, 1738-1742, Copyright 2009, with permission from Elsevier. (e) Current/oxygen-time profile for the electrolytic reduction of  $\text{TiO}_2$  using a  $\text{TiO}_2 \cdot \text{RuO}_2$  anode in a  $\text{CaCl}_2$ - $\text{CaO}$  melt and (f) photos and field emission scanning electron microscopy (FESEM) images of the  $\text{TiO}_2 \cdot \text{RuO}_2$  anode before (I) and after 24 h (II) electrolysis. Reprinted from [105].

in oxygen gas with a volume fraction of 98.87% in the gas product.

Xie *et al.* [109] added lunar regolith simulant MLS-1 and

metallic aluminum to a molten salt composed of 51.22wt%  $\text{NaF}$  and 48.78wt%  $\text{AlF}_3$ . After 4 h of aluminum thermal reduction at  $980^\circ\text{C}$ , the mass fractions of Al, Si, Fe, and Ti in

the obtained alloy were 79.71%, 12.03%, 5.91%, and 2.35%, respectively. Subsequently, electrolysis of the alumina-containing molten salt at 960°C with an Fe–Ni inert anode produced oxygen gas with a volume fraction greater than 99% in the gas product, achieving a current efficiency of 84.94%, and the cathode obtained an Al–Si–Fe–Ti–Ni alloy.

Liu *et al.* [110–111] performed molten salt electrolysis–aluminum thermal reduction of fly ash in a molten salt composed of 52.7wt% NaF and 47.3wt% AlF<sub>3</sub> at 960°C, obtaining an Al–Si–Ti–Fe alloy. The process flowchart for metal and oxygen production from lunar soil using inert anode electrolysis–aluminum thermal reduction is shown in Fig. 11(a). The procedure was followed by inert anode electrolysis, which produced oxygen gas with a volume fraction of 96.83% in the gas product. The reaction mechanism of aluminum thermal reduction of mixed oxides in cryolite molten salt and the experimental set-up for inert anode electrolysis are depicted in Fig. 11(b) and (c). When lunar regolith simulant NEU-1 was electrolyzed with an Fe–Ni inert anode, pure oxygen was evolved from the inert anode surface (Fig. 11(d)), and the cathode yielded an Al–Si alloy [112].

### 3.3.5. Molten regolith electrolysis

Molten regolith electrolysis is the most direct method for obtaining oxygen and metals from molten lunar regolith. It is

derived from the MOE process, which involves directly electrolyzing molten metal oxides to obtain metals and oxygen [113]. The principle involves heating the lunar soil to a molten state, where anions move toward the anode, and cations tend to move toward the cathode within the molten regolith. Through electrolysis, oxygen ions are discharged at the interface of the inert anode, resulting in the production of oxygen, while metals are deposited at the cathode, as depicted in Eqs. (28) and (29):



Temperature and electric potential are the primary factors in the decomposition of metal oxides (Fig. 12(a)) [114]. In theory, all metal oxides can be reduced using molten oxide electrolysis. However, beyond a certain degree of reduction, a very viscous spinel, primarily composed of MgAl<sub>2</sub>O<sub>4</sub>, is formed. This increases both the viscosity and liquidus point of the remaining melt, thereby preventing the extraction of all oxygen in practice [113]. At the anode, a reversible iron oxidation–reduction reaction may occur, where Fe<sup>2+</sup> is oxidized to Fe<sup>3+</sup> at the anode and then reduced again at the cathode, limiting the current efficiency of oxygen extraction. The oxygen obtained from the electrolysis of simulated molten regolith

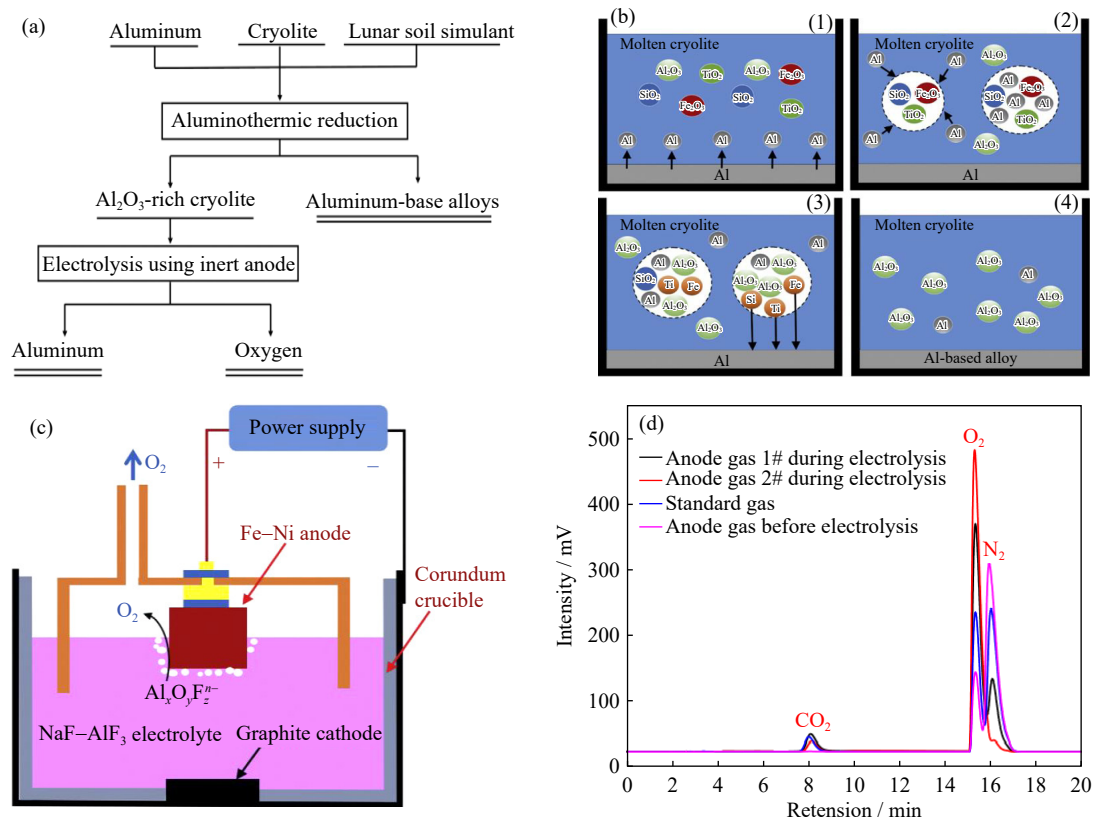


Fig. 11. (a) Flowchart of the metal and oxygen preparation from lunar soil through molten salt electrolysis using an inert anode and aluminothermic reduction, (b) graphical description of the aluminothermic reduction of mixed oxides in cryolite molten salts, and (c) schematic diagrams of the experimental apparatus for molten salt electrolysis [111]. Reprinted from *J. Alloys Compd.*, 718, A.M. Liu, Z.N. Shi, X.W. Hu, *et al.*, Production of metals and oxygen from coal fly ash by aluminothermic and electrochemical reduction process, 279–287, Copyright 2017, with permission from Elsevier. (d) Gas chromatograms recorded from the standard gas and outlet anode gas before and during electrolysis. Reprinted from A.M. Liu, Z.N. Shi, X.W. Hu, B.L. Gao, and Z.W. Wang, *J. Electrochem. Soc.*, vol. 164, H126–H133 (2017) [112]. © IOP Publishing. Reproduced with permission. All rights reserved.

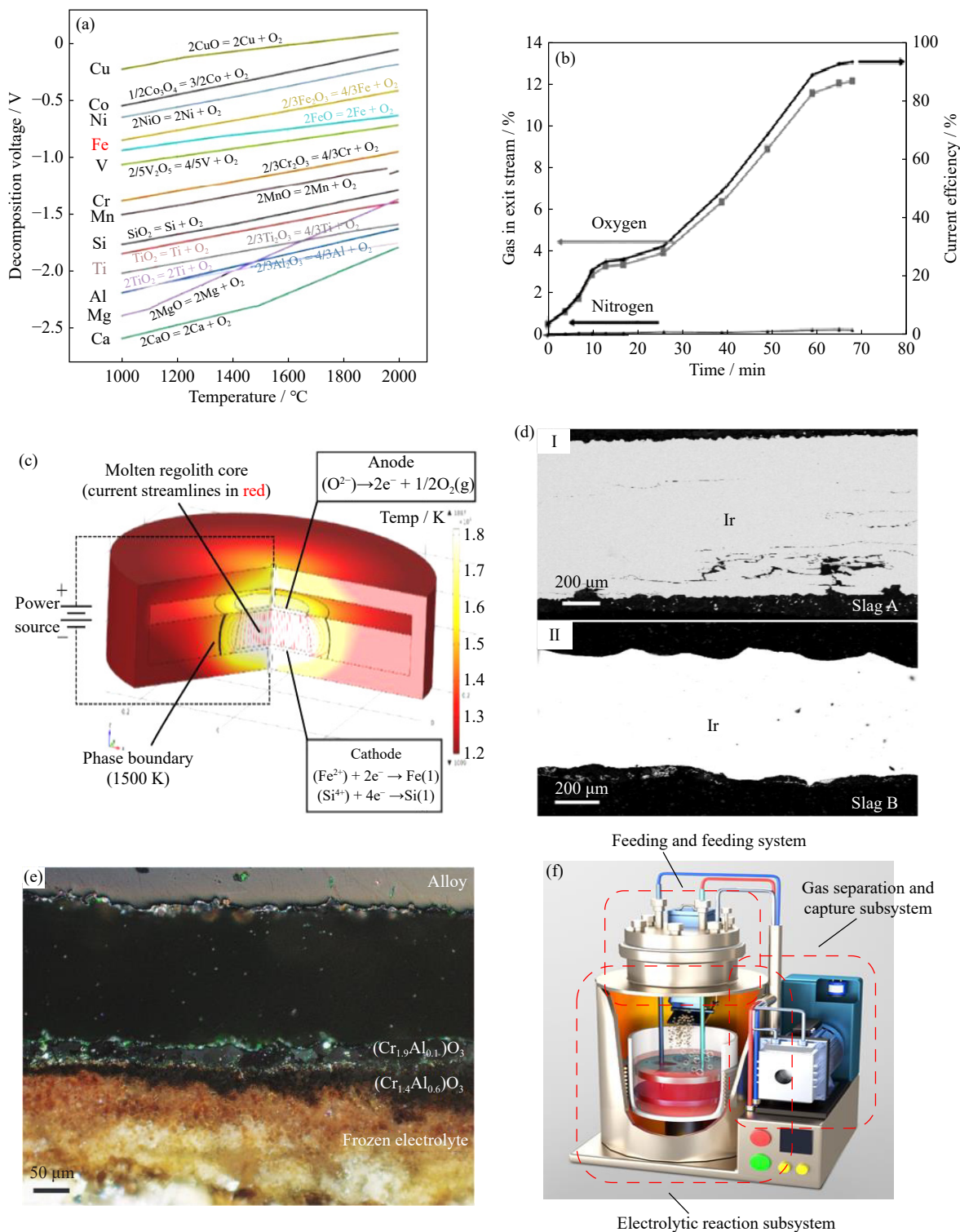


Fig. 12. (a) Decomposition voltages of various metal oxides. M.Y. Wang, H.D. Jiao, Z.H. Pu, et al., *Angew. Chem. Int. Ed.*, vol. 61, art. No. e202206482 (2022) [114]. Copyright Wiley-VCH Verlag GmbH & Co. KGaA. Reproduced with permission. (b) Oxygen obtained from simulated lunar regolith through molten regolith electrolysis. Reprinted from A.H. Sirk, D.R. Sadoway, and L. Sibille, *ECS Trans.*, vol. 28, 367-373 (2010) [115]. © IOP Publishing. Reproduced with permission. All rights reserved. (c) Schematic of a molten regolith electrolysis reactor that produces oxygen gas at the anode and molten metals at the cathode [117]. From Development of a molten regolith electrolysis reactor model for lunar *in-situ* resource utilization, S. Schreiner, L. Sibille, J. Dominguez, A. Sirk, J. Hoffman, and G. Sanders; reprinted by permission of the American Institute of Aeronautics and Astronautics. (d) SEM of Ir anode after electrolysis in (I) an acidic slag and (II) an alkaline slag. Reprinted from H. Kim, J.D. Paramore, A. Allanore, and D.R. Sadoway, *ECS Trans.*, vol. 33, 219-230 (2010) [119]. © IOP Publishing. Reproduced with permission. All rights reserved. (e) Optical micrograph of the anode/electrolyte interface with phases identified by EDS and XRD [122]. Reprinted by permission from Springer Nature: *Nature*, A new anode material for oxygen evolution in molten oxide electrolysis, A. Allanore, L. Yin, and D.R. Sadoway, Copyright 2013. (f) Schematic diagram of the lunar regolith melting electrolysis oxygen production payload.

lith is shown in Fig. 12(b) [115]. Sibille *et al.* [116] achieved electrolysis with 94% current efficiency at 1600°C, extracting 35 g of oxygen from 100 g of regolith simulant.

Molten regolith is chemically corrosive; to protect the reactor wall from corrosion, only the core of the regolith is melted by the Joule heat of electrolysis, with the outer layer remaining solid and in contact with the reactor wall. A schematic diagram of the molten regolith electrolysis reactor is shown in Fig. 12(c). The Joule heat generated by the molten regolith itself is sufficient to maintain the temperature of the electrolysis process above the liquidus line of the regolith. Additionally, the electrodes must have good corrosion resistance, stability at high temperatures, high mechanical strength, and good resistance to heat and electrical shock. Therefore, the electrodes can be composed of precious metals such as iridium, molybdenum, or platinum [117].

The temperature of molten regolith electrolysis reaches 1550°C, resulting in a very short lifespan for the anode material [118]. Kim *et al.* [119] and Wang *et al.* [120] explored the corrosion resistance of iridium anodes based on the melt composition, finding that a higher silica content in the melt reduces the corrosion rate of iridium (Fig. 12(d)). They also found that under a constant current of 3.5 A in high-calcium slag, the voltage stabilized at around 2 V, while in high-silica slag, the voltage stabilized at around 4 V [121]. Sadoway and his coworkers [122] proposed a new chromium-based alloy,  $\text{Cr}_{1-x}\text{Fe}_x$  ( $0 < x < 0.3$ ), which exhibited good inertness in the Molten oxide electrolysis (MOE) process, allowing for the production of oxygen at the anode while simultaneously extracting the metal. After electrolysis, a 100  $\mu\text{m}$ -thick oxide layer forms on the anode surface, consisting of a lower Al content solid solution ( $\text{Cr}_{1.9}\text{Al}_{0.1}\text{O}_3$ ) in the inner layer and a higher Al content solid solution ( $\text{Cr}_{1.4}\text{Al}_{0.6}\text{O}_3$ ) in the outer layer (Fig. 12(e)). Its outstanding performance makes it a promising anode material for molten regolith electrolysis. Therefore, there is an urgent need to establish an oxygen production facility adapted to the lunar surface environment during the lunar landing phase. This involves examining the technical parameters of the core reactor, scaling effects, service stability, and oxygen life quality to complete the modular and integrated design of the oxygen generator. During the exploration phase, a scaled oxygen production unit and integrated system should be established to ensure the supply of high-quality oxygen. A schematic diagram of the lunar regolith molten electrolysis oxygen production payload is shown in Fig. 12(f).

The advantage of the MOE method lies in its ability to utilize any weathered layer as a raw material for molten oxide electrolysis without the need for beneficiation and without requiring additional reagents, thus maximizing IS-RU. Furthermore, by controlling the potential, it is possible to selectively obtain desired metals based on the stability of metal oxides. Additionally, the abundant solar energy on the Moon provides the necessary energy for the electrolysis process. However, the drawbacks of the MOE method include the high operating temperatures, as well as the strong corrosiveness of silicate melts at these high temperatures, making it difficult to remove the spinel generated during the electrolysis process from the apparatus. Therefore, addressing the long-term stability of inert anodes and available reactors on the Moon still needs attention.

iveness of silicate melts at these high temperatures, making it difficult to remove the spinel generated during the electrolysis process from the apparatus. Therefore, addressing the long-term stability of inert anodes and available reactors on the Moon still needs attention.

### 3.4. Method comparison

To date, there are eight main methods for *in-situ* oxygen production from lunar resources, including hydrogen reduction, carbothermal reduction, hydrometallurgy, water-ice electrolysis, molten regolith electrolysis, molten salt electrolysis, high-temperature decomposition, and fluorination/chlorination. In the context of both domestic and international development of oxygen production technologies, the evaluation of oxygen production on lunar regolith should focus on the sources of raw materials (including the availability of oxygen-containing materials and oxygen production reagents on the Moon), energy sources, oxygen production efficiency, equipment feasibility, technological (difficulty) feasibility, and economic feasibility (including transportation costs). These four aspects will be comprehensively assessed, with a total score of 100 points allocated, allowing a maximum of 25 points for each unilateral evaluation, as detailed in Fig. 13 and Tables 2 and 3.

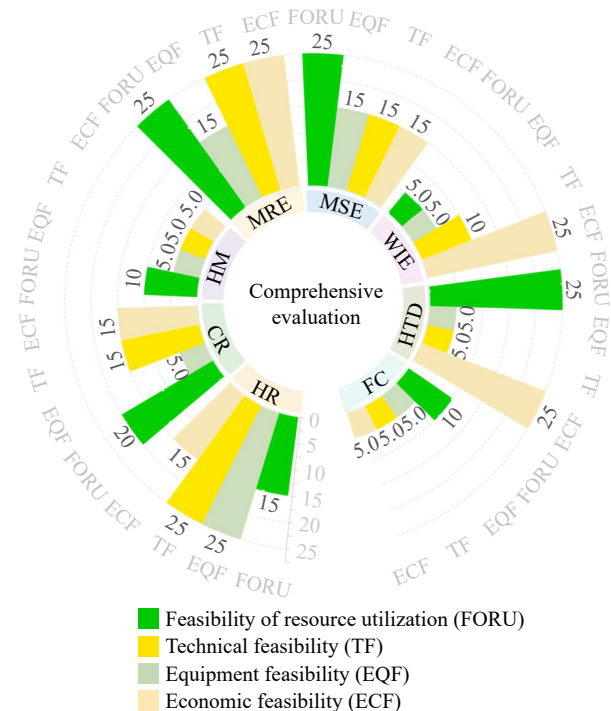


Fig. 13. Comprehensive evaluation chart of *in-situ* oxygen production methods for lunar resources (HR—Hydrogen reduction; CR—Carbothermal reduction; HM—Hydrometallurgy; MRE—Molten regolith electrolysis; MSE—Molten salt electrolysis; WIE—Water ice electrolysis; HTD—High-temperature decomposition; FC—Fluorination/chlorination).

## 4. Conclusion and outlook

Oxygen is a crucial resource for future human deep space exploration. To achieve sustainable lunar missions, numer-

**Table 2. Comprehensive evaluation of lunar resources oxygen production technology**

Oxygen production methods	Applicable minerals	Reaction temperature / °C	Engineering oxygen production efficiency	Feasibility of resource utilization	Equipment feasibility	Technical feasibility	Economic feasibility	Comprehensive evaluation
Molten regolith electrolysis	SiO <sub>2</sub> ; FeTiO <sub>3</sub> ; FeO (Similar to the composition of basalt)	Approximately 1600	About 20%–30%	25	15	25	25	90
Hydrogen reduction	FeTiO <sub>3</sub> (Not widely distributed)	Approximately 900	<5%	15	25	25	15	80
Molten salt electrolysis	SiO <sub>2</sub> ; FeTiO <sub>3</sub> ; FeO	950	39%–43%	25	15	15	15	70
High-temperature decomposition	SiO <sub>2</sub> ; FeTiO <sub>3</sub> ; FeO; Al <sub>2</sub> O <sub>3</sub> ; MgO; CaO; CaAl <sub>2</sub> Si <sub>2</sub> O <sub>8</sub>	>2000	About 20%	25	5	5	25	60
Carbothermal reduction	FeTiO <sub>3</sub> ; Fe <sub>2</sub> SiO <sub>4</sub> ; FeSiO <sub>3</sub> ; Mg <sub>2</sub> SiO <sub>4</sub> ; MgSiO <sub>3</sub> ; CaSiO <sub>3</sub>	>1600	About 10%	20	5	15	15	55
Water ice electrolysis	Water ice	Ordinary temperature	86%	5	5	10	25	45
Fluorination/chlorination	FeTiO <sub>3</sub>	846	—	10	5	5	5	25
Hydrometallurgy	FeTiO <sub>3</sub> ; TiOSO <sub>4</sub> ; FeSO <sub>4</sub>	—	—	10	5	5	5	25

ous processes have been developed to extract this valuable resource on site. This paper provides a comprehensive review of the current status and examines the advantages and disadvantages of these processes. We believe that the molten regolith electrolysis method holds significant technical advantages over other methods. The benefits of this method can be summarized in four key aspects. First, it exhibits strong resource adaptability, as lunar regolith is abundant on the Moon's surface and high in oxygen content, making it suitable for *in-situ* oxygen production at any lunar site. Second, it benefits from ample energy sources, with the lunar surface receiving abundant solar energy, providing direct power for regolith electrolysis oxygen production. Third, it demonstrates good technical and economic feasibility, with low reliance on Earth, thus promoting lunar self-sufficiency in *in-situ* oxygen production. Fourth, metal by-products can be obtained at the cathode while electrolyzing oxygen; they can serve as important raw materials for developmental activities on the Moon. However, issues regarding the long-term stability of inert anodes, available reactors, and effective oxygen separation under low-gravity conditions still need to be addressed on the Moon.

As the exploration of the Moon receives increasing attention, it is likely that new processes will emerge to improve the performance of existing methods. Simultaneously, we should recognize the differences between laboratory conditions and *in-situ* testing on the Moon. In the future, humans will need to overcome a series of challenges, such as high-temperature electrode materials, corrosion-resistant reactors, and the supply of reductants under reduced gravity.

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## Conflict of Interest

The authors declare no potential conflict of interest, and the manuscript is approved by all authors for publication.

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Table 3. Scoring standard of lunar resources oxygen production technology

Oxygen production methods	Feasibility of resource utilization	Equipment feasibility	Technical feasibility	Economic feasibility
Molten regolith electrolysis	***** (Abundant lunar regolith)	*** (The equipment needs to be resistant to high temperature and corrosion; Simple device integration; Oxygen separation is simple)	***** (Long term service inert anode needs to be developed)	***** (High efficiency; Low transportation costs)
Hydrogen reduction	*** (Only ilmenite is easily reducible)	***** (The operating conditions of the equipment are relatively mild; Complex equipment integration; Difficulty in oxygen separation)	***** (Special requirements for mineral composition and particle size; The fluidized bed reaction process is prone to cohesive loss of flow)	** (Low efficiency; High transportation costs)
Molten salt electrolysis	***** (Abundant lunar regolith)	*** (The equipment needs to be corrosion-resistant; Simple device integration; Oxygen separation is simple)	*** (Moon soil requires tedious pre-treatment, including pressing tiles and sintering them into tightly oxidized objects)	*** (High efficiency; High transportation costs)
High-temperature decomposition	***** (Abundant lunar regolith)	* (The equipment needs to be resistant to high temperature and corrosion; Simple device integration; Oxygen separation is simple)	* (High density energy sources are required, and equipment materials are demanding, which cannot be achieved in the short term)	***** (High efficiency; Low transportation costs)
Carbothermal reduction	*** (Abundant lunar regolith, Reducing SiO <sub>2</sub> is relatively difficult)	* (The equipment needs to be resistant to high temperature and corrosion; Complex equipment integration; Difficulty in oxygen separation)	*** (Reactions can easily lead to equipment nodulation; There are significant safety hazards in the transportation of carbon containing raw materials)	*** (Medium efficiency; High transportation costs)
Water ice electrolysis	* (Collecting water ice is exceptionally challenging)	* (Low equipment requirements; Simple device integration; Difficulty in oxygen separation)	** (Existing technology is difficult to achieve water ice mining)	***** (High efficiency; Low transportation costs)
Fluorination/ chlorination	** (Only applicable to ilmenite)	* (The equipment needs to be corrosion-resistant; Complex equipment integration; Oxygen separation is simple)	* (Significant safety hazards during transportation)	* (Low efficiency; High transportation costs)
Hydrometallurgy	** (Only suitable for raw materials with high ilmenite content)	* (The equipment needs to be corrosion-resistant; Complex equipment integration; Oxygen separation is simple)	* (Significant safety hazards during transportation)	* (Low efficiency; High transportation costs)

Note: One \* represents 5 points, with a total score of 100 points. \*\*\*\*\*—Highly; \*\*\*\*—Strong; \*\*\*—Average; \*\*—Poor; \*—Very poor.

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