Review of silicon recovery in the photovoltaic industry

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Abstract

The photovoltaic industry is developing rapidly to support the net-zero energy transition. Among various photovoltaic technologies, silicon-based technology is the most advanced, commanding a staggering 95% market share. However, the energy-intensive process of manufacturing silicon wafer raises concerns. In the photovoltaic supply chain, a substantial amount of photovoltaic secondary silicon-containing resource (PV-SSCR), including metallurgical-grade silicon refined slag (MGSRS), silicon fume (SF), silicon cutting waste (SCW) and end-of-life silicon solar cell (ESSC) from discharged modules, can be recycled. Recycling holds the potential to enhance economic value and reduce the overall environmental impacts associated with the lifecycle of silicon photovoltaics. This article offers a comprehensive overview of techniques and applications of four kinds of PV-SSCR: MGSRS, SF, SCW, and ESSC. Moreover, it also highlights challenges and opportunities for further research and development in this field.

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Introduction

In 2022, the global photovoltaic industry entered the terawatt (TW) era, with a cumulative installed capacity reaching 1,185 GW. Crystal silicon cells accounted for more than 95% of this capacity [1,2].
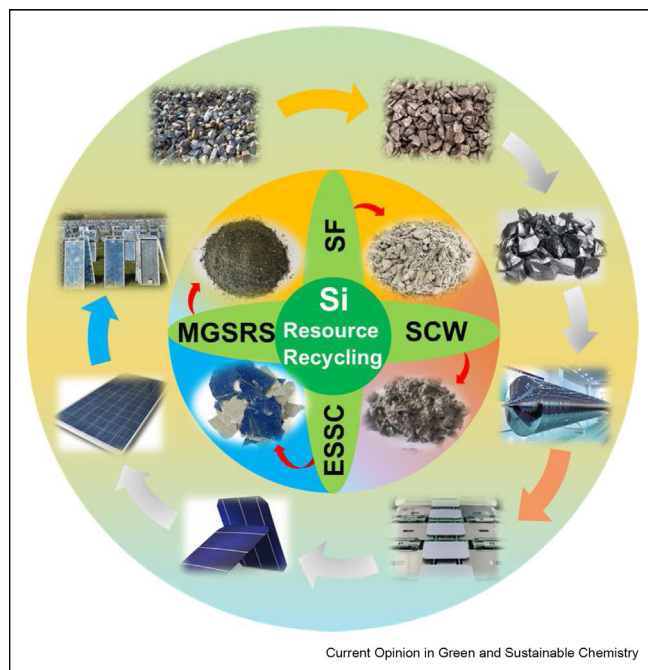
Figure 1 illustrates the value chain of the silicon photovoltaic industry, ranging from industrial silicon through polysilicon, monocrystalline silicon, silicon wafer cutting, solar cell production, and finally photovoltaic (PV) module assembly. The process of silicon production is lengthy and energy consuming, requiring 11−13 million kWh/t from industrial silicon to polycrystalline silicon [3]. A significant amount of photovoltaic secondary silicon-containing resource (PV-SSCR) is generated throughout the lifecycle. Metallurgical-grade silicon refined slag (MGSRS) and silicon fume (SF) are byproducts of industrial silicon production. Silicon cutting waste (SCW) is generated during silicon wafer cutting, and end-of-life silicon solar cell (ESSC). The proportion of silicon-containing solid waste generated in each step is calculated based on 2022 global industrial silicon production of 7.783 million tons, and the results are shown in Table 1.

This article aims to provide a comprehensive review of the advancements in silicon recovery research and development within the photovoltaic industry over the last decade. It synthesizes and examines key concepts, technologies, and challenges associated with PV-SSCR recovery and reuse. Furthermore, the cross-analysis of various silicon recovery methodologies offers crucial insights into addressing a pressing industry challenge: silicon recovery from ESSC.

Metallurgical-grade silicon refined slag (MGSRS)

The blowing refining converts process involves converting impurities into oxides within the silicon melt. The slag that forms on the surface of silicon melt is known as MGSRS. However, due to the high viscosity and poor fluidity of the slag, some silicon gets involved in the MGSRS [10]. MGSRS primarily contains silicon (Si), silicon carbide (SiC), silicon oxide (SiOx), aluminum oxide (Al_2O_3), calcium oxide (CaO), and R(SiO$_3$)n. In the industrial silicon refining process, $\sim$200 kg of industrial silicon slag are produced for every 2 tons of refined silicon. Given the global industrial silicon production of over 3 million tons annually, this results in $\sim$400,000 tons of silicon being produced as slag each year. Regrettably, $\sim$60,000 tons of silicon resources are lost annually without proper recovery [4].

Figure 1

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Flow diagram of silicon industry chain and silicon-containing secondary solid waste resources.

Scholars have utilized SiC in MGSRS as a blowing agent to create porous glass ceramics with an average pore size of 1.2 mm and a compressive strength of 4.6 MPa. MGSRS is used for applications like road paving and low-value products such as cement [11,12]. Despite its uses in glass ceramics and road materials, silicon recovery from MGSRS is considered a downgrading disposal method, necessitating the need for more sustainable recycling techniques. Present methods for pure Si recovery techniques include smelting and physical separation, as shown in Figure 2. The technology for silicon recovery through sintering encompasses high-temperature smelting [13], slag refining process [14], electromagnetic induction refining [15], and blowing refining [16]. Major methods for physically separating

and recovering silicon consist of manual sorting, electric sorting, and flotation [4]. The method of pure silicon recovery as shown in Figure 3.

Smelting method: The high-temperature melting method achieves silicon-slag separation by controlling temperature, holding time and stirring time. However, due to the high melting point of SiC and the viscosity of the slag, relocating the remelted silicon becomes challenging, affecting the separation efficacy [13]. The viscosity and surface tension of silicate in MGSRS can be reduced by adding a slagging agent. Han et al. [15] combined electromagnetic separation with slag-making refining technology to isolate purified silicon from MGSRS. However the process demands a significant amount of energy and results in substantial slag consumption. Zhang et al. [16] introduced blowing during the smelting process to accelerate melt flow. Furthermore, while the purity of silicon recovery reached 99.28%, the recovery rate remained low. Thus, the current smelting method for silicon recovery exhibits notable drawbacks, including low recovery rate, high energy consumption and substantial bulk slag auxiliary.

Physical separation method: Achieving complete separation of silicon and silicates cannot be achieved by through manual sorting is impractical. Some scholars have proposed electric sorting due to the differing properties of silicon and slag, but equipment limitations restrict this approach. E. Larsen et al. [17] used a two-step flotation method to extract silicon from slag; however, the need for hydrogen fluoride (HF) remains unresolved. Tan et al. [4] first introduced terpenic oil as a surfactant to improve the surface hydrophilicity of silicon and silicate. The recovery rate of solid silicon reached 77.42% $\pm$ 4.40%, with purity exceeding 76%. However, variations in silicon content in slag generated by different producers require suitable adjustments to the flotation methods.
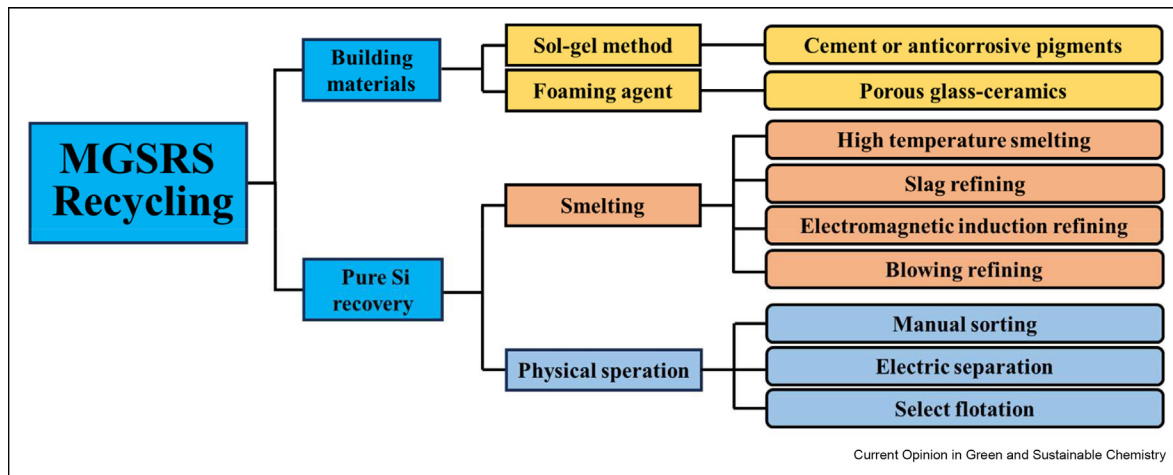
A gradual trend in MGSRS recovery is expected to emerge. After the acid leaching of MGSRS, oxides and silicates transform into soluble salts, while silicic acid enters the liquid phase. The solid remaining after

Table 1

Production of silicon-containing secondary solid waste.

Secondary resources	Proportion in production	Global production (t)	Element	Reference
MGSRS	10% of industrial silicon	778,300 (Forecast in 2022)	Si, SiC, SiOx, Al_2O_3, CaO, $R(SiO_3)n$	[4]
SF	5%−15% of industrial silicon	389,150−1,167,450 (Forecast in 2022)	SiO_2, C, CaO, Fe_2O_3, Na_2O, K_2O	[5]
SCW	30%−35% of crystalline silicon	162,000−189,000 (Forecast in 2021)	Si, Fe, Al, Ca, O, C, B, P	[6]
ESSC	5.4% of end-of-life PV module	3,240,000−4,212,000 (Forecast cumulative production in 2025)	Si, Al, Ag, Pb, B, P, O	[7−9]

Figure 2

Categories of MGSRS recycling methods.

separation comprises a mixture of Si and SiC. Silicic acid has the potential to produce hydrated silica and ultrafine silica. Mixtures of Si and SiC have been utilized in the negative electrode of lithium-ion batteries (LIBs). Victor Vanpeene et al. [18] proposed a high-value application, transforming Si and SiC from MGSRS into micrometric powder, implanting submicrometric SiC inclusions in a nanocrystalline/amorphous Si matrix. This material serves as an anode in LIBs, maintaining a capacity > 1000 mAh g^{-1}(> 3 mAh cm^{-2}) over 100 cycles. However, cycling exhibited a significant irreversible increase in the electrode mass and thickness attributable to the accumulation of solid electrolyte interphase (SEI) products. This implies that further work is required to enhance the SEI stability to achieve a viable high-capacity anode material.
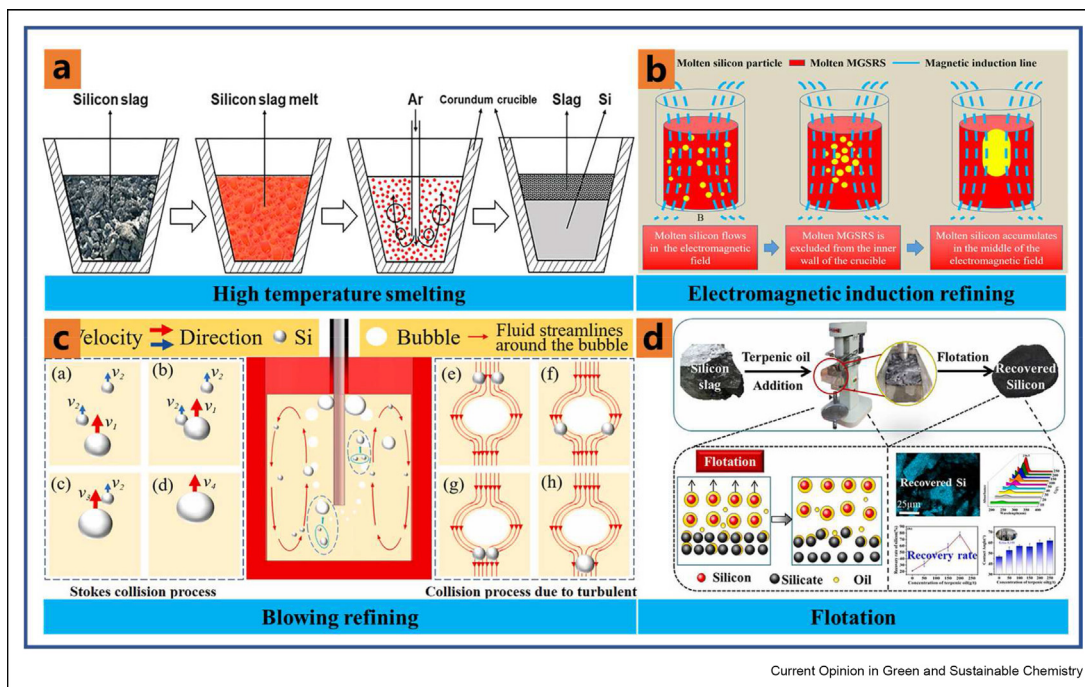
Silicon fume (SF)

In the process of smelting industrial silicon, SiO_2 in the high-temperature area of the furnace reacts with a carbon-reducing agent to produce SiO, carbon monoxide (CO), and a small amount of gaseous metal. The escaping gas rapidly oxidizes and condenses into spherical sub-micron amorphous silica particles, forming what is known as SF. The main impurities found in SF include carbon (C), CaO, ferric oxide (Fe_2O_3), sodium oxide (Na_2O), potassium oxide (K_2O), and other metal compounds. In Figure 4, SF is utilized in concrete [19], refractory materials [20], high-purity SiO_2 [21], adsorbing material [22,23] and other fields [24] owing to its fine particles, lightweight nature, large specific surface area, and high refractometry.

Scholars have extensively studied the preparation of concrete [25], and cement [26]. The SiO_2 in SF reacts with the lime in cement to form silicates, giving the

concrete a high elastic modulus, higher compressive strength, and increased corrosion resistance [25]. Martinez et al. [27] found that adding SF to Al_2O_3–MgO refractory castable resulted in a more silicate glass phase, reducing the bonding degree of castable, enhancing the mechanical strength, and yielding a denser structure after calcination. Wang et al. [22] prepared SSZ-13 zeolite with a high specific surface area (545.74 m^2/g). Ahmed et al. [23] utilized SF as a solid phase extraction agent, and the Si–O–Si and O–H functional groups in the SF enhanced the adsorption and binding of metal ions such as Zn(II) and Cd(II). Other avenues of study include additive materials [28], silica gel [29], and nano-silica [30].
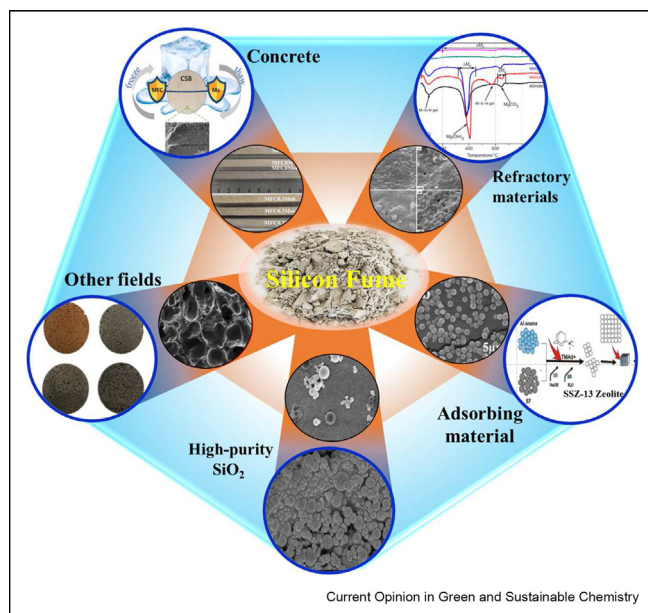
From 2022 to 2024, the demand for high-purity quartz sand is projected to reach 67,500 tons, 89,600 tons, and 11,100 tons, respectively. The price of high-purity quartz sand has risen from $\sim$ \$4,000 in 2021 to \$55,000 per ton at present. Enhancing the purity of SF will expand its application field and improve its value. Currently, the primary purification methods are calcination, acid treatment, alkali treatment, flotation, and more. M. Barati et al. [31] studied the removal of carbon impurities through calcination at 750 °C, achieving significant improvement. Sun [21] et al. utilized a mixture of acids (HF, HCl) to leach SF, resulting in an increased SiO_2 content from 98.606% to 99.9096%. He et al. [32] introduced pre-treated SF after acid leaching into sodium hydroxide (NaOH) solution to prepare a nano-silica sol with a particle size of 80 nm–100 nm and a purity of 99.91%. Scholars proposed the use of $C_{19}H_{42}BrN$ and $C_{18}H_{33}NaO_2$ as flotation agents, achieving an SF purity of 92.92%. However, the purification method does not yet meet the requirements for preparing 5N-grade quartz sand.

Figure 3

Method of pure silicon recovery from MGSRS: **(a)**High temperature smelting [13], **(b)**Electromagnetic induction refining [15], **(c)**Blowing refining [16] and **(d)**Flotation [4].
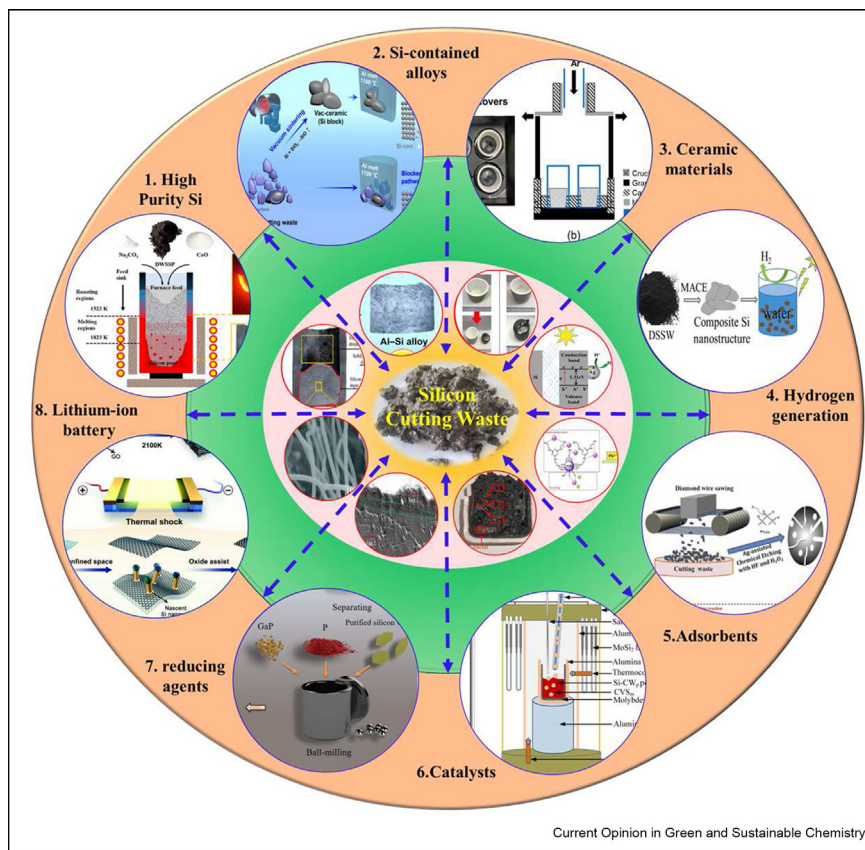
Figure 4

Recovery and recycling pathways for SF.

Characteristics of silica fume, including its large surface area, high silica content, low boron concentration, and small particle size, make it suitable as a feedstock for the production of high-purity silicon. Barati et al. [31] prepared crystalline silicon with a purity $> 2N$ through magnesiothermic reduction reaction and acid etching treatment on commercial SF (97.5% SiO_2). Due to the sub-micron size of SF, it is possible to prepare nanoporous silicon materials via metal thermal reduction for the negative electrode of LIBs. These attributes contribute to mitigating the volume expansion of the silicon anode of LIBs, reducing stress and strain, thus improving cycle stability. Carbon impurities present in SF can also enhance electrical conductivity.

Silicon cutting waste (SCW)

Approximately 30%–35% of SCW is generated when a diamond wire-cutting silicon rod is employed to prepare crystalline silicon wafers. In 2021, globally crystalline silicon consumption amounted to $\sim$540,000 tons, resulting in an estimated 162,000–189,000 tons of silicon wasted as SCW [6]. SCW is primarily composed of sub-micron flake particles of Si with a purity >90%, high crystallinity, and impurity elements including oxygen(O), C, iron (Fe), aluminum (Al), and nickel (Ni).

Figure 5

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Recovery and recycling pathways for SCW.

SCW can be recycled and reused in eight aspects as illustrated in Figure 5, including high-purity Si, Si-contained alloys, ceramic materials, hydrogen generation, adsorbents, reducing agents, and LIBs. A summary of the process methods and detailed recycling technology characteristics are presented in Table 2.

Yang et al. [33] recycled SCW to produce high-purity silicon via HCl leaching. However, surface oxidation hindered impurity removal, prompting the use of a mixed acid solution containing HF to improve SCW leaching efficiency [34]. Zhang et al. [53]. Proposed pyrometallurgy as a means to remove metal and non-metallic impurities such as C, O, boron (B), and phosphorus (P). The main pyrometallurgy methods for SCW are summarized in Table 2. Some researchers have combined acid leaching with pyrometallurgy, achieving silicon purity conforming to metallurgical grade standards (98.5%–99.5%). Furthermore, while acid leaching methods operate at lower temperatures, their removal efficiency and generation of waste acid are comparatively higher than high-temperature metallurgical processes. However, high-temperature metallurgical methods effectively remove most of the metals from Si.

Hence, the intended application fields of the product should be considered before purification to optimize the advantages of acid leaching or high-temperature metallurgical methods and improve efficiency.

Energy consumption and economic factors are important considerations for SCW recycling. Conversely, the development of comprehensive recycling technologies for green and sustainable practices should continue. Alternatively, expanding application fields is equally important. Wei et al. [40] proposed preparing Al–Si alloy, while Chen et al. [41] achieved a Si utilization rate of up to 95.03% via vacuum sintering to produce Al–Si alloy. Some scholars have also proposed using SCW to prepare SiNx, SiC, and SiC–AlN, suitable for ceramic materials such as diodes and special crucibles [54]. Kobayashi et al. [44] first reported that the fine silicon powder reacted with water to produce hydrogen, a reaction accelerated via photocatalysis [55] and porous silicon [45]. Chen et al. found that the SCW-prepared porous silicon exhibited effective adsorption of harmful heavy metal ions lead Pb(II) [46] and Cd(II) [56]. Zhang et al. [57] proposed silicon thermal reaction as a green recovery method for vanadium slag, reducing carbon emission.

Table 2

Different methods of SCW recycling application technology.

Technology target	Method	Characteristic	Reference
High purity Si-acid leaching (impurity removal efficiency%/Si purity %)	4M HCl	Al: 95.6	[33]
	5% HF + 5% HCl + 5% H_2SO_4/ ultrasonic	Ni:97.16, Fe: 99.62, Al: 93.3	[34]
High purity Si- Pyrometallurgy (impurity removal efficiency%/Si purity %)	Roasting + smelting	Al: 48.44/Si : 99.31	[35]
	Vacuum refining	P: 29.86, Al:82.73, Ca: 92.83/Si : 99.65	[36]
	Non-transfer arc-assisted vacuum smelting	C: 84.3	[37]
	Electroslag remelting refining	B: 80, P: 65	[38]
	Electromagnetic separation + slag treatment	Al: 91.67, B: 93.09/Si : 99.98	[39]
Si-contained alloys	Cryolite auxiliary smelting + agitation	Si recovery ratio (69.46%) (Si−Al alloy)	[40]
	vacuum sintering	The utilization rate of Si 95.03% (Si−Al alloy)	[41]
Ceramic materials	Incorporation of carbothermal nitridation and direct nitridation	Si_3N_4 ceramic density of 2.31 g cm^{-3} and a compressive strength of 109 Mpa	[42]
	Combustion synthesis	Si_3N_4 content higher than 90%	[43]
Hydrogen generation	Si nanopowder with water in the neutral pH	~55 ml/g hydrogen, pH 8.0	[44]
	Metal-assisted chemical etching	367.65 μmol $h^{-1}g^{-1}$	[45]
Adsorbents	Ag-assisted chemical etching + three-step grafting	On Pb(II) adsorption capacity 253.3 mg/g	[46]
Reducing agent	Silicothermic reduction	The dissolution kinetics of SiO_2 in SCW was determined	[47]
	Ferrochrome production of silicon instead of carbon	Chromium average yield of 93.4%	[48]
LIB	Ag-assisted chemical etching	972 mAh g^{-1} after 100 cycles at 1.0 A g^{-1}(pSi/ Ag/C/G)	[49]
	Cu etching crushed + Ag etching obtain porous	1409 mAh g^{-1} after 500 cycles at 0.2 A g^{-1} (PSi@SiOx/Nano-Ag)	[50]
	DC thermal plasma	Over 1000 mAh g^{-1} after 200 cycles at 1.0 A g^{-1}(PSi/Ag/C)	[51]
	Electrothermal shock	2381.7 mAh g^{-1} after 500 cycles at 1.0 A g^{-1}(Si nanowires)	[52]

Research in these extended application fields introduces new recycling concepts for SCW, holding significance from both waste recovery and economic perspectives. However, these concepts are still exploratory and exhibit a low Si consumption rate. The rapid growth of LIBs has led to a substantial demand for negative electrode materials. The core material, Si found in SCW, has a theoretical lithium storage capacity of 4200 mAh g^{-1} for LIBs. Additionally, the in-situ doping of B and P impurities in SCW is anticipated to enhance material properties. Xi et al. [50] prepared porous silicon via metal-assisted chemical etching. Zhang et al. [51] obtained submicron silicon through DC thermal plasma. Lu et al. [52] proposed obtaining Si nanowires via Electrothermal shock, showcasing enhanced cycle stability of 2381.7 mAh g^{-1} after 500 cycles at 1.0 A g^{-1}. This confirms the potential for utilizing SCW to produce anodes made with silicon material for LIBs.
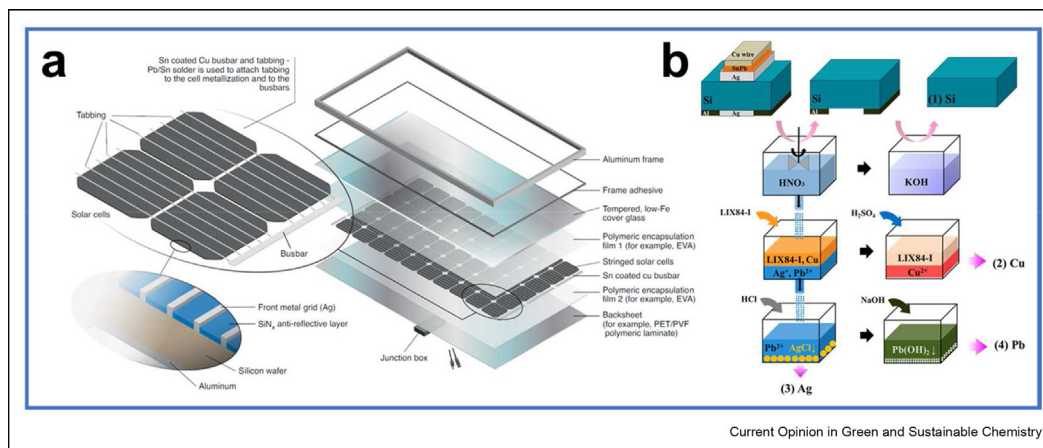
End-of-life silicon PV solar cells (ESSC)

According to forecasts by the International Renewable Energy Agency (IRENA), approximately 60−78 million tons of photovoltaic waste will be generated by the year 2050 [7,8]. At the core of the end-of-life crystalline silicon photovoltaic module lies the solar cell. It primarily consists of high-purity silicon, aluminum back electrodes, silver grid, and other valuable metals. Furthermore, while landfill solid waste is a common option, it is not environmentally friendly or conducive to recycling [58]. Therefore, there is a pressing demand for ESSC recycling. The schematic diagram of the c-Si PV module structure is shown in Figure 6a. The composition of ESSC includes Si at 90.185%, Al at 8.985%, silver (Ag) at 0.720%, Pb at 0.042%, Sn at 0.013%, and other elements at 0.055%. Various existing application technologies for ESSC recycling are outlined in Table 3, predominantly encompassing high-purity solar-grade silicon, refabricated solar cells, and silicon anode for LIBs.

These recycling processes can be classified into two categories, silicon purification (involving mechanical synergy with chemical leaching purification, heat combined with chemical leaching purification) and silicon material preparation (recycling ESSC for new solar cells and LIBs anode). Mechanical treatment typically

Figure 6

(a) Photovoltaic module structure diagram [8] **(b)**Process flow of chemical leaching for solar cells [61].

Table 3

Different methods of ESSC recycling application technology.

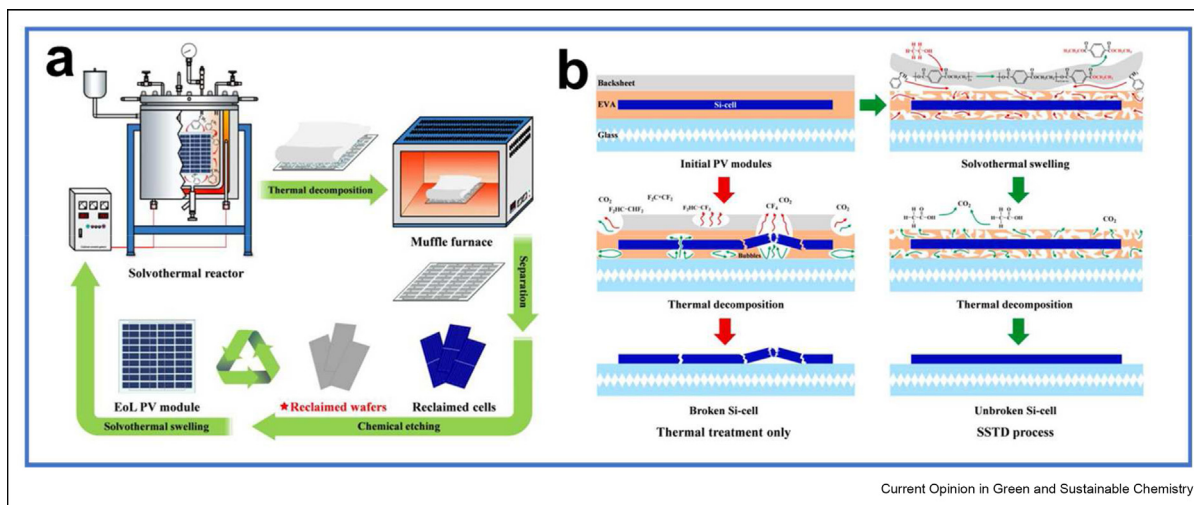| Technology target	Method	Characteristic	Reference
Silicon purification (remove Ag, Al)	0.35 M I2 + 0.7 M KI, liquid−solid ratio(10 : 1)	The reaction rate is close to HNO_3, the environmental influence is small	[69]
Silicon purification	48%HF, 70%HNO_3,97%H_2SO_4+99%CH_3COOH mixed etching, 20 min	The yield of silicon was 87% (high reaction rate)	[70]
	Electrodeposited Ag+11.4% HNO_3 + 20% NaOH + HNA	The yield of silicon was 99%, and silicon purity was 4N	[71]
	10 M NaOH, 63 °C, 5 min + 6M HNO_3, 70 °C, 5 min + H_3PO_4, 70 °C, 45 min	The yield of silicon purity was 99.9984%	[72]
Re-fabricated solar cell	20% HNO_3 + 20% HF remove Ag and Al	Efficiency of 18.5%	[66]
	60% HNO_3, indoor temperature, 5min + 80% KOH, 45 °C, 8min + H_3PO_4	The reconstructed solar cell has the same efficiency as the original	[73]
LIB (W-rAlAg@C)	Ball-milled + Remove Al, Ag + +NMP/CNTs	961.9 mAh/g after 50 cycles at 0.1 A/g	[67]
LIB (W−Si-rM@G)	Acid leaching + mixed with graphite and ball-milled	913 mAh g^{-1} after 200 cycles	[68]
LIB (Si/SiOx/Al_2O_3)	Ball-milled + 18 M H_2SO_4 and 16 M HNO_3 leaching + combined with a carbon fiber paper (CP) substrate	1603 mAh g^{-1} after 100 cycles at 200 mA g^{-1}	[74]
LIB (Si nanoparticles)	Via arc thermal plasma under Ar and N_2 mix gases	2920 mAh g^{-1} at 0.1 A g^{-1}	[75]
LIB (porous Si)	Ball-milled + etching removal Al, Ag, SiNx + molten salt electrolysis	2427.7 mAh g^{-1} after 200 cycles at 1.0 A g^{-1}	[76]

involves crushing or grinding waste modules into small particles or fine powders [59]. Subsequently, various etching processes using different types of chemicals, such as HNO_3, HCl, HF, H_3PO_4, and more are used to remove impurities (Ag, Al, SiN*x*, etc.) to obtain pure silicon. Acid leaching, depicted in Figure 6b, effectively removes metallic impurities [60], with Si remaining stable in acid solutions except for HF, while metallic impurities react with acid to form water-soluble metal salts.

Tammaro et al. [62] used heat treatment reaching 600 °C instead of mechanical treatment, to release ESSC from the PV module, followed by chemical leaching. Some scholars focus on pyrolytic separation to

achieve complete silicon extraction as shown in Figure 7, unbroken wafers also offer possibilities for value-added silicon wafer reuse [63].
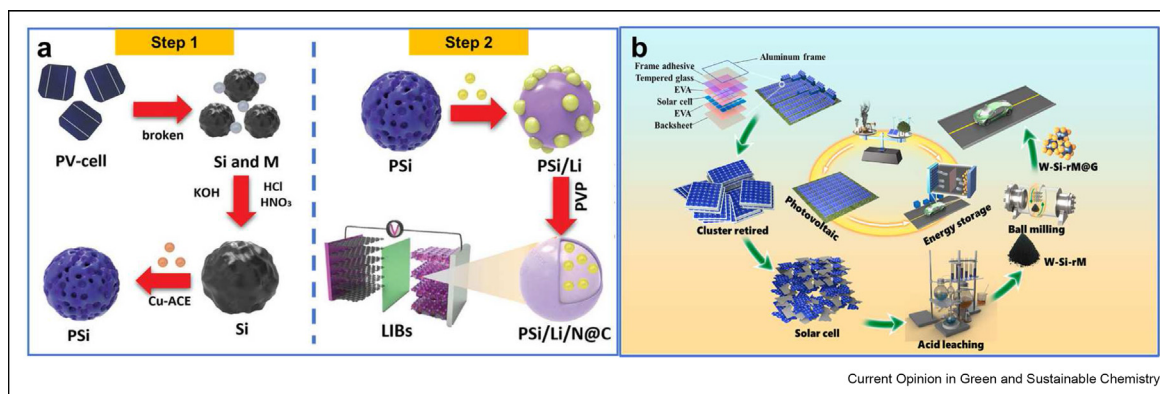
Jun-Kyu Lee et al. [65] researched ESSC recycling for refabricated solar cells, and the results indicated that the PV performance of the refabricated solar cell reached 18.5%, closely matching the performance of virgin cells [66]. Furthermore, Zhang et al. [67] and Liao et al. [68] prepared porous silicon-carbon composites and submicron silicon-carbon composites using impurity removal Si material from ESSC as raw materials. This was achieved using metal-assisted chemical etching (MACE) technology and high-energy ball milling methods as shown in Figure 8.

Figure 7

Schematic diagram **(a)** and mechanism diagram **(b)** of the Si-wafer nondestructive recovery system with solvothermal swelling-thermal decomposition technology [64].

Figure 8

(a) Schematic illustration of the synthesis of PSi/Li/N@C [67]. **(b)**Schematic illustration of the synthesis of W−Si-rM@G. [68].

Conclusion

The photovoltaic industry produces secondary silicon resources, which have been proved to be recyclable. This article offers a comprehensive review of the progress made in PV-SSCR recovery, focusing on critical areas within the silicon photovoltaic industry, including MGSRS, SF, SCW, and ESSC. Successful production of high-purity recycled silicon has been achieved. These methods often require substantial acid use or high temperatures.

Several recycled applications look promising, including potential reuse applications such as advanced construction materials, ceramics, alloys, and absorbents. Notably, the high capacity of silicon-based negative

electrodes for LIBs and their adaptability to impurities have garnered significant academic interest. Further research is needed to make these recycling methods more cost-effective and sustainable for their adoption within the industry.

Multiple studies aim to address volume expansion during silicon lithiation processes, although this field still faces significant challenges. The prospects for converting PV-SSCR into silicon-based anode materials for LIBs are outlined as follows:

1. The interface between Si and SiC in MGSRS exhibits instability, with SiC struggling to effectively inhibit volume expansion. To improve this interface

effect, it is suggested to combine the carbonyl group on the surface of Si and SiC within an effective medium. This approach is expected to significantly exert the inhibition effect of SiC on the volume expansion of silicon.

2. Research on the preparation of nano-porous silicon via magnesium thermal reduction of SF is relatively limited, nonetheless, delving deeper into this area holds substantial importance for porous silicon from sub-micron SF.

3. Addressing the challenge of separating silicon porous/nanomaterials prepared from SCW from the growth substrate while preserving their structure is a critical consideration.

4. ESSC for silicon-based negative electrode applications holds distinctive advantages in terms of doping and polymetallic composite. However, current research works in this field are limited, and the *in-situ* utilization of impurities holds promising potential.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this article.

Data availability

No data was used for the research described in the article.

Acknowledgments

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