

Innovative recycling of high purity silver from silicon solar cells by acid leaching and ultrasonication

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ABSTRACT

Precious and scarce silver (Ag) is used as a front electrical contact in silicon solar panels. With massive amounts of solar panel waste coming to end-of-life, it is imperative to recover all the Ag from these modules. In this paper, we propose a novel method to easily reclaim Ag from end-of-life silicon solar cells using low concentration sulfuric acid (H_2SO_4) leaching followed by ultrasonication. Our process simplifies the Ag recycling procedure by directly recovering the Ag contacts from solar cells, eliminating the need for secondary precipitation/electro-deposition. First, scanning electron microscopy (SEM) with energy dispersive X-ray spectroscopy (EDX) and X-ray photoelectron spectroscopy (XPS) was used to study the leaching and ultrasonication process on rejected solar wafers. Next, a solar panel from landfill was heated in a furnace to burn off the polymer encapsulant. The silicon wafers were then collected from the burning process and leached in 2 M H_2SO_4 at two different shaking speeds for 48 h at room temperature. Finally, the end-of-life silicon wafer pieces were collected, sonicated in water, and then the sonication water was centrifuged to show a proof-of-concept to recover the Ag contacts. Inductively coupled plasma optical emission spectroscopy (ICPOES) is used to monitor dissolved elements in the leachate as a function of time and shaking speed. XPS is used to evaluate the composition of the silicon cell wafer surface before and after H_2SO_4 leaching. SEM is used to image the recovered Ag contacts morphology and EDX confirms the recovered particles contain high amounts of Ag. A proposed schematic illustrates the authors' hypothesis for the peeling mechanism.

1. Introduction

With the need for renewable energy increasing and the price of silicon solar modules decreasing, the installation of solar technology has skyrocketed. However, mass deployment of solar panels over the last few decades is quickly leading to a huge pileup of waste by 2030 [1]. Not only do silicon solar panels contain hazardous materials such as lead (Pb) [2] and chromium (Cr) [3] but panels also contain precious silver (Ag). Ag is used in many industries such as jewelry, electronics, photography, and plastic production [4]. In solar panels, Ag is used on the front electrical contacts for its superior electrical conductivity. However, mining of precious materials such as Ag causes substantial damage to endangered ecosystems and often occurs in water-scarce communities [5]. Continued mining of Ag to supply the demand for

clean energy presents a juxtaposition that can be avoided through sustainable recycling of Ag. Unsustainable Ag consumption is also predicted to limit silicon solar panel growth [6], adding another reason recycling of Ag from end-of-life silicon solar modules is imperative.

A variety of chemistries have been explored for Ag recovery, such as deep-eutectic solvents [7] and nitric acid [2,3]. However, a sulfur (S)-containing chemical is a good choice for Ag removal from solar cells because silver's high affinity for both inorganic and organic S compounds leads to the formation of various complexes in aqueous solutions [8]. For example, Wang et al. used 1 M H_2SO_4 plus hydrogen peroxide to leach and electrowin Ag from spent batteries, with a recovery rate of 98.5% [9]. Oh et al. used a leaching solution of ammonium thiosulfate, copper sulfate, and ammonium hydroxide to selectively leach 100% Ag from printed circuit boards [10]. Yang et al. used methanesulfonic acid

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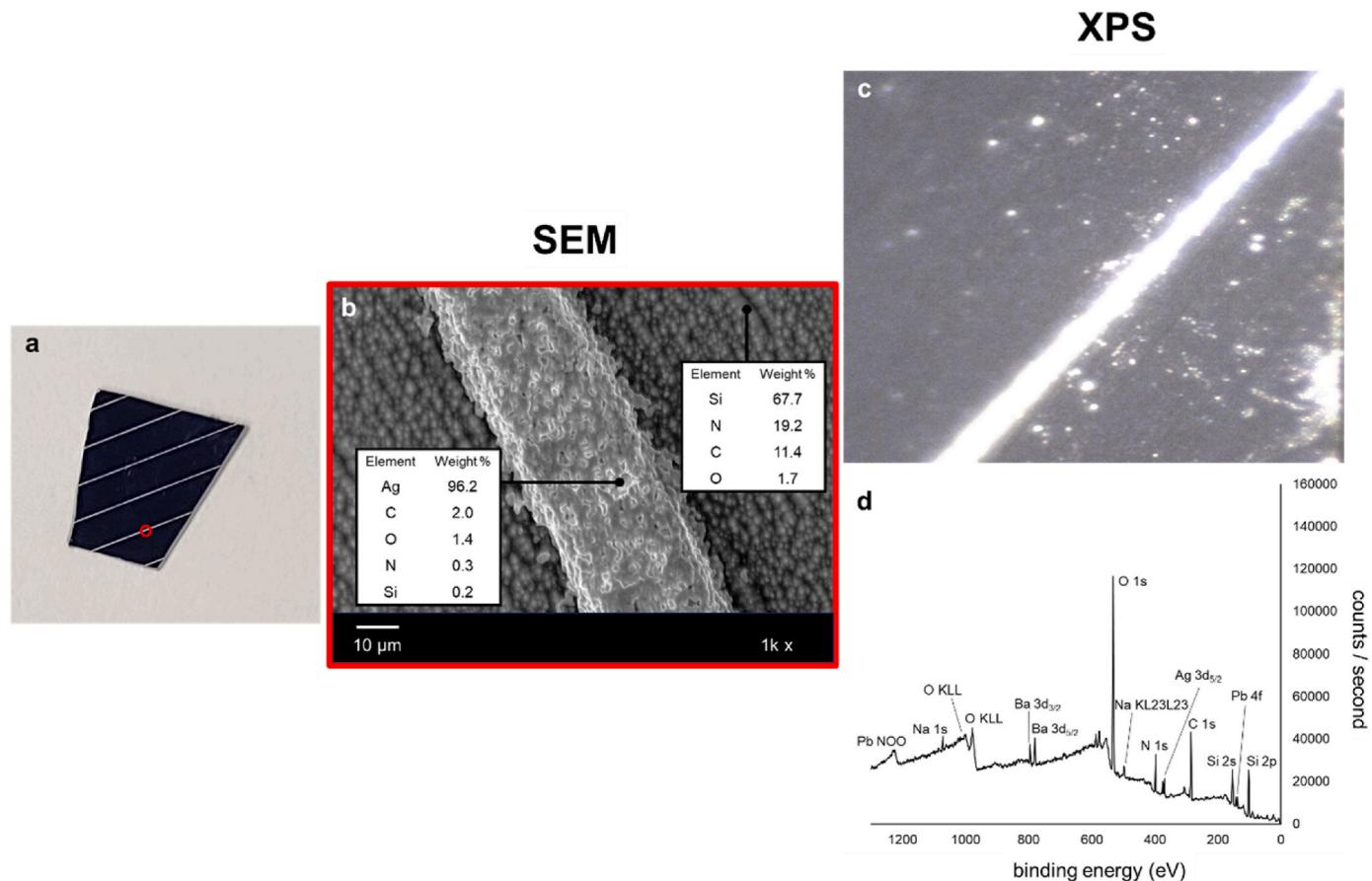


Fig. 1. (a) image of rejected solar wafer piece analyzed (no leaching or ultrasonication) (b) SEM image with EDX data (c) optical image of region investigated by XPS (d) XPS data showing elemental composition of the wafer piece.

+ hydrogen peroxide to leach Ag from solar cells [11]. In this paper we selected sulfuric acid (H_2SO_4) for Ag recovery from end-of-life solar cells because it is readily used in industry.

Also to optimize Ag recovery from silicon solar cells, it is important to understand Ag finger printing properties. Ag paste contains additives like glass and solvent, which play different roles in determining the properties of the Ag paste [12]. Wendling et al. investigated the effect of different chemicals, including H_2SO_4 , on the adhesion of silver fingers on silicon solar cell wafers for the application of the plating process in solar cell manufacturing. Their study hypothesized the surface interaction between the Ag contact and the Si wafer plays a critical role in adhesion [13]. Kraft et al. also tested metal cation effects on silver paste adhesion on solar cells in various plating mediums, including H_2SO_4 [14]. They found a correlation between the amount of glass in fired Ag pastes and paste adhesion, as well as a correlation between H_2SO_4 pH and Ag dissolution. In this paper, we combine low concentration H_2SO_4 solution followed by ultrasonication for the application of easy removal of Ag contacts from end-of-life silicon solar cells. What is unique about our process is that it allows for direct recovery of silver contacts; no secondary precipitation or electrochemical processing is necessary, adding to the novelty and environmental-friendliness of our process.

2. Materials and Methodology

To provide an in-depth understanding of the peeling mechanism, an experiment was performed using a rejected solar wafer sourced from a manufacturer. This wafer weighed 9.8 g and contained Ag contacts on the top, but no Al back contact. The wafer was crushed, and a piece was removed for scanning electron microscope (SEM) imaging and X-ray photoelectron spectrometer (XPS) sampling. A Zeiss Auriga was used for

SEM, an Oxford X-Max detector was used for energy dispersive X-ray spectroscopy (EDX), and a Kratos Supra was used for XPS. 1.2 g of the remainder of the wafer pieces was leached in 300 mL of ~2 M H_2SO_4 (Alfa Aesar, 95–98%) for 48 h at 350 rpm stirring. After this, the wafer pieces were rinsed, collected, and a sample was removed for SEM/XPS. The rest of the wafer pieces were sonicated in deionized (DI) water for 30 min. After sonication, the wafer pieces were removed from the DI water and allowed to air dry. A sample was collected for SEM/XPS.

To show this process would work on a real end-of-life silicon solar module, a module was sourced from landfill. The manufacturer was Neo Solar Power, and it had a max power rating of 285 W, open circuit voltage of 39.16 V, and short circuit current of 9.49 A. After the aluminum (Al) frame and junction box were removed, the cell was cut into strips. These strips were exposed to 550 °C for 30 min in a quartz reactor placed in a tube furnace (Nabertherm RT 50–250/13) with a constant flow rate of 1000 ± 10 mL/min air. At this temperature, the polymer decomposes [3] and the solar panel strips break into pieces. A water scrubber was used to capture halogenated fumes from the incineration process. After the samples cooled to room temperature, the Si wafers were separated from the broken glass panel and sonicated in DI water for 30–60 s, then allowed to dry in air before leaching.

Next 0.2 g of solar cell wafer pieces were leached in H_2SO_4 (98%) from MilliporeSigma diluted to ~2 M. The volume of this leachate was 50 mL. Two agitation speeds were tested: 160 and 350 rpm. A Heidolph DSG 304 orbital shaker was used to agitate the samples at 350 rpm, and a Grant Instruments PSU-10i orbital shaker was used for the 160 rpm experiments. The agitated leaching was carried out for 48 h. During this time, a ThermoFisher Scientific iCapPro XP inductively coupled plasma optical emission spectrometer (ICPOES) was used to record leaching concentrations at specific times. ICPOES standards were diluted with 1

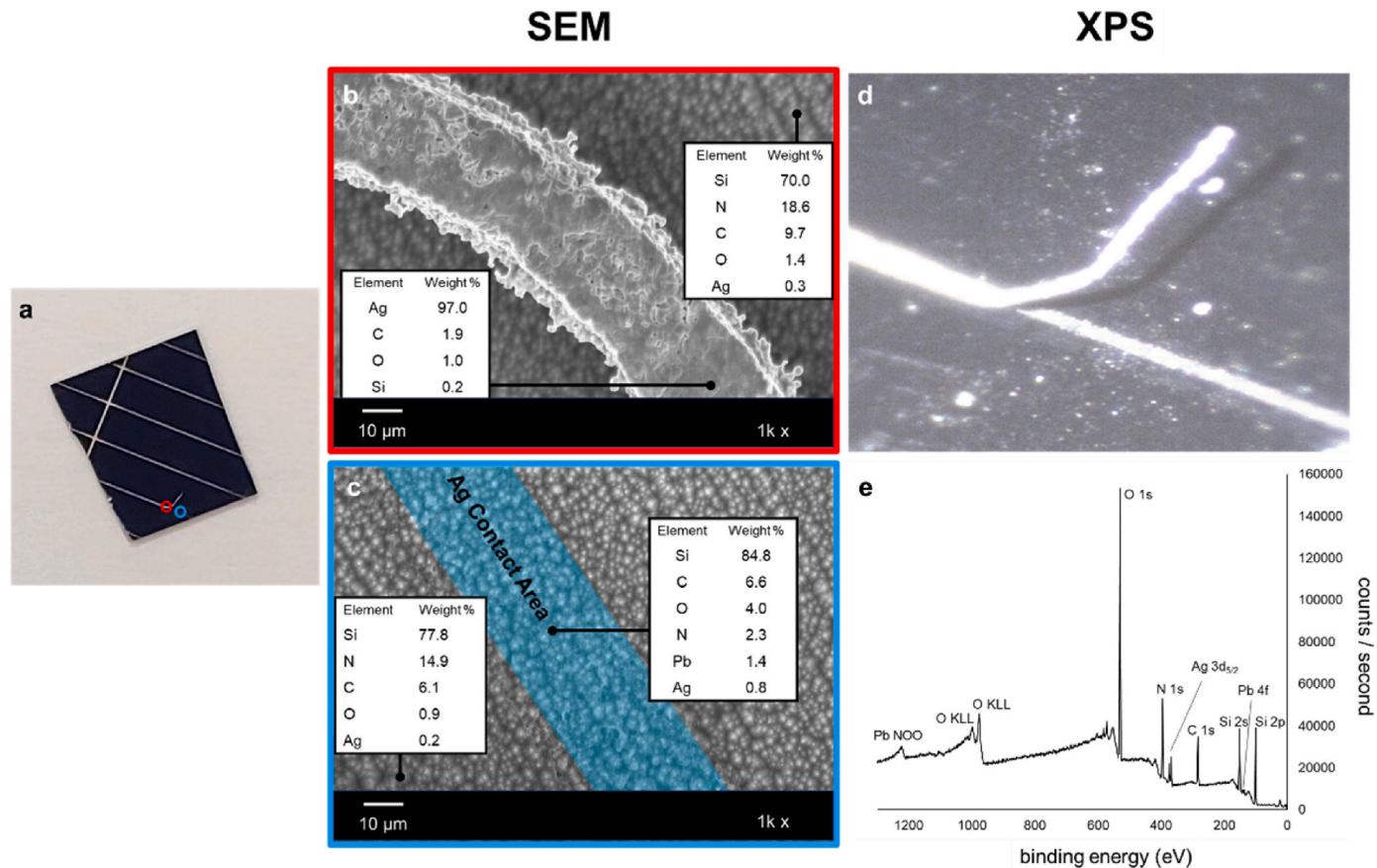


Fig. 2. (a) image of the rejected solar wafer piece analyzed (leaching, no ultrasonication) (b) SEM image with EDX data of Ag line (c) SEM image with EDX data of Ag contact region (d) optical image of region investigated by XPS (e) XPS data showing elemental composition of the wafer piece.

M H₂SO₄ to the following concentrations: 20 ppm, 10 ppm, 5 ppm, 2.5 ppm, 1.25 ppm, and 0.625 ppm. A 0 ppm solution was also included for the linear calibration curve. Three trials were repeated for each agitation speed. A FEI Quanta 200 FEG Environmental Scanning Electron Microscope (ESEM) was used for imaging the solar wafer surface at defined leaching times, and Energy Dispersive X-ray Spectroscopy (EDX) was used to quantify elemental composition of the cell wafers after defined leaching times.

Following leaching in 2 M H₂SO₄ at 350 rpm, these wafer pieces were sonicated in DI water for 15, 30, and 60 min. Leaching in 2 M H₂SO₄ at 350 rpm followed by sonication for 30 min was repeated three times. The sonication water from all three trials was collected and centrifuged to recover the Ag contacts. This Ag was analyzed using SEM and EDS. Finally, surface chemical composition of the cell wafer pieces before and after leaching was analyzed using a PHI 5000 Versa Probe III X-ray photoelectron spectrometer (XPS).

3. Results and discussion

3.1. Leaching and ultrasonication of rejected solar wafer pieces

A solar wafer rejected from the manufacturing line was crushed and treated in ~2 M H₂SO₄ for 48 h with stirring, then the pieces were ultrasonicated to recover the Ag contacts. At different stages in the process (no leaching or ultrasonication, post leaching, and post leaching + ultrasonication), rejected wafer pieces were removed and analyzed using SEM/EDX and XPS. The results of these analyses are shown in Fig. 1 - Fig. 3. Fig. 1 shows the results of analyzing the rejected wafer as received. The top contact is over 95% pure Ag according to EDX (Fig. 1b), with slight contamination of carbon (C) and oxygen (O), likely

from the organic binder used in the Ag paste [15]. The rejected wafer surface is silicon nitride (Si₃N₄) with C and O contaminants (Fig. 1b). XPS shows there is also trace sodium (Na), barium (Ba), and lead (Pb) present (Fig. 1d). The Na is likely from cross contamination, but the Ba is surprising. Pb is likely from the glassy additive in the Ag contact [16].

After leaching in ~2 M H₂SO₄, the peaks for Na and Ba disappear, and the peaks for Pb are much less pronounced (Fig. 2e). The Ag contact appears unaffected by the leaching process (Fig. 2b), other than it is starting to peel off the substrate (Fig. 2d). The area where the Ag contact used to reside was investigated by EDX and found to contain mostly Si with trace Pb and Ag (Fig. 2c). There is also very little nitrogen (N) present in this area, suggesting the region below the Ag contact is part of the Si wafer, as is reported in literature [17]. After 30 min of sonication, the majority of the Ag contacts have been removed from the rejected Si wafer surface or are peeled up (Fig. 3a and d). The Ag contact is chemically unaffected by the sonication process (Fig. 3b), and the area on the rejected wafer where the contact once resided contains majority Si with traces of C, N, Pb, O, and Ag (Fig. 3c). XPS suggests oxidation of the surface, and a small peak for Pb is visible (Fig. 3e). The calculated Ag recovery from this leaching and ultrasonication process is 0.14 g according to mass balance (Table 1S in Supplementary Material).

Fig. 4 shows SEM imaging of the boundary where the Ag contact was removed. The top portion of the image shows random pyramid texturing of the solar cell. Comparatively, the bottom portion of the image (where the Ag contact used to be) shows the texturing has been disrupted. This area is likely the remaining glassy layer, where trace lead oxide (PbO) from the Ag contact remains stuck to the rejected wafer surface (Fig. 1S in Supplementary Material). Along the boundary between the two distinct regions, tiny pores are visible (Fig. 4b). It is suspected these pores are pinholes in the glassy layer from Ag contact firing [17]. The

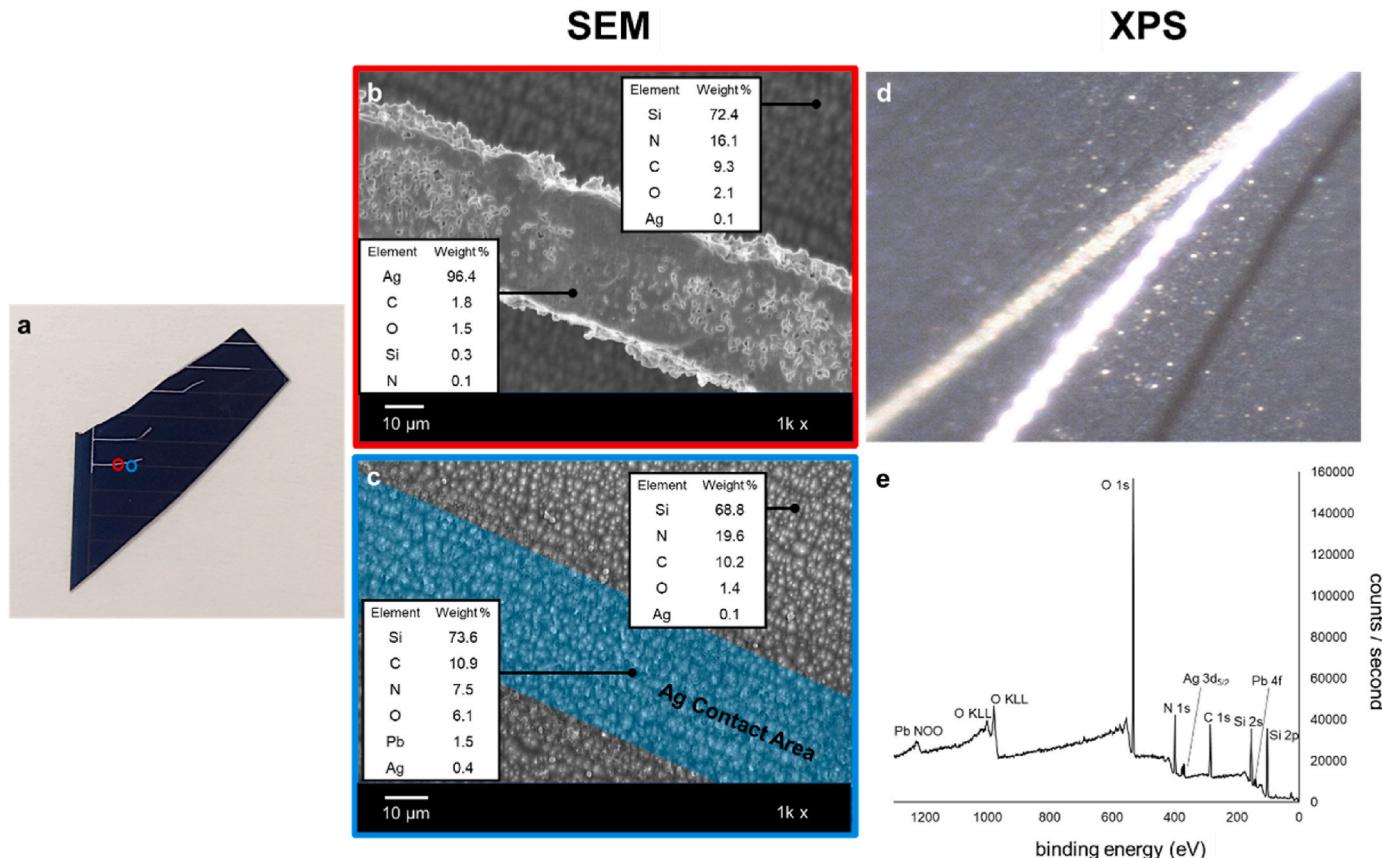


Fig. 3. (a) image of the rejected solar wafer piece analyzed (post leaching and ultrasonication) (b) SEM image with EDX data of Ag line (c) SEM image with EDX data of Ag contact region (d) optical image of region investigated by XPS (e) XPS data showing elemental composition of the wafer piece.

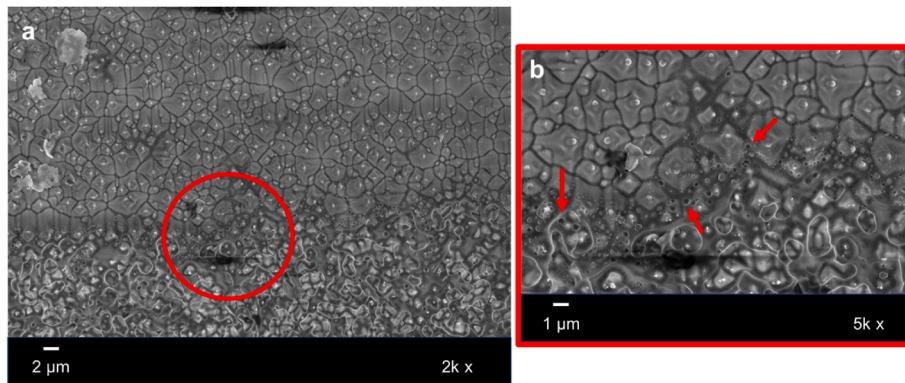


Fig. 4. SEM imaging of rejected Si wafer post leaching in 2 M H₂SO₄.

pinholes appear to be aggregated in a line along the boundary between the wafer and Ag contact, and could serve as weak points enabling Ag contact peeling.

3.2. Leaching and ultrasonication of end-of-life solar wafer pieces sourced from landfill

This data presented corresponds to the leaching of a solar panel obtained from landfill. Fig. 5 shows the average concentration of the leachate over time. The Al back contact leached at the highest concentration of all elements tested. This is not surprising, considering H₂SO₄ dissolves Al and has been investigated for Al recovery from fly ash [18, 19]. The maximum Al concentration in the 160 rpm tests was 609 ppm

(0.134 mg Al/mg cell) at 32 h. The maximum Al concentration in the 350 rpm tests was 641 ppm (0.139 mg Al/mg cell) at 32 h. When the standards were made, Si was found to precipitate slightly in H₂SO₄, which explains the large error bars for Si in Fig. 5. It should be considered that soluble fluorine from the burning stage could facilitate slight Si dissolution in solution, though further investigation is needed. The maximum Ag detected was 0.128 ppm (2.693E-05 mg Ag/mg cell) at 48 h in the 160 rpm tests, and 0.128 ppm (2.778E-05 mg Ag/mg cell) at 32 h in the 350 rpm tests. This shows the Ag fingers do not readily dissolve in the 2 M H₂SO₄. However, the H₂SO₄ leachate does disrupt the finger adhesion to the end-of-life wafers (Fig. 2S in Supplementary Material).

Fig. 6 presents the XPS survey spectra from the wafer portion of the end-of-life solar cells before and after H₂SO₄ leaching. The spectra show

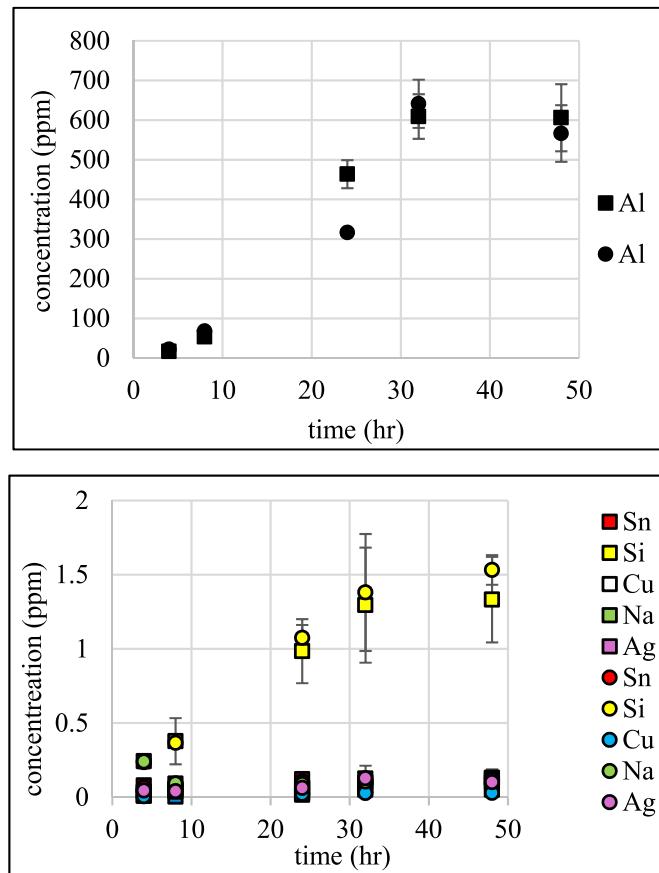


Fig. 5. ICPOES (top) high concentration and (bottom) low concentration data from leaching end-of-life solar wafer pieces in 2 M H₂SO₄ with stirring for 48 h. Squares represent 160 rpm stirring and circles represent 350 rpm stirring.

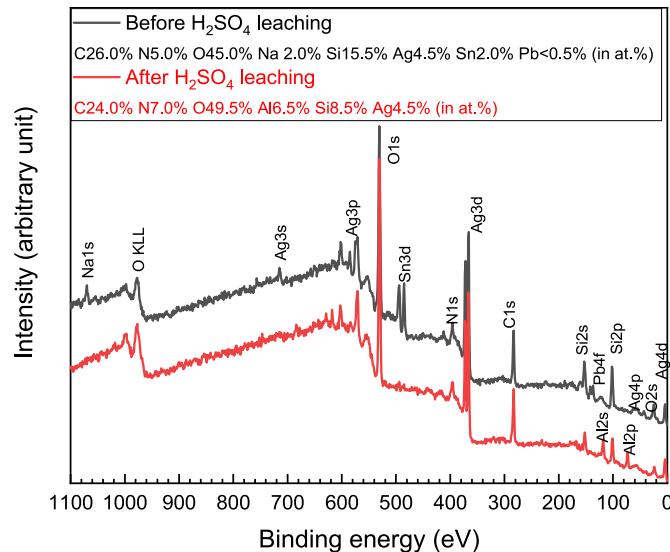
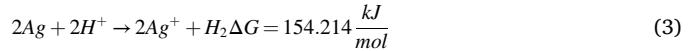
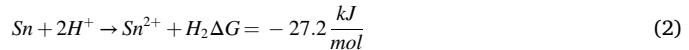


Fig. 6. XPS survey spectra from the cell wafers before and after H₂SO₄ leaching.

the change of surface composition on the wafer surface. Excluding the unavoidable surface contamination, i.e. adventitious C and the naturally developed native oxide layer, the major of the constituents of the end-of-life wafer before leaching include nitrogen (N), sodium (Na), silicon (Si), tin (Sn), and lead (Pb). After leaching, Na, Sn, and Pb on the end-of-life

wafer surface are removed, and the imbedded Al contacts are revealed. The trace Pb from the solder likely precipitates out of solution and forms lead sulfide (PbSO₄). The Si:N ratio of silicon nitride (Si₃N₄) decreases, suggesting a disruption of this layer. Using Gibbs free energy data [20] it can be seen that it is thermodynamically favorable for trace Pb and Sn to dissolve in H₂SO₄, but unfavorable for Ag dissolution, thus supporting the findings that Ag does not dissolve and the contacts can be reclaimed wholly:



End-of-life wafers treated in 2 M H₂SO₄ at 350 rpm were followed by sonication in DI water for 15, 30, and 60 min. Fig. 2S (in Supplementary Material) shows images of the end-of-life solar cells after various sonication times. The 15 min sonication image is shown in Fig. 7 for an example. The yellow is the back side of the Si wafer (Fig. 7a), as the Al back contact has been completely removed by H₂SO₄. After just 15 min of sonication, most of the Ag contacts have been dislodged, though a few remain attached to the end-of-life solar wafers. 100% of the Ag contacts were dislodged from the cells after 30 min of sonication. After 60 min, the Ag contacts have been broken up into small particles. Even before sonication, it is observed some of the Ag contacts are curling up from H₂SO₄ leaching and could be pulled up carefully using tweezers. Fig. 7b shows SEM imaging of the region where a Ag contact used to be. The faint line still present is likely the exposed Si wafer which is revealed once the Ag contact is peeled off [21]. This is confirmed in Figs. 2 and 3 where the majority of the contact area is Si with trace contaminants. Fig. 7c shows the back of the end-of-life solar wafer. Although the Al back contact is gone, Al contacts are still imbedded in the Si matrix. These imbedded Al contacts are the 'shiny' gray regions on the yellow backing of the Si wafer in Fig. 7a. The slight gray specks seen in Fig. 7c near the imbedded Al contact are slight residual Al from the back contact. EDX confirms both the contacts and gray specks are Al; no Ag was detected here. Additionally, both sides of the cell only contain slight oxidation.

Fig. 8a shows Ag particles collected post-leaching and sonication for 15 min. At 15 min, the particles are still large enough to be observed visually. The water from three rounds of 30 min sonication was collected and centrifuged (Fig. 8b). The Ag particles were collected from the water and characterized using EDX. Fig. 8c shows SEM imaging of the Ag particles after 30 min of sonication. The Ag fingers have been broken up into small, curled strips. EDX confirms these particles are majority Ag with slight oxidation and carbon contamination. Small chunks of Si wafer are mixed in with the Ag particles. However, because these chunks are not chemically bound to the Ag particles, they can be mechanically separated from the precious Ag by sifting or filtered during remelting of the Ag for new solar cell contacts.

Clearly from both sets of data, the 2 M H₂SO₄ + ultrasonication is shown to disrupt the boundary layer between the Ag contact and the Si wafer. Herein, a short discussion of possible explanations is provided along with a proposed mechanism in Fig. 9. Wendling et al. (2012) investigated Ag contact adhesion to a crystalline solar cell after exposure to different chemicals. They observed H₂SO₄ had a significant effect on Ag paste adhesion and presume this has to do with the lead oxide in the glass contained in the Ag paste [13]. One possible explanation could be the conversion of PbO to precipitate PbSO₄ [22]:



which, using available Gibbs energy data [20], is thermodynamically favorable. Kraft et al. (2013) also found H₂SO₄ appears to disrupt the

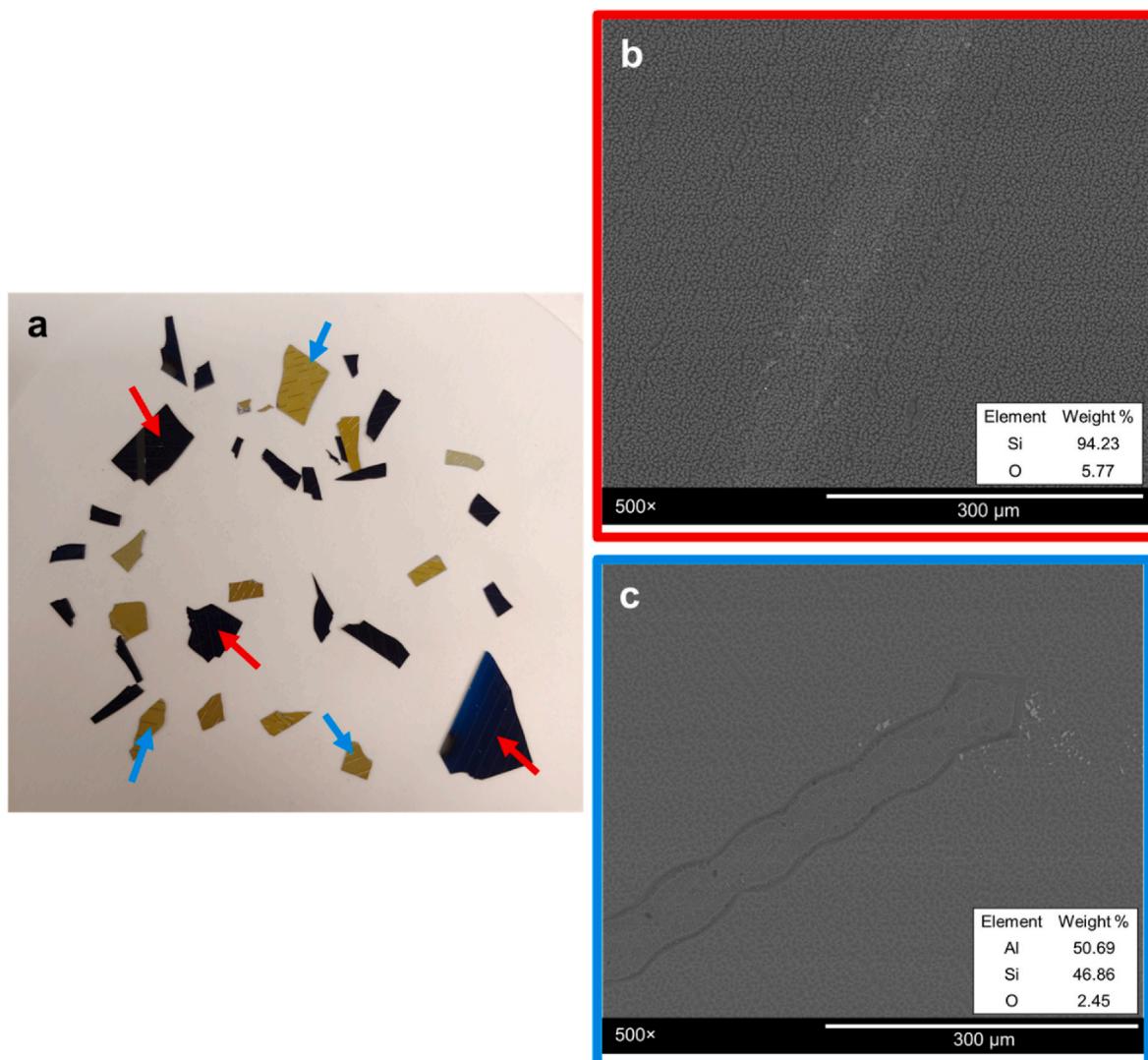


Fig. 7. (a) Image and (b,c) SEM image of end-of-life solar cell, post leaching in 2 M H₂SO₄ 48 h 350 rpm followed by 15 min sonication. No Ag is present where the finger used to be (b). Al contacts are still imbedded in the Si wafer (c).

glass additive in Ag pastes, as H₂SO₄ treatment at pH = 1 resulted in low contact adhesion. During Ag contact deposition, trace Pb additive also reacts as follows [15,16]:



through which etching of the Pb by H₂SO₄ could take place according to the reaction:



Therefore, we propose the Pb additive in the Ag contact plays a critical role in dislodgement of the Ag fingers during our process.

However, the change in the Si:N ratio presented in our XPS data (Fig. 6) suggests the silicon nitride layer might also play a role in finger dislodgement. Corrosion of silicon nitride in low concentration H₂SO₄ [23] and hydrolysis of silicon nitride [24] have both been reported. Monteverde et al. (2001) studied the effect of sintering aids in silicon nitride ceramics in 1.8 M H₂SO₄ and found evidence of grain boundary corrosion [25]. The corrosion of silicon nitride by water at elevated temperature and pressure has also been reported [26]. Therefore, we propose the silicon nitride layer around the Ag fingers might be disrupted by H₂SO₄ solution. It should duly noted we are proposing

disruption, not dissolution, of the silicon nitride layer, and this slight disruption is enough to help dislodge the Ag fingers from the wafer surface with ultrasonic assistance. Alternatively, the Si in solution could originate from leaching of the Si₃N₄ or silicon dioxide (SiO₂) glassy phase between the Ag contact and Si wafer due to trace F⁻ contamination from burning the cells. Besides disruption of the Si₃N₄ or SiO₂ by H₂SO₄, it is also possible the Si detected by ICPOES originates from solar wafer particles that were smaller than 0.45 μm and thus able to pass through the filters used before ICPOES.

The presence of pinhole voids along the Ag contact/wafer boundary (Fig. 4) also create weak points where acid and ultrasonication could penetrate the glassy layer boundary, severing the Ag contact from the wafer surface. Herein we propose a future investigation into the peeling mechanism is needed to decode the observed dislodging of Ag contacts from the wafer using low concentration acid followed by ultrasonication.

4. Conclusion

With massive amounts of silicon solar panels destined for landfill, it is imperative to recover 100% of the Ag contacts. In this paper, we demonstrate a low concentration of H₂SO₄ at room temperature with stirring can loosen the Ag contacts from the Si wafers. Sonication in

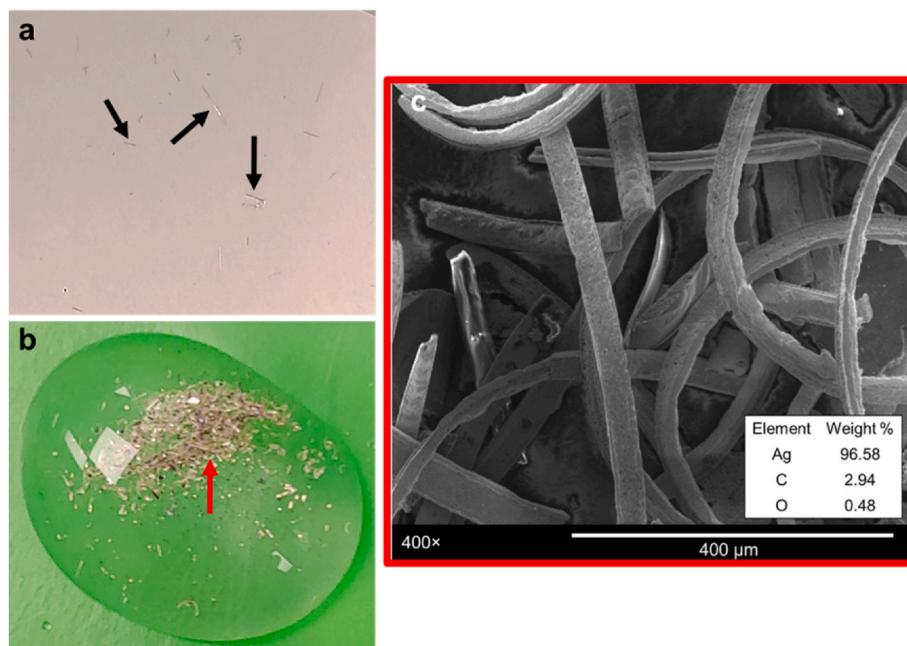


Fig. 8. (a) Image of Ag particles post leaching in 2 M H₂SO₄ 48 h 350 rpm followed by 15 min sonication. (b) Image and (c) SEM image of the Ag particles post leaching in 2 M H₂SO₄ 48 h 350 rpm followed by 30 min sonication.

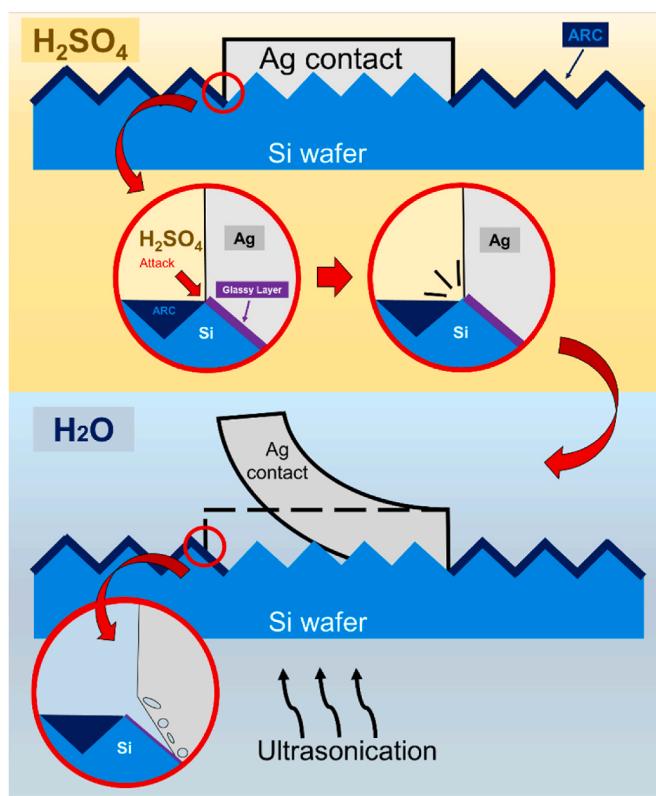


Fig. 9. Graphical schematic depicting a mechanism to describe Ag finger peeling post H₂SO₄ + ultrasonication process. It is proposed that the H₂SO₄ weakens the glassy layer boundary, and ultrasonication causes the Ag fingers to finish peeling.

water for 30 min following the acid leaching of Si wafers is enough time to completely remove the Ag fingers from the Si wafers. The Ag contacts can then be collected by centrifuging. In this way, 100% of the Ag contacts can easily be recovered from waste solar cells without the need

for secondary chemical processing, making our process more environmentally friendly. SEM/EDX and XPS quantify the peeling process and show residual Pb on the Si wafer surface where the Ag contact used to reside. Using XPS and ICPOES data, we provide a short discussion of the mechanism, though further investigation is needed to completely decode the Ag peeling process. Collectively, this research demonstrates a novel environmentally-conscious method for recovering 100% of Ag contacts from silicon cells.

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CRediT authorship contribution statement

Natalie Click: Writing – review & editing, Writing – original draft, Data curation, Conceptualization. **Ioanna Teknetzi:** Writing – review & editing, Methodology, Data curation. **Eric Pui Lam Tam:** Writing – review & editing, Data curation. **Meng Tao:** Writing – review & editing, Supervision. **Burçak Ebin:** Writing – review & editing, Supervision, Methodology.

Declaration of competing interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests:

Burçak Ebin reports financial support was provided by Swedish Energy Agency. Meng Tao reports financial support was provided by the U. S. Department of Energy's Office of Energy Efficiency and Renewable Energy (EERE) under the Advanced Manufacturing Office. Meng Tao reports a relationship with TG Companies that includes: equity or stocks. Natalie Click reports a relationship with TG Companies that includes: consulting or advisory. Meng Tao has patent #10,385,421 issued to Arizona State University. Meng Tao has patent SYSTEMS AND

METHODS FOR RECOVERING SCRAP SILVER pending to TG Companies. Meng Tao has patent CIRCULAR ELECTROCHEMICAL METAL RECOVERY pending to TG Companies. If there are other authors, they declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.solmat.2024.112834>.

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