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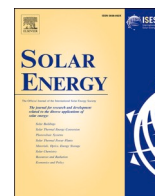
## **Silver cementation mechanism for leaching silicon solar cells in nitric acid**

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# Silver cementation mechanism for leaching silicon solar cells in nitric acid

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## ABSTRACT

Silicon solar panels are often overlooked in e-waste recycling technology, even though they contain precious silver (Ag). In order to help meet future global Ag demands and prevent contamination of the environment, all the Ag from end-of-life modules must be recovered instead of landfilled. The most mature Ag recycling recipes use high concentration nitric acid (HNO<sub>3</sub>) solutions often in combination with heating and agitation. After the Ag is leached, chemical precipitation or electrochemistry is used to recover metallic Ag. However, the process of Ag leaching in the HNO<sub>3</sub> system with competing elements from silicon solar cells is not well understood. In this paper, we investigate the thermodynamics governing Ag leaching in low-concentration HNO<sub>3</sub> without agitation or heating to expand fundamental knowledge in support of Ag recovery efforts from end-of-life solar panels. ICP-OES is used to quantify the amount of Ag leached in the HNO<sub>3</sub> solution over time. Scanning electron microscopy (SEM) and energy-dispersive spectroscopy (EDS) are used to study the changes on the silicon solar cell surface. Our results suggest when trace tin (Sn) is used in solar cell fingers, it causes Ag to cement in dendritic form.

## 1. Introduction

As technology is incorporated into more facets of life, the amount of global electronic waste (e-waste) will only continue to increase. Not only are resources wasted when e-waste is landfilled, but it is also known that improper disposal of e-waste poses many risks to human health and the environment [1]. Therefore, proper management of all e-waste is needed. One of the most overlooked types of e-waste is solar panels. For example, it is estimated that by 2050, the United States could see 10 million metric tons of solar module waste [2] and Italy could see 4.8 million tons [3]. Solar panels consist mostly of glass (70 % mass) and aluminum frame (18 % mass) [4]. The solar cells consist of just over 3.5 % mass of the entire module [4], yet this mass percentage contains precious silver (Ag). Ag is used for electrical contacts in silicon, copper-indium-gallium-diselenide, tandem, and heterojunction solar cells. Ag has a very low electrical resistivity [5], making it a good conductor and front contact Ag paste deposition is ubiquitous in the photovoltaic industry. Considering the waste amounts and Ag concentration in a module [2,6,7], it is estimated that around 4000 tons of Ag could be wasted in 2050, if modules in the United States are landfilled instead of recycled, similarly, it can reach 65,000 tons globally. Recycling of

precious Ag is necessary to help meet global demands [8,9] and prevent contamination of the natural world, as Ag<sup>+</sup> has long been known to be very toxic to fish [10].

Many different chemistries have been explored for Ag recovery from solar modules. Cho et al. [11] performed solvent extraction using tri-n-octylphosphine oxide (TOPO, C<sub>24</sub>H<sub>51</sub>OP) on a synthetic leaching solution. Yang et al. [12] used methanesulfonic acid (CH<sub>3</sub>SO<sub>3</sub>H) plus hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) to leach Ag from solar cell pieces. Zhang et al. [13] broke the panels into pieces, then leached Ag using deep-eutectic solvents. By far the most popular leachate explored in literature is nitric acid (HNO<sub>3</sub>). Dias et al. [14] used ~ 14 M HNO<sub>3</sub> at room temperature with agitation to achieve a Ag recovery rate of 94 %. Jung et al. [15] achieved a recovery rate of 90 % with 5 M HNO<sub>3</sub> at room temperature with agitation. Kuczyńska – Łażewska et al. [16] obtained over 99 % recovery with 3 M HNO<sub>3</sub>, 50 °C, and agitation. Sah et al. [17] only obtained 60 % recovery with 2 M HNO<sub>3</sub> at 70 °C with no agitation. Kastanaki et al. [18] report ideal leaching parameters of 2 N HNO<sub>3</sub> and 140 °C with real solar cells. Many HNO<sub>3</sub> leaching recipes require the use of high concentrations, heating, and sometimes agitation to achieve reasonable recovery rates [19]. However, in order to further advance Ag leaching from solar cells in HNO<sub>3</sub>, more fundamental research is needed.

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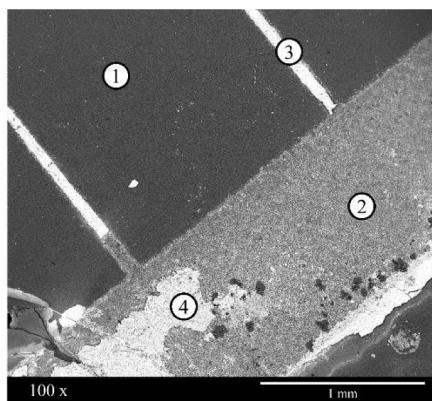


Fig. 1. SEM of end-of-life solar cell wafer #1 piece post incineration 550 °C and corresponding EDS results.

	Area 1	Area 2	Area 3	Area 4
Element	Weight %	Weight %	Weight %	Weight %
Ag	-	2.70	73.90	11.04
Cu	-	0.36	-	1.51
N	4.76	-	-	-
O	6.16	20.37	20.87	34.78
Pb	-	4.81	-	4.99
Si	87.89	66.73	0.67	1.19
Sn	1.18	5.38	4.57	46.49

In this paper, we present a comprehensive, thermodynamic study of Ag leaching in low-concentration  $\text{HNO}_3$  at room temperature with no agitation.

## 2. Materials and methodology

The following chemicals were used in these experiments: nitric acid ( $\text{HNO}_3$ ) (65 %) from MilliporeSigma or Sigma Aldrich and potassium hydroxide (KOH) from MilliporeSigma. The ratio of wafers to leachate was kept constant in all experiments at 1 g wafers: 250 mL leachate.

### 2.1. Investigation of $\text{HNO}_3$ leaching-wafer #1

To investigate Ag leaching from a Neo Solar Power solar module (max power rating: 285 W) sourced from landfill: the solar cell wafers (herein referred to wafer #1) were delaminated from the solar module by incineration at 550 °C in a quartz reactor placed in a tube furnace (Nabertherm RT 50-250/13) using air with a constant flow rate of  $1000 \pm 10$  mL/min. These were found to be the ideal parameters from tests in our group. Next, the solar wafer #1 pieces were sonicated in Milli-Q (MQ) water for 30–60 s and allowed to dry in the air. 0.01 g of these wafer #1 pieces were then placed in borosilicate beakers with 2.5 mL 2 M  $\text{HNO}_3$  for 5, 8, 24, 29, 48, and 72 h, in dark with no agitation at room temperature ( $21 \pm 2$  °C). This amount of mass was selected because the scale at which we could cut and incinerate the solar module was small. The 2 M concentration was selected as a mild concentration because it was shown previously to work well for Ag leaching [20]. A ThermoFisher Scientific iCapPro XP inductively coupled plasma optical emission spectroscopy (ICP-OES) was used for elemental analysis of the leachate. ICP-OES standards were diluted to the following concentrations: 20 ppm, 10 ppm, 5 ppm, 2.5 ppm, 1.25 ppm, and 0.625 ppm. Eight elements were included in the standards: Ag, Al, Cu, In, Na, Pb, Sn, and Si. The standards were diluted with 0.5 M  $\text{HNO}_3$ . A linear calibration curve was used for ICP-OES data fitting for each element. After leaching, the solar wafer #1 pieces were imaged with an FEI Quanta 200 FEG Environmental Scanning Electron Microscope (ESEM) and Energy Dispersive X-ray Spectroscopy (EDS) was used for elemental analysis. This leaching process was repeated with triplication for each leaching time, and the metal concentration in each triplication was recorded.

The following conditions were applied to carefully control the kinetics of this leaching: all the wafer #1 pieces were placed in the beaker with Ag fingers facing up. Only wafer #1 pieces containing fingers were selected for this leaching; no busbars were included. The beakers were covered with parafilm to prevent evaporation and placed in dark. Even though the volume to mass ratios were held constant for all trials, the active leaching area can differ slightly depending on the shape of the wafers. To quantify this, a surface area (S.A.) ratio between the wafers and leachate was determined for each trial using Equation (1):

$$\frac{S.A._{\text{wafers}}}{S.A._{\text{leachate}}} \times 100 \quad (1)$$

where  $S.A._{\text{wafers}}$  is the S.A. of the wafers in each trial and  $S.A._{\text{leachate}}$  is the S.A. of the liquid/wafer boundary. More information on the surface area calculations can be found in the [Supplementary Materials](#). HSC Chemistry 10 software was used for the thermodynamic modeling of observed Ag dissolution and precipitation.

### 2.2. Investigation of $\text{HNO}_3$ leaching-wafer #2

Because it was suspected that either Al or Sn could be the coupled redox reaction to Ag cementation; a different solar cell wafer, which did not contain Sn on the fingers (Fig. 2) but did contain an Al back contact, was leached. This wafer is herein referred to as wafer #2. The origin/manufacturer of the wafer is unknown. The glass front and fluorinated back sheet had been previously removed before arriving at Arizona State University, leaving behind the solar cell wafer #2 coated in ethylene–vinyl acetate (EVA). To liberate the wafer #2 pieces from the EVA, a portion of this wafer #2 was heated to 500 °C in a muffle furnace in air (Fisher Isotemp 550) for one hour, including ramp time, to remove the EVA. Next, 0.4 g solar cell pieces from wafer #2 were leached in 100 mL 2 M  $\text{HNO}_3$  for 29 h in dark with no agitation. 29 h was selected because it presented the clearly noticeable dendrite structure in the above experiment. After 29 h, the wafer #2 pieces were removed from the solution, rinsed with deionized water, and imaged on a Jeol JXA-8530F field emission electron probe microanalyzer.

### 2.3. Investigation of caustic leaching prior to $\text{HNO}_3$ leaching

Finally, an experiment was conducted in which the effect of alkaline KOH pre-treatment on Ag leaching was quantified. Alkaline pre-leaching for Al removal from solar cells is a popular recipe in the literature [15–17,21]. Additionally, because Sn is shown to dissolve in alkaline solutions [22,23], it is important to understand the alkaline pre-treatment's effect on Ag leaching and cementation. For this experiment: 0.05 g of solar cell wafer #1 were leached in 12.5 mL 5 M KOH at 75 °C and 250 rpm magnetic stirring for 10 min to remove the Al. Next, three individual batches each containing 0.01 g of these KOH-leached solar cell wafer #1 pieces were combined with 2.5 mL 2 M  $\text{HNO}_3$  in a borosilicate beaker, covered with parafilm, and placed in dark for 29 h with no agitation. 29 h was selected because it provided the optimal Ag dendrite structure in the 2 M  $\text{HNO}_3$  leaching with no KOH pre-leach experiments. After leaching for 29 h, the solar cell wafer #1 pieces were imaged with an FEI Quanta 200 FEG Environmental Scanning Electron Microscope (ESEM). A PHI 5000 Versa Probe III X-ray photoelectron spectrometer (XPS) was used to determine the chemical composition of the cell surface after KOH leaching.

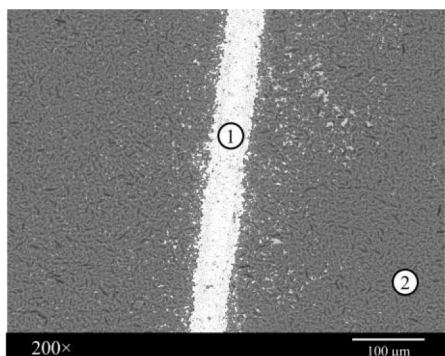


Fig. 2. SEM of end-of-life solar cell wafer #2 piece post incineration 500 °C and corresponding EDS results.

	Area 1	Area 2
<b>Element</b>	wt%	wt%
<b>Ag</b>	92.78	-
<b>Si</b>	0.69	100
<b>O</b>	5.66	-
<b>Cu</b>	0.87	-

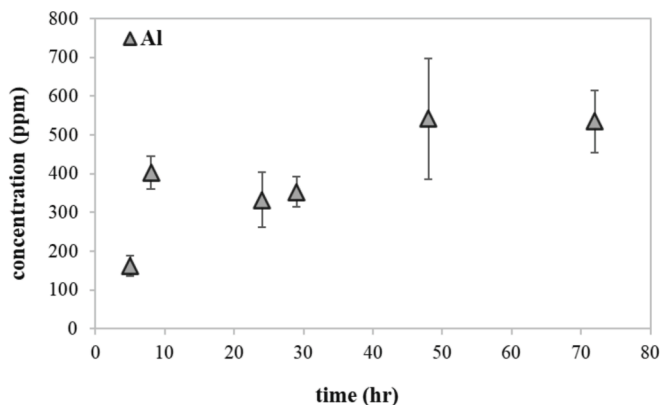


Fig. 3. ICP-OES high concentration data for 2 M HNO<sub>3</sub> leaching 5–72 h.

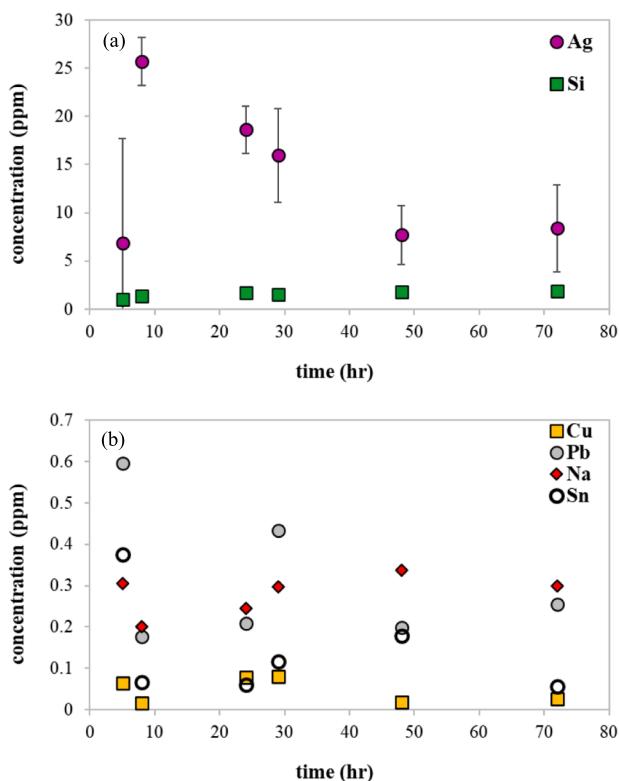


Fig. 4. ICP-OES low concentration data for 2 M HNO<sub>3</sub> leaching 5–72 h for (a) Ag and Si, and (b) Cu, Pb, Na, and Sn.

### 3. Experimental results and discussion

#### 3.1. Analysis of different solar cell wafer pieces

Before leaching, end-of-life solar wafer #1 pieces were analyzed using energy dispersive spectroscopy (EDS) (Fig. 1). On the finger, Sn was detected together with Ag. It is theorized that the Sn could have been used on the fingers as a coating to protect the Ag, or it could have been alloyed with the Ag to decrease the cost of cell manufacturing. Pb was also present on the fronts of the cells on the busbars, likely from solder. The O present indicates oxidation, most likely during incineration. Trace amounts of N detected (Fig. 1) is suspected to be from the silicon nitride (SiN) anti-reflective coating [24]. Cu is from the electrical contacts.

SEM/EDX has also performed on wafer #2 pieces. This solar cell did not have Sn on the fingers (Fig. 2); the fingers were over 90 % Ag with oxidation and trace Si and Cu. The wafer far away from the Ag finger was 100 % Si; likely the N was not able to be detected by EDX.

#### 3.2. Investigation of HNO<sub>3</sub> leaching-wafer #1

The S.A. ratios, as outlined by Equation (1), were between 4–7 % for these experiments. ICP-OES data from repeated leaching trials is shown below in Figs. 3 and 4. The data for the high elemental concentration in the leachate (Fig. 3) shows the Al readily leached into the acid. HNO<sub>3</sub> has also been used to dissolve Al from end-of-life solar modules in literature [25–27]. The existence of the other metals and ions in the solar cell leaching environment could also induce Al back sheet dissolution in HNO<sub>3</sub>, similar to the catalytic effect of mercury (Hg) on Al dissolution [28]. It is also hypothesized trace fluorine (F) (from burning the cells) or chlorine (Cl) (from the environment) could help facilitate Al dissolution in HNO<sub>3</sub> [29,30]. Cl contamination was present on the uncleaned cells according to XPS data, however to understand the exact mechanism, further investigation is needed.

In our study, the Al leaching rate plateaued at 48 h, where a maximum Al concentration of 541 ppm (0.13 mg Al per mg cell) was observed. Meanwhile, the Ag rapidly leached into solution up to 8 h, reaching a maximum concentration of 25 ppm/ 0.006 mg Ag per mg cell, then began precipitating out. This is observed by the decreasing trend-line in Fig. 4. Because Ag cementation is not reported elsewhere in literature to our knowledge, it was investigated further. Of the other elements tested, only Si had concentrations slightly above 1 ppm. Possible sources of Si in low-concentration aqueous acid are discussed in our previous paper [31].

Fig. 5 shows the SEM images of the wafer fingers at different time intervals. Maximum Ag leaching is achieved after 8 h in 2 M HNO<sub>3</sub> with no agitation at room temperature. However, after 24 h, dendritic growth is observed. At 29 h, large dendrites have grown on the existing finger paste (Fig. 6). At 48 and 72 h, the dendrites are gone, and solid particles



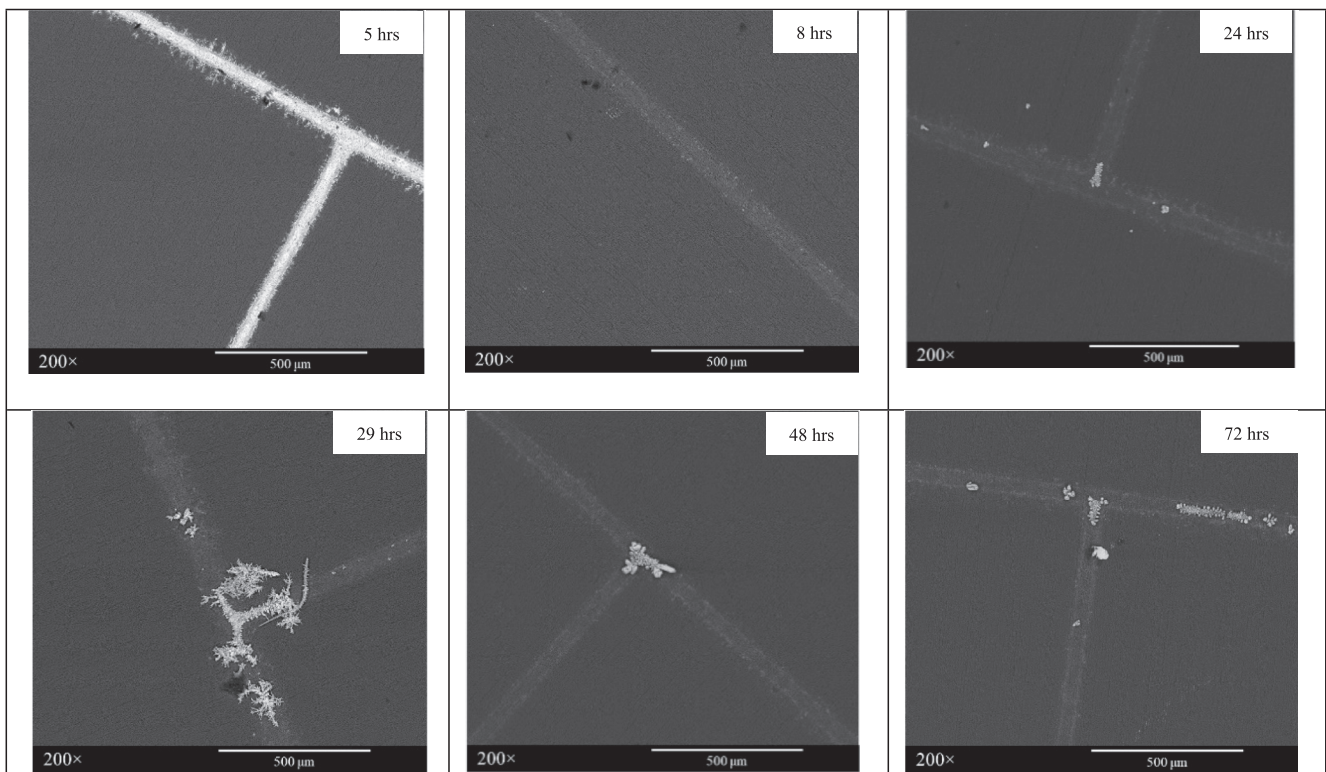


Fig. 5. SEM imaging (200 x) of Si wafers after leaching in 2 M HNO<sub>3</sub>, no agitation, for various times. Maximum Ag leaching is achieved after 8 h before cementation occurs.

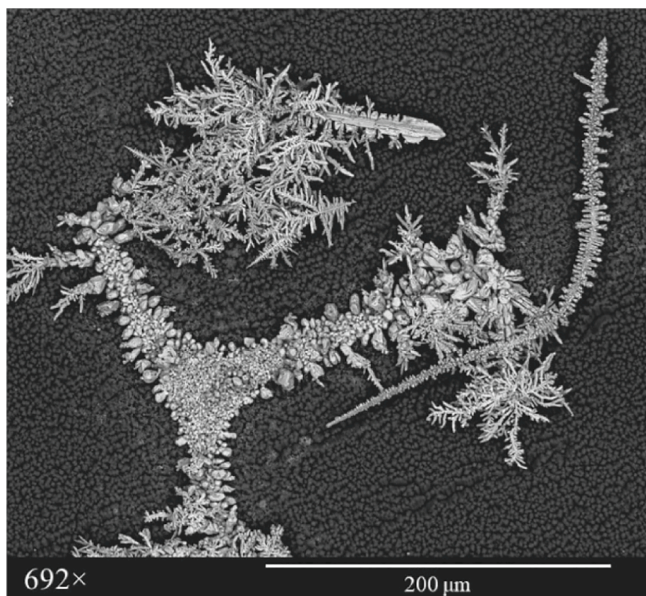


Fig. 6. SEM image of Ag dendrites growing on remaining fingers (wafer #1) after 29 h leaching in 2 M HNO<sub>3</sub>.

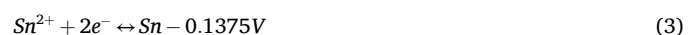
are left behind. During this time, the other element concentrations fluctuate as well. Namely, Al continues to increase in solution (Fig. 3), and the Sn concentration is constant at 0.06 ppm for 8, 24, and 72 h but almost twice as much was detected at 29 and 48 h (Fig. 4b).

The nucleation formation and growth of Ag cementation is shown best in Fig. 6, which is taken from 29 h leaching in 2 M HNO<sub>3</sub>. The remaining Ag paste is represented by globules that form a T shape facing the bottom left of the image. At the corners of the T, fresh Ag dendrites

are growing in the characteristic snowflake pattern. EDS shows these particles are 100 % Ag. Because this time produced the best dendrites, it was used for further investigation of the cementation mechanism.

### 3.3. Discussion of cementation thermodynamics

We propose two cementation suspects for the end-of-life solar wafer pieces: Al from the back contact, and trace Sn on the cell fingers (Fig. 1). Pb could also be a possible cementation agent, however since Pb was only located on the busbars, and no busbars were leached in the wafer #1 experiments, this is unlikely. Fig. 7 shows Gibbs free energy plotted against temperature for four possible cementation pathways using Al and Sn. Further cementation reaction and thermodynamic data (Gibbs free energy, enthalpy, entropy, and reaction constant (K)) of Ag using Al and Sn are given in Supplementary Material. It can be seen that electron exchange from metallic Al and Sn to Ag<sup>+</sup> is thermodynamically favorable, with Al being the most favorable. The standard reduction potentials (Eqs. (2)–(5)) also suggest that, if there are any species in the system with reduction potentials more negative than Ag, it will be favorable for Ag to remain in metallic form [32].



### 3.4. Investigation of HNO<sub>3</sub> leaching-wafer #2

In this experiment, solar wafer pieces from an end-of-life solar cell (which contained an Al back contact and no Sn on the front contacts) were leached in 2 M HNO<sub>3</sub> for 29 h, then imaged with SEM to look for the presence of dendrites. SEM of the wafer surface after leaching for 29

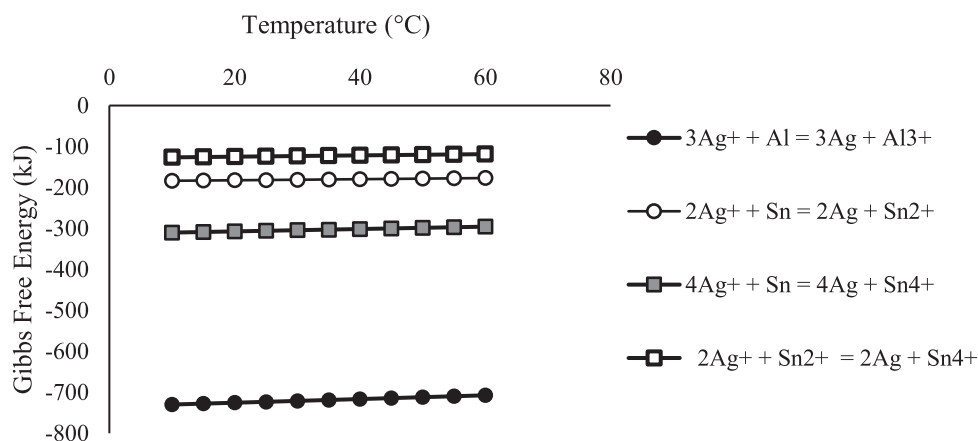


Fig. 7. Gibbs free energy versus temperature for various cementation processes.

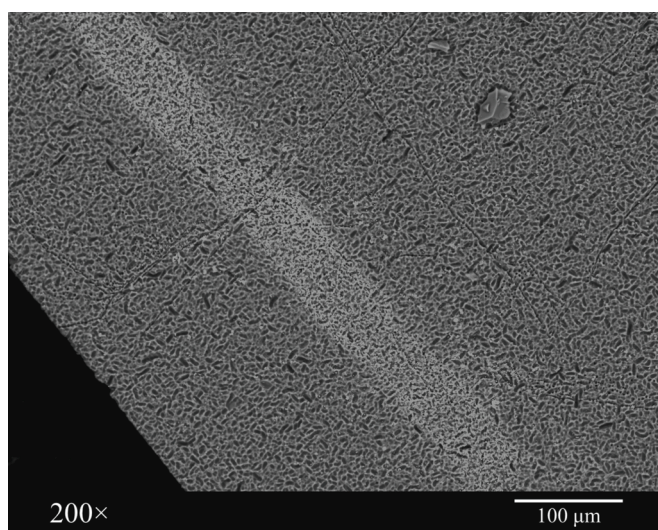


Fig. 8. SEM image of wafer #2 piece after leaching in 2 M HNO<sub>3</sub>, 29 h in dark, no agitation. No Ag dendrites are present; EDX suggested the area was 90 % Si.

h is shown in Fig. 8. Before leaching (Fig. 2), the front contact is over 90 % Ag, but after leaching, less than 1 % of the Ag remains in the area where the Ag contact used to be (faint diagonal line in Fig. 8). This area had an average Si weight percentage of 90 %, suggesting complete removal of the Ag finger. No Ag dendrites were observed, suggesting the cementation observed with wafer #1 samples is from trace Sn on the fingers.

### 3.5. Investigation of caustic leaching prior to HNO<sub>3</sub> leaching

A popular leaching recipe in literature is Al back contact removal using a caustic solution [15–17,21]. Caustic solutions are also known to leach Sn [22,23]. Therefore in this paper, we selected the use of KOH for the caustic pre-leach to remove the Al back contact and trace Sn from end-of-life solar cell wafer #1 pieces. Interestingly, XPS spectra obtained from a solar wafer piece leached in KOH (Fig. 9) suggests the formation of an Ag oxide layer. SEM image of the front contact still intact post leaching is shown in Fig. 10. With the formation of this oxide layer, none of the wafer #1 front contacts leached in the HNO<sub>3</sub> solution following caustic treatment, and no dendrites were observed. This suggests that although 2 M HNO<sub>3</sub> was suitable for Ag leaching of freshly-obtained solar cells, the caustic pre-treatment prevents Ag recovery using low-concentration HNO<sub>3</sub> or requires a longer duration to remove the protective layer and start Ag leaching. Sn was not detected by XPS, suggesting it leached into the KOH solution. The carbon (C) and bismuth

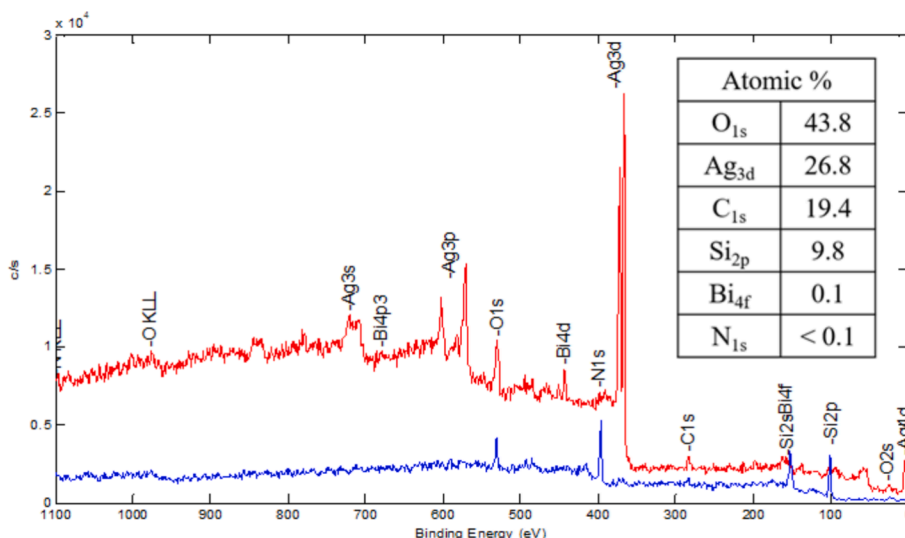


Fig. 9. XPS spectrum of Ag contact after KOH leaching. Ag:O = 1:1.5 suggesting the formation of an oxide layer over the Ag front contact.



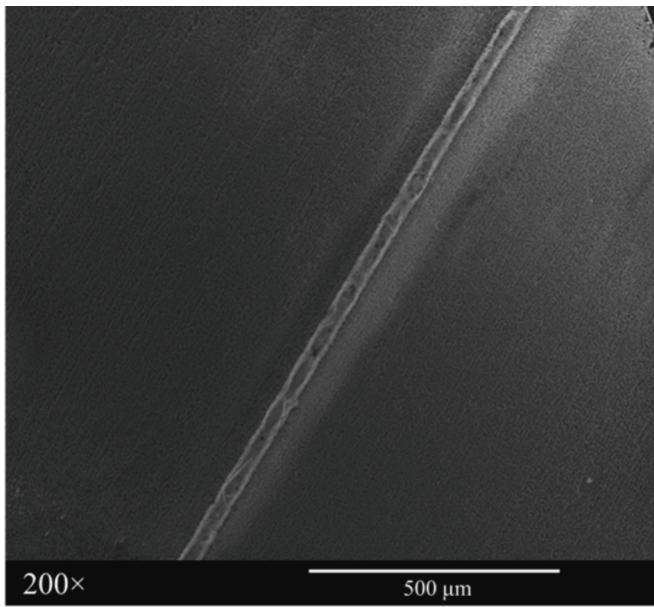


Fig. 10. Wafer #1 piece after KOH pre-leach, followed by 2 M HNO<sub>3</sub> leach for 29 h.

(Bi) in the XPS spectrum are likely contaminants, and the Si and nitrogen (N) are from the silicon nitride layer on the solar cells.

### 3.6. Proposed mechanism of Ag cementation by Sn

Because no Ag dendrites were present after wafer #2 pieces (which did not contain Sn on the front contacts) were leached, yet dendrites were present when Ag + Sn front contacts were leached (wafer #1), it is herein proposed that the trace Sn on the front contacts of the end-of-life solar wafer pieces (Fig. 1) causes Ag cementation. A schematic of the proposed mechanism is shown below in Fig. 11. Although tin is negligibly soluble in nitric acid, Mori et al. [33] found that the dissolution of

Sn in HNO<sub>3</sub> increased in the presence of Ag. Both metals dissolve in the experimental conditions, and it is proposed that Ag leaches faster than Sn, and once in solution, the Ag<sup>+</sup> ions are electrochemically cemented by residual Sn to form Ag dendrites. Once in solution, Sn is known to form a precipitate in HNO<sub>3</sub> mediums [34,35], which explains why very little Sn was measured on ICP-OES. Fig. 7 shows Sn is thermodynamically able to reduce Ag, supporting this claim.

## 4. Conclusions

In this paper, the thermodynamics of Ag leaching in low concentration HNO<sub>3</sub> at room temperature without agitation is explored, and a mechanism describing observed cementation is proposed. First, trace Sn is discovered on the front contacts of the cells. Experimental and thermodynamic data suggest this Sn in the front contacts causes Ag cementation in 2 M HNO<sub>3</sub> solution at room temperature and pressure with no agitation. Maximum Ag leaching occurs after 8 h, after which the cementation mechanism begins to dominate. Unleached front contact particles act as excellent nucleation sites for Ag dendrite growth, achieving maximum dendrite formation after 29 h of leaching. Lastly, when the Al back contact is first removed using KOH, no Ag leaches and no dendrites form after sequential leaching in 2 M HNO<sub>3</sub>. XPS data suggests the formation of a passivation layer on the front contacts. Sn does appear to leach into the KOH solution. Collectively, this paper demonstrates potential complications with Ag recovery from solar panels, illustrating the need for better communication from manufacturers about the contents of their modules as well as advancements in recycling technologies.

### CRediT authorship contribution statement

**Natalie Click:** Writing – original draft, Methodology, Investigation, Formal analysis, Data curation. **Ioanna Teknetzi:** Writing – review & editing, Methodology, Investigation. **Randall Adcock:** Writing – review & editing, Data curation. **Meng Tao:** Writing – review & editing, Project administration, Funding acquisition. **Burçak Ebin:** Writing – review & editing, Supervision, Project administration, Funding acquisition, Conceptualization.

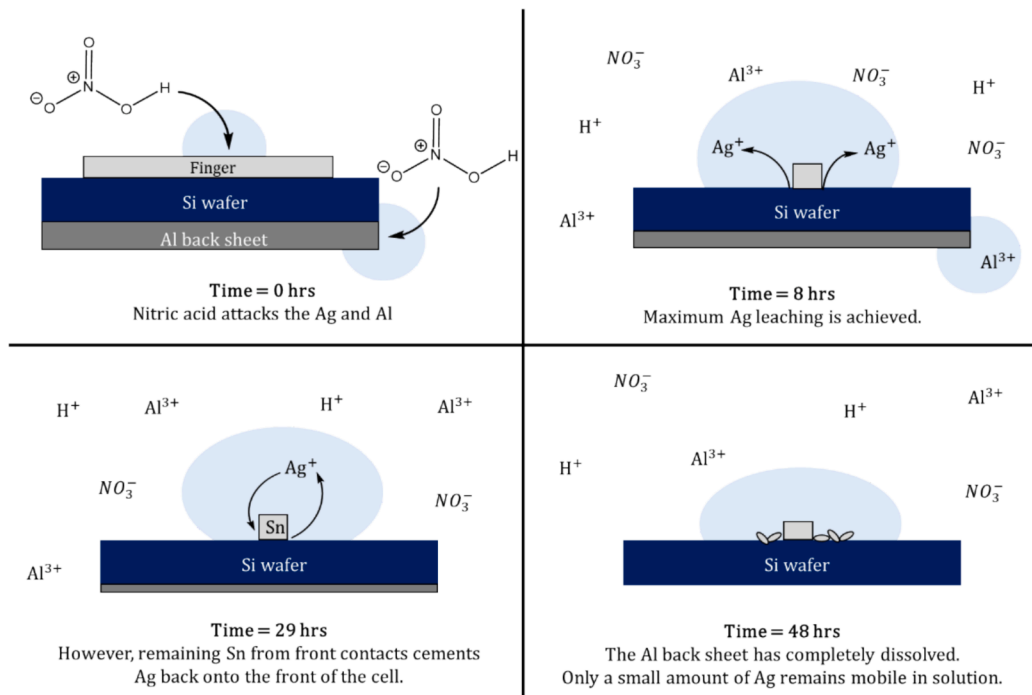


Fig. 11. Schematic for proposed cementation mechanism. Trace Sn on the front contacts is proposed to cause the Ag cementation.

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## Declaration of competing interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: Burcak Ebin reports financial support was provided by Swedish Energy Agency. Meng Tao reports financial support was provided by Office of Energy Efficiency and Renewable Energy. 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 #10385421 issued to Arizona State University. 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.

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## Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.solener.2024.113009>.

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