DISCLAIMER

Final Technical Report (FTR)

Cover Page

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Mool. C. Gupta *12/05/2024*

Signature of Certifying Official *Date*

By signing this report, I certify to the best of my knowledge and belief that the report is true, complete, and accurate. I am aware that any false, fictitious, or fraudulent information, misrepresentations, half-truths, or the omission of any material fact, may subject me to criminal, civil or administrative penalties for fraud, false statements, false claims or otherwise. (U.S. Code Title 18, Section 1001, Section 287 and Title 31, Sections 3729-3730). I further understand and agree that the information contained in this report are material to Federal agency's funding decisions and I have any ongoing responsibility to promptly update the report within the time frames stated in the terms and conditions of the above referenced Award, to ensure that my responses remain accurate and complete.

1. Acknowledgement:

"This material is based upon work supported by the U.S. Department of Energy's Office of Energy Efficiency and Renewable Energy (EERE) Solar Energy Technologies Office (SETO) under the High Efficiency Recycled Silicon Solar Cells Award Number(s) [DE-EE0010244].

2. Disclaimer:

3. Executive Summary:

The project, High-Efficiency Recycled Silicon Solar Cell, addresses the critical challenge of end-of-life (EoL) photovoltaic (PV) waste, which is expected to reach 78 million tons by 2050. Traditional recycling methods for recovering valuable materials like silver from silicon solar cells are inefficient, environmentally harmful, and economically unfeasible. Therefore, presently waste solar cells end up as landfills. This project aimed to develop a green, scalable, laser-based method for silver recovery, avoiding hazardous chemicals and minimizing operational costs. This research contributes to the circular economy in the renewable energy sector by enhancing material recovery from PV waste.

Goals:

1. Develop a laser ablation system for silver recovery from recycled silicon solar cells.
2. Develop a laser ablation system for the recovery of silver in the nanoparticle.
3. Optimize laser parameters for maximum recovery efficiency and control over the purity.
4. Characterize recovered silver nanoparticles for the composition and morphology of the nanoparticles.

5. Automate the recovery process using image processing and Convolution neural network (CNN) to improve precision and scalability.
6. Demonstrate the economic feasibility of the method for large-scale applications.
7. Demonstrate the nonchemical method that has no environmental concerns.

Accomplishments and Findings:

1. Technical Advancements in Silver Recovery: The project demonstrated the successful recovery of silver from solar cells using laser ablation and debonding. The UV 355 nm picosecond laser produced high-value silver nanoparticles, while the IR 1070 nm CW laser facilitated bulk silver recovery. This laser-based approach offers a clean and efficient alternative to traditional chemical methods.
2. Optimization of Laser Parameters: Laser parameters were optimized, with key settings including a 355 nm wavelength, 15 picosecond pulse duration, 500 mm/sec scanning speed, and 20 watts of power. These conditions produced spherical silver nanoparticles while maintaining silicon substrate integrity, ensuring both high purity and recovery speed.
3. High-Purity Silver Nanoparticles: The laser ablation method generated silver nanoparticles with 93.5% purity, confirmed through scanning electron microscopy (SEM) energy dispersive spectroscopy (EDS) analysis. The nanoparticles ranged from a few nanometers to less than 1 micron in size, providing economic value.
4. Economic Feasibility: A cost analysis showed that the processing cost for silver nanoparticle recovery is around $0.2 per gram, much lower than the market price of silver ($5 to $10 per gram). The ability to generate high-value nanoparticles from waste demonstrates the method's even higher economic viability.
5. Automation of the Laser Process: The use of image processing techniques and CNNs to automate the detection of silver electrodes significantly reduced manual labor and increased processing efficiency. The system successfully detected and helped align lasers with silver electrodes, improving precision and throughput.

Contribution to Understanding the Field: This project advances the field of PV waste management by developing sustainable, laser-based methods for material recovery. It demonstrates the versatility of laser ablation for producing high-purity silver nanoparticles and laser debonding for recovering bulk silver. The research offers valuable insights into optimizing laser parameters for material recovery, providing a cleaner alternative to chemical methods. This demonstrates the applicability of the laser-based method for the recovery of other materials.

Public Benefits: This project offers significant environmental and economic benefits by reducing reliance on landfills and minimizing the need for newly mined silver. The laser-based recovery method avoids hazardous chemicals, reduces environmental risks, and generates economically valuable silver nanoparticles, contributing to the circular economy in the solar energy industry. The process promotes sustainable practices while providing materials for electronics, catalysis, and medicine applications.

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4. Background:

In solar cell recycling, multiple techniques—mechanical, chemical, thermal, combined thermal-chemical, high-voltage pulse (HVP), and ultrasound-assisted—have been explored extensively to recover valuable materials like silver, especially in crystalline silicon photovoltaic (PV) technology [1-17].

Mechanical Methods: These approaches utilize physical forces to separate materials, including techniques like crushing or electro-hydraulic fragmentation (EHF), where high-voltage electrical discharges create shock waves that help break apart materials. Mechanical methods are generally environmentally safe since they avoid hazardous chemicals, yet they often fall short in isolating silver and other embedded metals within PV layers due to limited access to deeply bound materials [4].

Thermal Methods: Thermal processes rely on elevated temperatures to degrade encapsulating materials in PV cells, which releases embedded metals. This method, although effective at separating materials, is energy-intensive and may result in harmful emissions [14]. In large-scale applications, this environmental impact and energy demand make thermal methods less ideal for sustainable recycling practices.

High Voltage Pulse (HVP) Technology: HVP methods employ high-voltage electrical discharges within a fluid medium, creating shock waves that fragment PV cells. This technique is useful for separating fine metal particles while reducing dust and contamination levels. However, HVP is energy-demanding and requires specialized equipment, making it a less accessible choice for widespread adoption [9,15-16].

Ultrasound-Assisted Processes: An emerging alternative for silver recovery, ultrasound-assisted methods combine ultrasonic cavitation with mild chemical leaching. The ultrasonic waves help dislodge silver from PV surfaces while using relatively low concentrations of acids like nitric or sulfuric acid. This approach is advantageous as it minimizes hazardous chemical use and enhances recovery efficiency by providing a physical boost to the leaching process. Studies have shown it to be effective in recovering high-purity silver from solar cells offering a promising balance between efficiency and environmental safety [8,17].

Chemical Processes: Chemical leaching involves dissolving metals with specific reagents, such as hydrofluoric acid (HF), nitric acid (HNO_3), and sulfuric acid (H_2SO_4), to selectively recover silver and other valuable metals. While these methods can be highly effective at achieving high recovery rates, they carry significant environmental risks because of the hazardous nature of the chemicals involved. In addition to creating waste that requires careful disposal, these processes also have high operational costs due to the expenses associated with handling toxic reagents [3,8,12-13]. Despite the effectiveness of these chemical processes, they present several notable disadvantages. First, the use of hazardous chemicals and strong acids poses environmental risks, requiring careful handling and disposal to ensure worker safety and prevent contamination. Additionally, the procurement and disposal of such chemicals significantly contribute to the overall operating costs of the recycling process. These methods often involve multiple processing steps, heating, and prolonged reaction times, which lead to increased energy consumption and reduced operational efficiency. For instance, Zante et al. (2014) reported that silver recovery through nitric acid leaching at room temperature incurs costs of approximately $1.88/kg, with leaching times ranging from 10 to 20 minutes, depending on the acid concentration [18]. Lower nitric acid concentrations, while safer, require higher energy input due to the need for elevated temperatures or ultrasound assistance. Moreover, the use of alternative solvents like choline chloride brine costs around $17.52/kg, making chemical methods costly due to the large volumes required [18].

These economic and environmental drawbacks of chemical methods highlight the importance of exploring alternative silver recovery techniques, such as **laser ablation**. Laser ablation, as described in this project, offers a green and efficient alternative to chemical leaching. By using a focused laser beam, silver can be selectively removed from solar cells without the need for hazardous chemicals, high temperatures, or prolonged soaking periods. This method simplifies the process, reducing energy consumption and operational costs while ensuring the recovery of high-purity silver nanoparticles. In this study, the silver contact lines of silicon solar cells were successfully ablated in a water medium, producing silver nanoparticles. Water serves as a cooling and dispersant medium, preventing the aggregation of nanoparticles and aiding their collection.

Similarities to This Project

Physical Methods for Material Recovery:

Like this project's laser-based approach, many other existing methods for silver recovery from solar cells rely on physical processes rather than chemical reactions. Techniques such as thermal processing, mechanical separation, high voltage pulse (HVP) methods, and ultrasound-assisted processes also focus on the physical disruption or separation of materials to recover valuable metals like silver. In this regard, these methods share the overarching goal of physically extracting silver without relying on chemical dissolution.

Avoidance of Harsh Chemicals:

Some existing physical recovery methods, such as HVP and ultrasound-assisted techniques, also avoid the use of harsh chemicals like hydrofluoric acid (HF) and nitric acid (HNO_3), which are common in chemical leaching processes [8,17]. These methods, like laser ablation, emphasize environmental sustainability by minimizing chemical hazards and reducing the risks associated with chemical handling, disposal, and contamination. The shared focus on non-chemical approaches aligns with this project's goal of achieving green and sustainable silver recovery.

Minimizing Environmental Impact:

Both this project and the published methods discussed above share the common objective of reducing environmental harm. Whether through mechanical, thermal, or high-voltage processes, existing research strives to develop recycling methods that limit waste, energy consumption, and pollution. This project continues that focus by using laser ablation, which eliminates chemical waste altogether and offers an energy-efficient solution to the recovery of valuable silver from end-of-life solar cells.

Differences from This Project

This project introduces several key differences from the previously published methods for silver recovery from solar cells:

Laser-Based Recovery Method:

While prior research predominantly focuses on chemical, mechanical, and other physical methods for silver recovery, this project is the first to employ a laser-based method for this purpose. No previous studies have utilized laser ablation and laser debonding to recover silver, making this approach unique in its precision and control over the material removal process.

Complete Avoidance of Hazardous Chemicals:

Unlike most existing methods, which rely on hazardous chemicals such as hydrofluoric acid (HF) or nitric acid (HNO_3) for silver dissolution, this project avoids the use of any chemical agents entirely. The laser-based approach eliminates the risks associated with chemical handling and disposal, resulting in a cleaner, safer, and more environmentally friendly process.

No Need for High-Temperature Processing:

Many chemical methods require high temperatures to accelerate silver dissolution and recovery. This project, in contrast, operates at ambient conditions, with no need for elevated temperatures. The laser ablation and debonding processes are conducted at room temperature, reducing energy consumption and further enhancing the project's environmental sustainability.

No Prolonged Leaching Time:

Chemical methods typically involve long leaching times, where the silver must be soaked in acid solutions for extended periods to achieve adequate recovery. This project eliminates the need for such time-consuming steps, as laser ablation provides instantaneous material removal, greatly reducing the overall processing time.

Production of High-Value Nanoparticles:

Unlike traditional methods that recover silver in bulk form or as dissolved silver ions, this project produces high-value silver nanoparticles directly during the laser ablation process. These nanoparticles are of commercial interest due to their wide-ranging applications in industries such as electronics, medicine, and catalysis, making this method more economically advantageous compared to conventional silver recovery techniques.

Less Damage to the Silicon Substrate:

Chemical and mechanical processes often result in significant damage to the silicon substrate, making it difficult to reuse. This project's laser-based approach minimizes damage to the underlying silicon substrate, preserving its integrity and enhancing the possibility of reusing or repurposing the silicon wafers after silver recovery. This advantage further differentiates the laser method from more invasive recovery techniques.

Leveraging Previous Research to Accelerate and Improve This Project:

The published research on laser ablation in a water medium for the production of silver nanoparticles has been instrumental in accelerating the timeline and improving the quality of this project. Existing literature has extensively documented the use of laser ablation to produce nanoparticles of silver in liquid media [20]. These studies provided valuable foundational knowledge about how laser energy interacts with materials to generate nanoparticles, ensuring a higher degree of purity while maintaining material integrity. This project leverages these insights but applies them to solve the unique challenges posed by photovoltaic recycling, specifically focusing on recovering silver from solar cell electrodes.

Targeting Silver Nanoparticles from Complex Structures:

Unlike traditional laser ablation studies, which often focus on solid silver targets, this project uses laser ablation to recover silver from 75-micron-wide silver contact lines on screen-printed silicon solar cells. This introduces new geometrical constraints that are not present in prior research. The recovery process must be not only efficient but also produce high-purity nanoparticles and avoid damaging the silicon substrate. Published work on laser ablation in different materials helped inform this study's approach to optimizing laser parameters such as wavelength, pulse duration, and repetition rate to precisely ablate the silver while leaving the silicon intact.

Generation of Nanoparticles from Silver Electrodes Containing Mixed Materials:

Another key challenge addressed by this project is the recovery of silver from screen-printed electrodes, which consist of silver microparticles embedded in a matrix of secondary materials like lead-glass frits. This differs from typical laser ablation studies focused on pure metal targets. By leveraging the principles of nanoparticle generation from prior research, this study adapted the laser parameters and medium (water) to efficiently ablate silver nanoparticles while navigating the complexity of mixed-material electrodes. This process ensures that high-purity nanoparticles are generated, even from structurally complex surfaces.

Separation of Silver and Silicon Nanoparticles:

A crucial aspect of improving the quality of this project is the ability to separate silver from silicon impurities. During laser ablation, both silver and silicon nanoparticles are produced, requiring meticulous separation to maintain the purity of the recovered silver. Existing studies provided insights into nanoparticle stabilization in liquid media, which helped inform the decision to use water as the ablation medium [20]. This project leverages the knowledge of filtration and centrifugation techniques to separate silver and silicon nanoparticles, ensuring that the recovered silver maintains its high purity while avoiding contamination from silicon.

Application of Laser Debonding for Bulk Silver Recovery:

In addition to nanoparticle recovery, this project also uses laser debonding to recover bulk silver from the silver contact lines. This process, not commonly reported in prior studies, applies thermo-mechanical principles to debond the silver from the silicon substrate without causing thermal damage [20]. By adapting laser debonding techniques from other fields, this project efficiently separates bulk silver.

Maintaining Processing Speed and Purity:

One of the unique aspects of this project is the balance between maintaining high-purity silver recovery and ensuring that the process is efficient and scalable for industrial applications. Published work provided the basis for understanding how laser parameters (such as power and scan speed) impact nanoparticle size and purity [20]. By integrating this knowledge, the current project optimized laser ablation to recover silver nanoparticles quickly, without sacrificing quality or purity, thereby improving the feasibility of large-scale recycling operations.

Avoiding Damage to Silicon Substrate:

Unlike most other physical methods, which often damage the underlying silicon due to the mechanical or thermal stress involved in metal recovery, this project leverages laser ablation's precision to avoid damage to the silicon substrate. This difference is crucial for ensuring that the silicon can be reused or repurposed after silver recovery, further enhancing the overall sustainability of the recycling process.

In conclusion, by leveraging existing research on laser ablation and nanoparticle recovery while adapting these techniques to the unique challenges posed by photovoltaic recycling, this project has accelerated development and improved process efficiency. This comprehensive approach paves the way for more refined, scalable, and sustainable methods of silver recovery, ensuring high-purity nanoparticles and minimizing damage to other components of the solar cell structure.

5. Project Objectives:

This project aims to contribute to national clean energy goals and economic growth by developing sustainable, cost-effective methods for recycling silver from end-of-life (EoL) solar cells. As solar energy continues to expand, the disposal of photovoltaic (PV) modules, which have a lifespan of 25-30 years, becomes a critical environmental issue. By 2050, global PV waste is expected to reach approximately 78 million tons. This project addresses the need for efficient, environmentally friendly recycling methods, focusing on the recovery of valuable silver from solar cells through innovative laser-based techniques.

Significance and Innovation: The project introduces a novel, laser-based silver recovery process that offers a significant advancement over traditional recycling methods. Unlike chemical approaches that rely on hazardous acids and high-temperature treatments, laser ablation and laser debonding techniques enable the precise recovery of silver from solar cells without damaging the silicon substrate. This innovation not only increases recovery efficiency but also minimizes environmental impact. Additionally, the automation of the process using image processing and convolutional neural networks (CNNs) enhances accuracy, reduces manual labor, and accelerates processing time, making it suitable for large-scale implementation.

Expected Outcomes:

1. Development of a process for the recovery of high-purity silver nanoparticles and bulk silver from discarded solar cells using laser technology, eliminating the need for harmful chemicals.
2. Optimization of laser parameters to ensure efficient silver removal while preserving the silicon substrate, allowing the potential reuse of silicon in future applications.
3. Automation of the silver recovery process through the use of CNN-based models to detect silver electrodes, increasing processing speed and reducing labor costs paving the way for commercial and large-scale recovery of silver from solar cells.

Impact on Clean Energy Progression and Economic Benefit: The recycling of end-of-life (EoL) solar panels presents a significant opportunity, both environmentally and economically. If managed properly, the recycling of solar panels could unlock a $1 billion market opportunity [21]. By 2050, effective recycling techniques are projected to recover valuable materials worth approximately $15 billion, enabling the production of nearly 2 billion new solar panels using entirely recycled components [21]. This would not only conserve critical resources but also contribute to a more sustainable and circular economy.

This project directly supports these goals by offering an innovative, sustainable approach to recovering silver, a crucial material in solar panel production. The laser-based methods developed in this project offer a cleaner, more efficient alternative to traditional recycling techniques, reducing reliance on hazardous chemicals and minimizing energy consumption. The recovered silver nanoparticles and bulk silver will have high market value, offering additional economic benefits by reducing the demand for newly mined silver and providing materials for industries such as electronics, medicine, and catalysis [19]. By addressing the growing issue of PV waste, this project aligns with national clean energy goals, helping to mitigate the environmental impact of discarded solar panels while generating significant economic returns through resource recovery. As it is a small innovation project, it does not have the milestones and no/ no go points defined.

Task 1: Experimental Setup for Laser Fabrication of Ag Nanoparticles

Objective:

Establish a laser ablation system to recover silver nanoparticles from silicon solar cells.

Task 2: Fabrication of Ag Nanoparticles and Role of Laser Parameters

Objective:
Study the effect of different laser parameters (power, wavelength, pulse duration, scan speed) on silver ablation and nanoparticle formation.

Task 3: Composition, Morphology, Size, and Impurity Characterization of Ag Nanoparticles

Objective:
Characterize the size, shape, composition, and impurities of the silver nanoparticles generated by laser ablation.

Task 4: Automation of Laser Alignment through Image Processing and CNN-Based Detection

Objective:
Automate the detection of silver electrodes on silicon solar cells using image processing and convolutional neural networks (CNNs).

Task 5: Cost Analysis and Comparison with Other Methods

Objective:
Conduct a detailed cost-benefit analysis of the laser-based silver recovery process and compare it to traditional methods like chemical leaching.

Task 6: Report, Presentations, and Publications

Objective:
Document the project's progress and disseminate findings through timely submissions of reports, presentations, and journal publications.

6. Project Results and Discussion:

This section provides a high-level quantitative comparison of anticipated project outcomes against realized results. Quality metrics are clearly stated, and the progress against award milestones is assessed. Each task is evaluated with a clear sense of progress, referencing Go/No-Go decision points and comparing anticipated goals with realized outcomes. Confidence in the results is discussed, along with necessary improvements and future directions.

Task 1: Experimental Setup for Laser Fabrication of Ag Nanoparticles

Anticipated Outcome: The objective was to establish an experimental laser ablation system to recover silver nanoparticles from silicon solar cells. The anticipated outcome involved setting up the laser system with proper parameters and verifying the system's efficiency in removing silver without causing damage to the silicon substrate.

Milestone:
The milestone was achieved with high realization confidence. The system setup was successfully completed, and the silver removal from solar cell contact lines was confirmed.

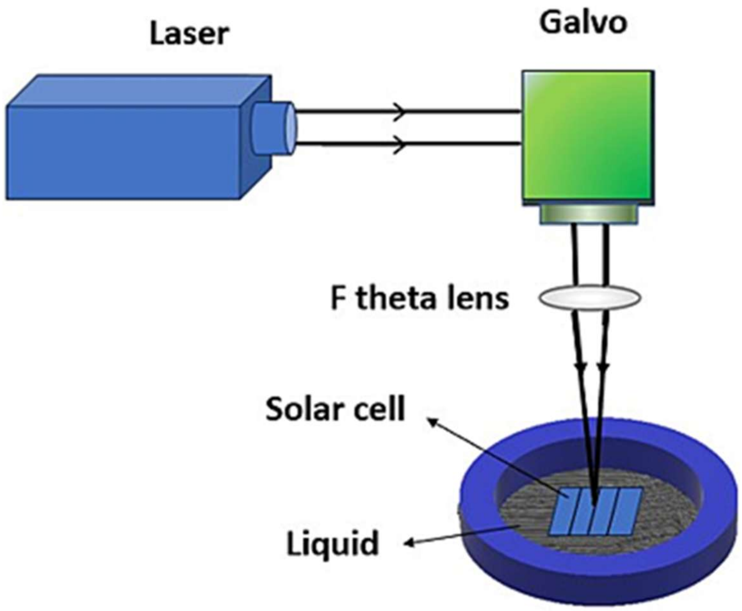

Fig. 1. Schematic diagram of the experimental setup.

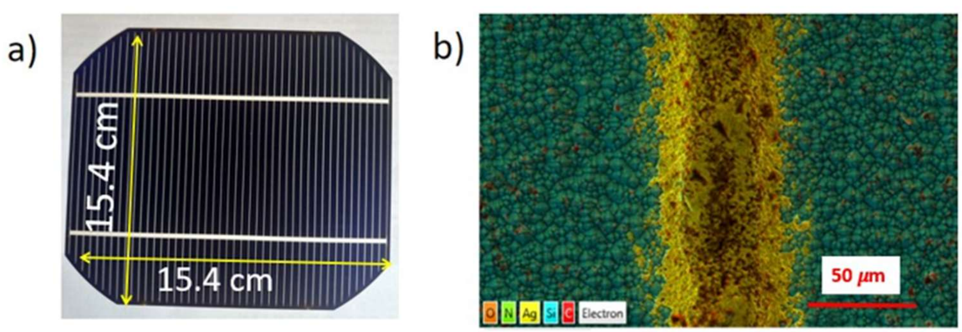

Fig. 2. (a) is the photograph of the solar cell with the silver lines used in the experiment. (b) Top-view EDS mapping of a solar cell with a silver contact line before laser ablation.

Realized Outcome: The experimental system was successfully established. The experimental setup, as shown in Figure 1, involved immersing the cleaned solar cell samples in deionized water within a Petri dish. The samples (shown in Figure 2) were cleaned using an ultrasonic cleaner to remove surface contaminants like dust. During laser ablation, the samples were maintained at the beam's focal point, with the water layer above the cells kept at a thickness of 1.8 mm. A galvanometer scan head (SCANcube 14 by SCANLAB), along with EZCad software and an F-theta lens, was used to precisely guide the laser beam along the silver lines. A visible reference laser and cameras were employed to ensure accurate positioning of the laser beam. After ablation, the Ag nanoparticles formed in the solution were collected via ultrasonication for further analysis.

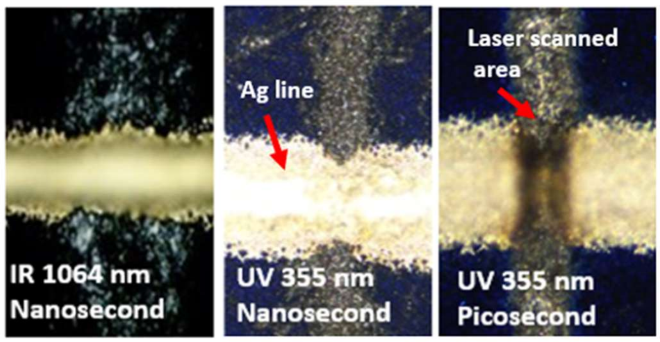

Fig. 3. Top-view optical images of a solar cell indicating (a) the effect of the type of laser used (laser scanning speed was kept at 500 mm/sec).

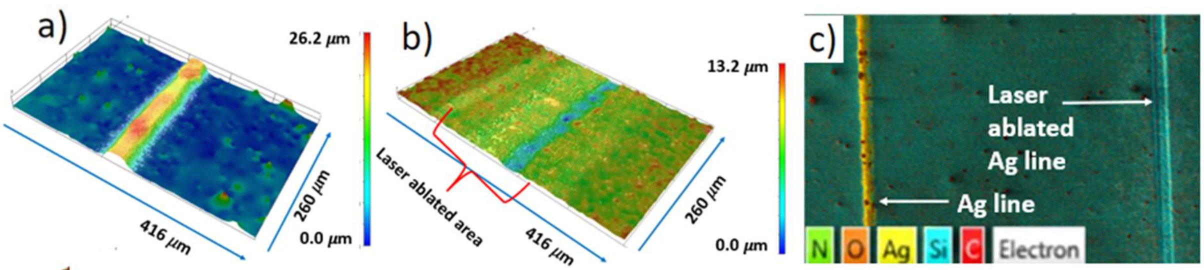

Fig. 4. Optical microscopy image of the (a) solar cell with Ag line and (b) solar cell with laser ablated Ag line. The color scale indicates the height at different locations. (c) EDS mapping illustrating the bare Ag line and laser ablated Ag line indicating the complete removal of Ag after laser ablation.

Various lasers were tested to determine the most effective parameters for silver removal as shown in Figure 3. Among the lasers tested, the UV 355 nm picosecond laser proved to be the most efficient in ablating the silver contact lines with minimal impact on the silicon substrate. It required only 15 passes to fully remove the silver, as opposed to the 120 passes needed for the nanosecond laser, significantly reducing processing time. Height measurements taken after the ablation process confirmed that the silver was entirely removed, leaving the surface level nearly identical to the surrounding silicon areas, with no significant substrate damage (Figure 4). Precise alignment using a visible reference laser, and a camera ensured that the laser beam targeted only the silver lines, avoiding the surrounding silicon, further minimizing any unintended damage. This setup allowed for the effective recovery of silver nanoparticles while preserving the integrity of the silicon substrate.

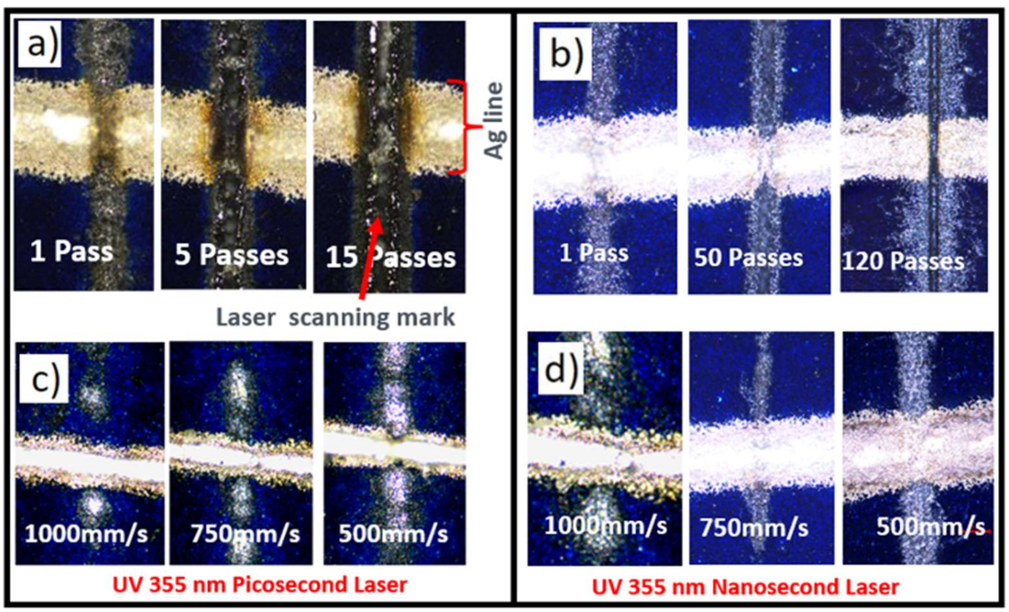

Fig. 5. Top-view optical images of a solar cell indicating the effect of the number of passes with (a) UV355 nm picosecond and (b) nanosecond laser with laser scanning speed of 500 mm/sec. Effect of laser scanning speed with UV355 nm (c) picosecond (d) nanosecond laser.

Task 2: Fabrication of Ag Nanoparticles and Role of Laser Parameters

Anticipated Outcome:

The goal of this task was to fabricate silver nanoparticles from solar cells and explore the effect of various laser parameters, including power, wavelength, pulse duration, and scan speed, on silver and silicon ablation.

Milestone:
Realization confidence is high, as the project successfully fabricated nanoparticles and optimized the laser parameters for effective silver ablation and nanoparticle formation.

Realized Outcome:

The study explored the impact of various laser types and parameters on the ablation of silver contact lines from silicon solar cells. The comparison between the UV 355 nm nanosecond and picosecond lasers revealed that the picosecond laser achieved complete removal of silver contact lines after just 15 passes, whereas the nanosecond laser required approximately 120 passes for the same result (Figure 5). The picosecond laser operated at a power of 18 watts, which contributed to its superior performance compared to the 8-watt nanosecond laser.

The scanning speed was found to be a crucial factor in determining the efficiency of the silver ablation process. Experiments were conducted with various scan speeds to find the optimal balance between removal efficiency and uniformity. A scanning speed of 500 mm/sec was identified as optimal for both the nanosecond and picosecond lasers, ensuring that silver contact lines were ablated evenly across the solar cell surface (Figure 5). This speed corresponded to 98% beam overlap, given the laser beam's diameter of 35 µm, ensuring a high level of uniformity in the ablation process. Speeds higher than 500 mm/sec resulted in uneven ablation, leaving portions of the silver line unprocessed.

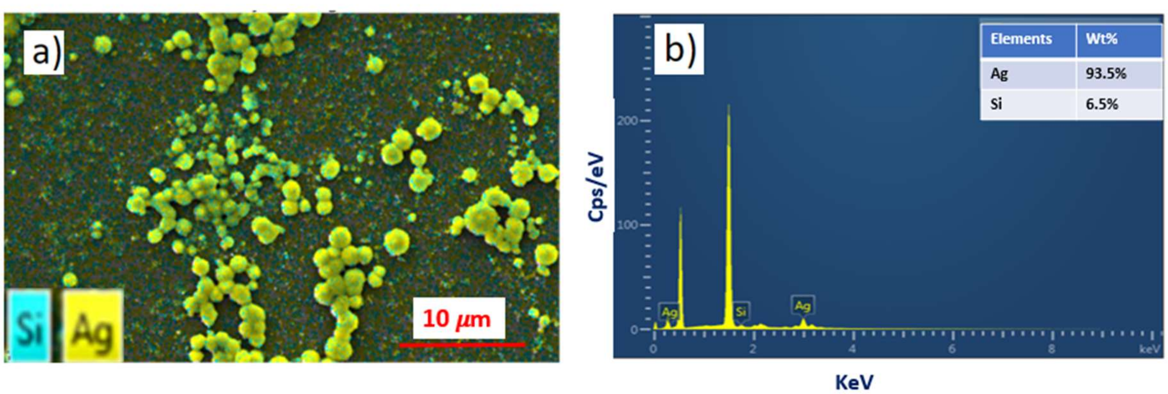

Fig. 6. a) EDS mapping of silver nanoparticles after filtering and centrifugation for improving purity. b) elemental compositional data of mapping in (a).

Task 3: Composition, Morphology, Size, and Impurity Characterization of Ag Nanoparticles

Anticipated Outcome: A detailed characterization of the silver nanoparticles generated through laser ablation, including their composition, morphology, size distribution, and impurity levels.

Milestone:
Partially Met. While the task successfully characterized the composition, morphology, and size distribution of the nanoparticles, the purity of the silver nanoparticles was still below the target of >99 wt%. The achieved purity of 93.5 wt% (shown in Figure 6) was improved through post-processing techniques such as filtration and centrifugation. Further refinement of laser alignment and ablation control is needed to reduce the silicon content and reach the desired purity levels.

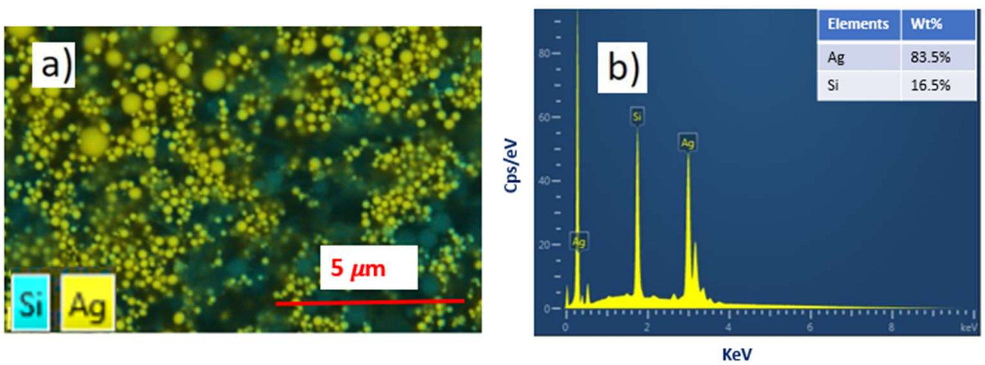

Fig. 7. (a) SEM-EDS mapped images of ablated Ag nanoparticles obtained from UV 355 picosecond laser. (b) Elemental compositional data of laser-ablated nanoparticles in (a).

Realized Outcome: Following the completion of the laser ablation experiments, ultrasonication was applied to the solution containing the ablated nanoparticles to facilitate the separation of the nanoparticles from the solar cell surface. The transformation of the solution from transparent to a yellow hue provided a visual indication of the presence of silver nanoparticles in the solution.

SEM and EDS analyses were conducted to study the size, morphology, and purity of the produced nanoparticles before post processing as shown Figure 7. The SEM results demonstrated that the nanoparticles exhibited a distinct spherical morphology, with no visible signs of agglomeration or clustering as shown. EDS confirmed that the nanoparticles were predominantly composed of silver, but silicon impurities were detected, accounting for approximately 16.5% of the total particle weight. The silicon impurities were attributed to laser misalignment during the ablation process, which caused ablation of areas adjacent to the silver contact lines, as well as some ablation beneath the contact lines.

Optimization of Laser Parameters and Water Thickness: To further optimize the nanoparticle formation and reduce silicon contamination, various experimental parameters, including water level and laser power, were adjusted. As shown in the Figure 8, it was observed that water thickness and laser power had a more pronounced effect on the size and shape of silicon particles than on silver particles, which retained their spherical shape under most conditions. When the water thickness was maintained at 1.8 mm above the sample and the laser power was set at 20 watts, the silicon particles formed large plate-like structures, while the silver nanoparticles remained small and spherical. At lower laser powers (below 15 watts), the silicon particles exhibited more regular shapes, but as the laser power increased, the formation of irregularly shaped silicon particles occurred due to plasma and shock wave effects.

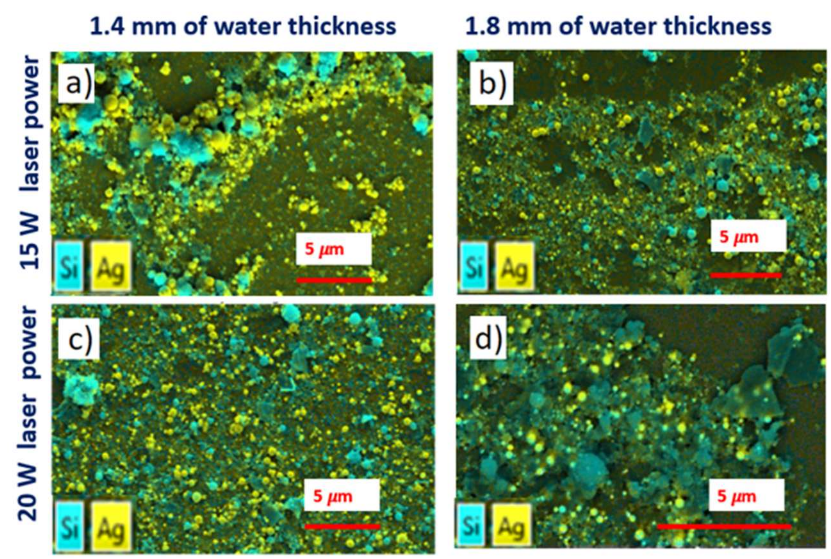

Fig. 8. (a-d) EDS mapping of ablated nanoparticles, indicating the morphology and the composition of ablated nanoparticles for different experimental conditions indicated in upper horizontal and left vertical writings.

Improving Silver Nanoparticle Purity: To enhance the purity of the recovered silver nanoparticles, a combination of filtration and density-based centrifugation was applied. Initially, the solution was passed through a 450 nm particle filter, which effectively blocked the larger silicon plate-like particles, allowing the smaller silver particles to pass through. However, due to the size distribution of the silicon particles, smaller silicon particles still remained in the solution.

In the second step, density-based centrifugation was employed, exploiting the density difference between silver and silicon. The solution was centrifuged at 10,000 rpm for three minutes, causing the heavier silver nanoparticles to settle as sediment. This process successfully isolated the silver nanoparticles from the silicon particles. Post-centrifugation, SEM and EDS analyses confirmed that the purity of the silver nanoparticles had increased to 93.5%, significantly reducing the presence of silicon impurities (Figure 6).

Task 4: Automation of Laser Alignment through Image Processing and CNN-Based Detection

Anticipated Outcome: Automate the detection of silver electrodes on silicon solar cells using image processing and convolutional neural networks (CNNs) to improve the precision and efficiency of the laser ablation process.

Milestone:
Met. Automation of silver electrode detection was successfully implemented, significantly improving the accuracy of identifying silver electrodes. Realization confidence is high for silver electrode detection, but further improvements are needed in real-time detection and throughput analysis for large-scale implementation.

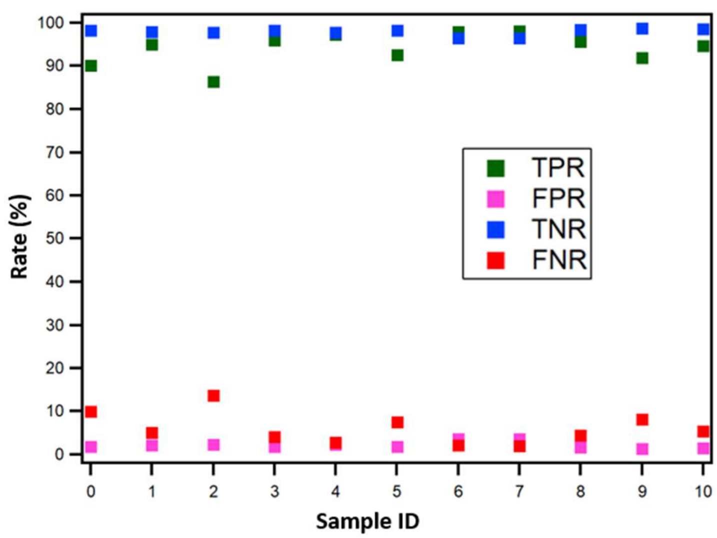

Fig. 9 True positive rate (TPR), false positive rate (FPR), true negative rate (TNR), and false negative rate (FNR) obtained for the testing dataset.

Realized Outcome:

Image Processing for Silver Electrode Detection: A MATLAB code was developed to automate the detection of silver electrodes through classical image processing techniques, including edge detection, Hough transform, and line length and angle analysis. The code efficiently converted input images to grayscale, applied adaptive thresholding, and detected straight lines representing the silver electrodes. By setting criteria for line length, angle, and distance between lines, the code minimized noise and enhanced detection accuracy. A key feature of the code was its ability to generate vector files compatible with EZCad software, enabling automated laser scanning of the detected silver electrodes (Figure 9).

However, traditional image processing methods faced challenges under variable lighting conditions, image contrasts, and complex backgrounds, which led to occasional misidentification of electrodes, limiting the overall accuracy and efficiency of the detection process.

Transition to CNN-Based Electrode Detection: To address the limitations of classical image processing, convolutional neural networks (CNNs) were introduced for detecting silver electrodes. The CNN model was based on a U-Net architecture, optimized for semantic segmentation tasks, and trained using a dataset of silver electrode images with manually labeled electrodes. The dataset included diverse scenarios, such as images with different geometries, lighting conditions, and backgrounds, as well as solar cells submerged in water. Data augmentation techniques, including image rotation, stretching, and contrast adjustments, were applied to expand the dataset, enhancing the model's robustness in real-world scenarios.

The trained CNN model successfully generated accurate binary segmentation masks representing the presence of silver electrodes on the solar cells. These masks were converted into vector files for laser scanning, improving the precision of laser ablation by aligning the laser with the detected silver lines and avoiding silicon areas. This automation effectively removed the need for manual alignment and significantly reduced processing time.

Validation and Performance Metrics: The CNN model was validated using a separate dataset, and the performance was measured using metrics such as True Positive Rate (TPR), False Positive Rate (FPR), True Negative Rate (TNR), and False Negative Rate (FNR) as shown in Figure 9. The model demonstrated strong performance with a TPR of 94.10%, an FPR of 2.13%, and a TNR of 97.87%, ensuring highly accurate detection of silver electrodes and minimal misidentification of background areas (Figure 14). The False Negative Rate (FNR) remained low at 5.90%, indicating that the model effectively captured the majority of the silver electrodes.

Task 5: Cost Analysis and Comparison with Other Methods

Objective:

To conduct a detailed cost-benefit analysis of the laser-based silver recovery process by collecting market data, estimating operational costs, and comparing these results to traditional methods like chemical leaching.

Milestones: Met.

1. Gathered market data for silver and nanoparticle prices.
2. Estimated the cost of operating lasers and the electricity required.

The cost estimation excludes the cost of raw materials, such as end-of-life (EoL) solar cells, labor costs (as automation is expected for commercial-scale laser processing), and logistics-related costs. This analysis focuses on process operational expenses. Extrapolated the results for large-scale production to assess scalability.

Realized Outcome:

Market Data for Cost Calculation:

Market data was successfully collected, showing that as of 2024, the price of silver nanoparticles ranged between $5-$10 per gram, while bulk silver was valued at over $900/kg. This provided a reference for evaluating the economic viability of the laser-based recovery process.

Approximation of Laser and Electricity Costs:

The cost analysis included electricity prices obtained from the U.S. Energy Information Administration (EIA), with a commercial rate of $0.13/kWh [22]. The operational cost of the laser systems, which have an expected lifespan of up to 10 years or 100,000 hours, was estimated at $0.50 per hour based on a purchase price of $50,000. These calculations confirmed the cost efficiency of the laser process, especially when compared to chemical leaching, which incurs higher costs due to the use of hazardous chemicals and extended reaction times.

Yield and Cost Calculation:

With optimized laser parameters, including a scanning speed of 500 mm/sec, it was determined that approximately 110 seconds were required to scan the silver electrodes on a 15x15 cm^2 solar cell containing 90 mg of silver. This translated into a recovery rate of up to 3 grams of silver nanoparticles per hour. For bulk silver recovery using infrared continuous wave (IR CW) lasers, a yield of 1 gram of silver per hour was achieved. The combination of efficient silver recovery and low operational costs demonstrated the laser method's viability compared to traditional methods such as chemical leaching.

Parameter	Estimated Value	Details
Market Price of Silver Nanoparticles	\$5–\$10 per gram	As of 2024, market data for evaluating the economic viability of silver nanoparticle recovery.
Market Price of Bulk Silver	\$900 per kilogram	The reference price for bulk silver to compare recovery methods.
Laser System Cost	\$50,000	Operational lifespan: 10 years or 100,000 hours.
Maximum Laser power consumption	300 W/hr.	Based on the power ratings lasers and other instruments including computer, chiller, galvanometer etc., used during the experiments.
Electricity Cost	\$0.13/kWh	Based on U.S. Energy Information Administration data.
Operational Cost per Hour	\$0.54	Includes electricity and laser system operation.
Silver Recovery Rate (Nanoparticles)	Up to 3 grams/hour	Based on smaller-scale experimental results and calculations, the total silver recovered is approximately 0.06 mg/cm, with a scanning speed of 500 mm/sec where three parallel laser lines were drawn to cover the area of silver lines, we extrapolate for the 1 hr. of operation.
Silver Recovery Rate (Bulk)	Up to 1 gram/hour	Using infrared CW laser.
Processing Cost for Nanoparticles	\$0.20 per gram	Estimated for laser-based recovery. The cost estimation excludes the cost of raw materials, such as discarded solar cells, any labor costs (as automation is generally implemented in commercial-scale laser processing), and logistics-related costs, focusing solely on the process's operational expenses.

Processing Cost for Bulk Silver	$0.54 per gram	

Table 1. Cost calculations for the silver recovery

Task 6: Reports, Presentations, and Publications

Objective:

To document the project's progress and disseminate findings through timely submission of reports, presentations, and journal publications.

Presentations:

The project findings were presented at prestigious conferences, such as:

1. 52nd IEEE Photovoltaic Specialists Conference (PVSC 52), Seattle, USA.
2. 7th International Conference on Advanced Nanoparticle Generation & Excitation by Lasers in Liquids (ANGEL - 2024), Charlottesville, USA.
3. 2024 SETO Peer Review, Solar Energy Technologies Office, Washington DC.
4. 50th IEEE Photovoltaic Specialists Conference (PVSC 50), San Juan, Puerto Rico.

Manuscript Submissions:

1. Khetri, M., & Gupta, M. C. (2024). Recycling of Silver from Silicon Solar Cells by Laser Debonding. Solar Energy, 270, 112381.
2. Khetri, M., Kanaujia P. K. & Gupta, M. C. (2024). Laser Recycling of Silver from Silicon Solar Cells. (Under Preparation).
3. Khetri, M., & Gupta, M. C. (2024). Recycling of Silver from CIGS Solar Cells Using Laser Ablation and Debonding (Under Preparation).

7. Significant Accomplishments and Conclusions:

Significant Accomplishments:

1. **Establishment of Laser Ablation System for Silver Recovery**: One of the key accomplishments was the successful setup and optimization of the laser ablation system for silver recovery from end-of-life (EoL) silicon solar cells. The use of a UV 355 nm picosecond laser, operating at a scanning speed of 500 mm/sec, enabled efficient removal of silver from contact lines without damaging the silicon substrate. This accomplishment represents a significant improvement over traditional methods, such as chemical leaching, which are less efficient and

environmentally hazardous. The laser ablation system proved to be a more sustainable and scalable solution, contributing to the advancement of photovoltaic (PV) recycling efforts. In addition, the optimized conditions yielded the laser debonding for the bulk silver recovery. However, for the laser ablation process to be effective, it is crucial that the solar cells are delaminated, meaning the cells must be separated from their encapsulating materials (such as glass and EVA layers) to expose the silver electrodes. This step is essential to ensure direct laser access to the silver contact lines, as the encapsulation materials would otherwise impede the ablation process. The need for delaminated solar cells poses a current challenge, but ongoing research into efficient delamination techniques will help make this process more practical and scalable.

2. **Fabrication of High-Purity Silver Nanoparticles**: Another major outcome was the fabrication of silver nanoparticles with 93.5% purity after post-processing, which included filtration and density-based centrifugation. This purity level demonstrates the laser ablation system's capacity to recover valuable materials with minimal contamination. Although the purity target of >99% was not fully achieved, this result still shows promise for the scalability of the process. The recovery of silver nanoparticles, which have applications across multiple industries, adds significant economic value to the recycling process. In the case of laser debonding, the recovered silver has the same purity as the electrodes.

3. **Automation Using CNN Models for Silver Electrode Detection**: The integration of convolutional neural networks (CNNs) into the laser ablation process automated the detection of silver electrodes on silicon solar cells. This innovation significantly improved laser alignment precision and reduced manual intervention, allowing for a more efficient process. The CNN model achieved a True Positive Rate of 94.10% and a False Positive Rate of only 2.13%, ensuring accurate electrode detection. This accomplishment demonstrates the feasibility of scaling the process while reducing labor costs and increasing throughput.

4. **Comprehensive Cost-Benefit Analysis:**
   A detailed cost-benefit analysis demonstrated that the laser-based silver recovery process is economically viable. The estimated cost of recovering silver using this method is significantly lower than traditional chemical leaching methods, especially when considering the environmental impact and operational costs. The laser-based approach eliminates the need for hazardous chemicals, significantly reducing environmental and disposal costs, which are substantial in chemical processes.
   The cost estimation, however, excludes the cost of raw materials such as end-of-life (EoL) solar cells, labor costs (as automation is generally implemented in commercial-scale laser processing), and logistics-related costs, focusing solely on the process's operational expenses. Despite these exclusions, the analysis

highlights that the laser-based method offers a faster, cleaner, and more scalable solution compared to traditional chemical leaching, with significantly reduced waste handling and disposal requirements. The combination of lower operational costs and minimal environmental impact positions the laser process as a competitive and sustainable alternative for large-scale silver recovery.

Challenges and Lessons Learned:

1. **Silicon Impurities in Recovered Silver:**
   - A significant challenge encountered was the presence of silicon impurities in the recovered silver nanoparticles, which initially accounted for up to 16.5% of the total weight. This issue was mainly due to laser misalignment during ablation, causing unintended ablation of adjacent areas or underlying silicon. Although the project successfully improved purity through post-processing techniques like filtration and centrifugation, this added complexity to the process. The lesson learned here is that greater precision in laser alignment, coupled with more refined process control, will be essential for future applications.
2. **Control Over Nanoparticle Size Distribution:**
   - While the project achieved nanoparticle fabrication, controlling the size distribution of the nanoparticles posed a challenge. Laser parameters, such as power and water thickness, had a considerable effect on the size and morphology of the produced nanoparticles. The need for further optimization in this area became clear, as consistent control over nanoparticle size is crucial for applications in high-value markets, such as electronics or medicine.
3. **Limitations in Real-Time Automation:**
   - Although the CNN-based model was successful in detecting silver electrodes with high accuracy, the system faced challenges in scaling to real-time detection. Addressing these limitations will be critical for fully automating the silver recovery process on a larger, industrial scale.

8. Path Forward:

Fully Automated Processing to Improve Yield and Purity: A primary focus for future development is achieving fully automated processing of silver recovery. Current advancements in image processing and CNN models have already demonstrated increased precision in electrode detection and laser alignment, but further automation is needed to fully optimize the yield and purity of recovered silver nanoparticles.

Automation will reduce manual intervention, improve consistency, and increase throughput, ultimately leading to better yield and enhanced purity while minimizing operational costs.

1. **Testing and Scaling for Large-Scale Recovery:** The next logical step is to extrapolate the experimental conditions to test large-scale recovery using high-power lasers. By increasing the laser power and optimizing throughput, the process can be scaled to recover silver from larger batches of EoL solar cells. High-power lasers can significantly cut down processing time and costs, making the method more viable for industrial applications. Large-scale recovery would not only address the increasing solar panel waste but also enhance the economic feasibility of the process.

2. **Balancing Size Control and Ablation Yield:** While size control of nanoparticles is well-documented in the literature, achieving both precise size control and high ablation yield with high purity is a complex challenge. There is often a trade-off between purity and yield, as optimizing one can impact the other. For future work, refining the laser parameters to maintain a balance between these factors will be critical. Overcoming this challenge will be essential to achieving commercially viable results without compromising the quality or quantity of the recovered silver.

There are clear opportunities for technology transfer and commercialization, especially in the solar recycling and materials recovery sectors. The laser-based method offers a cleaner, more efficient, and scalable alternative to chemical leaching, which is both more environmentally harmful and costlier. The ability to recover high-purity silver nanoparticles and bulk silver, with minimal damage to the silicon substrate, adds value, as the silicon could potentially be reused in new solar cells, promoting a circular economy.

The developed laser-based method for silver recovery is inherently suited for processing production waste or damaged solar cells that are not encapsulated, as these cells allow direct access to the silver contact lines without the need for additional preprocessing. In contrast, end-of-life (EoL) solar cells require delamination to separate the intact solar cells from their encapsulating materials, such as glass and ethylene-vinyl acetate (EVA), to expose the silver electrodes for laser processing. Presently, significant research efforts are focused on improving delamination techniques to address these challenges. Emerging methods aim to achieve efficient, cost-effective, and environmentally friendly delamination, ensuring the structural integrity of the recovered cells. With continued advancements in this area, it is expected that delaminated EoL cells will become readily available in the near future, facilitating the seamless application of the developed laser-based recovery method to EoL solar panels and enhancing the overall scalability and sustainability of photovoltaic recycling processes.

9. Products:

Publications/Papers:

1. Khetri, M., & Gupta, M. C. (2024). *Recycling of Silver from Silicon Solar Cells by Laser Debonding*. *Solar Energy*, 270, 112381.
2. Khetri, M., & Gupta, M. C. (2024). *Laser Recycling of Silver from Silicon Solar Cells*. (Under Preparation).
3. Khetri, M., & Gupta, M. C. (2024). *Recycling of Silver from CIGS Solar Cells Using Laser Ablation and Debonding*. (Under Preparation).

Conferences/Presentations:

1. Khetri, M., & Gupta, M. C. (2024). *Laser Recycling of Silver from Silicon and CIGS Solar Cells through Bulk by Laser Debonding and Nanoparticles by Laser Ablation*. 52nd IEEE Photovoltaic Specialists Conference (PVSC 52), Seattle, USA (Oral Presentation).
2. Khetri, M., & Gupta, M. C. (2024). *Laser Generation of Silver Nanoparticles from Recycled Solar Cells*. 7th International Conference on Advanced Nanoparticle Generation & Excitation by Lasers in Liquids (ANGEL - 2024), Charlottesville, USA (Oral Presentation).
3. Khetri, M., & Gupta, M. C. (2024). *Laser Recycling of Silver from Waste Silicon Solar Cells*. 2024 SETO Peer Review, Solar Energy Technologies Office, Washington DC (Poster Presentation).
4. Khetri, M., Kanaujia, P. K., & Gupta, M. C. (2023). *Recycling of Silver from Waste Silicon Solar Cells*. 50th IEEE Photovoltaic Specialists Conference (PVSC 50), San Juan, Puerto Rico, USA (Oral Presentation; Best Student Presentation Award Finalist).

Inventions/Patents:

- [Patent Pending] *Laser-Based Method for Recycling Silver from Solar Cells*, co-inventors: Khetri, M., & Gupta, M. C.

10. Project Team and Roles:

- **Dr. Mool C. Gupta (Professor, University of Virginia)**
  Role: Principal Investigator (PI) for the project.
  Contribution: Principal Investigator

- **Mahantesh Khetri (Graduate Research Assistant, University of Virginia)**
  Role: Graduate Research Assistant.
  Contribution: Researcher

- **Puneet Kumar Kanaujia (Researcher, University of Virginia)**
  Role: Researcher.
  Contribution: Researcher

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