

Production of Solar Reflective Materials Using a Laboratory- Scale Roll Coater

**Final Subcontract Report
September 30, 2004**

R. Smilgys
*Science Applications International Corporation
McLean, Virginia*



NREL

National Renewable Energy Laboratory
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Operated for the U.S. Department of Energy
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Abstract

This report summarizes work performed by Science Applications International Corporation (SAIC) in fulfillment of a contract with the National Renewable Energy Laboratory. The work described here is a continuation of work performed on an earlier contract (YAR-5-15005-01). The goal of the work has been to demonstrate that it is possible to produce a durable low-cost reflector for solar-thermal-electric power systems. The core technology is a technique called ion-beam-assisted physical vapor deposition to produce a silvered reflector with a protective alumina coating. In the previous contract, SAIC optimized the coating process for batch coating. In this contract, we transitioned the coating process from batch coating to roll coating. In the course of the contract, we successfully designed and built a laboratory-scale web handling machine; integrated the machine into the existing SAIC coating chamber; and roll-coated material using an alumina deposition rate as high as 20 nm/s. For a set of optimized parameters, the reflective material was highly reflective and well adhered.

Executive Summary

Solar-thermal-electric power systems use large solar reflectors to concentrate sunlight to generate electricity. The widespread application of solar-thermal-electric power generation depends on developing a durable, low-cost reflector. The National Renewable Energy Laboratory (NREL) is developing reflectors for the U.S. Department of Energy Concentrating Solar Power (CSP) Program. The goals are specular reflectance above 90% for at least 10 years under outdoor service conditions and a large-volume manufacturing cost of less than \$10.8/m² (\$1.00/ft²). Currently, the best candidate materials for solar reflectors are silver-coated glass and silvered-polymer films. Polymer reflectors are lighter in weight, offer greater system design flexibility, and have the potential for lower cost than glass reflectors. These materials may meet the current reflector goal, but it is questionable whether they can meet a longer lifetime goal of 30 years of outdoor service.

This SAIC program, funded by NREL, was initiated to continue the development of a promising low-cost “ultra-thin glass” reflector combining the best of both thin-glass and silvered-polymer reflectors. The reflector consists of a leveled steel substrate coated with a copper layer, followed by a layer of silver, and finally, by a protective optically transparent alumina top coating (Fig. 1). Earlier, this basic design was recognized as having high potential, but the difficulty has been to find a durable top coating. The alumina top coating is essential to sustain high reflectance in outdoor service. The coating is deposited by a technique called ion-beam-assisted physical vapor deposition (IBAD).

A previous contract (#YAR-5-15005-01) sponsored by NREL to develop an advanced solar reflective material (ASRM) was highly successful. The goal was to develop a process that could produce a durable low-cost reflector for solar-thermal-electric power systems. SAIC expanded on original work performed by Armstrong World Industries using IBAD to produce a silvered polymer reflector with a protective alumina coating. The first samples of reflector were produced at Armstrong and showed the highest performance of any polymer-based reflector ever tested at NREL. A cost analysis performed by the Materials Systems Laboratory (MSL) at Massachusetts Institute of Technology

showed that the reflector could be produced within NREL's cost goal if the reflector were produced in a roll coater with an alumina deposition rate of 30 nm/s. SAIC built an in-house coating system to meet these technical objectives in steps. By the end of the contract, SAIC had increased the deposition rate to over 20 nm/s, and the sample size to 76.2cm x 152.4 cm (30" x 60"). The technical understanding of the coating process was advanced to the point where SAIC was ready to integrate roll-handling hardware into its coating system. This would be a major step toward demonstration of a coating process compatible with commercial production of solar reflector.

The period of performance for this contract spanned more than four years, but all sample production was performed within the first two-and-a-half years. The thrust of the program was to transition the IBAD coating process from batch coating to roll coating. The motivation for the transition is that a roll-coating process has the potential to meet NREL's cost goals for an advanced solar reflective material.

Work began in the spring of 2000 on the web-handling machine (Task 1). We originally intended to subcontract the design and building of the machine to a vendor expert in roll coating. The recommended vendor was Frontier Industrial Technology. However, while under negotiation with Frontier over the requirements for the machine, it became clear that SAIC itself was better able to meet the cost, schedule, and technical requirements than Frontier. The main reason was that we needed a custom machine, not the off-the-shelf machine offered by Frontier. The unusual requirements for this program were high web tension, slow web speed, cryogenic drum cooling, and integration into the SAIC coating chamber.

SAIC began designing the web-handling machine in May 2000 and finished construction by October. The machine was assembled in the coating chamber because it was too heavy (400 lbs) to handle without a crane. Integration and testing of the machine in the chamber continued through March 2001. We note here a few of the important activities. The machine was suspended from a cart on the chamber's overhead rails to enable movement along the axis of the chamber. The cart included a second set of orthogonal rails to enable lateral movement. In this way, the machine could be moved throughout the chamber in a Cartesian coordinate system. The two evaporation sources were placed along a lateral line in the center of the chamber. The lateral axis on the cart was motorized so the machine could be remotely positioned over either evaporation source. The ion source was positioned to point above the rod-fed evaporator. The cooling drum was filled with ethyl alcohol and tested down to -60°C.

We encountered difficulties that were eventually overcome. The original drive-motor vacuum box was too weak and burst. We built a new box with thicker walls and reinforcement ribs. The pulse signal from the encoder mounted to the drive motor could not be read as a digital signal because of electrical noise, so we digitized the signal and performed a fast Fourier transform (FFT) to extract the pulse frequency. The commercial core chuck we purchased slipped under modest web tension, so we machined a keyway to prevent slippage. We replaced brass bushings with bearings to stop excessive wear in the rotary feedthrough of the drive-motor box.

An issue not resolved when the contract began was the choice of web material. Under the previous contract, samples were batch-coated onto polymer substrates. NREL

noted that a reflector on a stainless-steel substrate would cost less to install than one on a polymer substrate. In addition, the steel substrate withstands higher process temperatures compared to PET. Unfortunately, as produced, a steel substrate has a rough surface finish unsuitable to be a specular reflector. First, the surface must be leveled, which is a process whereby the surface is made specular. Under Task 5, SAIC unsuccessfully sought a vendor for leveled stainless steel. Some of the leveling methods investigated were electropolishing, electroplating, and polymer coatings. The closest substitute we found was a carbon steel substrate with a mirror-like surface finish manufactured by American Nickeloid. The finish is produced by a chrome electroplate. The substrate is available in coils 36 inches wide and as thin as 8 mils. We used this material as the web in Tasks 4 and 6.

In the original statement of work, once the roll coater was working, SAIC was to begin deposition of alumina at 20 nm/s under Task 4 (see Task Chronology). While testing the roll coater, it became apparent that it would be wiser to begin deposition at 10 nm/s and work up to 20 nm/s. For this reason, NREL modified the contract to add Task 6, included a hiatus period to test the durability of the materials produced in Task 6, and extended the period of performance. Six deposition runs were performed under Task 6 in May and June 2001. In the first run, silver was deposited directly onto the substrate, which resulted in flaking of the alumina coating deposited later. Subsequent runs used the structure of substrate / copper / silver / alumina, and adhesion was excellent.

Work resumed on Task 6 in May 2002. After a one-year hiatus in ASRM work, there were difficulties in achieving a quality ASRM. The main problem was flaking of the alumina coating. In retrospect, the cause of the flaking was nonuniformity of process conditions across the web—both the alumina deposition rate and the ion-beam assist. For example, we discovered that the alumina deposition rate was uniform over a band only 6 inches wide on the web. Also, on May 15, the working distance was reduced between the ion source and the web. This led to a more concentrated ion beam on the 6-inch-wide band.

Work began on Task 4 in June 2002 to increase the alumina deposition rate from 10 nm/s to 20 nm/s. The starting point for the scale-up was to linearly scale the ion-beam power from the best value found under Task 6 (i.e., 323 V/323 mA @ 8 nm/s). We also reduced the working distance between the rod-fed evaporator and the web from 24 inches to 11.5 inches to help increase the deposition rate. By the second run, we discovered that the steel substrate was damaged by a deformation later called “ripples,” centered along a band where the ion source and evaporator were most intense. From a test run on June 19, we deduced that the ripples form when the ion source nonuniformly heats the substrate. To solve this problem, we backed off the ion source to a longer working distance and took steps to increase web cooling. Specifically, we deposited several mils of indium onto the drum, and increased the web tension to crush the backside of the web against the indium film. These changes, plus adjustment of the ion assist, led to a good reflector for the run on August 13. The run on August 15 was excellent and proved that it was possible to produce a clear, well-adhered ASRM using a 20 nm/s alumina deposition rate.

An important piece of proprietary knowledge is the reactive gas used in the ion source. SAIC acquired this knowledge from Armstrong World Industries, who first developed IBAD alumina as a protective coating for plastic. Armstrong personnel discovered that alumina coatings on plastic were cracked when produced with oxygen.

Their investigation showed that a particular reactive gas produced coatings without cracks. The reactive gas might be the basis for the excellent adhesion of alumina on silver, when the process parameters have been optimized. Under this contract, SAIC investigated whether an improvement in adhesion was possible using the reactive-gas chemistry. Specifically, a chemical is available that is the product of reaction between the proprietary gas and alumina. In certain runs, we deposited a thin film—called the proprietary binding layer (PL)—of this chemical at the interface between silver and alumina. These runs can be found in Table 3. SAIC was unable to evaluate whether the PL improved alumina adhesion on silver because of insufficient time and money.

Task Chronology

Task 1. Design and build web-handling machine

Performed at SAIC, April-October 2000

Task 2. Install, integrate, and test web-handling machine in SAIC chamber to make a laboratory-scale roll coater

Performed at SAIC, August 2000-March 2001

Task 3. Automate control of deposition process for advanced solar reflective material

Performed at SAIC, April 2000-February 2001

Task 4. Optimize deposition process at 20 nm/s or highest rate using roll coater for advanced solar reflective material

Performed at SAIC, June-August 2002

Task 5. Evaluate coatings and processes for leveling a stainless-steel web

Performed at SAIC, April-June 2000; October-December 2003

Task 6. Optimize deposition process at 10 nm/s using roll coater for advanced solar reflective material—(Task added after subcontract initiated)

Performed at SAIC, May-June 2001; May-June 2002

Task 7. Integrate NREL ion source into SAIC coating system—(Task added after subcontract initiated)

Performed at SAIC, July-August 2002

Task Results

Front surface alumina reflectors produced by IBAD technology exhibit highly promising experimental results. The primary goals of the new subcontract were to transition the technology from a batch to a continuous process and to optimize the deposition rate at 20 nm/s. A continuing specific goal was to establish whether this material is a "viable" solar reflector material candidate with the potential to meet or exceed the reflector performance criteria and the potential to be manufactured for less than \$10.76/m² (\$1/ft²). Specific objectives to achieve this goal include: design and build a web-handling machine; install, integrate, and the test web-handling machine in SAIC's chamber to make a laboratory-scale roll coater; automate control of the deposition process; optimize the deposition process at 20 nm/s using the roll coater prepare and submit to NREL for testing twelve (12) 20.3 cm x 1 m (8"x3') samples of ASRM; and evaluate coatings and

processes for leveling a stainless-steel web. After the subcontract was initiated, it was realized that the deposition process should be optimized at 10 nm/s using the roll coater and the resulting ASRM samples should be prepared and submitted to NREL for testing prior to optimizing at 20 nm/s. In addition, in case the SAIC ion gun was insufficient for optimizing at 20 nm/s, NREL loaned SAIC their ion gun.

Task 1. Design and build web-handling machine

The essential feature of this contract was the web-handling machine. The design and building of the machine was performed at SAIC. This was the most timely and cost effective means to fabricate the custom machine needed to perform Tasks 4 and 6. A complete set of mechanical drawings is included with this report. See Fig. 3 for pictures of the assembled web-handling machine in the SAIC coating chamber. Here, we point out a few of the web-handling machine's custom qualities.

One essential quality of the web-handling machine is superior cooling of the web in the deposition zone. An IBAD alumina coating is evaporated from a molten pool of alumina at over 2000°C. At the same time, a flux of energetic ions bombards the coating. The result is a heat load of several watts per square centimeter on the web. The exact heat load depends on the alumina deposition rate and the ion source parameters. Every web-handling machine wraps the web around a cooled drum that removes the heat by conduction through the backside of the web. The special aspect of the SAIC drum is that it was designed to operate at a lower temperature than a standard commercial drum. For example, we received a quote from Frontier Industrial Co. for their coldest operating drum, only -20°C. During performance on Task 4, it proved to be critical to operate the SAIC drum at -65°C. The lower operating temperature was achieved through innovative design.

The cooling drum is a stainless-steel cylinder filled with ethyl alcohol. It consists of a 32.39-cm (12.75-in) outer-diameter (OD) hollow cylinder with a wire-sealed flange on one end. The other end is welded shut. A 6.03-cm (2.375-in) OD tube was welded into the center of the welded end. The drum rotates on a bearing on the smaller tube. At the far end of the smaller tube, a custom-made block with a pair of O-rings fits over the tube. The O-rings allow the smaller tube (and drum, by extension) to rotate while sealing its pressurized core against vacuum. The custom block remains fixed. A flange attached to the custom block passes two liquid nitrogen lines (LN2) and two thermocouple lines—one in the alcohol and one above the alcohol. A band heater was placed between the LN2 lines and the O-rings to keep the O-rings from freezing.

Task 6 was successfully performed with the drum having a simple electropolish finish; the alumina deposition rate was 10 nm/s. Under Task 4, when the deposition rate was increased to 20 nm/s, more-efficient heat conduction became necessary. To accomplish this, we evaporated a coating of indium a few mils thick onto the drum. The purpose of indium was to conform and fill the gap between the back of the steel web and the drum surface. In 2003, we sent the drum to New Brunswick Plating, Inc., and had a fresh coating of indium electroplated in anticipation of performing additional deposition runs.

Closely related to a cold drum is the need for very high web tension. When a coating of indium is present, a substantial pressure is needed to crush the indium coating between

the web and the drum. The web-handling machine was designed to deliver a maximum of 41,000 Pa (6 psi). Because the drum has 903.2 square centimeters (140 in²) of web in contact, this amounts to a total web tension of 381 kg (840 lbs) across a 30.5-cm (12-in) - wide web. Most commercial machines do not deliver this much tension because a polymer web would tear.

The SAIC machine was also capable of very slowly advancing the web. A commercial web machine designed to coat PET with aluminum measures web speed in feet per second. The SAIC machine instead ran as slow as inches per minute. This was needed because the slowest step in ASRM processing is alumina deposition. Suppose the goal is a 2000-nm thick alumina coating deposited at 20 nm/s. When a single ion source was used, the deposition zone was no wider than 15.2 centimeters (6 in). Therefore, the web must advance 15.2 cm (6 in) in 100 seconds or 9.1 cm (3.6 in) per minute. While depositing silver or copper, the web could be advanced by as fast as 78.7 cm (31 in) per minute.

Finally, it was important to design the machine so it could be integrated into the existing SAIC coating chamber. One issue was the drive motors. Most commercial machines mount their drive motors outside vacuum because ordinary motors are not vacuum compatible. In particular, the gear reduction motors we used were packed with heavy gear oil. To prevent contamination of the vacuum, we sealed the motors in a vacuum-tight box with rotary, electrical, and cooling feedthroughs.

Task 2. Install, integrate, and test web-handling machine in SAIC chamber to make a laboratory-scale roll coater

Task 2 is closely related to Tasks 1 and 3. The three tasks together encompass all of the activities needed to construct a laboratory-scale roll coater. We define the roll coater as the web-handling machine integrated in the SAIC coating chamber. The major activities were reinforcement of the overhead rail system, assembly of the web-handling machine on the rail system, installing a motor to permit lateral drive remotely, sealing the drive motors in vacuum, building the motor-drive box and interfacing it to the optical encoder, and leak checking and low-temperature checkout of the cooling drum. These activities are discussed in more detail as part of Tasks 1 and 3.

Task 3. Automate control of deposition process for advanced solar reflective material

The transition from batch coating to roll coating increased the number of experimental parameters the operator has to control or monitor during a deposition run. The purpose of Task 3 was to automate some of these parameters to improve process uniformity and reproducibility. The parameters specified for automation were chamber gas pressure, ion source gas flow, ion source power, web speed, evaporator power, deposition shutter, and microbalance thickness monitor.

The chamber pressure is the result of a balance between pumping speed and gas load. We read the total pressure using a Balzers pressure gauge (TPG251). The principle gas load is outgassing from molten alumina, proprietary gas flow to the ion source, and oxygen gas flow to the oxygen injection line. The later two gas sources are regulated by MKS mass flow controllers. We interfaced the Balzers gauge and the MKS mass flow readout to the laboratory PC through a National Instruments multifunction I/O card.

Typically, during a deposition run, the gas flow to the ion source was held constant, and the flow to the oxygen injection line was regulated to maintain a constant total pressure.

The ion-source power supply is a Commonwealth Scientific Corp. IBS1200. It has a RS232 computer interface, but we decided to leave the supply under manual control. The reason is that the supply already self-regulated to hold a constant beam current and voltage, which is the desired state during a deposition. We considered there might be value to interface the supply for automatic shutdown when the operator was not present, but the coating system was not stable enough to leave it running unattended for long, so this automation control was dropped.

The web speed is an important parameter because when the deposition rate is constant, the coating thickness is proportional to the web speed. To achieve uniform coating thickness, the web speed must be constant. The speed of one motor was monitored using an optical encoder attached directly to the motor shaft. The signal from the encoder was digitized and processed in the PC to determine the web speed. We built a motor control box using Minarik motor drivers. The speed of the driving motor was regulated via a control voltage from the PC. We wrote a Labview VI program that created a feedback loop between the encoder and motor driver to maintain a constant web speed.

The evaporation rate of alumina to first order depends on the emission current fed into the alumina melt. Ideally, one would establish a feedback loop between the deposition rate monitor and the emission current. This is difficult when evaporating alumina because the material only melts in a local hot spot. If the beam is not moved, it burrows a hole there and the evaporation distribution changes. Because the monitor is far off axis, the measurement of the rate reads falsely low.

Task 4. Optimize deposition process at 20 nm/s or highest rate using roll coater for advanced solar reflective material

Task 4 was performed after Task 6. Task 6 was added after the contract was initiated, leading to non-sequential task numbering, but was performed prior to Task 4. See Task 6 for a general description of the deposition process. Here, we focus on the specifics of reaching a 20 nm/s alumina deposition rate.

Sixteen deposition runs were performed from June to August 2002. The runs are identified in Table 1. The deposition structure was copper / silver / alumina on chrome-plated steel foil, except for one run, in which we substituted titanium for copper (27Aug02).

Overall, we found that the scale-up from 10 nm/s to 20 nm/s was very difficult. Most of the substrates in Table 1 are poor examples of ASRM. We struggled to find process parameters to create a clear, well-adhered mirror. This was finally achieved on 15Aug02 over the entire substrate and over portions of 13Aug02 and 22Aug02. We encountered the problem of balancing ion-beam assist with the deposition rate. In addition, we encountered an entirely new problem. The first run on 11June02 and some subsequent runs showed a regular pattern of deformation to the steel-foil substrate. No run performed at lower rate showed this type of damage. We still do not fully understand the cause, but we found an approach to minimize it.

For the first run at 20 nm/s (11June02), the ion-assist power was doubled from previous successful runs at 10 nm/s as a starting point. Because the alumina coating appeared brown, we believed that the ion-assist power was too low, and insufficient oxygen was being incorporated into the growing film. For the next run, the ion-assist power was increased. Once again, the alumina coating appeared brown, but more significantly, the steel foil was roiled with a regular pattern of deformations down the center of the substrate. We call this damage “ripples” (Fig. 4). A closer inspection of the 11June02 substrate also revealed ripples, albeit at a much lower level.

The purpose for runs 19June02 and 20June02 was to determine the cause of the ripples. Because deformation is an energetic process, we speculated that either the ion beam or evaporator was responsible. In 19June02, we processed short lengths of substrate with ion-assist alone, evaporator alone, and both together. The result was that ripples only formed when the ion beam was present. Because the ion beam is stopped at the surface, where its energy is converted to heat, we next sought to reduce the heat load on the substrate. This could also be the cause of the alumina coating being brown, if the coating were too hot for sufficient oxygen to incorporate. In 20June02, the drum was cooled to a lower temperature and the web speed was increased. Ripples did not form when the ion-beam voltage was 500 V. When the deposition rate was 20 nm/s, the alumina coating turned brown, but there were no ripples.

From these tests, we decided that it was necessary to cool the substrate better. Because the drum was already nearly as cold as we could achieve, the only other possibility was to increase the thermal conductivity between the drum and substrate. As designed, the drum’s surface was simply electropolished stainless steel. The backside of the steel-foil substrate is not specular. This combination does not lead to intimate contact and efficient heat transfer. To increase the thermal conductivity, we evaporated about 50 microns of indium onto the drum.

The purpose for runs 23July02 and 26July02 was to test if the indium film was successful in removing ripples. In the 23July02 run, the substrate was deposited with an ion beam up to 800 V. Although the ripples were greatly reduced compared to the 11June02 run, they were not eliminated. In the 26July02 run, four process conditions were tested. We discovered that no ripples formed if the substrate was untensioned and uncooled. However, the alumina coating was colored dark brown, so it is not a viable processing method. More importantly, we discovered that holding the web under higher tension against the drum removed ripples, while giving a transparent alumina coating. At the highest tension, the web is forced against the drum with 41,000 Pa (6 psi) of pressure.

To reach a 20 nm/s alumina deposition rate, we were forced to reduce the working distance between the rod-fed evaporator and the web to 29.2 cm (11.5 in). This distance is so short that the deposition rate varies by more than 40% across the width of the web. We also moved the ion source close to the drum with the intention of balancing the ion-assist and deposition rate in a central band about 15.2 cm (6 in) wide. The result was that the ion source nonuniformly heated the center of the web.

In summary, from these tests, we concluded that ripples form when the web is under tension and heated nonuniformly by the ion beam. The method to reduce ripples was two-fold. First, keep the web as cool as possible by using high web tension, a film of in-

dium between the drum and web, and a cold drum. Second, use a broad ion beam that covers the full width of the drum to prevent nonuniform heating.

Of the remaining runs in August, three (13Aug02, 15Aug02, and 22Aug02) gave an ASRM that passed the initial test of being a clear, well-adhered mirror. The other runs were less successful for various reasons. For example, on 7Aug02, the partial pressure of oxygen was increased too high. A higher evaporation power was needed to compensate for increased beam scattering. This led to more splatters and heat load on the web. On 12Aug02, the run was performed without pause, and the drum temperature warmed gradually during the course of the run, which led to browning and flaking. For subsequent runs, the deposition was paused whenever the web warmed. These conditions then gave the three satisfactory runs. On 23Aug02, the alumina deposition rate was 20% higher and not optimized. On 27Aug02, because of its poor adhesion titanium was an unsuccessful substitution as an alternative for the copper back layer.

Task 5. Evaluate coatings and processes for leveling a stainless-steel web

A stainless-steel foil is a durable, robust, web material for constructing a solar facet. However, the surface finish of stainless steel is not specular, which is necessary to achieve high reflectivity of a silvered surface. Leveling is the process by which a rough surface finish is made specular. The purpose for this task was to investigate coatings and processes to level stainless-steel foil to prepare it for coating. Under this task, SAIC investigated three possibilities: a proprietary polymer coating (Valophene, manufactured by Ferro Corporation), electropolishing, and electroplating.

SAIC had Ferro coat several sheets of stainless steel with Valophene. The coating is sprayed on and cured at 240°C. The coating adheres strongly to steel and yields a glassy-like surface; however, heating to 120°C, typical of deposition conditions, softens the coating. Four deposition runs were performed to evaluate Valophene (Table 2). We discovered that evaporated copper and silver do not adhere well to Valophene unless a chrome binding-layer is present.

SAIC had three sheets of stainless-steel foil (7"×12", 4 mils thick) electropolished by Able Electropolishing Corp. Electropolishing can be thought of as reverse electroplating. Instead of material being deposited, an electric current drives the removal of material. The technique is able to polish because local high points on a surface experience a higher electric field and so etch more quickly. Although the surface specularity was much improved, it was still not sufficient to be a mirror. Consequently, SAIC abandoned electropolishing as a solution.

We were unsuccessful at finding a vendor able to electroplate a stainless-steel web. However, we discovered that American Nickeloid mass marketed an electroplated carbon steel substrate with a mirror-like finish. Nickeloid stocks the substrate up to 36 inches wide and down to 8 mil thick. The excellent finish is achieved by a copper strike, followed by nickel, then chrome.

After reviewing the options, SAIC decided that the 8-mil substrate from American Nickeloid was the best available material for ASRM coating. Valophene was abandoned because the coating was not available on a web without process development. SAIC pur-

chased several hundred lineal feet of Nickeloid substrate and used it for all deposition runs under Tasks 4 and 6.

After Tasks 4 and 6 were complete, NREL reported that the Nickeloid substrate rusted during outdoor exposure. In 2003, SAIC looked again for a leveled stainless-steel foil. Two new possibilities were discovered. One is a company called Mirror Metals (www.mirrormetals.com) that markets stainless-steel sheet with an excellent mirror finish. The finish is achieved by mechanical polishing. The company does not sell a thin foil, so a web would need to be developed. The other possibility is a product called Presbrite Chrome, which is manufactured by Plated Strip International Ltd. The product is an electroplated stainless-steel substrate with a mirror finish. We requested, but never received, samples to submit for testing at NREL.

Task 6. Optimize deposition process at 10 nm/s using roll coater for advanced solar reflective material

Task 4 was performed after Task 6. Task 6 was added after the contract was initiated, leading to non-sequential task numbering, but was performed prior to Task 4. SAIC performed 12 deposition runs to optimize the coating process using a 10 nm/s alumina deposition rate (Table 3). Here, we describe the process:

With the chamber up to atmosphere, the protective cover sheet is removed from the Nickeloid substrate and the substrate is washed with isopropyl alcohol and clean-room wipes. Then, the exposed substrate is wound onto the take-up spool to screen it from dust during pump down. Loose chunks of alumina are poured into the rod-fed evaporation source.

The chamber is pumped to high vacuum using two CTI-10 cryopumps. The next day, the loose alumina is melted. After two days, the pressure is 8×10^{-4} Pa (6×10^{-6} torr).

On the day of the run, the first step in the deposition process is ion-beam cleaning of the substrate. The web-handling machine is driven laterally over the rod-fed source, because this is where the ion source points. The recipe for cleaning is 300 V, 300 mA running on a mixture of argon, oxygen, and/or proprietary gas, with a web speed of 4 in./min. Each section of the web is cleaned for two minutes. Typically, two loops are cleaned while the web is being wound backwards. The chamber pressure is about 9.3×10^{-3} Pa (7×10^{-5} torr).

The next step is deposition of copper. The web-handling machine is driven laterally over the four pocket sources, because this is where the copper and silver pots are located. With the shutter open, the pot of copper is heated until a rate begins to show. Then, the web is advanced forward at the maximum speed under manual control [79 cm/min (31 in/min)]. The copper rate is stabilized at 1 nm/s on the microbalance. This corresponds to 3 nm/s at the web when the tooling factor is accounted for. At this rate, 50 nm is deposited onto the web in one pass.

The next step is deposition of silver. The same procedure is followed as for copper, except the web is wound backwards. The silver rate is stabilized at 6 nm/s at the web to deposit 100 nm in one pass. The chamber pressure is about 2.7×10^{-3} Pa (2×10^{-5} torr).

The final step is deposition of alumina. The web-handling machine is moved laterally over the rod-fed evaporator. During or just after deposition of silver, LN2 is forced into

the drum of the web-handling machine. The temperature of the drum is measured by two thermocouples—one immersed in the alcohol and one above the alcohol. The lowest temperature recorded was -65°C . The valve to the LN2 is not under computer control. It is switched opened periodically to keep the alcohol temperature below -10°C . A second tank of LN2 is used to cool two cryopanel located on either side of the web-handling machine. The flow is adjusted manually to keep frost from forming on the exhaust.

The shutter is closed over the rod-fed source while the evaporation rate is increased to 3 nm/s. The working distance between the molten alumina and web surface is 61 cm (24 in). The ion source is turned on and brought up to the desired operating current and voltage. The web speed is brought to the set point under computer control. The shutter is opened to the source, and the evaporation rate is increased to 10 nm/s. The mass flow control of oxygen to the injector is turned on under computer control. The chamber pressure stabilizes at 0.02 Pa (1.5×10^{-4}) torr and the deposition process begins.

Under favorable circumstances, the rate remains steady for 5-10 minutes. As the beam burrows into the rod, the rate begins to drop. Then, the electron beam is manually “walked” around to level the rod surface, and the power is adjusted to restore the rate. The shutter is left open during this process because it only takes a few minutes.

The progress of a run is measured in loops of material wound onto the take-up spool. Each loop marks 101.6 cm (40 in) of substrate processed. The goal is two loops in a run or just over 1.8 m (6 ft) of substrate.

When a microbalance crystal becomes overloaded, it reads erratically. When a single crystal is continuously exposed, the total deposit of alumina is recorded (about 6,500 nm \times 3 = 19.5 microns, using a 3x tooling factor). When 2.03 m (80 in) of substrate is processed with an 20.3-cm (8-in)-wide deposition zone, the coating is 6,500 \times 3 \times (8/80) = 1.95 microns thick in one spot.

Task 7. Integrate NREL ion source into SAIC coating system

Ion-beam assist is essential to deposit a clear, well-adhered alumina coating at high rate. The ion-beam parameters—namely, beam voltage and current—must be optimized at a particular deposition rate. The SAIC coating system has a single ion source (Commonwealth Scientific Corp., 16-cm Kaufman high output). Under Task 4, we were concerned that the SAIC ion source would lack sufficient power to optimize the alumina coating. Given the uncertainty and short-term need for a second ion source, NREL agreed to lend an ion source to SAIC. The purpose of Task 7 was to prepare for the worst-case scenario, whereby a second ion source would be needed to complete Task 4.

Task 7 was performed in parallel with Task 4 during the summer of 2002. The NREL ion source was installed in the SAIC coating system, but not operated because it proved unnecessary to complete Task 4.

Conclusions and Recommendations

About 10 years ago at Armstrong World Industries, Dr. Jeffrey Ross made the first sample of a solar reflector based on IBAD alumina. That sample showed high specular reflectance and extraordinary durability in accelerated testing at NREL. Since then,

SAIC and NREL have strived to realize the potential of the technology to meet NREL's cost and performance goals for an ASRM. The major achievement under this contract was the production of ASRM using a roll coater. The conceptual bridge between batch coating and roll coating has been crossed. The coated substrates produced under deposition runs 13Aug02 and 15Aug02 were bright, well-adhered reflectors. These samples proved that it is possible to roll-coat an ASRM using IBAD alumina. They probably do not represent the best that is possible in making an ASRM because SAIC ran out of time before these samples could be reproduced or further optimized.

SAIC has not operated the roll coater in two years. Last year, the cooling drum was freshly plated with indium. This year, the web-handling machine was disassembled and stored. Nonetheless, the SAIC coating chamber is in regular operation. One recent improvement was the installation of a two-axis screw drive under stepper motor control for mounting fixtures within the chamber for coating. The screw drive is compatible with the web-handling machine, so it would not be a difficult matter to reassemble and drive the web-handling machine once again inside the chamber. With the screw drive, lateral motion of the web-handling machine would actually be simpler. SAIC still owns the rod-fed evaporator. However, it is outside the chamber in storage. The four-pocket evaporator previously used for copper and silver evaporation, as well as the 16-cm ion source, remain in the chamber and are available.

The question is if further development work is warranted at SAIC. Two topics of interest are ASRM cost reduction and material performance. Because the alumina deposition rate is the slowest step, a higher deposition rate would lower cost. Unfortunately, we do not think the existing equipment at SAIC is able to operate sustainably above 25 nm/s. Therefore, a substantial increase in rate would not be possible at SAIC. Another source of cost reduction is switching to a lower-cost grade of alumina. SAIC could evaluate this. To enhance performance, SAIC could also evaluate adhesion promotion and anti-soiling layers. Furthermore, the ion-beam assist parameters used on August 15 could likely be further improved. In the past two years, NREL has had the opportunity to perform accelerated and outdoor testing of material already produced and delivered by SAIC. From these tests, we understand that the Nickeloid substrate is problematic as a web material because it rusts. A further investigation of web materials is warranted. An appropriate place to start might be to inquire if American Nickeloid could electroplate a more corrosion-resistant substrate. The advantage is that the company already knows how to manufacture a substrate with mirror-like qualities.

Another approach is to enlist a commercial company with roll-coating expertise to continue the development of the ASRM. If NREL establishes a relationship with such a company, SAIC is willing to consult and share its technical expertise.

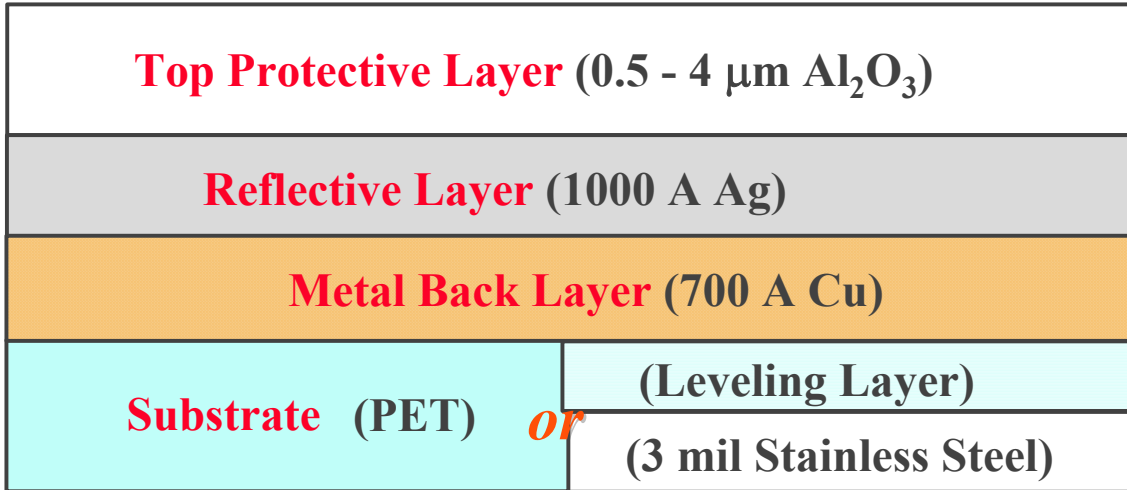


Figure 1. Advanced solar reflective material.

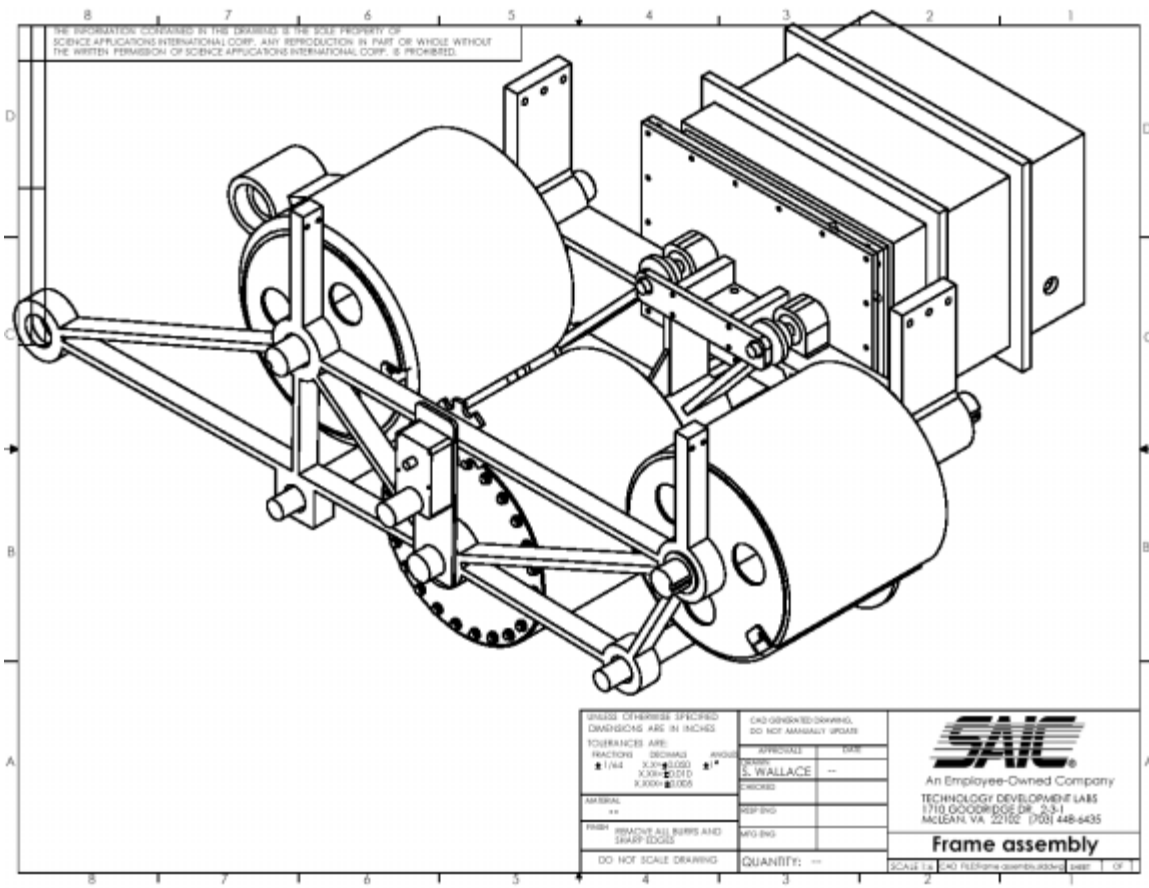


Figure 2. Schematic of web-handling machine assembly.

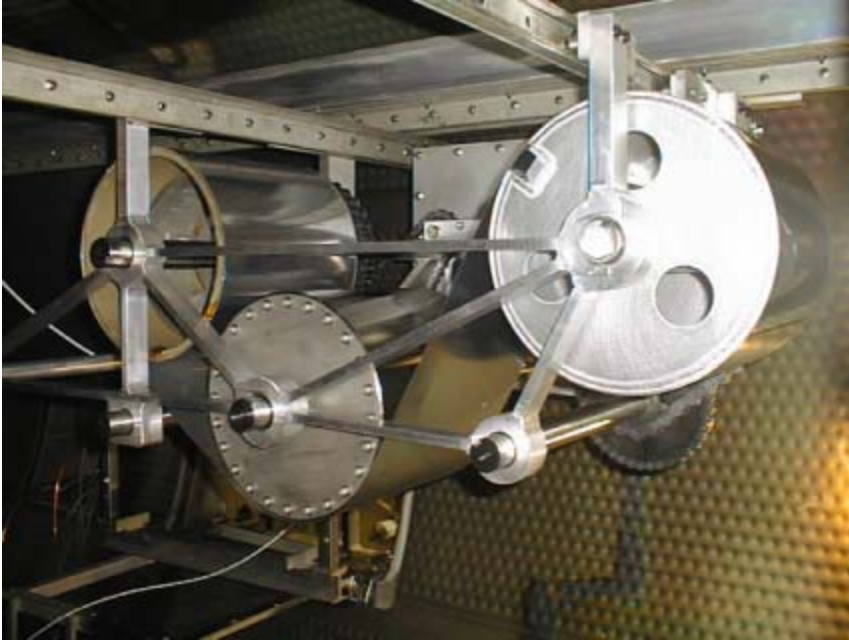


Figure 3a. Front view of web-handling machine inside the chamber. A Nickeloid substrate is wound around the cooling drum in the middle.

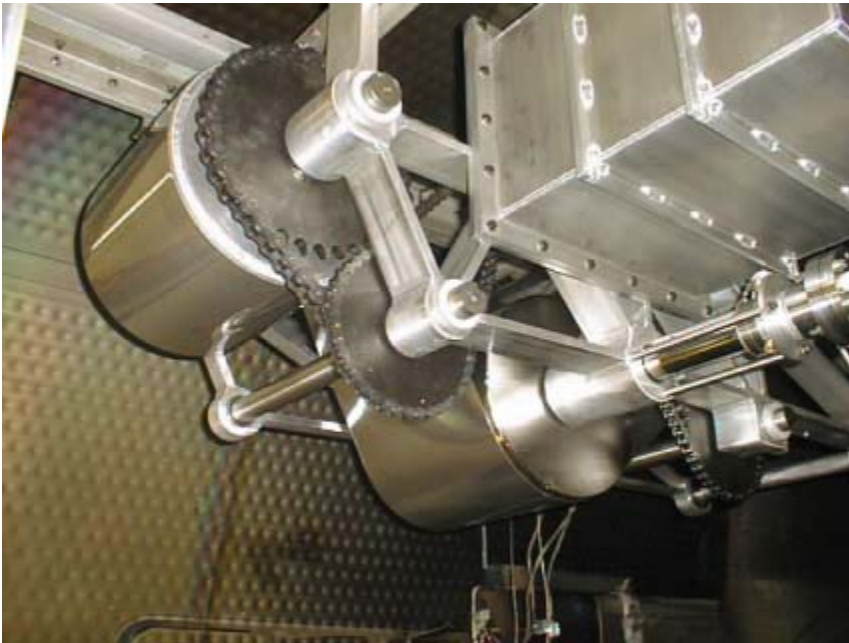
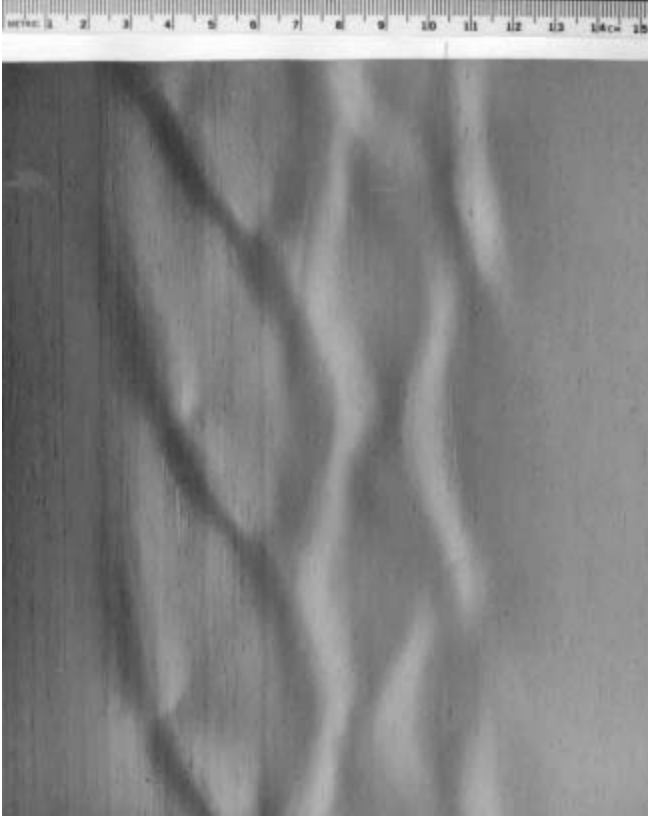


Figure 3b. Backside view of web-handling machine inside the chamber.



*Figure 4. Backside view of Nickeloid substrate with “ripples.”
The web was advanced in the vertical direction.*

Table 1. Deposition runs performed under Task 4.

Run Date	Coating Structure	Position [in.]	Ion Assist [V,mA]	Speed [pps]	Drum Temp [°C]	Comment
2Aug02	Al ₂ O ₃ /Ag/Cu	0-20	710 /710	100	-23	no ripples or flaking
	Al ₂ O ₃ /Ag/Cu	20-40	775 /775	100	-15	alumina flaking
	Al ₂ O ₃ /Ag/Cu	40-60	840 /840	100	-26	bad flaking, slight ripples
	Al ₂ O ₃ /Ag/Cu	60-80	750 /800	100	-4	bad flaking, bad ripples
7Aug02	Al ₂ O ₃ /Ag/Cu	0-40	710/710	100	-34	pressure too high/ poor quality
	Al ₂ O ₃ /Ag/Cu	40-70	760/760	100	-20	
	Al ₂ O ₃ /Ag/Cu	70-80	650/650	100	-8	
12Aug02	Al ₂ O ₃ /PL/Ag/Cu	0-20	680/680	90	-40	
	Al ₂ O ₃ /PL/Ag/Cu	20-40	710/710	90		flaking alumina
	Al ₂ O ₃ /Ag/Cu	40-60	710/710	90	-24	
	Al ₂ O ₃ /Ag/Cu	60-80	680/680	90	-16	
13Aug02	Al ₂ O ₃ /PL/Ag/Cu	0-20	650/650	140	-47	good mirror
	Al ₂ O ₃ /PL/Ag/Cu	20-40	600/600	140	-32	some tint
	Al ₂ O ₃ /Ag/Cu	40-60	600/600	145	-42	some tint
	Al ₂ O ₃ /Ag/Cu	60-80	550/550	145	-44	good mirror
15Aug02	Al ₂ O ₃ /PL/Ag/Cu	0-25	670/750	200	-55	good mirror
	Al ₂ O ₃ /PL/Ag/Cu	25-40	710/710	200	-45	good mirror
	Al ₂ O ₃ /Ag/Cu	40-60	710/710	200	-42	good mirror
	Al ₂ O ₃ /Ag/Cu	60-80	670/750	200	-28	good mirror
19-21Aug02	Al ₂ O ₃ /Ag/Cu	0-40	710/710	150	-50	poor adhesion
	Al ₂ O ₃ /Ag/Cu	40-80	670/750	145	-32	poor adhesion
22Aug02	Al ₂ O ₃ /PL/Ag/Cu	0-40	670/750	144	-44	spot flaking
	Al ₂ O ₃ /Ag/Cu	40-80	670/750	200	-40	some tint

Run Date	Coating Structure	Position [in.]	Ion Assist [V, mA]	Speed [pps]	Drum Temp [°C]	Comment
23Aug02	Al ₂ O ₃ /Ag/Cu	0-40	750/750	200	-41	higher rate/
	Al ₂ O ₃ /Ag/Cu	40-80	750/750	200	-30	some tinting/ good mirror/
27Aug02	Al ₂ O ₃ /Ag/Ti	0-40	670/750	150	-14*	poor adhesion
	Al ₂ O ₃ /Ag/Ti	40-80	710/710	200	-2*	poor adhesion

Position identifies section of substrate measured in inches from start of run. This is an approximate measure because there is a transition region as wide as the deposition zone after conditions are changed.

Speed is a “pulses per second” reading from the optical encoder. The actual substrate speed in inches/minute is found by multiplying the encoder reading by 0.0162.

PL ≡ proprietary binding layer

* Readings probably in error due to faulty drum thermocouple.

Table 2. Deposition runs performed under Task 5.

Date	Structure	Substrates
21April00	50 nm Cu/100 nm Ag/ 0.5 μm Al_2O_3	PET/SS, Valophene (2" \times 2")
28April00	2 nm Cr/ 50 nm Cu/100 nm Ag/ 0.5 μm Al_2O_3	PET/SS, Valophene (2" \times 2")
2May00	2 nm Cr/ 50 nm Cu/100 nm Ag/ 0.5 μm Al_2O_3	PET/SS, Valophene (2" \times 2")
10May00	2 nm Cr/ 50 nm Cu/100 nm Ag/ 0.5 μm Al_2O_3	PET/SS, Valophene (2" \times 2")
12June00	50 nm Cu/100 nm Ag/ 0.5 μm Al_2O_3	PET/SS, Cr-plated steel substrate
19June00	100 nm Cr / 1 μm Al_2O_3	PET/SS, Cr-plated steel substrate
7July00	100 nm Ag/ 1 μm Al_2O_3	Cr-plated steel substrate

Table 3. Deposition runs performed under Task 6.

Date	Loop	Structure	Rate [nm/s]	Appearance	Web Speed [in./min]	Ion Source	O ₂ Injection
29May01	1+2	$\text{Al}_2\text{O}_3/\text{Ag}$	6.5	flaking	1.25	oxygen, argon	Yes
1June01	1	$\text{Al}_2\text{O}_3/\text{Ag}/\text{Cu}$	5	clear, adhered	1.25	proprietary gas	Yes
6June01	1	$\text{Al}_2\text{O}_3/\text{Ag}/\text{Cu}$	5	adhered	1.25	proprietary gas	Yes
	2	$\text{Al}_2\text{O}_3/\text{Ag}/\text{Al}_2\text{O}_3$	5	lightening tracks	1.25	proprietary gas	Yes
10June01	1+2	$\text{Al}_2\text{O}_3/\text{Ag}/\text{Cu}$	10	adhered	2.5	proprietary gas	Yes
20June01	1+2	$\text{Al}_2\text{O}_3/\text{Ag}/\text{Cu}$	7-10	adhered	2	proprietary gas	Yes
25June01	1	$\text{Al}_2\text{O}_3/\text{Ag}/\text{Cu}$	10	adhered	2	proprietary gas	Yes
	2	$\text{Al}_2\text{O}_3/\text{Ag}/\text{Cu}$	10	adhered	2	oxygen	Yes
12May02	1+2	$\text{Al}_2\text{O}_3/\text{Ag}/\text{Cu}$	10	flaking	1.6	proprietary gas	Yes
15May02	1+2	$\text{Al}_2\text{O}_3/\text{Ag}/\text{Cu}$	10	flaking	2	proprietary gas	Yes
16May02	1+2	$\text{Al}_2\text{O}_3/\text{Ag}/\text{Cu}$	10	flaking	2	proprietary gas	Yes
29May02	1	$\text{Al}_2\text{O}_3/\text{Ag}/\text{Cu}$	10	flaking	1.3	proprietary gas	Yes
31May02	1+2	$\text{Al}_2\text{O}_3/\text{Ag}/\text{Cu}$	10	flaking	1.3	proprietary gas	Yes
4June02	1+2	$\text{Al}_2\text{O}_3/\text{Ag}/\text{Cu}$	10	variable	1.6	proprietary gas	

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14. ABSTRACT (Maximum 200 Words) This report summarizes work performed by Science Applications International Corporation (SAIC) in fulfillment of a contract with the National Renewable Energy Laboratory. The work described here is a continuation of work performed on an earlier contract (YAR-5-15005-01). The goal of the work has been to demonstrate that it is possible to produce a durable low-cost reflector for solar-thermal-electric power systems. The core technology is a technique called ion-beam-assisted physical vapor deposition to produce a silvered reflector with a protective alumina coating. In the previous contract, SAIC optimized the coating process for batch coating. In this contract, we transitioned the coating process from batch coating to roll coating. In the course of the contract, we successfully designed and built a laboratory-scale web handling machine; integrated the machine into the existing SAIC coating chamber; and roll-coated material using an alumina deposition rate as high as 20 nm/s. For a set of optimized parameters, the reflective material was highly reflective and well adhered.						
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