

# **Durability of Solar Reflective Materials with an Alumina Hard Coat Produced by Ion-Beam-Assisted Deposition**

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# Durability of Solar Reflective Materials with an Alumina Hard Coat Produced by Ion-Beam-Assisted Deposition

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A promising low-cost reflector material for solar concentrating power (CSP) generation is a silvered substrate protected by an alumina coating several microns thick. The alumina hard coat is deposited under high vacuum by ion-beam-assisted-deposition (IBAD). Samples of this material have been produced both by batch and continuous roll-coating processes. The substrate materials investigated were polyethylene terephthalate (PET), PET laminated to stainless-steel foil, and chrome-plated carbon steel strip. The advantage of steel strip compared to PET is that it withstands a higher process temperature and lowers the final product installation costs. In this paper, we compare the durability of batch and roll-coated reflective materials with an alumina deposition rate as high as 10 nm/s. In general, the durability of the samples is found to be excellent. Comparisons between accelerated and outdoor exposure testing results indicate that these front-surface mirrors are more susceptible to weather conditions not simulated by accelerated tests (i.e., rain, sleet, snow, etc.) than other types of solar reflectors. For long-term durability edge protection will be necessary and durability could be improved by the addition of an adhesion-promoting layer between the silver and alumina.

## INTRODUCTION

CSP systems convert thermal energy collected from sunlight concentrated by large solar reflectors into electricity. Their widespread application depends in part on developing a durable, low-cost reflector. The goal is to produce a reflector having a specular reflectance that remains above 90% for at least 10 years under outdoor service conditions, and a large-volume manufacturing cost of less than \$10.80/m<sup>2</sup> [1]. The National Renewable Energy Laboratory (NREL) has funded Science Applications International Corporation (SAIC) in McLean, VA, since 1995 to develop a promising low-cost reflector. The advanced solar reflective mirror (ASRM) under development consists of an optically transparent alumina coating deposited over a silvered polymer or metal-foil substrate, as shown in Fig. 1. A copper film is used to increase adhesion between the silver film and the substrate. The dense morphology of the alumina top coating, which is essential for sustained high reflectance during outdoor service, is produced by IBAD.

<b>Top Protective Layer (0.5-4 <math>\mu\text{m}</math> <math>\text{Al}_2\text{O}_3</math>)</b>	
<b>Reflective Layer (100 nm Ag)</b>	
<b>Metal Back Layer (50 nm Cu)</b>	
<b>Substrate (PET)</b>	<b>Chrome-Plated Steel (203 <math>\mu\text{m}</math>, 8 mils)</b>

Fig. 1: Structure of advanced solar reflective material.

Solar reflective materials are optically characterized prior to exposure testing and periodically remeasured as a function of exposure time to assess optical durability. Spectral hemispherical reflectance of samples is measured using dual-beam ultraviolet-visible-near infrared (UV-VIS-NIR) spectrophotometers. An integrating-sphere attachment allows the absolute reflectance to be measured as per ASTM E903-82 with a secondary reflectance standard (traceable to the National Institute of Standards and Technology) [2]. The solar-weighted hemispherical reflectance,  $\rho_{2\pi}$ , weighted across the entire solar spectrum ( $\lambda=250$  to  $2500$  nm), is a meaningful single measure of optical performance, where the spectral measurement is convolved with and normalized by the terrestrial solar spectrum [3]. The samples were subjected to outdoor and accelerated exposure testing until their hemispherical reflectance falls below 20% of their initial values. Typically, outdoor exposure testing (OET) was only done at NREL in Golden, CO. In some cases, in addition to NREL's OET work, OET was performed at four other outdoor exposure sites: Tempe, AZ (APS); Miami, FL (FLA); Sacramento, CA (SMUD); and Fort Davis, TX (TX). Operational exposure sites are fully equipped with appropriate meteorological and radiometric instrumentation and data-logging capability to allow monitoring of site-specific environmental stress conditions experienced by weathered samples. OET samples are frequently measured as received (i.e., dirty) and after washing (i.e., clean) which results in a saw-tooth pattern of performance vs. time of exposure. Accelerated exposure testing (AET) occurred in an Atlas Ci65 WeatherOmeter (WOM) (Ci65), Atlas Ci5000 WOM (Ci5000), and a 1-kW solar simulator (1 kW-SS). The accelerated weathering chambers allow control and monitoring of light intensity, relative humidity (RH), and temperature (T). Typical conditions are  $T = 60^\circ\text{C}$  and  $\text{RH} = 60\%$  for the Ci65 and Ci5000. Each chamber can accommodate a large number (~200-300) of samples (roughly 67 mm x 44 mm) at the same time with simulated solar irradiance levels of roughly 1-2X. A single day of testing (24 hours) is roughly equivalent to three times the outdoor exposure in terms of light intensity for the Ci65 and six times for the Ci5000. The solar simulator uses a filtered xenon-arc light source and can achieve intensities of about 2 - 5 times the outdoor exposure in a wavelength band between 300 and 450 nm. The 1 kW-SS operates at  $80^\circ\text{C}$  and 80% RH and can accommodate four 25.4-mm x 25.4-mm or eight 12.7-mm x 25.4-mm samples. A sample whose durability has been well characterized, ECP300/Al, was included in the 1kW-SS as a control.

## DURABILITY OF BATCH-COATED SAMPLES

The first samples of solar reflective material were produced on PET film (76.2  $\mu\text{m}$  thick) substrate in a 66-cm-wide box coater by batch processing with an alumina deposition rate of 1 nm/s. The details of the sample production and testing have been previously reported [2]. An important piece is the reactive gas used in the ion source. Alumina coatings crack when produced with oxygen, but alumina coatings produced with proprietary reactive gas do not crack.

The samples have maintained a high (95%) hemispherical reflectance even after 3600 hours of accelerated exposure testing in a 1 kW-SS exposure chamber, and after 5 years of outdoor exposure at NREL and Ci65 exposure testing. The reflectors with the thickest alumina coating (4  $\mu\text{m}$ ) exhibited the greatest durability.

A high deposition rate is necessary to reduce production costs [3]. The goal was to increase the deposition rate while maintaining optical durability. During 1998 and 1999, a series of samples were batch coated in a larger coating chamber. The alumina deposition rate was increased for these samples from 1 nm/s to 22.5 nm/s. Many samples prepared with a deposition rate as high as 10 nm/s are well adhered and transparent. These have maintained high hemispherical reflectance for 36 months of accelerated Ci65 exposure testing (Fig. 2). Samples prepared with properly optimized deposition parameters, have maintained high hemispherical reflectance for 42 months of outdoor exposure at NREL (Fig. 3). Funding constraints prevented optimization of the deposition parameters at each incremental deposition rate. Consequently, the outdoor reflector durability was poor for about half of the samples shown in Fig. 3. For example, in the 7 nm/s deposition rate case, the samples had insufficient ion assist and the durability was unsatisfactory. The 11.5 nm/s sample was not completely oxidized, as indicated by lower hemispherical reflectance. The alumina coating on the 22.5 nm/s sample was tinted brown at the center due to insufficient oxidation, but clear at the edge. The process was not optimal because there were also tensile cracks in the coating. This is an indication of excessive residual tensile stress. Despite the brown tint, the 22.5 nm/s samples have maintained high hemispherical reflectance after 9 months of exposure outdoors at NREL and in the Ci65. Comparing Figs. 2 and 3, samples produced with non-optimal process conditions show reduced durability outdoors, but display no significant durability loss in the Ci65.

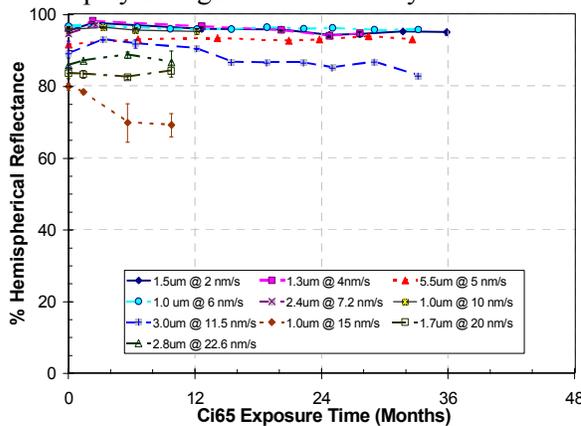


Fig. 2: Solar-weighted hemispherical reflectance of batch-coated alumina front-surface reflectors deposited at increasing deposition rates as a function of Ci65 exposure.

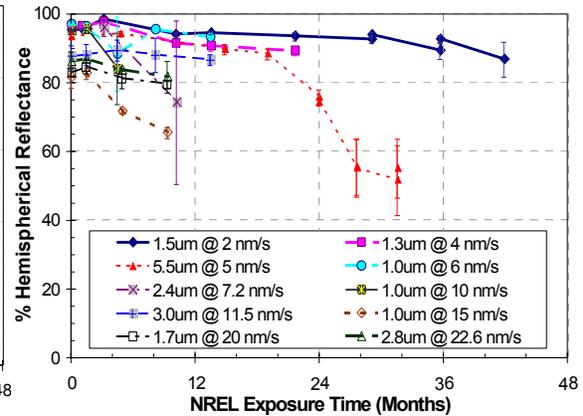


Fig. 3: Solar-weighted hemispherical reflectance of batch-coated alumina front-surface reflectors deposited at increasing deposition rates as a function of outdoor exposure at NREL.

All of the samples were inadvertently exposed without edge protection, a condition known to be extremely harsh. Outdoor samples that failed began to fail due to flaking around the edges, particularly after snowstorms. The outer 1-2 mm of alumina coating chipped off, which exposed the silver and allowed it to corrode; subsequent snowstorms then further chipped the alumina away until only a small center area remained.

Samples were also exposed outdoors at APS, FLA, SMUD, and TX; results are shown in Figs. 4 and 5. These samples were also exposed without edge protection, and the samples that failed also flaked around the edges. The durability of the alumina as a function of exposure, from

best to worst, is: 1 kW-SS, Ci65, TX, APS, NREL, SMUD, and FLA. APS, TX, and the 1 kW-SS are similar in that no degradation is observed in the hemispherical reflectance of the ASRM. This is quite unusual compared to other reflectors (e.g. polymer, glass, and aluminum). Typically, accelerated exposure is more severe than outdoor exposure [i.e., 1 kW-SS (~5xNREL), Ci5000 (~6xNREL), and Ci65 (~3xNREL)]. Normally, the most severe outdoor sites are FLA and APS; TX is intermediate, and SMUD and NREL are the least severe sites (Fig. 6) [6]. FLA has the highest total precipitation and relative humidity of the OET sites, shown in Table 1, followed by SMUD and NREL; TX and APS have the lowest. NREL has the lowest minimum monthly ambient temperature and the largest difference between the maximum and minimum monthly ambient temperature. In contrast to polymer and glass reflectors, because of this combination of humidity, precipitation, temperature extremes, and snow, outdoor exposure at NREL, SMUD, and FLA is more stressful for front-surface reflectors than AET, as can be seen in Figs. 4 and 5.

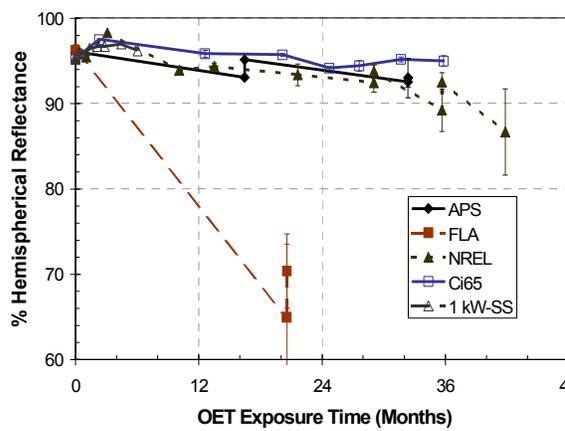


Fig. 4: Solar-weighted hemispherical reflectance of batch-coated alumina front-surface reflectors with 1.5- $\mu\text{m}$   $\text{Al}_2\text{O}_3$  deposited at 2 nm/s as a function of accelerated 1 kW-SS and Ci65 and outdoor exposure at APS, FLA, and NREL.

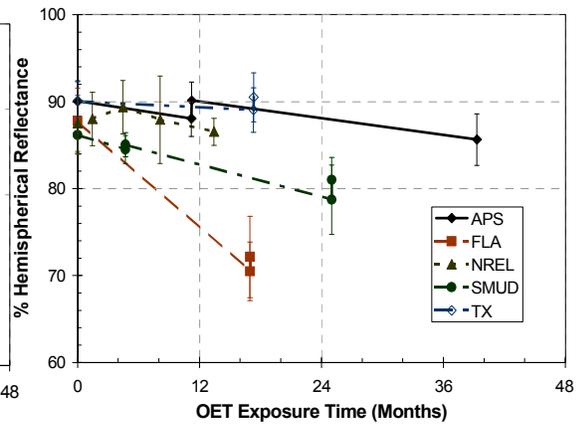


Fig. 5: Solar-weighted hemispherical reflectance of batch-coated alumina front-surface reflectors with 3- $\mu\text{m}$   $\text{Al}_2\text{O}_3$  deposited at 11.5 nm/s as a function of and outdoor exposure at APS, FLA, NREL, SMUD, and TX.

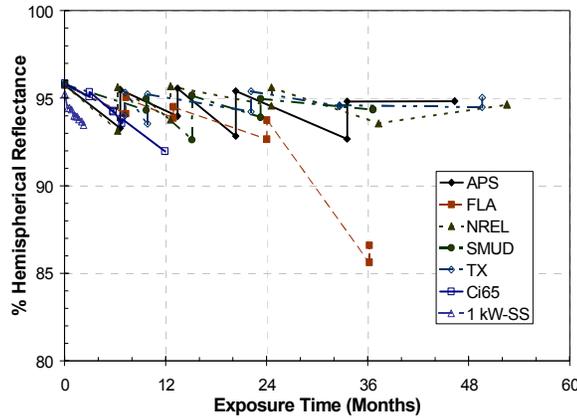


Fig. 6: Solar-weighted hemispherical reflectance of 3M ECP-305+ polymer reflectors as a function of accelerated 1 kW-SS and Ci65 and outdoor exposure at APS, FLA, NREL, SMUD, and TX.

Table 1: Summary of OET Meteorological Data [6, 7]

Site	Average Yearly Total Precip. (mm)	Average Max, Min Monthly Precip. (mm)	Average Day, Night Yearly RH (%)	Average Yearly $T_{\text{ambient}}$ (deg C)	Average Max, Min Monthly $T_{\text{ambient}}$ (deg C)	Average Yearly Total Solar (MJ/m <sup>2</sup> )	Max, Min Monthly Total Solar (MJ/m <sup>2</sup> )	Yearly Total UV (MJ/m <sup>2</sup> )
APS	195.6	25.4 2.54	50 23	22.6	34.2 12.0	5759.1	1056.4 144.9	219.9
FLA	1419.9	236.2 45.7	83 61	24.4	28.2 19.6	3731.8	631.6 304.6	164.5
NREL	391.2	60.7 12.7	67 40	10.11	25.8 -1.7	6124.7	701.6 452.6	277.3
SMUD	444.5	94.0 2.5	83 46	16.0	24.3 7.3	4074.7	771.9 93.9	194.7
TX	223.5	43.2 7.6	56 28	17.3	27.9 6.0	4245.8	754.2 346.5	129.6

Environmental stress factors that cause degradation have been identified from outdoor and accelerated exposure tests [8]. For most solar mirrors, exposure during service to sunlight (particularly ultraviolet wavelengths), temperature, and moisture can lead to loss in reflectance. The relative severity of these stresses is generally in the order they were mentioned above. Degradation can also result from synergistic effects (e.g., photothermal, photohydrolytic). The unintentional failure to protect the edges was fortuitous, as the resulting flaking revealed a weakness in the ASRM structure (i.e., insufficient adhesion between alumina and silver) and probable solution (i.e., edge protection and adhesion-promoting layer), which had not been indicated by the accelerated or outdoor (with edge protection) exposure testing used to date. In general, the durability of the alumina reflectors appears to depend on the type and amount of precipitation at the site (Table 1) and not on the solar radiation; therefore, outdoor exposure is more stressful to front-surface reflectors than accelerated exposure. The accelerated test chambers do not provide qualitative simulation of outdoor test results for front-surface reflectors. An accelerated test that incorporates cyclic precipitation or a humidity-freeze-thaw cycle (believed to be an important stress factor for these materials) needs to be developed. Edge protection will be necessary for long-term durability, and an adhesion-promoting layer between the silver and the alumina could improve the durability of the reflector.

#### **DURABILITY OF 10-nm/s ROLL-COATED SAMPLES**

During 2001, the transition was made from batch to roll-coating sample preparation. A web-handling machine was incorporated into the high-vacuum chamber previously used for batch coating of ASRM to make a laboratory-scale roll-coater. The basic design of the web-handling machine is two reels and a cooled drum. A coil of substrate material up to 35.5 cm wide unwinds from a payoff reel, wraps over a cooled drum, and winds onto a take-up reel (Fig. 7). The construction and operation of the laboratory-scale roll-coater was previously presented [9]. The substrate material chosen for the roll-coater was chrome-plated steel strip (from American Nickeloid Company). One of its advantages is a higher threshold to thermal damage than for

example, PET. The material is available in 0.92-m-wide rolls as thin as 203  $\mu\text{m}$  (8 mils), and costs about \$7.60/m<sup>2</sup> in volume.

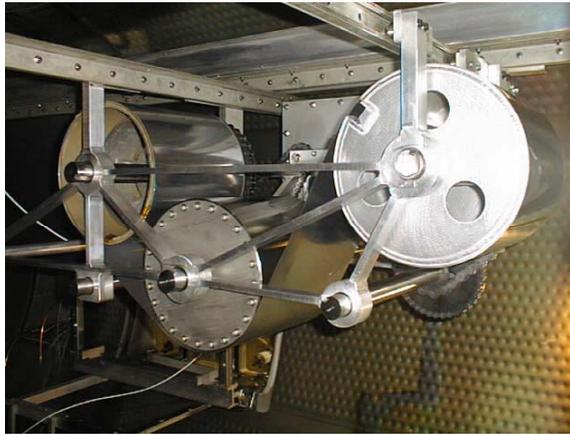


Fig. 7: Web-handling machine mounted inside high-vacuum chamber. A substrate material is wound over the cooled drum in the center. The take-up reel on the right is about 33 cm in diameter.

Six deposition runs were performed using the roll-coater with an alumina deposition rate of 10 nm/s. The goal was to achieve the same durability as earlier batch-coated samples. A summary of these runs is listed in Table 2. For each run, a length of strip equivalent to two loops of the take-up reel was coated. On June 6 and June 25, the first and second loops had different deposition conditions. Alumina film thickness was calculated by fitting measured spectrophotometer reflectance spectra to predicted reflectance values from a thin-film analysis program. The 29May01 sample demonstrated that good adhesion of silver to chrome-plated steel required an adhesion-promoting layer. Copper was used in all cases except the 25June01-2 sample, where alumina was used instead.

Sample adhesion is characterized by hand flexure of a narrow strip of material. When adhesion is excellent, the entire coating stack remains adhered. When adhesion is poorer, coating flakes are removed. If a flake curls with the alumina side exposed, the coating is under compressive stress. If the copper side is exposed, the coating is under tensile stress. By adjusting the ion-beam assist parameters, the desired neutral stress of the alumina coating can be achieved. Because earlier samples had demonstrated the need for edge protection, replicate samples were cut from the strips of materials for durability testing and several edge-protection concepts were used for samples exposed outdoors at NREL and in the Ci500 WOM. One approach was to apply 3M Tedlar tape around the periphery of the mirror as a protectant. After review of the literature and discussions with window and glass mirror vendors, a CPFilm product (Spectraseal ACL1307) intended as window sealant was recommended as a promising edge protectant. The third edge protection (“none”) was used as a benchmark to compare with earlier testing to determine the relative effectiveness of edge-protection strategies.

Table 2: Samples Produced in Roll-Coater

Run Date	Coating Structure	Alumina Deposition Rate [nm/s]	Web Speed [cm/min]	Ion Source Gas	Stress	Adhesion	Appearance
29May01	Al <sub>2</sub> O <sub>3</sub> /Ag	6.5	3.2	O <sub>2</sub> /Ar		Bad	Flaking
01June01	Al <sub>2</sub> O <sub>3</sub> (1.4μm)/Ag/Cu	5	3.2	PG	++	Good	Clear
06June01-1	Al <sub>2</sub> O <sub>3</sub> (2μm)/Ag/Cu	5	3.2	PG	0	Good	Clear
06June01-2	Al <sub>2</sub> O <sub>3</sub> /Ag/Al <sub>2</sub> O <sub>3</sub>	5	3.2	PG	0	Poor	Discharge damage, brown tint
10June01	Al <sub>2</sub> O <sub>3</sub> /Ag/Cu	10	6.3	PG	+	Excellent	Clear, brown tint area
20June01	Al <sub>2</sub> O <sub>3</sub> (1.6μm)/Ag/Cu	7-10	5	PG	+	Excellent	Brown tint
25June01-1	Al <sub>2</sub> O <sub>3</sub> (3μm)/Ag/Cu	10	5	PG	0	Good	Clear
25June01-2	Al <sub>2</sub> O <sub>3</sub> (3μm)/Ag/Cu	10	5	O <sub>2</sub>	-	Poor	Clear

Ion Source Gas: PG=propriety gas

Residual Stress: (+)=compressive, 0=neutral, and (-)= tensile

Adhesion: Bad=delaminated; Poor=flaking; Good=adhered but flakes when flexed; Excellent=no flaking when flexed.

The initial optical performance of roll-coated samples matched earlier batch-coated samples. The initial solar-weighted hemispherical reflectance was 95%-96%, except for 06June01-2 and 25June01-2 samples. Both of these samples had lower initial reflectance and also were less durable in accelerated and outdoor exposure, as shown in Figs. 9, 10, and 11. The variable performance of these two samples, demonstrated by the large error bars, is consistent with the poor adhesion exhibited by these samples. Sample 06June01-2 was produced with an alumina adhesion layer and was tinted brown with discharge damage. Discharge damage caused by the electrical discharge breakdown of the plasma appears as white streaks similar to lightening. Sample 25June01-2 was produced using oxygen as the ion-source feed gas. Both samples showed a drop in performance after 3.5 months outdoors in Colorado, with the largest drop shown by the 06June01-2 sample. Between 3 and 7 months, the performance of the 01June01, 10June01, and 20June01 samples degraded, and the 20June01 sample had the poorest performance outdoors. The 06June01-1 and 25June01-1 samples were unchanged after 7 months of outdoor exposure. The 25June01-2 sample was the only one to show a drop in performance after 2 months of exposure in the Ci5000. There are small changes in performance after 7 months of exposure in the Ci5000 for the 10June01, 25June01-1, and 06June01-1 samples. The performance is relatively unchanged for the samples exposed in the 1 kW-SS exposure chamber, continuing to demonstrate that for front-surface reflectors, durability is more significantly stressed outdoors. The next-highest stress is in the Ci5000, then the 1kW-SS. The samples deposited at 10 nm/s on the roll-coater that showed the best durability were those made with the proprietary gas. However, it should be noted that the backsides of the samples exposed outdoors started to rust. In the case where the alumina had a pinhole, rust would erupt on the front surface at the pinhole. The cause of the pinholes is being pursued. The chrome-plated carbon steel strip may not be durable enough for long-term use; chrome plating both sides of the carbon steel or substituting a stainless-steel foil substrate might prevent the rust. To date, the beneficial results of the edge protection schemes are inconclusive but edge tape and no edge protection are usually better than the CPFilm product.

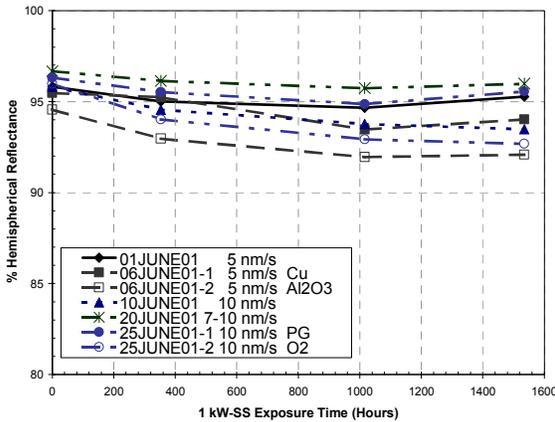


Fig. 8: Solar-weighted hemispherical reflectance of roll-coated alumina front-surface reflectors as a function of 1-kW solar simulator exposure.

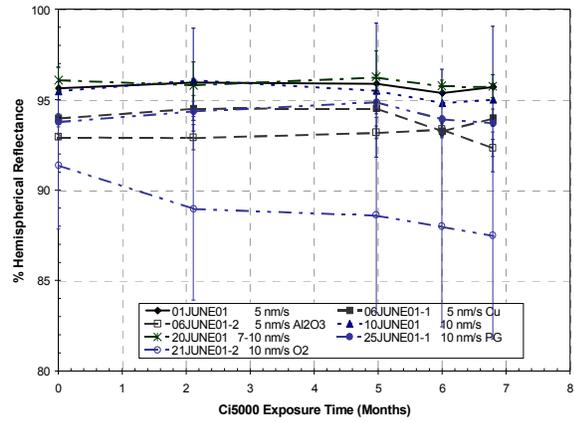


Fig. 9: Solar-weighted hemispherical reflectance of roll-coated alumina front-surface reflectors as a function of Ci5000 exposure.

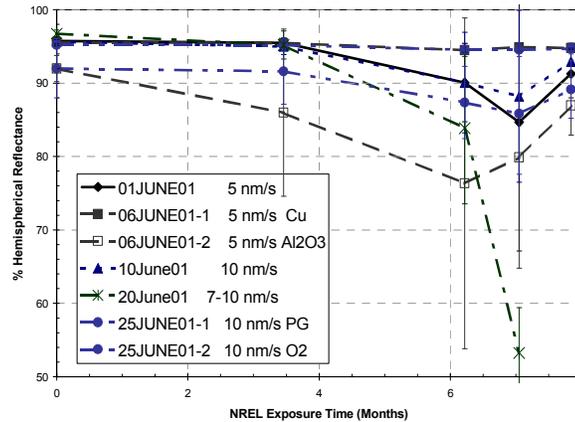


Fig 10. Solar-weighted hemispherical reflectance of roll-coated alumina front-surface reflectors as a function of outdoor exposure at NREL.

## SUMMARY AND FUTURE PLANS

Significant progress has been made in developing a new type of solar-reflective material. The ASRM under development has the potential to deliver high performance at a manufacturing cost lower than thin glass. However, before the material can be commercially viable, we must demonstrate that durable material can be produced cost effectively. To date, the durability of some samples produced by roll coating is excellent and equivalent to samples produced by the batch process, when the deposition conditions have been optimized. Samples deposited at 10 nm/s on the roll-coater that showed the best durability and adhesion were those made with the proprietary gas and low residual compressive stress determined by the ion assist. These results are preliminary, and durability testing is ongoing. Comparisons between accelerated and outdoor exposure tests indicate that this ASRM is more susceptible to weather conditions not simulated

by accelerated tests (e.g., rain, sleet, and snow). An accelerated test that simulates a greater number of relevant outdoor conditions needs to be developed. Edge protection will be necessary for long-term durability. An adhesion-promoting layer between the silver and alumina could improve durability. Most recently, the alumina deposition rate has been increased to 20 nm/s in roll-coated samples. The analysis of this material has just begun. Other interests are to incorporate an anti-soiling layer, metal adhesion layers other than copper, an adhesion-promoting layer, and edge-protection schemes. Future plans also include to increase the deposition rate and to deploy the material in an existing concentrating solar power system.

A cost model developed seven years ago showed that the deposition rate and thickness of the alumina coating strongly influence the unit cost of the solar-reflector material. It motivated us to transition from batch to roll coating, and increase the alumina deposition rate to 20 nm/s. The switch from PET to a steel substrate for the ASRM is a significant change from the original model. The cost of adhesion and anti-soiling layers were not included, and certain equipment costs were understated. To help commercialize the technology, NREL has initiated a subcontract to update the cost analysis.

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13. ABSTRACT ( <i>Maximum 200 words</i> ): A promising low-cost reflector material for solar concentrating power (CSP) generation is a silvered substrate protected by an alumina coating several microns thick. The alumina hard coat is deposited under high vacuum by ion-beam-assisted-deposition (IBAD). Samples of this material have been produced both by batch and continuous roll-coating processes. The substrate materials investigated were polyethylene terephthalate (PET), PET laminated to stainless-steel foil, and chrome-plated carbon steel strip. The advantage of steel strip compared to PET is that it withstands a higher process temperature and lowers the final product installation costs. In this paper, we compare the durability of batch and roll-coated reflective materials with an alumina deposition rate as high as 10 nm/s. In general, the durability of the samples is found to be excellent. Comparisons between accelerated and outdoor exposure testing results indicate that these front-surface mirrors are more susceptible to weather conditions not simulated by accelerated tests (i.e., rain, sleet, snow, etc.) than other types of solar reflectors. For long-term durability, edge protection will be necessary, and durability could be improved by the addition of an adhesion-promoting layer between the silver and alumina.				
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