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Final Letter Report

Advanced Process Development for High Reflector Coatings on Solar Concentrator Panels

**United Solar Technologies and Pacific
Northwest National Laboratory CRADA**

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**PM Martin
CD Stewart
WD Bennett
JW Johnston**

October 1996

**DOE Office of Energy Research
Laboratory Technology Research Program**

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**Pacific Northwest National Laboratory
Richland, Washington 99352**

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Final Letter Report

CRADA with United Solar Technologies and Pacific Northwest National Laboratory Advanced Process Development for High Reflector Coatings on Solar Concentrator Panels

**PM Martin
DC Stewart
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October 1996

**Prepared for U.S. Department of Energy
under Contract DE-AC06-76RLO**

**Pacific Northwest National Laboratory
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Advanced Process Development for High Reflector Coatings on Solar Concentrator Panels

Purpose/Objective

The objectives of this project were as follows:

- Develop and demonstrate the manufacturing process for vacuum deposition of low-cost thin-film high reflectance coatings onto large solar concentrator panels.
- Demonstrate thin-film deposition processes for commercialization of this technology by United Solar Technologies (UST)
- Apply reflective coatings to solar concentrator panels for prototype application by UST.

Summaries of Activities Performed

Phase 2 project activities were initiated in January, 1995. United Solar Technologies (UST) is developing solar concentrator modules for domestic active and passive solar energy generation. Their objective is to manufacture and market these modules. The solar concentrator consists of fourteen parabolically-shaped pressure formed aluminum (Al) panels. To perform to design specifications, the panels must be highly reflective to the solar spectrum. UST tasks for this CRADA were:

- build the facility to manufacture the Al panels,
- manufacture the Al panels,
- apply a polymer smoothing underlayer to the panels,
- characterize the reflectance and optical scatter of the panels after PNNL applies the reflective coatings,
- and assemble the coated panels into the concentrator modules.
- field test solar concentrator modules and dishes

The tasks assigned to PNNL were:

- define the manufacturing process for deposition of the reflective coating

- develop a polymer smoothing underlayer for the reflective coating
- apply the solar reflective coatings onto solar concentrator panels supplied by UST
- evaluate coating performance

PNNL's primary responsibilities were to develop the manufacturing process for low-cost high reflective coatings on 8-ft solar concentrator panels, apply these coatings onto panels supplied by UST, and to assist UST with development of the prototype deposition chamber. Due to funding cut backs, the last task was not performed, but is now being performed in a follow-on SBIR program with EDTEK (a sister company with UST), Sandia National Laboratory and NREL. All coatings were deposited using PNNL's large-optics coating facility.

Magnetron sputtering was the deposition technique for the reflective coating. UST's primary responsibilities were to build the facility to manufacture the solar concentrator panels, supply panels for PNNL to coat, assemble the concentrator dish, and field test the dish.

The ultimate goal of this, and the follow-on work, was to demonstrate a low-cost thin-film process for application of the reflective/protective coatings on solar concentrator panels. To accomplish this, PNNL is developing the high-rate magnetron-sputtering process, and a new polymer-multilayer coating technology, which EDTEK will evaluate in the follow-on work with SNL and NREL. Polymer coatings can be deposited at much lower costs and higher rates than conventional magnetron-sputtered coatings. Because of the relative low funding level, only a small part of PNNL activities was directed at evaluating the use of polymer layers in the solar reflective coatings.

Experimental

PNNL's work was divided into three tasks:

- 1) Manufacturing Process Development
- 2) Reflective Coating Deposition
- 3) Process Improvement

Task 1. Manufacturing Process Development

United Solar Technologies, Inc. (UST) fabricated the parabolic aluminum solar concentrator panels by a pressure forming process [1]. Low-cost sheet aluminum was used. After forming, the panels had scratches, pits, bumps, and stains over the surface. During the first year of this CRADA, it was determined that application of a smoothing underlayer (polymer or urethane) was the best and lowest-cost technique to eliminate optical scattering due to the defects in the panels, and achieve a specular reflective coating. Polishing the panels was too costly and time consuming.

The objective of this task was to develop and define the manufacturing process for deposition of reflective coatings onto UST's 8-ft solar concentrator panels. This included:

- chamber configuration and deposition equipment
- coating materials
- smoothing layer materials and applications
- chamber jiggling
- identifying process steps

Fabrication of the reflective coatings on the 8-ft solar-concentrator panels involved evaluation of various smoothing layers and reflective coatings on test coupons, application of a smoothing layer to the bare panels, and coating deposition by reactive magnetron sputtering.

The smoothing layer was critical for achieving a specular surface for the reflective coating. Sputtered materials replicate the surface features of the substrate, and the sputtering process therefore could not be used to smooth the rough surface of the panel, and create a specularly reflective surface. Urethane and acrylate polymers demonstrated the best smoothing and adhesion to the Al panels in previous test evaluations [2,3]. Dipping, painting, and spraying processes were evaluated for application of the smoothing layer. Best results were achieved for the urethane smoothing layer formed by a dipping process. The spray process introduced small bubbles, and painting introduced ridges and thickness nonuniformities. In this work, the acrylates were both successfully sprayed by a local auto body shop, and deposited by vacuum flash evaporation of an acrylate monomer. Urethane and acrylates with thicknesses between 50 and 100 μm were used initially as the smoothing layer for the panels discussed here because of ease of application. However, the acrylates showed significant potential for improved adhesion, performance and cost reduction. One of the panels was mechanically buffed after application of the acrylate. Even though the small ripples were eliminated, this process was ruled out because of the high cost of an additional process step. In many cases the panel was primed with an adhesion promoter. This appeared to improve smoothing layer adhesion, but again was ruled out because of the extra process step and incompatibility with the vacuum deposition process. In future work, an adhesion promoter will be incorporated into the smoothing layer [4].

To determine the effectiveness of the smoothing layers, a test matrix consisting of 3.8-cm-square Al sheet coupons was constructed. The matrix included bare Al coupons, Ag and Al layers on bare Al coupons, Ag and Al layers on an Al coupon with a urethane smoothing layer, and $\text{Al}_2\text{O}_3/\text{AlN}$ -overcoated Ag and Al layers on an Al coupon with a urethane smoothing layer. Ag and Al were also deposited on mylar and polypropylene (PP) substrates with and without acrylate smoothing layers. The specular reflectance of the samples was measured between 200- and 1100-nm wavelengths with Perkin Elmer Lambda 12 and Beckman UV/VIS 5270 dual beam spectrophotometers.

The improvement in specular reflectance resulting from the polymer smoothing layer was particularly dramatic when used with bare metal substrates. The rms surface roughness of the bare Al ranged from 0.5 to 1 μm . Appendix 1 shows the specular reflectance of the 3.8-cm-square Al coupons for a) bare metal, b) Ag on bare metal, c) Ag on urethane undercoat, and d) $\text{Al}_2\text{O}_3/\text{Ag}$ on urethane undercoat. Before application of a urethane smoothing layer, the specular reflectance of an Ag-coated Al substrate (curve b) was below 20% at visible wavelengths, not a significant improvement over that of a bare Al. The coating was hazy, which indicated a high amount of optical scattering (diffuse reflectance). The specular reflectance of the Ag on the urethane smoothing layer (curve c) ranged from 82% at 400-nm wavelength to 98% at 800-nm wavelength. While this is lower than the reflectance of Ag on glass [3], it is a significant improvement over the untreated substrate, and acceptable for concentrator applications. Application of the Al_2O_3 layer (curve d) formed an enhanced metal reflector, and increased the reflectance to near 99% at wavelengths between 400 and 900 nm. The performance demonstrated in curves c and d could not be achieved without the smoothing layer.

As a first step in determining the stability and durability of the coatings, the test coupons were placed in a Ransco Model 16607 environmental chamber and subjected to a relative humidity of 95% and temperature of 50°C for 24 hr. They were also cycled between -62°C and 71°C, holding at these temperatures for two hours. The ramp rates for heating and cooling were 4°C/min and -1.6°C/min respectively. They were also subjected to the testing per MIL-F-48616 for adhesion and abrasion resistance. Because other relevant testing is not available at this facility, further testing is planned at the National Renewable Energy Laboratory (NREL), which will expose the coatings to intense solar radiation, accelerated environmental tests, and abrasion tests.

Task 2. Reflective Coating Deposition

The reflective coatings were deposited onto 8-ft solar concentrator sections supplied by UST. UST initially supplied twelve panels, and was to apply the polymeric and urethane smoothing layers. They applied urethane smoothing layers to six of these. The urethane layers debonded during delivery. To facilitate coating the panels, PNNL either applied acrylate smoothing layers or coordinated with local suppliers to apply the smoothing layer onto these and the balance, as described in the previous section. PNNL utilized significant funds, not originally set aside for this process step, to apply the smoothing layers.

Appendix 2 shows the 3-m vacuum chamber used for coating deposition. The chamber was originally used to develop and fabricate high-performance multilayer laser-mirror coatings on large substrates. The facility is unique in that precision-sputtered multilayer optical coatings can be applied to curved and rotationally-asymmetric substrates as large as 2.5 m in diameter. This chamber is capable of simultaneously or sequentially depositing four different coating materials, dielectric, metals, or semiconductors, for multilayer designs. Up to four 15-cm x 76-cm magnetron sputtering cathodes (VacTec), and three ion sources can be located in the base of the chamber. The panel was located and rotated with simple planetary motion in the top section. The center of the concentrator panel was 1.83 m from the sputtering source, with each end located symmetrically 1.65 m from the sputtering source.

Table 1 summarizes the deposition conditions used for each layer of the high reflectance coating. Ag, AlN, and Al₂O₃ coatings were deposited onto the Al panels using an Ag target for the Ag layer, and an Al target for the dielectric layers. The minimum purity of all targets was 0.99999. The nonreactive gas was Ar in all cases.

The AlN layer prevented oxidation of the Ag layer during deposition of the top Al₂O₃ layer. For cathode input powers shown in Table 1, the deposition rate (coating thickness per unit time of deposition) was 150 Å/min for AlN, 98 Å/min for Al₂O₃, and 500 Å/min for the Ag layer. Because the substrate was opaque and reflective coatings are being deposited, coating thickness was controlled by optical monitoring in the reflectance mode. Before coating deposition, all substrates and panels were ion etched with a beam voltage of 100 V and an ion current (at the aperture) of 2 A.

Table 1. Deposition Conditions for Reflective Coating

Layer	Thickness (μm)	Target Power (kW)	Ar Partial Pressure (mTorr)	Reactive Gas	Reactive Gas Partial Pressure (mTorr)
Ag	0.2	7.85	0.41		
AlN	0.05	6.0	0.29	N ₂	0.12
Al ₂ O ₃	1.0	6.0	0.21	O ₂	0.20

Appendix 3 shows a picture of four UST panels with smoothing and reflective layers. The coatings were very reflective and durable. Appendix 4 shows the reflectance spectrum of a solar panel coated with a urethane smoothing layer and the Al₂O₃/AlN/Ag layers. The reflectance, measured by a fiber-optic diode-array spectrophotometer, is comparable to that of the Al coupon (curve d, Appendix 1) treated with these thin-film materials. The uniformity of optical performance of the coating over the entire 2.5-m panel was better than ±5%.

The primary concentration ration of the panel was measured by United Solar Technologies to be 800:1 [1]. The design concentration ration was 500:1 [1]. This ratio was determined by the profile of the reflected beam at the focus of the parabolic panel. This measurement indicated that the diffuse reflectance and figure (slope and contour) error of the coated panel was significantly lower than expected.

Initial durability and environmental testing performed on the coating was encouraging. The Al₂O₃-protected coatings survived MIL-F-48616 environmental tests for adherence, humidity, moderate abrasion, severe abrasion, and temperature cycling. The actual environment which the concentrator experiences is more severe. Additionally, the coating is exposed to high intensity solar radiation, rain erosion, and sand/dust abrasion for extended periods. Extended lifetime tests are being planned at the National Renewable Energy Laboratory (NREL) to further evaluate the durability of the coatings. Four of the panels were delivered to UST, and are now in the field being tested by UST in a solar concentrator module [1].

Task 3. Process Improvements

Improvements were realized in the materials for and application of the smoothing layers. This was also the motivation for follow-on work to utilize the polymer multilayer process for deposition of the smoothing layer, as discussed in a following section. We were able to apply a polymeric smoothing layer to the entire 8-ft concentrator sections. Adhesion of the polymeric layers applied by UST was a problem. This poor adhesion was attributed to poor cleaning of the surface of the aluminum panel, and the lack of a bonding layer or adhesion promoter. PNNL reworked all the UST panels, and applied acrylate smoothing layers. Adhesion of this smoothing layer on the reworked panels was excellent, and was superior to that of the urethane.

The smoothing layer provided a specular surface on which the reflective coating was applied. The resulting reflective coating had very high reflectance, very near the reflectance of bare aluminum on a glassy surface [3].

Significant Accomplishments

The significant accomplishments of this project were:

- PNNL coated and delivered 12 solar concentrator panels to UST
- development of an adherent polymeric smoothing layer for the large area solar concentrator panels
- demonstration of highly reflective coatings on 8-ft solar concentrator panels
- achievement and exceeding of UST specifications for reflective coating and panel performance
- apparent high environmental durability of the reflective coatings
- demonstration of the potential for a low cost deposition process for reflective coatings on solar concentrator panels

Reflective coatings were applied to twelve solar concentrator panels supplied by UST. The coated panels were delivered to UST.

UST applied urethane smoothing layers onto the first six panels they delivered to PNNL. All coatings delaminated during delivery. Delamination was due to poor cleaning and priming. We found that acrylate polymers demonstrated good adhesion to the aluminum panels with the proper bonding layer and cleaning of the panel. Heat curing of the polymer was also critical. Panels that were baked demonstrated good adhesion, while adhesion was variable on those that were cured with an infrared heat source. Also, when the polymer smoothing layer is deposited in vacuum, an adhesion promoter can be incorporated into the acrylate to ensure good adhesion.

As shown in Appendix 4, the reflectance of the coatings on the solar concentrator panels was very close to that of the bare aluminum on a glassy surface. This is a very significant accomplishment considering the initial surface roughness of the bare panels.

All UST's reflectance and optical scattering specifications were exceeded, which was demonstrated in their achieving an 800:1 concentration, compared to the expected 500:1 ratio.

The PNNL coating and PML processes, when combined with the UST/EDTEK panel forming process, could significantly lower the cost of solar concentrator modules, and residential solar energy usage [1].

A technical publication based on this work will appear in the Journal of Vacuum Science and Technology [3].

Significant Problems

As with the first year CRADA with UST [2], the major problem in executing this CRADA was the delays UST experienced in delivering the solar concentrator panels for coating, and preserving a schedule for coating depositions. These delays and "short fuses" were a constant source of frustration.

Proper curing and adhesion of the polymeric smoothing layer to the aluminum panels were significant technical problems. This occurred initially for the urethane due to poor cleaning of the panels by UST. UST applied these layers to uncleaned and contaminated panels. If the surface of the panel is clean and free of grease, we expect the adhesion to be good. This was demonstrated by the panels on which PNNL applied the polymeric layers. Adhesion was excellent to these panels.

If the smoothing layer was not cured properly, outgassing of solvents occurred when the panel was placed under vacuum before the coating application. The outgassing caused small bubbles to form in the smoothing layer, intermittent delamination of the smoothing layer, and reaction of the silver metal layer with the polymer.

Industry Benefits Realized

UST and a sister company EDTEK now have access to a process to apply highly reflective/protective coatings to the solar panels they manufacture. They also have access to the PML technology being developed and licensed by PNNL. These processes show significant promise for low-cost fabrication of solar concentrator modules, and have the potential to make UST and EDTEK competitive in the residential solar energy market.

Recommended Follow-On Work

EDTEK, a sister company with UST, and PNNL are cooperating in Phase 1 of a SBIR with SNL and NREL to develop a low-cost fabrication process for a solar concentrator dish.

Fabrication is based on the coating technology developed at PNNL, and EDTEK's pressure forming process for the aluminum panels.

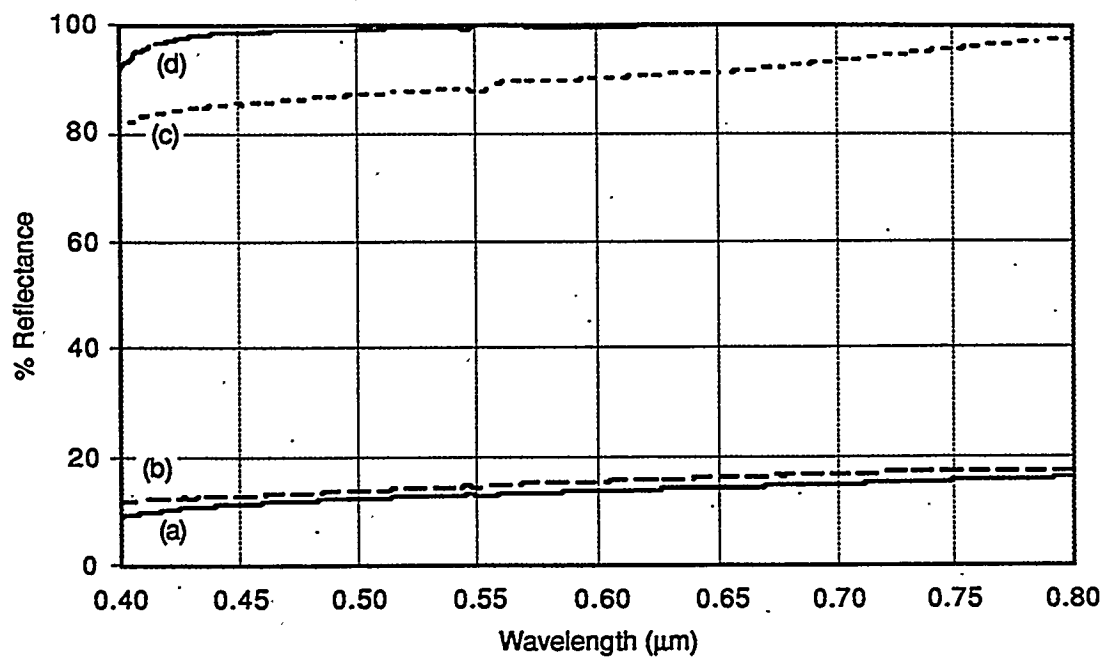
Follow-on work should also address incorporation of the PML process into the fabrication process, and the application of PML barrier coatings being developed at PNNL. These coatings would supply a very high level of environmental and abrasion protection to the metal reflective layer [5].

Potential Benefits from Pursuing Follow-On Work

If successful, the UST/EDTEK solar-concentrator system has the potential to revolutionize domestic energy generation and management. UST has recently manufactured a complete solar-concentrator system, and this system is in the field being tested. The follow-on work would also assist UST/EDTEK in manufacturing the entire concentrator system. PNNL would benefit from the technology transfer, and possible licensing of the PML technology for manufacture of low-cost solar concentrators.

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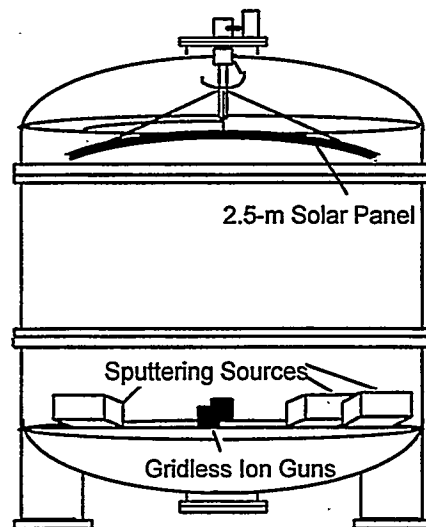
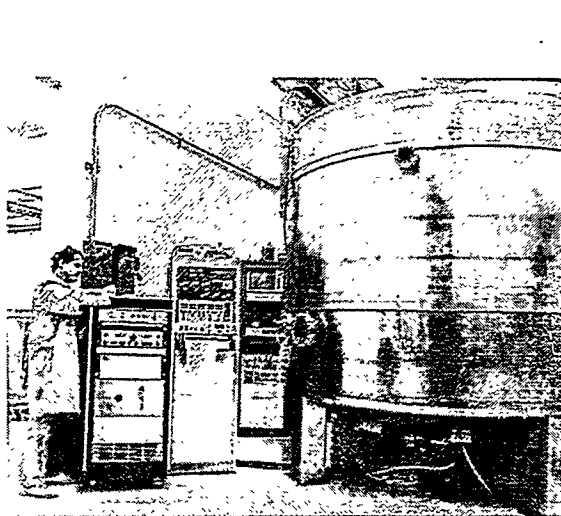


APPENDIX 1

Reflectance Spectra of Coatings on 3.8-cm Al Test Coupons

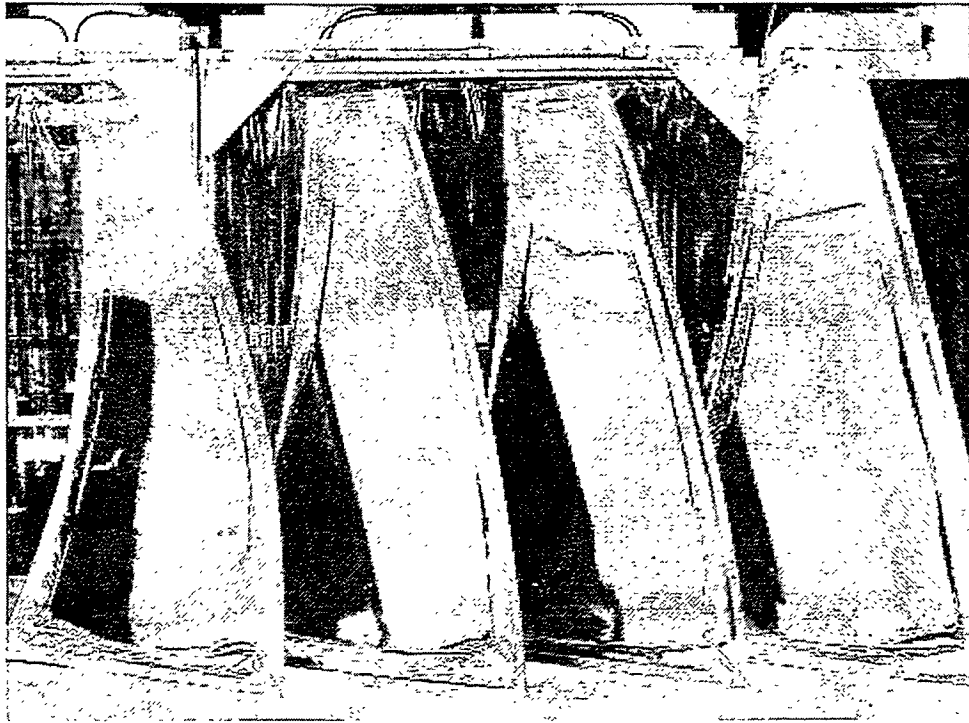
Key:

- (a) = bare metal
- (b) = Ag on bare metal
- (c) = Ag on urethane undercoat
- (d) = Al₂O₃/Ag on urethane undercoat



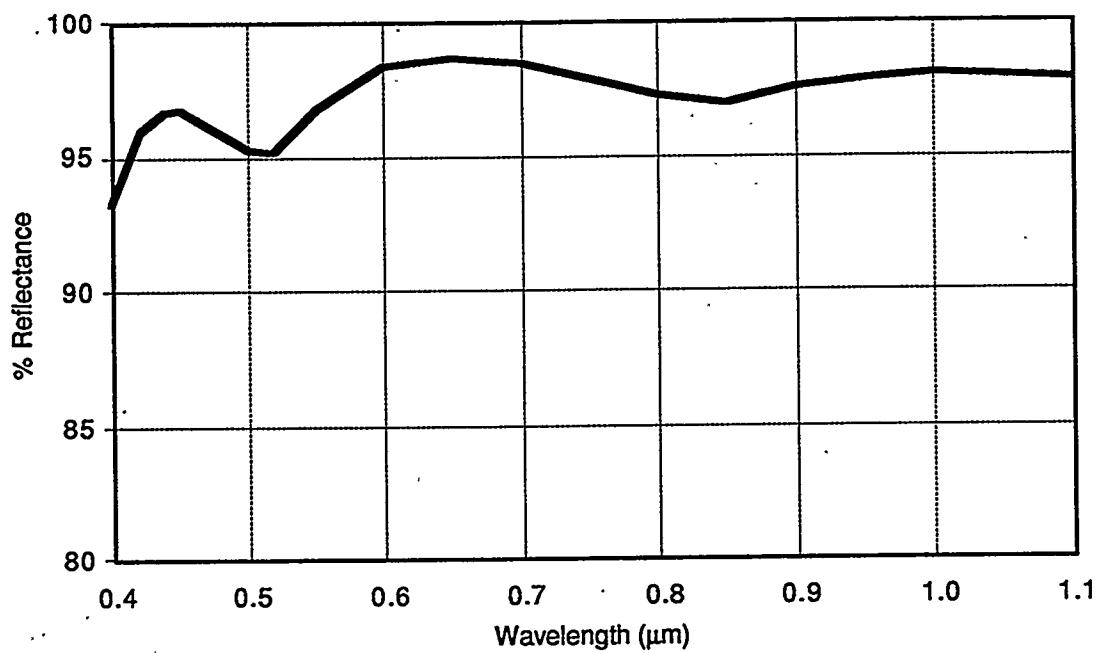
APPENDIX 2

PNNL 3-m Coating Chamber



APPENDIX 3

Picture of Four UST Panels with Solar Reflective Coatings



APPENDIX 4

Reflectance Spectrum of a Coated Solar Concentrator Panel

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Germantown, Maryland 20874

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