

* PROGRESS TO DEVELOP AN ADVANCED SOLAR-SELECTIVE COATING

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Abstract

Increasing the operating temperature of parabolic trough solar fields from 400°C to >450°C can increase the overall solar-to-electricity efficiency and reduce the cost of electricity from parabolic trough power plants. Current solar-selective coatings do not have the stability and performance necessary to move to higher operating temperatures. The objective is to develop new, more-efficient solar-selective coatings with both high solar absorptance ($\alpha > 0.96$) and low thermal emittance ($\epsilon < 0.07$ at 450°C) that are thermally stable above 450°C, ideally in air, with improved durability and manufacturability, and reduced cost. Using computer-aided optical design software, multilayer solar-selective coatings with optical properties exceeding the goals (absorptance of 0.959 and emittance of 0.070 at 450°C) and lower thermal losses than the commercial coatings were modeled for materials with high thermal stability. This exceeds the specified goal by about 1% overall, because 1% in emittance equates to about 1.2% in absorptance. The key issue is depositing the modeled coating. To deposit the individual layers and the prototype modeled solar-selective multilayer structure, ion-beam-assisted (IBAD) electron-beam (e-beam) co-deposition was used because of its versatility and lower material costs. Experimental work has focused on modeling high-temperature, solar-selective coatings; depositing the individual layers and modeled coatings; measuring the optical, thermal, morphology, and compositional properties and using the data to validate the modeled and deposited properties; re-optimizing the coating; and testing the coating performance and durability. The progress to develop a durable advanced solar-selective coating will be described.

Keywords: absorber, electron-beam, emittance, optical modeling, oxidation resistance, solar selective

1 Introduction

Trough solar systems use parabolic, single-axis tracking mirrors to concentrate sunlight 30–60 times onto an evacuated receiver tube, at the focal point of the reflector, thus heating the heat-transfer fluid (i.e., synthetic oil) flowing through the receiver. Electricity is generated by passing the heated oil through a heat exchanger and using the steam generated to drive a turbine generator. Parabolic-trough solar technology has been demonstrated by nine utility-scale plants installed between 1984 and 1991 in California's Mojave Desert. These plants, referred to as Solar Electric Generating Systems (SEGS), represent 354 MW of installed electric-generating capacity that operate daily, providing power to the electricity grid. The technology had been dormant since 1993 due to low energy prices, but a new 64-MW trough plant recently began producing power and four new projects are being developed or constructed. The SEGS experience has proven the parabolic-trough technology to be a robust and reliable power technology in an industrial-utility operating environment. Its key advantages are proven performance, manufacturing simplicity, use of standard equipment and materials, improvement in cost effectiveness via incremental steps, and low technical or financial risk to the investor.

The current levelized cost of electricity (LCOE) from these plants is \$0.10–\$0.12 per kWh (real 2004 dollars). The goal of the U.S. Department of Energy (DOE) Office of Energy Efficiency and Renewable Energy (EERE) is to reduce the cost to \$0.06–\$0.08 per kWh by 2012. The cost of the solar-collector technologies needs to be reduced by half to achieve the long-term goal of developing parabolic-trough power plants capable of competing on a cost-competitive basis with conventional fossil power technologies as dispatchable intermediate power generation in the wholesale power market (LCOE of \$0.04–\$0.06/kWh).

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The overall solar-to-electric efficiency of parabolic-trough solar power plants can be improved and the cost of solar electricity can be reduced by improving the properties of the selective coating on the receiver and increasing the solar-field operating temperature to $>400^{\circ}\text{C}$. New, more-efficient selective coatings will be needed that have both high solar absorptance and low thermal emittance at elevated temperatures. Although the coatings are designed to be used in evacuated environments, the coatings need to be stable in air in case the vacuum is breached. Current coatings do not have the stability and performance desired for moving to higher operating temperatures. For efficient photothermal conversion, solar absorber surfaces must have low reflectance ($\rho \approx 0$) at wavelengths (λ) $\leq 2 \mu\text{m}$ and a high reflectance ($\rho \approx 1$) at $\lambda \geq 2 \mu\text{m}$. The cutoff may be higher or lower depending on the operating temperature. For parabolic-trough applications, the spectrally selective surface should be thermally stable above 450°C , ideally in air, and have a solar absorptance (α) greater than 0.96 and a thermal emittance (ϵ) below 0.07 above 450°C . Achieving the improved properties is very important if the parabolic troughs are going to move to higher temperatures.

Figure 1 shows the influence that the receiver technology can have on the cost of electricity. Assuming a solar-field working temperature of 400°C , the relative cost of electricity for current receiver selective coatings decreases with improvements in the optical properties: moving from the original black chrome, to the original Mo-Al₂O₃ cermet (cermets are highly absorbing metal-dielectric composites consisting of fine metal particles in a dielectric or ceramic matrix), to the newer-generation improved Al₂O₃-based receivers both as-tested properties (t) compared to specified properties (s) from the first manufacturer and the new Mo-Al₂O₃ cermet from the second manufacturer [1,2]. The figure also shows the reduction in the cost of electricity that could be achieved by reaching the DOE goals. The improved receiver as-tested results represent the DOE baseline technology assumption for near-term future parabolic-trough plants. The DOE long-term goal represents a 10% improvement in the cost of electricity. The uncertainties in the values shown in Fig. 1 have not been carefully quantified at this point, but are thought to be on the order of several percent of the values shown.

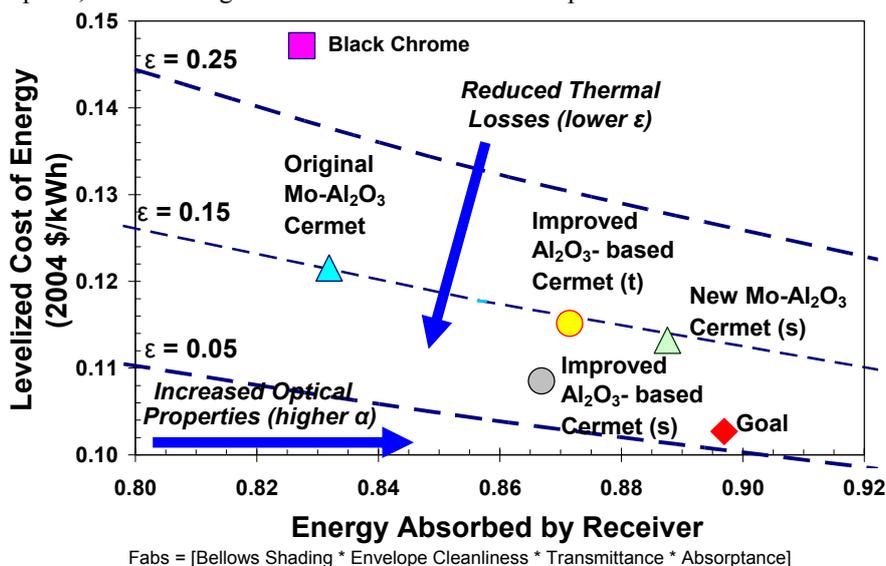


Figure 1. Influence of receiver technology on levelized cost of electricity as a function of energy absorbed by the receiver (i.e., = bellows shading x glass envelope cleanliness x glass envelope transmittance x solar selective coating absorptance) for a receiver with improved optical properties [i.e., higher absorption (α)] and reduced thermal losses [lower emittance (ϵ)] for as-tested properties (t) compared to specified properties (s) for a DOE parabolic-trough 2005 reference power plant configuration, 100-MWe solar-only SEGS, 6 h of thermal-energy storage. The dashed lines show the receiver analysis lines of constant emittance.

Increasing the operating temperature of the power plant and increasing its efficiency will lower the LCOE of parabolic troughs. This requires increasing the absorption, lowering the emittance, and increasing the durability and oxidation resistance of the solar-selective coating. To achieve our goals, the following were proposed:

- Review the literature for potential high-temperature solar-selective coatings.
- Review the high-temperature oxidation-resistance properties of potential materials for high-temperature solar-selective coatings.
- Use modeling software to optically model solar-selective designs or refine existing ones and extract optical constants of the steel substrate used in the design.

- Develop high-temperature solar-selective coatings using multiple cermet layers by ion-beam-assisted (IBAD) electron-beam (e-beam) physical vapor deposition (PVD).
- Verify functionality through material characterization, high-temperature optical characterization, and durability testing.

2 LITERATURE REVIEW OF HIGH-TEMPERATURE SOLAR-SELECTIVE COATING

2.1 *Potential High-Temperature Solar-Selective Coating*

To identify potential high-temperature absorbers, the literature was reviewed for medium- to high-temperature absorber coatings [3]. Several materials have the appropriate optical properties and should be durable at operating temperatures above 500°C. Various transition metals—particularly those formed from the refractory metals of groups IVA, VA, and VIA and their binary and ternary compounds—have been suggested for high-temperature applications because of their high melting point and chemical inertness [4]. The titanium, zirconium, or hafnium metal boride, carbide, oxide, nitride, and silicide materials have some of the highest melting points in nature, with HfC having the highest melting point at 3316°C. These materials also have a high degree of spectral selectivity, high hardness, and improved wear, corrosion, and oxidation resistance [5,6]. A double-cermet film structure has been developed that has higher photothermal conversion efficiency than surfaces using a homogeneous cermet layer or a graded film structure [7]. Surface texturing is a common technique to obtain spectral selectivity by the optical trapping of solar energy. The emittance can be adjusted (higher or lower) by modifying the microstructure (microcrystallites) of the coatings with ion-beam treatments [8]. However, there is a trade-off between a highly absorbing coating and one with low emittance. Highly absorbing coatings appear rough, porous, and absorb solar energy; coatings with low emittance are very smooth, dense, highly reflective, and mirror-like to thermal energy. Combining several concepts, a high-temperature solar-selective coating could be developed from materials with intrinsic solar selectivity and high-temperature stability using multiple cermet layers, along with appropriate surface texturing and incorporating multiple antireflective (AR) coatings. The optical properties of the refractory metal compounds have a high degree of flexibility; with further research, multiple-layer cermets with noble metals could be viable high-temperature absorbers for the Concentrating Solar Power (CSP) program.

2.2 *High-Temperature Oxidation-Resistant Properties*

At this point, none of the existing coatings have proven to be stable in air at 400°C. Achieving the goal of a solar-selective coating that is stable in air at temperatures greater than 450°C requires high thermal and structural stabilities for both the combined and individual layers, excellent adhesion between the substrate and adjacent layers, suitable texture to drive the nucleation and subsequent growth of layers with desired morphology, enhanced resistance to thermal and mechanical stresses, and acceptable thermal and electrical conductivities. Other desirable properties are good continuity and conformability over the tube, as well as compatibility with fabrication techniques. The material should have a low diffusion coefficient at high temperature and be stable with respect to chemical interactions with the oxidation product, including any secondary phases present, over long periods of time at elevated temperature. Selecting materials with elevated melting points and large negative free energies of formation can meet these objectives. Stable nanocrystalline or amorphous materials are the most desirable (and practical) for diffusion-barrier applications, especially in light of material and process limitations. However, there will be a trade-off in the microstructure between a highly oxidation-resistant coating (i.e., amorphous or nanocrystalline) and a solar-selective coating with both high absorption (i.e., columnar or porous microstructure) and low emittance (i.e., smooth or highly dense). High thermal stability is manifested by high melting points, single-compound formation, and lack of phase transformations at elevated temperature.

The material properties of candidate materials for the solar-selective coating were reviewed, materials with low thermal stability and high reactivity were down-selected, and materials with more-suitable properties were modeled. For example, based on their melting points, the metals W, Mo, Ir, Os, and Ta are prime candidates for high-temperature applications; but W, Mo, Os, and Ta have very poor oxidation resistance, and molybdenum oxide is very volatile.

3 Optical Model

Designs of solar-selective coatings with multiple layers were optically modeled using thin-film design software [9]. Multilayer coatings were modeled because they were significantly easier to model than cermets, with the expectation of later converting the best multilayer design into a cermet. The absorptance was increased and the

number of layers was reduced from the initial design by further modeling to a construction that is significantly easier to deposit. Solar-selective coatings with optical properties exceeding the goals (absorptance of 0.959 and emittance of 0.070 at 450°C) were modeled for materials with high thermal stability (Table 1). The modeled performance exceeds the goal specification by about 1% overall, as 1% in emittance is about equivalent to 1.2% in absorptance. The comparison between the modeled reflectance for NREL 6A and the measured reflectance of the commercially manufactured improved Al₂O₃-based cermet with an ideal solar-selective coating is shown in Fig. 2. Spectral absorptance and emittance can be expressed in terms of total reflectance $\rho(\lambda, \theta)$ for opaque materials from Kirchoff's law,

$$\alpha(\lambda, \theta) = 1 - \rho(\lambda, \theta) \quad (1)$$

$$\varepsilon(\lambda, T) = \alpha(\lambda, T) \quad (2)$$

NREL is pursuing the intellectual property of the modeled coatings. Incorporating improved AR coatings, cermets, and texturing the surface should further improve the solar-selective coating; however, trade-offs exist between simultaneously obtaining both low emittance and high absorptance. There is significant uncertainty regarding the real property values of the modeled selective coatings, and the key issue has become making the coating and testing its actual properties.

Table 1. Theoretical optical properties for NREL's modeled prototype selective coating compared with actual optical properties of existing commercial selective coatings.

	Original Mo-Al ₂ O ₃ Cermet	Improved Al ₂ O ₃ -based Cermet	NREL#6A
Solar α	0.938	0.954	0.959
Thermal ε @			
25°C	0.061	0.052	0.027
100°C	0.077	0.067	0.033
200°C	0.095	0.085	0.040
300°C	0.118	0.107	0.048
400°C	0.146	0.134	0.061
450°C	0.162	0.149	0.070
500°C	0.179	0.165	0.082

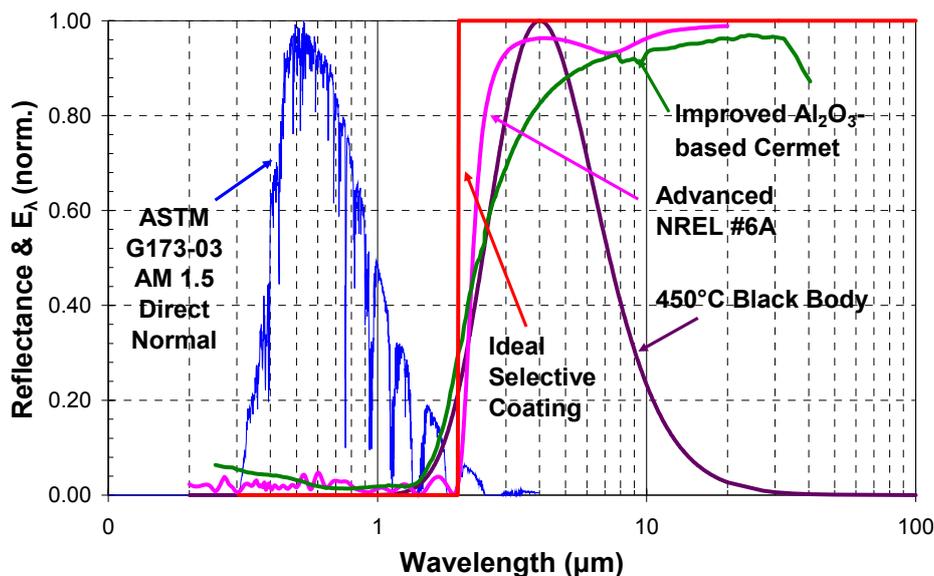


Figure 2. Modeled data for NREL #6A coating.

4 Deposition of Modeled Coating

Deposited solar-selective coatings that reproduce the modeled coatings are likely to be successful and meet the CSP optical and durability requirements. To begin solar-selective prototyping, the IBAD e-beam chamber in the

NREL multichamber vacuum system was used because of the versatility and lower cost for initial material prototyping. The e-beam gun uses 7 cubic centimeter (cc) crucibles that hold small volumes of materials to deposit six materials sequentially or co-deposit two different materials simultaneously. Dielectrics can be evaporated directly or reactively, and an ion gun can be used to improve the quality, composition, and density of the coating.

AR coatings are typically one-quarter-wavelength stacks of dielectrics of alternating high and low refractive indices. Layer thickness control is therefore critical to the optical properties, and deposition parameters are critical to the microstructure, which defines the mechanical and thermal properties. Accordingly, pneumatic shutters were installed and wired to the quartz-crystal thin-film controller to allow control of the critical layer thicknesses, and the thin-film controller was calibrated. The easiest, simplest, lowest-cost solution of evaporating compounds was tried first, because co-deposition would require further modification of the deposition chamber by installing the co-deposition plate with two e-beam guns and purchasing a second e-beam sweep control, additional quartz-crystal sensors, and shutters.

Individual layers in the architecture were reactively evaporated from substoichiometric starting compounds with increasing amounts of reactive gas and ion assist. To determine the appropriate level of ion assist for a dense adherent coating, different levels (high, low, and none) of ion assist were examined. The individual layers for the NREL#6A architecture were deposited and their properties were characterized: step profilometry for thickness; ellipsometry for thickness and index of refraction; ultraviolet-visible-near-infrared (UV-Vis-NIR), infrared (IR), and Fourier transform infrared (FTIR) spectrophotometry for optical properties; scanning electron microscopy (SEM) and atomic force microscopy (AFM) for morphology and surface roughness, and X-ray photoelectron spectroscopy (XPS) analysis for stoichiometry. SEM and AFM confirmed that the layers' morphology moved from columnar to a dense structure with increasing levels of ion assist. The optical properties, stoichiometry, and optimum reactive gas flow and partial pressure for the individual layers were determined. XPS verified that stoichiometric oxide layers were deposited from substoichiometric starting compounds. More importantly, the XPS results showed that the method of directly evaporating the binary-compound reflective layers from substoichiometric compounds would not give highly absorbing, low-emittance coatings because the compounds evaporated preferentially into layers of A/mixed AO_x oxides/B/mixed BO_x oxides from bottom to top. [It should be noted that because NREL is pursuing the intellectual property of the modeled solar-selective coating, the binary compound in the deposited coatings are designated as AB (stoichiometric) or A_xB_y (substoichiometric), and likewise the oxides are AO_x or A_xO_y and BO_x or B_xO_y .] While the individual layers were characterized, the modeled NREL#6A architecture structure was deposited and characterized. The optical performance of the compound NREL#6A was quite encouraging, despite having deposited the layered structure instead of a compound for the reflective layer, and systematically missing the modeled layer thicknesses from -1.3% to +26.1%. These results were not unexpected; but the easiest, lowest-cost solution of evaporating compounds was tried prior to the more expensive and more equipment-intensive method of co-deposition.

From these results, it was determined that the compound reflective layer and the solar-selective coating would need to be deposited by co-deposition to produce a solar-selective coating closer to that modeled. This required extensive upgrades/modifications to the deposition system, including the following: installing the co-deposition plate with two e-beam guns; installing a lift for the e-beam deposition plate for safety considerations; installing a second crystal sensor; replacing the e-beam sweep and installing a second e-beam sweep; installing shutters for each e-beam gun and the substrate; modifying the concomitant wiring and plumbing; upgrading the cooling-water manifold; upgrading the residual-gas analyzer (RGA) and software; upgrading/repairing the gas flow controller; rebuilding a roughing pump; and starting automation of the deposition process.

After the major modifications were completed, the individual layers of the architecture were evaporated from elemental starting materials by reactive e-beam co-deposition. By manually varying the power and deposition rate of the two materials and/or increasing the amounts of the reactive gas, compounds with various compositions were produced. The individual layers and the modeled NREL#6A architecture were deposited by directly and reactively evaporating the compound layers by elemental co-deposition and were then characterized. The coating was dark blue and the absorption was improved; but the absorption was lower and the emittance was higher than the modeled coating. The results were quite encouraging that the coating will perform as modeled, even though the measured optical performance of the deposited coatings was lower than the modeled performance; this result is because of thickness and compositional errors known to have occurred during the coating deposition.

From error analysis, errors in layer thickness lead to errors in the absorption. The layer thicknesses for the co-deposited solar-selective coating were overshoot by up to 52.7% or undershot by up to 10.3% because of manual control. An optical coating is only as good as the optical monitoring and control during deposition. In most cases, to achieve high yield for demanding requirements, it is necessary to minimize the optical thickness errors below 1%, particularly at sensitive turning points. NREL's initial monitoring and control errors greatly exceeded 5% for the deposited thin-film thickness; therefore, the measured performance for coatings was inferior to the modeled coating, as expected. To resolve the thickness errors, the deposition monitor and control will be upgraded with the addition of an optical monitor, providing positive feedback between the quartz-crystal monitor and the optical monitor, and automating the coating process. Automation will remove human error from the coating deposition process. Also, by providing steering and cutting at sensitive turning points, mid-course corrections can be made for any thickness errors in order to deposit a coating that matches the model.

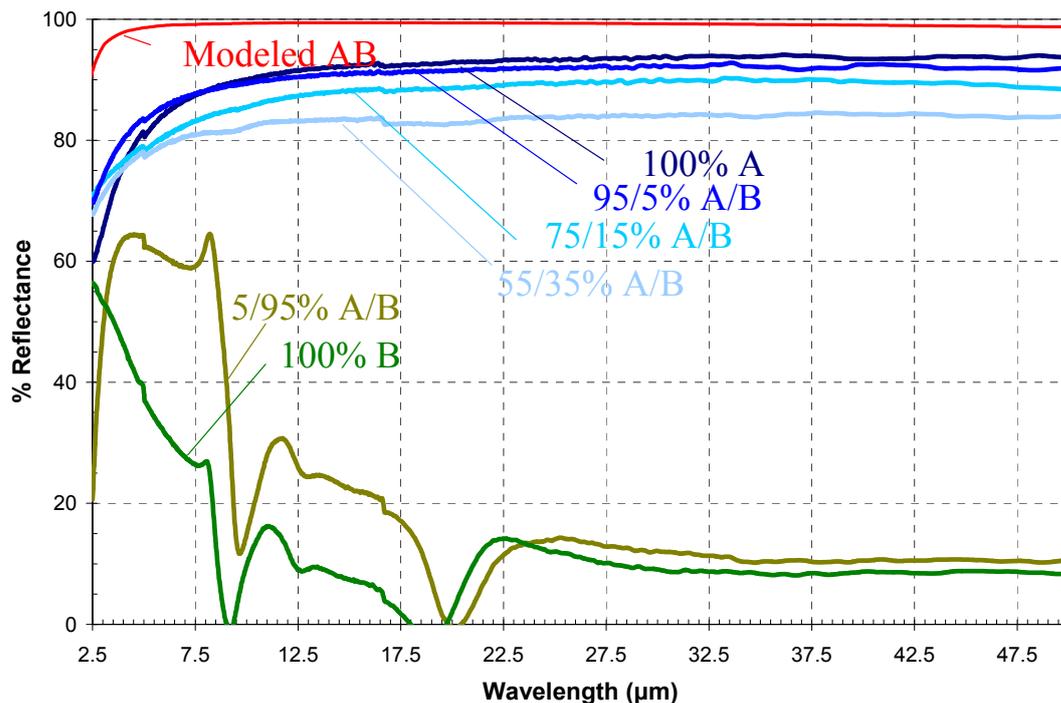


Figure 3. Spectral reflectance of co-deposited AB binary compound reflective layer on glass with different A and B composition ratios (measured on PE 883 IR spectrophotometer from 2.5 to 50 μm at 25-nm intervals)

From error analysis, errors in the stoichiometry cause errors in both absorption and emittance. The primary reflective layers of the solar-selective design are binary compounds. The thickness of the individual layers and the composition was determined during deposition using a quartz-crystal microbalance. Shown in Fig. 2, the spectral reflectance of the binary compound varies significantly in the NIR-IR range depending on the ratios of the two constituent materials. A couple of key items were observed. First, the titanium-rich compositions have a higher reflectivity in the IR—corresponding to a low emittance—and a sharper band edge. Second, the deposited coating as measured with the XPS had a poor correlation with the predicted composition from the quartz-crystal microbalance during the deposition run. The coating stoichiometry of the primary reflective layers was not optimized, which caused differences between the modeled and deposited coating. To resolve the compositional errors, the optimal compound stoichiometry and the thin-film phase-formation sequence will be determined. In addition, by varying the ion assist, the optimal coating density will be determined that gives both high absorption and low emittance with excellent oxidation resistance. Depositing these optimized individual materials in the solar-selective coating without the thickness and composition errors should result in a deposited solar-selective coating closer to that modeled.

5 Characterization and Stability of Solar-Selective Coating

Developing spectrally selective materials also depends on reliable characterization of their composition, morphology, and physical and optical properties. The key for high-temperature usage is low ϵ . NREL has been developing the protocols and building the capability for accurate, precise measurements of the thermal/optic

properties of the selective coating. Therefore, a round-robin experiment was conducted with two commercial laboratories and NREL, with IR reflectance and high-temperature capabilities. The three laboratories measured two improved cermet samples: the original Mo-Al₂O₃ cermet sample, and a black Ni at room temperature. One commercial laboratory measured improved cermet samples at incident angles of 15°, 30°, 45°, and 60° at room temperature from 0.3 to 26 μm. NREL's FTIR laboratory and the second commercial laboratory made measurements on the improved Al₂O₃-based cermet samples from 1.5 to 26 μm at 200° and 400°C, and again, at room temperature after being heated. Preliminary results comparing the high- and low-temperature measurements and between the two commercial laboratories showed that the measurements were within measurement error. However, the NREL FTIR measurements were very noisy at the transition around 2000 nm because of FTIR equipment issues, and there was a large discontinuity at 2500 nm between the FTIR and UV-VIS-NIR results. In addition, the results took significant time to receive. For more rapid progress, a Perkin-Elmer (PE) 883 IR spectrophotometer (883) was purchased to furnish the capability to immediately measure the optical properties of deposited films and provide more timely feedback to the deposition process. This allows the individual layers and solar-selective coatings to be deposited, optical properties measured with the 883, results of the optical measurements inputted back into the model and deposition process, and further refinements made to the model and deposited coating. Solar-selective samples measured with the 883 had a very smooth transition, and the discontinuity at 2500 nm between the IR and the UV-Vis-NIR measurements was reduced to less than 4%. However, the IR reflectance spectra measured on the 883 are lower than the spectra of the same cermet samples measured on newer instruments at Surface Optics Corporation (SOC), shown in Fig. 4. Therefore, the optical properties of the co-deposited NREL 6A coating are probably better than were measured. Recently, the 883 was calibrated and new National Institute of Standards and Technology (NIST) traceable gold IR standards and a 3X Beam Condenser Specular Reflectance accessory were purchased to more accurately measure the samples with the 883. We need to resolve the problem with the low reflectance measurements, purchase a more-accurate instrument, or subcontract the measurements. Until that time, the PE 883 IR spectrophotometer can be used to evaluate relative improvements in the deposited coating, but not to determine the final optical properties of the coating.

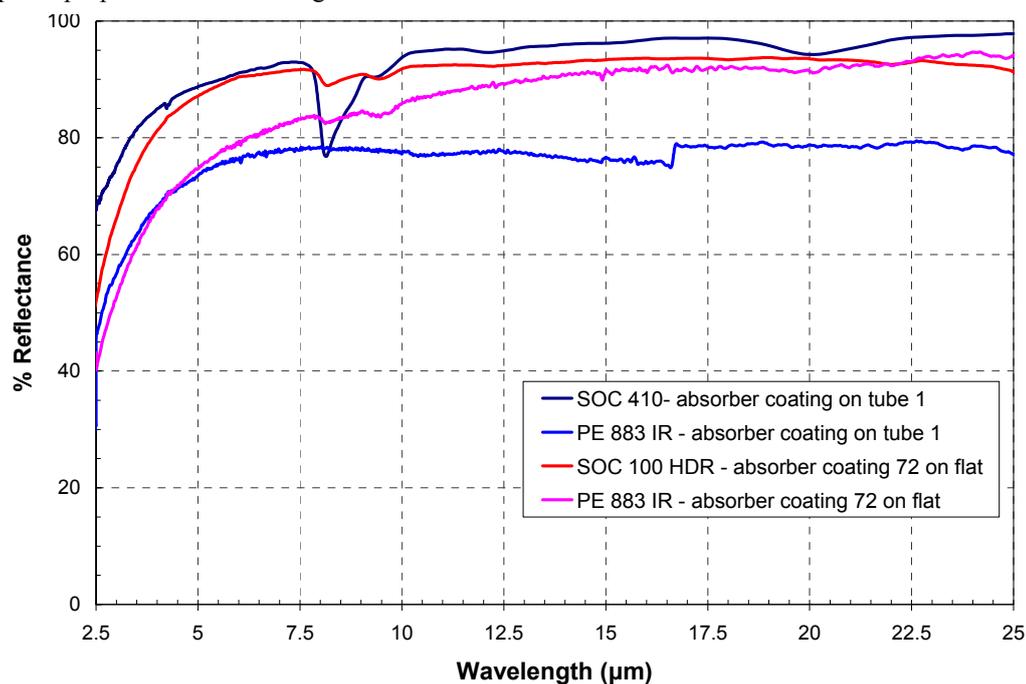


Figure 4. Comparison of NREL PE 883 IR spectrophotometer measurements and SOC 410 Directional Hemispherical Reflectometer and 100 Hemispherical Directional Reflectometer IR measurements for two different cermets on flat and tubular substrates.

The long-term thermal stability above 450°C, ideally in air, is an important requirement for the solar-selective coating. Compared to the desired lifetime (5–30 years), durability or thermal stability is usually tested by heating the selective coating—typically in a vacuum oven, but sometimes in air—for a relatively short duration (hundreds of hours). To determine the durability and thermal stability of commercial and NREL's spectrally selective coatings under development, a high-temperature inert-gas oven was purchased that allows the coatings to be exposed at their operating temperatures and conditions for long periods of time. As data become available,

development can begin of a criterion for high-temperature selective surfaces applicable for concentrating applications.

6 Future Work

Using computer-aided design software, a solar-selective coating with $\alpha = 0.959$ and $\varepsilon = 0.070$ at 450°C composed of materials stable at high temperature has been successfully modeled optically. This exceeds the goal specification by about 1% overall, because a 1% improvement in emittance is equivalent to about 1.2% in absorptance. Producing the modeled coating prototype is now imperative.

The individual layers were deposited by directly and reactively evaporating the compound layers from substoichiometric compounds and by elemental co-deposition. But because of thickness, compositional, and measurement errors, the optical performance of the deposited coating did not match the modeled performance. To resolve the compositional errors, the optimal compound stoichiometry and the thin-film phase-formation sequence must be determined. To resolve the thickness errors, the optical monitoring must be upgraded and the coating process needs to be further automated. In addition, by varying the ion assist, the optimal coating density must be determined that gives both high absorption and low emittance with excellent oxidation resistance. NIST-traceable gold IR standards and a 3X Beam Condenser Specular Reflectance accessory will be used to more accurately measure the samples with the 883 to evaluate relative improvements in the deposited coating. To determine the final optical properties of the coating, measurements will be subcontracted until more-accurate instruments are purchased.

Further modeling refinements and the effect of errors during deposition will be investigated with the software. The entire heat-collection element (air/glass/AR coating, vacuum, AR coating/solar-selective coating/stainless steel) structure will also be modeled. The optical properties of the deposited film layers will be measured, optical constants extracted, and constants applied back into the model. Modeling a cermet solar-selective coating and incorporating texturing will follow. Improvements are expected by incorporating improved AR coatings, cermets, and textured surfaces. However, trade-offs exist between low emittance and high absorptance. If needed, we will try methods to texture the surface.

Future plans include converting the model from a multilayer to a cermet, depositing the cermet architecture, and trying methods to texture the surface. A hydrogen barrier and AR layers may be added to the steel substrate and glass envelope, respectively. In thin-film deposition, a good general rule of thumb is the following: the lower the process pressure and the more energetic the process, the more control exists over the film properties but the higher the cost of the equipment and coating. PVD e-beam/IBAD techniques were selected to give greater control over the film properties. The intention is that after a viable high-temperature solar-selective coating is developed by e-beam, then the coating will be demonstrated by the lower-cost more-commercial method of sputtering, the feasibility of lower-cost methods will be explored, and coating industry experts will perform a rigorous cost analysis.

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