













Development and Testing of Abrasion Resistant Hard Coats for Polymer Film Reflectors

Preprint

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Presented at SolarPACES 2010 Perpignan, France September 21-24, 2010

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Conference Paper NREL/CP-5500-49273 October 2010

Contract No. DE-AC36-08GO28308

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DEVELOPMENT AND TESTING OF ABRASION RESISTANT HARD COATS FOR POLYMER FILM REFLECTORS

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Abstract

Reflective polymer film technology can significantly reduce the cost of solar reflectors and installed Concentrated Solar Power (CSP) plants by both reduced material cost and lower weight. One challenge of polymer reflectors in the CSP environment pertains to contact cleaning methods typically used with glass mirrors. Such contact cleaning methods can scratch the surface of polymer reflectors and thereby reduce specular reflectance. ReflecTech, Inc. (a subsidiary of SkyFuel, Inc.) and the National Renewable Energy Laboratory (NREL) initiated a cooperative research and development agreement (CRADA) to devise and develop an abrasion resistant coating (ARC) suitable for deposition onto polymer based mirror film. A number of candidate ARC products were identified as candidate formulations. Industrial collaborators prepared samples having their ARCs deposited onto ReflecTech Mirror Film pre-laminated to aluminum sheet substrates. Samples were provided for evaluation and subjected to baseline (unweathered) and accelerated exposure conditions and subsequently characterized for abrasion resistance and adhesion. An advanced ARC product has been identified that exhibits outstanding initial abrasion resistance and adhesion to ReflecTech Mirror Film. These properties were also retained after exposure to the various accelerated stress conditions. This material has been successfully manufactured as a 1.5 m wide roll-to-roll construction in a production environment.

Keywords: Abrasion resistant hardcoat, ARC, polymer film reflector, specular reflectance, coating adhesion

1. Introduction

The desired ARC properties are high transparency and optical clarity, weatherability, abrasion resistance, strong adhesion to the base mirror film substrate, compatibility with high volume roll-to-roll production, and low material and manufacturing costs. A number of candidate ARC products such as UV-cured thermoset acrylates, polyurethanes, and vacuum deposited inorganic oxide hardcoats were identified as candidate formulations. Industrial collaborators prepared samples having their ARCs deposited onto ReflecTech Mirror Film pre-laminated to aluminum sheet substrates (~10 cm x 10 cm size). ARC coated samples were provided and were evaluated for baseline (unweathered) abrasion resistance and adhesion to the polymer film reflector material. Additional samples were also subjected to a variety of accelerated exposure conditions and subsequently tested for abrasion resistance and adhesion. Once the optimal ARC formulation was ascertained, additional laboratory batch samples were prepared, roll-to-roll pilot and production scale materials were produced and further weathering tests were conducted.

2. Experimental

A comprehensive test plan was devised to evaluate and demonstrate the abrasion protective properties of candidate hardcoats both as initially formulated/applied and as a function of expected in-service environmental conditions. Appropriate aspects of the plan included measurement of properties directly of

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interest (e.g. specular reflectance at several acceptance angles), use of applicable/relevant substrates (ReflecTech Mirror Film laminated to standard mill-finish aluminum), and exposure of candidate samples to the types of conditions expected in service both in terms of weathering and contact cleaning.

2.1. Screening Tests

In addition to demonstrating that the initial optical properties of the hard-coated ReflecTech Mirror Film are satisfied, it was important to confirm that abrasion resistance of unweathered materials is maintained after representative weathering has been experienced. For screening purposes, accelerated test exposures included UV light, condensation cycling, and thermal cycling. Candidate samples were subjected to UV exposure in an Atlas Ci5000 WeatherOmeter[®] as per ASTM G155 [1]. A xenon arc lamp source was filtered to provide a light intensity of about 2X the terrestrial solar spectrum. Temperature and relative conditions were held constant at 60°C and 60% respectively. A Q-Lab QUV chamber was used to expose samples to cyclic condensation according to ASTM D4587 [2]. A UVA-340 light source was used having a 290-340 nm UV light spectrum. Samples were cycled between dry exposure at 60°C with the light source on for 4 hours and 100% relative humidity (condensation) at 30°C in the dark for 4 hours. ASTM D6944 (Method B) was used to perform thermal cycling tests of candidate samples [3]. Each cycle held the samples at -5°C for 16 hours and then 50±3°C for 8 hours; 30 cycles were completed.

A modified version of ASTM Standard D4060 was used to quantify abrasion resistance [4]. Abrasive stress was applied using a Taber abraser unit and a Devices and Services Specular Reflectometer was used to optically assess abrasive damage to the candidate ARCs and thereby monitor the performance parameter of direct interest namely, specular reflectance, as a function of the number of cycles. Taber abrasion is a severe stress compared to normal cleaning measures used in solar fields but it does allow a standardized protocol to obtain meaningful intercomparisons of candidate materials samples. In terms of available standardized test procedures, the Taber abraser test is reported to have the highest correlation with the appearance of hardcoated films in actual use [5]. A minimum 250 g arm weight was used. Specular reflectance was measured at 660 nm, $\rho_s(\theta,\lambda=660)$, at $\theta=7.5$ and 12.5 mrad (0.43°, and 0.72°) half acceptance angles. Specular reflectance was measured at four spots on each 10 cm x 10 cm sized sample before abrasion, after 10 cycles, and after 30 cycles.

In addition to maintaining abrasion resistance, it is important that the ARC retain good adhesion to the ReflecTech Mirror Film underlayer before, during, and after weathering. Coating adhesion was measured using the ASTM D3359 cross-hatch tape peel test [6].

2.2. Additional Accelerated Weathering Tests

Beyond the requisite baseline abrasion resistance and adhesion properties demonstrated in screening tests, intrinsic UV weatherability is an essential requirement of organic-based materials (such as polymer mirror films and UV-cured acrylate coatings) that are expected to exhibit extended lifetimes under outdoor service conditions. The performance and durability properties of the standard ReflecTech Mirror Film product have been previously documented [7]. The most promising candidate ARC samples that passed the screening tests were subsequently subjected to further accelerated weathering tests to determine their resistance to UV exposure. Two accelerated UV exposure chambers were used. The first was an artificial solar simulator light source that uses a 1.4 kW xenon arc lamp to provide a ~2X terrestrial solar spectrum in the 300-500 nm bandwidth [1]. Four sets of identical samples are concurrently exposed under constant irradiance in each of four chamber quadrants that experience four combinations of temperature and relative humidity. This allows the effects of these stresses to be determined in parallel with resistance to UV.

Samples were also exposed at NREL's ultra-accelerated weathering system (UAWS) [8]. The UAWS uses special optical facets to reflect primarily the UV wavelengths of terrestrial natural sunlight to avoid overheating the samples during exposure. The UAWS facets use interference coatings deposited onto glass. These consist of multiple alternating layers of materials with high and low refractive indexes to selectively reflect the UV. The near infrared and visible wavelengths are attenuated to reduce the thermal loading of the material under test, since otherwise the intense heat would render the testing invalid. ARC coated

ReflecTech Mirror Film samples were exposed at 100X concentration of natural UV sunlight for an equivalent of over 10 years outdoor exposure at 30° and 60° C.

To supplement Taber abrasion testing, another ASTM test (D2486) was also performed [9]. This test uses linearly articulated scrub brushes that are cycled in a back-and-forth motion 37 times per minute across the surface of ARC coated ReflecTech Mirror Film samples. A Byk Model PB5005 wet abrasion scrub tester was used to perform this test. Although designed to quantify the abrasion resistance of paint products, the test can simulate the most aggressive of solar mirror field cleaning processes.

As a further test of coating adhesion, samples of ARC coated ReflecTech Mirror Film laminated to aluminum sheet (36 cm x 76 cm) were immersed in deionized water for extended timeframes. The coating was inspected for visual delamination and the cross hatch test [6] was used to further quantify durability.

3. Results

3.1. Screening Test Results

Abrasion resistance of candidate hardcoat formulations was screened by comparing the measured specular reflectance after 30 Taber abrasion cycles with measurements made before abrasion. Several dozen prospective vendors were contacted and requested to submit samples of ARC coated ReflecTech Mirror Film for evaluation. Seven hardcoat companies provided samples; these are designated as B through H in Table 1. Several of these prepared more than one formulation indicated by the numeric suffix in Table 1. Uncoated ReflecTech Mirror Film was included for comparative purposes and is denoted as vendor-formulation A-1.

		Change in Specular Reflectance [Δρ _s (θ=12.5 mrad)] after 30 Taber Cycles as a Function of Type of Weathering					
Vendor -			Ci5000	Condensation Cycles	Thermal Cycles		
Formulation	Coating Type	None	(925 MJ/m ² UV)	(215 cycles)	(30 cycles)		
A-1	None	43.5					
B-1	TiO ₂	43.9					
C-1	UV Cured Acrylate	1.1	2.6	7.2	2.5		
C-2	UV Cured Acrylate	0.2	2.9	10.3	2.5		
C-3	UV Cured Acrylate	1.5	3.2	4.7	1.7		
C-4	UV Cured Acrylate	1.6	3.1	1.4	1.6		
D-1	UV Cured Acrylate	8.0	0.2 ^b	2.8	1.8		
D-2	UV Cured Acrylate	3.1	4.1 ^b	7.5	3.8		
E-1	UV Cured Thermoset Acrylate	7.2	17.3 ^b	9.1	4.6		
F-1	PECVD SiO _x N _y	13.0°					
G-1	Polyurethane	18.5					
G-2	UV Cured Acrylate	0.0	b				
H-1	Polyaspartic				28.3		
H-2	Polyaspartic		С				

a After 10 Taber cycles

Table 1. Abrasion resistance of candidate ARC coated ReflecTech Mirror Film

Candidates B-1, F-1, and G-1 had unacceptable unweathered abrasion resistance, exhibiting over 10% loss in specular reflectance at 12.5 mrad half acceptance angle after 30 Taber cycles. The loss in specular reflectance of the unweathered E-1 sample was also quite high, and this sample also had poor abrasion resistance after weathering. In addition, E-1 severely yellowed after UV exposure equivalent to about 3 years outdoors (925 MJ/m² UV). Coating G-2 demonstrated excellent unweathered abrasion resistance but severely yellowed after Ci5000 exposure. Candidate H-1 was not tested prior to weathering but exhibited poor abrasion resistance after thermal cycling. The H-2 coating formulation reduced the unweathered solar-weighted hemispherical reflectance to unacceptable values (~86%). The two formulations provided by vendor D

^b Severe yellowing after UV exposure

Coating unduly degrades unweathered reflectance

included a commercial product (D-1) and an experimental coating (D-2). D-2 showed marginal abrasion resistance unweathered and also after the various exposure conditions. The abrasion resistance of D-1 was excellent. The manufacturer claimed a 2 year useful outdoor lifetime. After two years equivalent outdoor exposure in the Ci5000, candidate D-1 had not degraded. However, after 3 years equivalent exposure the coating had severely yellowed.

Samples C-1 through C-4 were provided by Red Spot Paint & Varnish Company, Inc. These all demonstrated excellent unweathered abrasion resistance. C-1 and C-2 were existing ARC formulations used by Red Spot for other products and applications. C-3 and C-4 were new formulations offered by Red Spot that were tailored to the particular specifications for polymer film mirrors. The Red Spot samples all demonstrated very good abrasion resistance after 3 years equivalent outdoor UV exposure in the Ci5000 WeatherOmeter. Importantly, the C-3 and C-4 samples were returned to Ci5000 exposure for additional UV weathering. After a cumulative ~6.2 equivalent outdoor exposure these materials showed no yellowing or loss in spectral hemispherical reflectance. Formulation C-4 was also successfully re-formulated to withstand loss in abrasion resistance after condensation cycles, as indicated in Table 1. This formulation was chosen as the top candidate to scale up through pilot and production trials to a commercial product.

The abrasion resistance of the C-4 material is shown visually in Figure 1. The picture on the left (Figure 1a) illustrates the severe abrasion (circular track) experienced by uncoated ReflecTech Mirror Film after 30 Taber abraser cycles. When ReflecTech Mirror Film is coated with the C-4 formulation the effect of abrasion is nearly imperceptible (Figure 1b). Also evident in the right-hand photo is the cross-hatch pattern used to test coating adhesion. None of the Red Spot samples exhibited loss of adhesion before or after weathering.

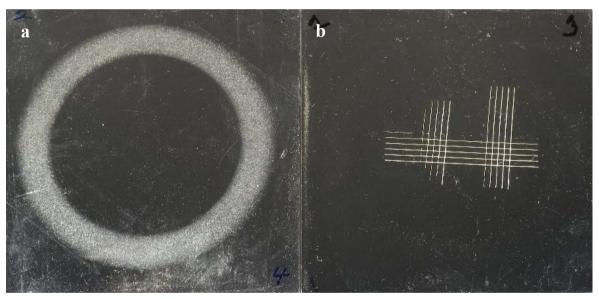


Figure 1. Digital image after 30 Taber abraser cycles for polymer film reflector samples (a) without and (b) with abrasion resistant coating. The crosshatch pattern on right resulted from test of coating adhesion.

Figure 2 presents measured specular reflectance data for the corresponding abraded ReflecTech Mirror Film and abraded ARC-coated ReflecTech samples. Without the ARC hardcoat, specular reflectance severely degrades after 10 and then 30 Taber abrasion cycle (Figure 2a). When coated with the C-4 ARC, the specular reflectance at 7.5 and 12.5 mrad half acceptance angles are virtually unchanged from the unabraded uncoated ReflecTech Mirror Film values (Figure 2b).

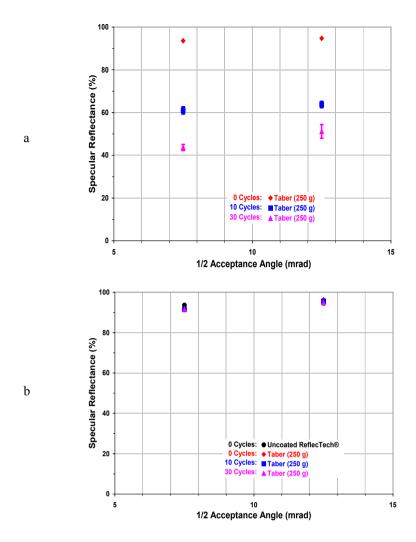


Figure 2. Specular reflectance measurements after 0 (initial), 10, and 30 Taber abrasion cycles of abraded regions of polymer film reflector samples (a) without and (b) with abrasion resistant coating.

3.2. Additional Accelerated Weathering Test Results

Screening tests discussed above identified an advanced ARC product from Red Spot Paint & Varnish Company, Inc. that exhibits outstanding initial abrasion resistance and adhesion to ReflecTech Mirror Film. These excellent properties are also retained after exposure to various other accelerated stress conditions. Figure 3 demonstrates the outstanding UV resistance of the ARC-ReflecTech material. After the equivalent of over 10 years UV exposure in NREL's UAWS there is no significant spectral loss in hemispherical reflectance. The small absorption band at ~1900 nm occurs at a region in the solar spectrum where very little solar resource is available. These results are for samples

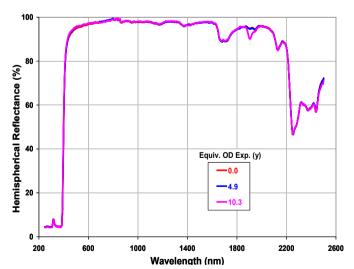


Figure 3. Spectral hemispherical reflectance of ARC coated ReflecTech samples after 0 (initial), 4.9, and 10.3 years equivalent UV exposure. Samples exposed at 30°C.

exposed at 30°C. Samples of this hardcoat have also demonstrated similar weatherability when exposed at the UAWS at a highly elevated temperature of 60°C.

Samples have also been exposed in NREL's solar simulator chamber. Figure 4 presents results for samples being exposed at 35°C and 5% relative humidity and at 60°C and 5% relative humidity. These samples have withstood a cumulative UV equivalent to nearly 8 years outdoors in without loss solar-weighted hemispherical reflectance. Companion samples being exposed at the same temperatures, but at 70% relative humidity, were discontinued for routine chemical analysis after vears equivalent outdoor UV exposure without degraded reflectance.

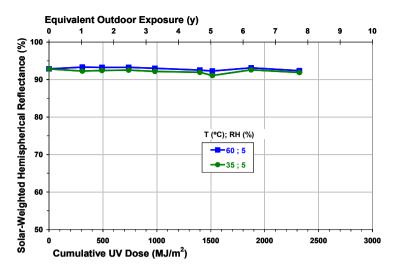


Fig 4. Accelerated UV exposure of ARC coated ReflecTech in the NREL Solar Simulator chamber at 60°C, 5% RH & 35°C, 5% RH.

Taber abrasion is a particularly harsh test for abrasion resistance and is not representative of the type of abrasive stresses that solar mirrors might experience during reflector cleaning. It has, however, provided a

standardized protocol that has allowed candidate competing ARC formulations to be compared and ranked. A slightly more realistic (although less aggressive) abrasion test subjects samples to scrub brush induced wear. This test was applied to the production version of ARC coated ReflecTech Mirror Film. As can be seen in Figure 5, no loss in specular reflectance ($\theta = 7.5$ and 12.5 mrad) has occurred after over 50,000 back-andforth scrub brush cycles which simulates more than 50 years of wear. Furthermore, no scratches are visibly evident.

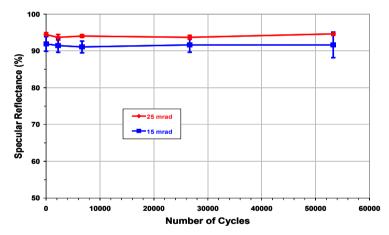


Figure 5. Abrasion resistance exhibited by production run application of ARC to ReflecTech in terms of scrub brush cycles.

Samples of ARC coated ReflecTech Mirror Film laminated to aluminum panels were immersed in deionized water for 30 days. No visual indication of coating delamination was evident. The cross hatch tape peel test was also performed and no loss of adhesion was found.

3.3. Scale-Up Results

Based on screening test results and further accelerated testing, the Red Spot ARC formulation was selected to be used in a manufacturing environment. Prospective hardcoat vendors were contacted and evaluated regarding their experience and ability to apply the ARC hardcoat to ReflecTech Mirror Film in a roll-to-roll production condition. Laboratory samples were subsequently scaled up to pilot and ultimately production trials. At each step in this process sample materials were retested for their abrasion resistance, adhesion, and weatherability. Specular reflectance after abrasion for the Red Spot laboratory-prepared, pilot, and production trials have all been excellent. As shown in Table 2, for the batch laboratory prepared ARC sample 30 Taber abraser cycles typically resulted in a change in specular reflectance at both 7.5 and 12.5 mrad half

acceptance angles of ~1.6%. This same excellent abrasion resistance was maintained by comparable samples during the pilot run. All five pilot trials resulted in minimal specular reflectance loss with abrasion (~1-1.5% at 15 and 25 mrad). For samples prepared during the production trial the sample performance was again similar to the lab and pilot prepared samples with $\Delta \rho_s(\theta=15,25 \text{ mrad}) \sim 1.5\%$.

Sample Preparation	Change in Specular Reflectance [Δρ _s (θ)]			
	after 30 Taber Cycles as a Function of Type of Weathering			
	$\theta = 7.5 \text{ mrad}$	$\theta = 12.5 \text{ mrad}$		
Laboratory	1.6	1.6		
Pilot	0.4	1.4		
Production	1.3	1.5		

Table 2. Maintenance of abrasion resistance from laboratory to pilot to production sample preparation.

4. Conclusions

An advanced ARC product applied to ReflecTech Mirror Film has demonstrated excellent abrasion resistance, adhesion, and weatherability. A UV curable acrylate formulation from Red Spot Paint & Varnish Company, Inc. withstands Taber and scrub brush abrasion to the extent that visual affects are nearly imperceptible and measured specular reflectance is virtually unchanged. Resistance to Taber abrasion is maintained after exposure to UV light, condensation cycling, and thermal cycling. The hardcoat has demonstrated outstanding weatherability that includes highly accelerated exposure to over 10 years equivalent outdoor UV. This material has been successfully scaled up from batch laboratory samples through pilot conditions to being manufactured as a 1.5 m wide roll-to-roll construction in a production environment.

Acknowledgements

Dick Burrows and Christa Loux made many of the measurements discussed herein. Weathering of samples at the UAWS was performed by Carl Bingham at NREL. We especially thank Pat Peach and Kristy Wagner at Red Spot Paint and Varnish who provided their candidate ARC hardcoat formulations and invaluable advice and technical support. NREL's part of this CRADA work was performed under DOE contract DE-AC36-99-GO10337.

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	October 2010	С	onference Paper	•				
4.	TITLE AND SUBTITLE					TRACT NUMBER		
	Development and Testing of A		n Resistant Hard	d Coats for	DE-	AC36-08GO28308		
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					5c. PRO	GRAM ELEMENT NUMBER		
6.	AUTHOR(S)				5d. PROJECT NUMBER			
	Gary Jorgensen, Randy Gee,	and M	ichael DiGrazia		NREL/CP-5500-49273			
					5e. TASK NUMBER			
					TC07.5011			
					5f. WOF	RK UNIT NUMBER		
7	PERFORMING ORGANIZATION NA	ME(S) /	ND ADDRESS(ES)			8. PERFORMING ORGANIZATION		
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9	SPONSORING/MONITORING AGEN	ΙΟΥ ΝΔΙ	ME(S) AND ADDRES	SS(ES)	10. SPONSOR/MONITOR'S ACRONYM(S)			
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12.	2. DISTRIBUTION AVAILABILITY STATEMENT National Technical Information Service							
	U.S. Department of Commerce							
	5285 Port Royal Road							
	Springfield, VA 22161							
13.	SUPPLEMENTARY NOTES							
14.	14. ABSTRACT (Maximum 200 Words)							
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15	production environment. 15. SUBJECT TERMS							
10.	Abrasion resistant hardcoat; ARC; polymer film reflector; specular reflectance; coating adhesion							
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