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"Invited Paper"

Exposure Testing of Solar Absorber Surfaces*

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Abstract

The Los Alamos National Laboratory has been involved in supporting, monitoring and conducting exposure testing of solar materials for approximately ten years. The Laboratory has provided technical monitoring of the ITRI¹, DSET², Lockheed³, and Berry⁴ contracts and has operated the Los Alamos exposure Facility for over five years. This report will outline some of the past exposure testing, the testing still in progress, and describe some of the major findings. While this report will primarily emphasize solar absorber surfaces, some of the significant findings relative to advanced glazing will be discussed.

Introduction

The determination of materials durability is equally if not more important than initial performance or cost in establishing overall cost effectiveness. For most applications, rates and degree of degradation are important factors in determining cost effectiveness. Although past exposure testing has included both mechanical and optical property evaluations, recent funding limitations have created focus on the most easily obtained, i.e., optical testing and visual effects. This paper will concentrate on the optical results obtained.

Many developmental materials were generated under funding by the DOE Program Research and Development Announcement (PRDA) and Request for Proposal (RFP) processes. Many of these materials and products have been included in the Los Alamos exposure testing program in an attempt to identify those that might evolve into durable commercial products. Other materials being exposed and evaluated are continuations of the Los Alamos efforts to develop inexpensive selective absorber coatings, like the chemical conversion coatings or selective paints. Promising advanced coatings are being included as they are developed.

Long term real time data is also needed in which to compare accelerated test methods. The Los Alamos project will attempt to provide the long term exposure data upon which failure mechanisms and accelerated testing correlations can be based.

History of types of testing

There has been a wealth of durability testing conducted on solar materials over the past ten years. Table 1 describes, in matrix form, some of the investigators and the types of tests that have been conducted. This is not intended to be a complete list, however, it does cover many of the major contributors that have received DOE support. Many additional tests have been conducted by commercial organizations or testing that has been contracted for and done by commercial testing laboratories that is not available to the general public.

Much effort has been exerted to bring solar standards and standard test methods into effect. The ASTM Subcommittee E-44.04 has been the forerunner in this activity.

Los Alamos testing program

The Los Alamos Exposure Test Program is presently focused on outdoor real time exposure testing and the determination of optical changes on physical changes and failures.

Los Alamos performs two basic functions in exposure testing:

1. research and development materials exposure and evaluation,
2. and commercial materials exposure.

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The research and development role is a continuation of our efforts to determine the long-term durability of some of the more promising materials developed in DOE-funded programs. In addition to determining durability effects and providing manufacturer's guidance for these materials, any new potentially promising materials have been included for exposure and evaluation.

Table 1. Exposure Durability Tests

Type of Test	Berry ⁴	DSET Labs. ²	Honeywell ⁵	ITT ¹	LANL ⁶	Lockheed ³	ABS ⁷
1. Outdoor Stagnation Tests of Full collectors		X	X		X	X	X
2. Outdoor Stagnation Tests of Material Coupons	X	X		X	X	X	X
3. Outdoor Accelerated	(a)	X	(a)	(a)	(a)	(a)	(a)
4. Thermal Testing					X	X	X
5. Temperature Cycling	X	X				X	X
6. Temperature and Humidity	X				X	X	
7. Temperature and UV		X				X	X
8. Weatherometer		X	X				

(a) These investigators subcontracted accelerated outdoor testing to DSET laboratories.

Los Alamos continues to fund and provide technical monitoring of the DSET program that is focused on evaluating commercial materials. The Los Alamos exposure site serves as the high altitude exposure facility, which represents one of four exposure sites for this program. This investigation includes glazing and reflector materials in addition to absorber materials.

A photo of the overall exposure facility is shown in Figure 1 and the DSET commercial exposure experiments are shown in Figure 2. The Los Alamos exposure boxes are based upon the ASTM Standard Practice E781-81. The various types of exposure boxes are described in Table 2 and examples of the temperature excursions are shown in Figure 3. Peak temperatures are higher than those shown, where the single glazed selective surface (SGSS) test box exceeds 400°F during peak periods.

Optical measurements are made using the following equipment:

1. Beckman 5270 Spectrophotometer Equipped with a Beckman Integrating Sphere.
2. Gier Dunkle DB-100 Infrared Reflectometer.
3. Devices and Services Solar Spectrum Reflectometer.
4. Additional equipment available but not routinely used for this program include: Gier Dunkle MS-251 Solar Reflectometer, Beckman and Perkin Elmer Far Infrared Detection Systems.

Although the majority of the data presented in this report is centered around integrated measurements, spectral distributions have been studied for many of these materials. Much of this data is described in more detailed reports⁵. Detailed changes can normally first be detected from the spectral distributions, however, it is the integrated data that defines the materials solar performance capabilities.

Research and development conting

Table 3 describes the exposure results obtained for the development class of materials. These were selected from various suppliers during the early stages of their coatings development programs. There could be upgrading and product improvements that may have improved these products since their procurement. They were, however, representative of the particular type of coating at the time they were included in the program. Some of the more interesting results and observations of these materials are as follows:

Figure 1. Los Alamos exposure facility

Figure 2. DSET experiments at the Los Alamos exposure facility

Table 2. Exposure Test Boxes

Box No.	ID Code	Description
1	OPEN	Unglazed, flat black absorber, no insulation between absorber plate and back of box.
2	SGFB	Single glazed, flat black absorber; insulated.
3	DGFB	Double glazed, flat black absorber; insulated
4	SFGSS	Single Tedlar-film glazed, selective absorber no insulation between absorber plate and back box.
5	SGSS	Single glazed, selective absorber, insulated

Figure 3. Exposure-box temperature profiles

1. The Alcoa coating has changed considerably in properties over this three year exposure period. The initial optical properties $\alpha_g/\epsilon = 0.934/0.988$ for this coating represented a selective coating with only moderate properties. As the exposure of this coating progressed the emittance continued to decrease, improving the overall thermal performance of the coating. This trend continued to the two years sampling period, when it became apparent that the coating was being transformed into a powdery, rather fragile, coating. When removed for the three year optical measurements, the properties had changed to $\alpha_g/\epsilon = 0.888/0.184$ and an extremely fragile coating. Some of the reduction in absorptance may be attributed to handling damage. In a case where good initial handling properties are required for manufacturing purposes and which are not of particular importance once it is sealed in a collector and installed, this would still be a viable coating.
2. The SHOWA non-overcoated coatings appear to have maintained their properties over the three year exposure period with optical properties of $\alpha_g/\epsilon = 0.86/0.05$. Overcoating of the samples has resulted in increasing both α_g and ϵ to $0.92/0.20$. This particular overcoat is showing considerable change with the emittance decreasing from the 0.20

range to 0.10 making it a better coating since the absorptance appears to be relatively constant. Only additional testing will determine if other problems will begin appearing.

3. The Ferro porcelain enamels are quite stable after three years of exposure. The SnO_2 overcoated sample having initial selectivity of α_s/ϵ of 0.945/0.204 has shown a very slight change to 0.934/0.211 after three years of exposure. A slight degradation was noted early in the sample that appeared to be a spalled area. This defect has not increased in size or changed appreciably in properties.
4. The Berry developmental coatings on aluminum have shown rather insignificant changes in optical properties in the glazed exposure boxes. Only very small reductions in α_s , approximately two percent, have been noted in an unglazed exposure box.
5. The ULVAC coating has now shown an approximately two percent decrease in α_s in the single glazed selective surface exposure box after two years of exposure.
6. Ergenics Maxorb has shown exceptional stability under glass or cover films. When exposed in an open box it has shown an approximately 20 percent reduction in α_s .
7. Ergenics Skysorb has shown good stability in all exposure boxes, including the open box. It is unfortunate that initial α_s and ϵ are not better, i.e. $\alpha_s \sim 0.85$ and $\epsilon \sim 0.15$.
8. The 3M selective surface, now replaced by a newer product, has shown good stability in the covered exposure boxes, but poor durability in the open box.

Selective paints

Exposure testing of selective paints began with the inclusion of the Honeywell selective paints into the Los Alamos exposure program in 1982. The Honeywell program⁵ sought to develop both thickness sensitive selective paint (TSSP) and thickness insensitive selective paint (TISP). As industrial firms began their investigations into these coatings, their prototype products were also brought into the program. The original coating investigations were centered around aluminum for a substrate materials. Collector manufactures using other substrate materials, i.e., copper and stainless steel, and showing interest in the low cost selective paints brought about the durability testing of these materials.

Tables 4 through 6 describes the long term effects of exposing the selective paints to outdoor exposure testing. Most of these materials were removed and measured at three month, six month, one year, two year, and three year intervals. For simplification only the highest exposure results, the three year data, are tabulated for comparison with the original base line data. These tables describe the exposure results for the three year paint samples and some more recently added samples that have only accrued 16 and 24 months of exposure. More detail of the selective paint programs can be found in Solar Energy Materials^{8,9}. Some of the general results of this project are as follows:

1. Honeywell thickness sensitive selective paint (TSSP) based upon the G.E. Co. SR-125 silicone resin and the rotogravure print process have all shown excellent durabilities. Those produced earlier by the reverse roll coat process show a three to five percent increase in emittances. The FEP overcoated material also appears to be very stable. The open exposure box tests for one sample has shown seven percent emittance increases, while remaining stable for another set. The samples produced with the Cargill 6247 resin and exposed under glazing or film show decreases in ϵ , which makes it a better coating, however, this may be signaling a long term degradation problem. The open box samples show large decreases in both α_s and ϵ .
2. Honeywell thickness insensitive selective paints (TISP) have shown one to two percent decreases in α_s while ϵ has remained relatively constant.
3. The Thermo-250 paints on stainless steel and on aluminum appear to have only minor changes in properties, none that reduce the performance of the coating.
4. Dulite coatings exposed under glass also appear to be quite stable for the heavy or medium coating thicknesses. The light thickness coatings are showing a slight reduction in α_s . Paint samples exposed in the open box had the painted surface nearly completely removed by a hailstorm.
5. The Zuel coatings for all thicknesses have shown good stability in all of the exposure boxes. The only significant change is a 3% decrease in α_s for the light coating in the open unglazed exposure box.

6. An early selective paint by Solec, Hi/Sorb-I has shown a durability problem with heavy coatings and considerable change in medium and light coatings. This coating was changed and improved with a later product.
7. The Solkote Hi/Sorb-II has now been exposed for 16 months and is showing good stability on aluminum and stainless steel substrates. This coating on copper is showing changes in both α_s and ϵ , indicating that diffusion and oxidation of the copper substrate may be influencing the properties.
8. The Colt selective paint on copper has shown a large increase in ϵ in the open exposure box and a slight increase in the glazed boxes.

Chemically converted coatings

As part of a Los Alamos program to develop and investigate low cost selective surfaces, selective surfaces produced by chemical conversion were investigated. LA-9842-MS¹⁰ describes the various coatings investigated. Base line materials and some of the more promising coatings that evolved from this program were incorporated into the exposure test program. The objective is to determine their durability and ultimately determine if any have commercial potential. Samples of substrate materials and the completed converted samples were exposed in order to determine exposure effects. Some coatings may offer additional protection for marginally durable substrates. Some of the coatings being tested and the optical results are shown in Table 7 through 10 for zinc, galvanized and zinc/aluminum, nickel, aluminum and copper substrates. Some of the important results are:

1. Zinc, galvanized and zinc/aluminum substrate materials

The substrate materials results are showing relative large increases in α_s without too great an increase in ϵ when exposed in the open box. It is doubtful if they will even reach the point where they are relatively good intrinsic selective absorbers, however, this appears to be the trend. This is particularly true for Galvalume smoothed and large spangle. Most zinc base selective coatings are not showing good stability in the open box, however, some are showing good stability in the covered boxes.

2. Nickel Substrate Materials

Good stability is shown for the Patclin Chemical Co. Patox #1 conversion coatings and the Metex Black Nickel on nickel sulfamate in the covered boxes. All show poor stability in the open exposure box.

3. Aluminum Substrate Materials

The substrate results show that the oxide build-up in the open box is having only a small effect on the as received aluminum. The etched aluminum samples likewise are changing only a small amount in the SGSS exposure box. The A-15 coating is highly degraded in the open box, but has reasonably good stability in the covered boxes, showing only about two percent decrease in α_s .

4. Copper Substrate Materials

All samples are showing considerable degradation in properties. The Vestar overcoat appears to be stabilizing the coating from an emittance standpoint; however, large decreases in α_s are occurring.

Transparent materials exposure testing

Potential solar glazing materials are being exposed at the Los Alamos exposure facility in two forms, i.e., small samples exposed to direct UV or outdoor weathering and full size glazings or collectors. Spectral distributions are measured for the small samples and their weighted air mass 1.5 solar transmittances determined from these distributions. Total solar transmittances are determined for the full size glazing materials using the pyranometer method.

The glazing materials, both full size and small samples, that are undergoing exposure testing at Los Alamos include: AR-treated etched glass, heat mirror films/full size glazings, AR-treated film/glass units, scrim reinforced PVC's, fiberglass and cloth reinforced silicones, Kynar, PAN film, and Tedlars. In addition to these research or developmental materials, DSET Laboratories, operating under contract to Los Alamos, is exposing and evaluating commercial solar glazing materials at four exposure sites. These are located in Arizona, California, Florida, and New Mexico and provide exposure environments of desert, industrial, coastal and high altitude.

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Table 3. Developmental or Advanced Selective Coatings
Results, Selective Surface Single Glazed Exposure Box

Coating	Description	Original α_s , (a)	3-Year Exposure α_s/ϵ (a)
Alcoa	Aluminum Substrate	0.934/0.588	0.888/0.184 (b)
SHOWA Sample A Overcoated	Nickel-pigmented anodic Al_2O_3	0.920/0.200	0.920/0.093
SHOWA Suniodize overcoated 29	Nickel-pigmented anodic Al_2O_3	0.899/0.214	0.890/0.052
SHOWA Sample G-28	Nickel-pigmented Anodic Al_2O_3	0.850/0.051	0.862/0.048
ULVAC	Cr-CrO _x composite film on aluminum	0.903/0.043	0.894/0.075
Berry	Developmental coating on heavy aluminum sheet	0.932/0.091	0.937/0.081
Ergenics Maxorb	Graded composite NiO-Ni-Cr/NiCrO _x produced by chemical conversion	0.960/0.075	0.959/0.068
Ergenics Skysorb	Colored stainless steel	0.828/0.166	0.843/0.158
3M	Graded composite NiO-Ni-Cr/NiCrO _x	0.938/0.072	0.928/0.080
Ferro XS-111 Porcelain	High α_s porcelain	0.934/0.839	0.937/0.840
Ferro XS-111	SnO ₂ overcoated	0.945/0.204	0.934/0.211 0.926/0.294 (c)
Sunstrip Int'l.	Aluminum Substrate	0.916/0.151	0.922/0.139 (d)

(a) Absorptance, α_s , has been determined by using the D&S Solar Spectrum Reflectometer weighted for air mass 1.5. Emittance, ϵ , was determined by using the Gier Dunkle DB-100 Infrared Reflectometer.

(b) Coating becoming powdery and difficult to handle.

(c) Spalled area

(d) Two-year exposure

Table 4. Selective Paints

Paint	Process	Substrate	Exposure Box/Side	Original α_s/ϵ	3-Year Exposure α_s/ϵ
Honeywell TSSP	Dip-Coated sheet w/FEP overcoat	Aluminum	5/F	0.930/0.178	0.936/0.164
Honeywell TSSP	Reverse-roll coat production simulation	Aluminum foil	1/F	0.857/0.104	0.840/0.158
			4/F	0.862/0.108	0.873/0.131
			5/F	0.875/0.110	0.891/0.147
Honeywell rotogravure print w/SR-125 resin	Preproduction run	Aluminum foil	5/F	0.922/0.236	0.929/0.258

Table 4. Selective Paints (Cont'd)					
Honeywell rotogravure print w/6247 resin	Preproduction run	Aluminum foil	5/F	0.918/0.310	0.933/0.226
Honeywell rotogravure print w/SR-125 resin	Production simulation	Aluminum foil	1/F	0.889/0.092	0.866/0.090
			2/F	0.890/0.093	0.900/0.093
			3/F	0.883/0.076	0.895/0.065
			4/F	0.894/0.090	0.903/0.080
			5/F	0.880/0.102	0.902/0.083
Honeywell rotogravure print w/6247 resin	Production simulation	Aluminum foil	1/F	0.897/0.134	0.548/0.050
			2/F	0.899/0.144	0.907/0.090
			3/F	0.898/0.140	0.910/0.083
			4/F	0.900/0.143	0.910/0.078
			5/F	0.902/0.142	0.912/0.080
Los Alamos-applied Honeywell TISP on Al	Light coat	Aluminum sheet	5/F	0.865/0.166	0.850/0.159
	Med. coat		5/F	0.890/0.262	0.876/0.261
	Heavy coat		5/F	0.874/0.253	0.855/0.248
Los Alamos-applied Honeywell TISP on primed steel	Light coat	1010 mild steel (primed)	5/F	0.908/0.679	0.899/0.686
	Med. coat		5/F	0.902/0.473	0.884/0.474
	Heavy coat		5/F	0.881/0.392	0.873/0.384
Los Alamos-spray- applied Thermo-250 on stainless steel	Bare	34% stainless steel	5/F	0.472/0.142	0.554/0.133
	Light Spray		5/F	0.865/0.540	0.885/0.540
	Heavy Spray		5/F	0.871/0.810	0.883/0.814
Los Alamos-spray- applied Thermo-250 on aluminum	Bare	Aluminum sheet	5/F	0.074/0.030	0.067/0.031
	Light		5/F	0.800/0.490	0.820/0.491
	Heavy		5/F	0.868/0.790	0.882/0.797
Los Alamos-spray- applied Dulite (a)	Heavy spray	Aluminum sheet	5/F	0.954/0.867	0.956/0.840
	Medium spray		5/F	0.957/0.844	0.956/0.823
	Light spray		5/F	0.939/0.796	0.894/0.772

(a) Better properties could probably have been obtained with greater optimization and better process control; however, these samples were used to determine changes that may have been occurring. Dip-coated samples produced α_s/ϵ of 0.950/0.694.

Table 5. Selective Paint				
Sample	Substrate	Exposure Box/Side	Original α_s/ϵ	24 month α_s/ϵ
Zuel Solar Coat 100 Heavy Coating	Aluminum	1/F	0.955/0.474	0.946/0.547
		2/F	0.954/0.463	0.956/0.440
		3/F	0.955/0.468	0.961/0.485
		4/F	0.955/0.472	0.959/0.463
		5/F	0.955/0.477	0.958/0.468
Medium Coating	Aluminum	1/F	0.942/0.365	0.934/0.369
		2/F	0.948/0.354	0.949/0.362
		3/F	0.931/0.285	0.936/0.271
		4/F	0.935/0.282	0.937/0.263
		5/F	0.935/0.262	0.932/0.245
Light	Aluminum	1/F	0.867/0.140	0.835/0.136
		2/F	0.828/0.120	0.811/0.118
		3/F	0.811/0.121	0.810/0.108
		4/F	0.807/0.113	0.745/0.102
		5/F	0.761/0.098	0.741/0.083
Solkote by Solec Hi-Sorb (Discontinued)(b) Heavy Coating	Aluminum	1/F	0.959/0.713	--- / ---(a)
		5/F	0.959/0.702	--- / ---(a)
Medium Coating	Aluminum	1/F	0.908/0.407	--- / ---(a)
		5/F	0.906/0.388	0.879/0.352

Table 5. Selective Paints (Cont'd)				
Tight Coating	Aluminum	1/F	0.764/0.209	0.368/0.122 ^(a)
		2/F	0.774/0.204	0.793/0.196
		3/F	0.784/0.197	0.790/0.180
		4/F	0.778/0.180	0.786/0.173
		5/F	0.764/0.388	0.782/0.166

(a) Paint flaked from surface - discontinued test

(b) This was an early formulation that was changed when adhesion and durability problems developed.

Table 6. Selective Paints				
Sample	Substrate	Exposure Box/Side	Original α_S/ϵ	16 Month α_S/ϵ
Solkote Hi/Sorb II 00058-10	Cu	1/F	0.934/0.483	0.949/0.446
		4/F	0.934/0.483	0.934/0.510
		5/F	0.934/0.483	0.925/0.331
Solkote Hi/Sorb II 00058-2	Al	1/F	0.937/0.468	0.945/0.335
		4/F	0.937/0.468	0.928/0.338
		5/F	0.937/0.468	0.937/0.462
Solkote Hi/Sorb II 00058-13	S.S.	1/F	0.925/0.470	0.939/0.405
		4/F	0.925/0.470	0.926/0.464
		5/F	0.925/0.470	0.930/0.475
Colt #5	Cu	1/F	0.881/0.161	0.894/0.600
		4/F	0.837/0.117	0.863/0.128
		5/F	0.847/0.134	0.856/0.180

Table 7. Zinc Base Materials				
Conversion Coating	Substrate	Exposure Box/Side	Original α_S/ϵ	3-Year Exposure α_S/ϵ
Birchwood Casey Aluma Black A-15	Galvalume Extra Smooth	1/F	0.921/0.235	0.708/0.490
		4/F	0.916/0.222	0.914/0.262
		5/F	0.906/0.165	0.885/0.211
Enthone Ebonol Z80	Electroplated Zinc	1/F	0.878/0.185	0.942/0.333
		4/F	0.847/0.227	0.849/0.294
		5/F	0.867/0.258	0.843/0.790
Mac Dermid Metex Black MACro 866	Udylite Electro- plated Zinc	1/F	0.908/0.114	0.897/0.171
		4/F	0.895/0.090	0.852/0.113
		5/F	0.901/0.087	0.906/0.374
Vestar Coated		5/F	0.960/0.259	0.942/0.409
Heatbath Durocoat #20 and 21	Electroplated Zinc	5/F	0.710/0.207	0.889/0.503
Mitchell-Bradford Black Magic RT-Z40	Electroplated Zinc	1/F	0.942/0.091	0.945/0.288
		4/F	0.940/0.082	0.917/0.121
		5/F	0.918/0.069	0.880/0.183
None	Galvalume Smoothed	1/F	0.199/0.045	0.501/0.065
		5/F	0.209/0.050	0.300/0.078
None	Duraskin	1/F	0.433/0.117	0.524/0.172
		5/F	0.401/0.106	0.407/0.147
None	Galvalume Large Spangle	1/F	0.200/0.064	0.520/0.113
		5/F	0.182/0.071	0.301/0.115
Birchwood Casey Zinc Black Zn-3	Electroplated Zinc	5/F	0.946/0.066	0.828/0.160

Table 8. Nickel Materials

Conversion Coating	Substrate	Exposure Box/Side	Original α_S/ϵ	3-Year Exposure α_S/ϵ
None	Nickel Sulfamate	1/F	0.356/0.054	0.418/0.053
None	Bright Nickel	5/F	0.333/0.038	0.359/0.035
Metex Black Nickel	Bright Nickel	1/F	0.915/0.098	0.429/0.038
		5/F	0.906/0.072	0.909/0.075
Metex Black Nickel	Nickel Sulfamate	1/F	0.964/0.157	0.606/0.109
		5/F	0.922/0.103	0.942/0.101
Patclin Chem. Co. Patox #1	Bright Nickel	1/F	0.937/0.076	0.439/0.046
		5/F	0.937/0.092	0.940/0.070
Patclin Chem. Co. Patox #1	Nickel Sulfamate	1/F	0.903/0.128	0.472/0.046
		5/F	0.924/0.098	0.931/0.080

Table 9. Aluminum Substrate Materials

Conversion Coating	Substrate	Box/Side	Original α_S/ϵ	3-Year Exposure α_S/ϵ
None	Aluminum as received	1/F	0.193/0.041	0.308/0.044
None	Aluminum Etched	5/F	0.072/0.032	0.036/0.029
Birchwood Casey Aluma Black A-15	Aluminum Etched	1/F	0.918/0.245	0.198/0.045
		4/F	0.932/0.258	0.910/0.245
		5/F	0.943/0.299	0.928/0.260
Birchwood Casey Aluma Black A-15, Vestar Overcoated	Aluminum Etched	1/F	0.907/0.271	0.240/0.076

Table 10. Copper Substrate Materials

Conversion Coatings	Substrate	Exposure Box/Side	Original α_S/ϵ	3-Year Exposure α_S/ϵ
Birchwood Casey Brass Black M-24 (Front only) Coated	Copper	1/F	0.751/0.097	0.909/0.679
		4/F	0.766/0.129	0.882/0.174
		5/F	0.775/0.116	0.877/0.493
MacDermid MACuBlack (Both Sides) Coated	Copper	1/F	0.862/0.052	0.924/0.847
		4/F	0.872/0.056	0.812/0.124
		5/F	0.825/0.044	0.741/0.046
Vestar Overcoated		5/F	0.890/0.106	0.711/0.115