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**The Effect of Exposing Two
Commercial Manufacturers'
Second-Surface Silver/Glass
Mirrors to Elevated-
Temperature, Mechanical
Loading, and High-Humidity
Environments**

L. S. Dake
M. A. Lind

April 1982

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MANUFACTURERS' SECOND-SURFACE SILVER/GLASS
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SUMMARY

A preliminary examination of the effect of three accelerated exposure parameters on second surface silver/glass mirrors was performed. The variables studied were temperature (elevated and sub-zero), humidity and mechanical loading. One test consisted of exposing mirror coupons to dry heat (80°C) and heat plus water vapor (80°C, ~100% RH) environments. Another test consisted of subjecting mechanically loaded mirror strips to sub-zero temperature (-20°C), dry heat (80°C), and heat plus water vapor. Samples were evaluated qualitatively using dark field microscopy (1X and 100X). Quantitative determination of the effects of exposure testing on the mirrors was done with spectrophotometer spectral hemispherical and diffuse reflectance measurements.

Degradation that was progressive with time was observed for mirrors exposed to dry heat and heat plus water vapor. The degradation did not have the same visual appearance for the two environments. Mechanical loading at -20°C produced no degradation after three months' exposure time. Mechanical loading in dry heat and heat plus water vapor environments resulted in mirror degradation that was the same as that found in unloaded mirrors exposed to the same temperature and humidity.

These preliminary tests indicate that the dry heat and heat plus water vapor accelerated tests may provide useful information about mirror degradation, while the mechanical load tests do not. The microscopy and spectrophotometer reflectance measurements were both useful techniques for determining the extent of degradation.

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INTRODUCTION

Second surface silver/glass solar mirrors for field use require long lifetimes in order to be cost effective. The discovery that mirrors in some locations show severe degradation after only one or two years of outdoor exposure has prompted a great deal of research into the mechanisms of the degradation and ways to improve the lifetime and durability of these mirrors.⁽¹⁾ A persistent problem has been the development of test procedures that will reliably predict the performance of mirrors outdoors, or that will provide a relative ranking of the durability of different types of mirrors. Since real-time outdoor exposure can take a year or longer to produce significant changes in the mirrors, some type of accelerated exposure testing is needed. An advantage of simulated exposure testing is that specific environmental stresses that may be contributing to the mirror degradation can be isolated and evaluated. The effect of different combinations of stresses can be determined. This may contribute to a basic understanding of the degradation mechanisms. The main disadvantage of accelerated testing is the difficulty in an "a priori" determination of the effects of the absolute level of the applied stress and the relevant synergisms.

A statistically designed test matrix was formulated to provide a systematic approach to the development of reliable accelerated tests for mirrors. This approach is called the "Matrix Approach for Testing Mirrors" (MATM) and is discussed in detail in reference 2. Initially, six environmental stresses that were believed to contribute to mirror degradation were identified (humidity, temperature, mechanical loading, thermal cycling, ultra-violet radiation, and environmental pollutants). A preliminary evaluation of the effects of elevated and sub-zero temperature, humidity and mechanical loading on mirrors are presented in this work. The purpose of this study was to identify which of the above accelerated tests provide consistent and reproducible degradation on an acceptable time scale that differs in type or extent for different mirror types. As a minimum it was hoped that viable screening procedures for different mirror types could be developed. In addition, several techniques for objectively evaluating the degraded mirrors were also examined.

ELEVATED TEMPERATURE AND HUMIDITY TESTS

EXPERIMENTAL

Commercial mirrors manufactured by two different companies were used in the experiments. The mirrors were produced using the standard industrial wet chemistry process that has been described elsewhere⁽³⁾. The resulting mirror consists of a soda-lime silicate float glass substrate coated with approximately 70 nm of silver. A layer of copper approximately 30 nm thick is deposited on on top of the silver, followed by a layer of paint ~.1mm thick. The mirrors were cut into 5 X 5 cm (2 X 2 in.) squares for the elevated temperature and humidity tests.

The samples subjected to heat were baked in air at 80°C with relative ambient humidity of less than 40%. Samples were baked for specified times up to 675 hours.

The samples exposed to heat and water vapor were placed in the test fixture shown in Figure 1. A neoprene gasket was fitted over ceramic cups partially filled with de-ionized water. The mirror coupons were fitted over the neoprene gasket and were held in place by a top plate. The entire fixture was placed in an oven and baked in air at 80°C for the predetermined times. The relative humidity inside the ceramic cups rapidly approached 100%. In this manner, the paint side of the mirror was exposed to water vapor. The glass side was not exposed to the vapor, so remained undegraded and allowed a clear view of the silver/glass interface. All data was recorded at the center of the exposed area to avoid possible effects of the neoprene seal.

RESULTS

All the sample coupons were photographed through the glass side at 100X using standard darkfield techniques. Light areas in the photographs indicate the presence of scattering centers in the silver layer. Scattering centers may be caused by debonding at the silver/glass interface, agglomeration of the silver layer or some other surface roughening phenomena.

a.

b.

FIGURE 1. The Water Vapor Exposure Test Fixture Shown Broken Down (a) and Assembled with Mirror Coupons in Place (b)

The micrographs for the two mirror types after exposure for the specified amounts of time are shown in Figures 2 through 5. The 24-hour heat only micrographs are representative of the original condition of the two mirror types before testing. Clearly, the two environments had a different effect on the mirrors. In general, the degradation in both environments was more severe with increased exposure times.

Some caution must be used in interpreting the photographic data. There is significant sample to sample variation in new mirrors from both manufacturers. Therefore, small differences in the photographs are not significant, since different mirror coupons were used for each exposure time. Also, photographic exposure times and lighting conditions may not have been consistent for all the micrographs. Nevertheless, there are some trends in the data that are at least qualitatively repeatable in successive experiments. There is a significant difference between the heat only and the heat + water vapor experiments for each manufacturers mirror coupons. There are also significant differences apparent between the different manufacturers' mirrors. It may or may not be significant that the mirrors from manufacturer B showed measurably more degradation than those from manufacturer C and manufacturer C has an industry wide reputation for producing the highest quality mirrors.

The solar weighted hemispherical reflectivity of new mirrors and of the more severely degraded mirrors was measured. The results are shown in Table 1.

TABLE 1. HEMISPHERICAL SOLAR WEIGHTED REFLECTIVITY
REFLECTIVITIES

<u>Hours Exposed</u>	Manuf. B		Manuf. C	
	<u>Heat</u>	<u>Heat & Vapor</u>	<u>Heat</u>	<u>Heat & Vapor</u>
0	.918	.918	.918	.918
336	.915		.915	
504	.891	.904	.894	.898
672	.896	.901	.904	.915

DARKFIELD MICROGRAPHS (100X)

24 HOUR

48 HOUR

HEAT

HEAT + VAPOR

HEAT

HEAT + VAPOR

MANUFACTURER
B

MANUFACTURER
C

FIGURE 2. 100X Darkfield Micrographs Exposed for 24 and 48 Hours in Heat and Heat - Water Vapor Environments at 80°C.

DARKFIELD MICROGRAPHS (100X)

96 HOUR

168 HOUR

HEAT

HEAT + VAPOR

HEAT

HEAT + VAPOR

MANUFACTURER

B

MANUFACTURER

C

FIGURE 3. 100X Darkfield Micrographs Exposed for 96 and 168 Hours in Heat and Heat + Water Vapor Environments at 80°C.

DARKFIELD MICROGRAPHS (100X)

192 HOUR

336 HOUR

HEAT

HEAT + VAPOR

HEAT

HEAT + VAPOR

MANUFACTURER

B

MANUFACTURER

C

FIGURE 4. 100X Darkfield Micrographs Exposed for 192 and 336 Hours in Heat and Heat + Water Vapor Environments at 80°C.

DARKFIELD MICROGRAPHS (100X)

FIGURE 5. 100X Darkfield Micrographs Exposed for 504 and 672 Hours in Heat and Heat + Water Vapor Environments at 80°C.

The measurement error is estimated to be less than $\pm .005$ reflectance units. The repeatability in solar weighted values is $\pm .001$ units. The degraded mirrors do show reflectivity losses. The technique does not appear to be as sensitive as the photomicroscopy, but it does supply complementary, quantitative data. Diffuse reflectivities were also measured for selected mirrors. The diffuse reflectance did increase in the degraded mirrors (indicating an increase in scattering sites), but only by about 1/2%.

Spectral hemispherical reflectance data was recorded for selected samples over the wavelength region 300-2500 nm. Representative curves for new and degraded mirrors of one manufacturer are shown in Figure 6 and 7. In comparing the curves for new and degraded mirrors, it appears that the most significant changes in reflectivity are occurring in the ultra violet-visible region near the silver plasmon absorption band. This is confirmed by the plot of the difference of the degraded and new mirror reflectance curves shown in Figure 8.

MECHANICAL LOAD TESTS

EXPERIMENTAL

An experiment was done to determine the effect of mechanical loading on mirror degradation in sub-zero, dry heat, and heat plus water vapor environments.

Mirror samples used in the mechanical load test were of the same type as those described in the previous section. The mirrors were cut into 3.8 x 30.5 cm (1.5 X 12 inch) strips. The strips were loaded using the standard four point technique as shown in Figure 9. Two extruded aluminum angle irons separated by 25.4 cm (10 in) supported each mirror strip. A 2.7 kg (6 lb) weight rested on top of the mirror on two more angle irons separated by 6.1 cm (2.5 in). 2.7 kg was approximately 40% of the ultimate yield load of the mirrors. The resulting stress profile on the mirror is shown in Figure 10.

The silver layer was under compression when the weight rested on the coated side of the mirror and tension when the weight rested on the glass side of the mirror.

FIGURE 6. Typical Spectral Hemispherical Reflectivity of a Mirror Coupon Prior to Testing.

FIGURE 7. Typical Spectral Hemispherical Reflectance of a Mirror after 504 Hours in a 80°C Moist Environment.

FIGURE 8. Spectral Difference for a Mirror Coupon Before and After Testing. The Erratic Data at Wavelengths Greater than 700nm is Due to Instrumentation Noise.

FIGURE 9. Test Fixture for Mechanical Loading Tests

FIGURE 10. Stress Profile in Test Fixture

The loaded mirrors were placed in one of three environments: -20°C ; 80°C ; and 80°C plus $\sim 100\%$ relative humidity. The low temperature test was performed primarily to ensure that stress relaxation at the higher temperatures was not effecting the results. Both compression and tension loaded samples from both manufacturers were tested in each of the three environments. In addition, mirror samples with no mechanical load were tested along with the loaded samples in each of the environments. By using the unloaded samples as control samples, the effect of mechanical loading could be separated from the effect of the environment alone.

RESULTS

No visible degradation at 1X and 100X was observed in either the loaded or unloaded mirrors after 3 months' exposure to the -20°C environment. Degradation was observed in the mirrors in the dry heat and heat plus water vapor environment, but in all cases, the degradation for the loaded mirrors was the same as that for the unloaded mirrors. No degradation that could be attributed to the magnitude of mechanical load was observed after up to one month of exposure time.

CONCLUSIONS

On the basis of these preliminary experiments, the heat and heat plus water vapor environmental stress tests may be useful for evaluating mirrors and warrant further study. Both environments cause degradation in mirror samples in acceptable time periods (one month). The two different mirror types were distinguishable. The degradation was different in type and extent in the two environments.

The mechanical stress test does not appear to be useful, since the mechanical loaded mirrors degrade in the same manner as unloaded mirrors in the same environment. Sub-zero temperatures do not affect the mirrors after three months of exposure time.

The measurement techniques used were all effective. Microscopic examination at 1X and 100X provides a good qualitative technique for comparing the effects of different environments on the different mirrors. The spectrophotometer hemispherical and diffuse reflectance data give important quantitative information on the effect of degradation on the performance of the mirrors. In addition, the spectral data indicates wavelength regions where changes are occurring.

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