
Electroless Nickel and Ion-Plated Protective Coatings for Silvered Glass Mirrors

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April 1982

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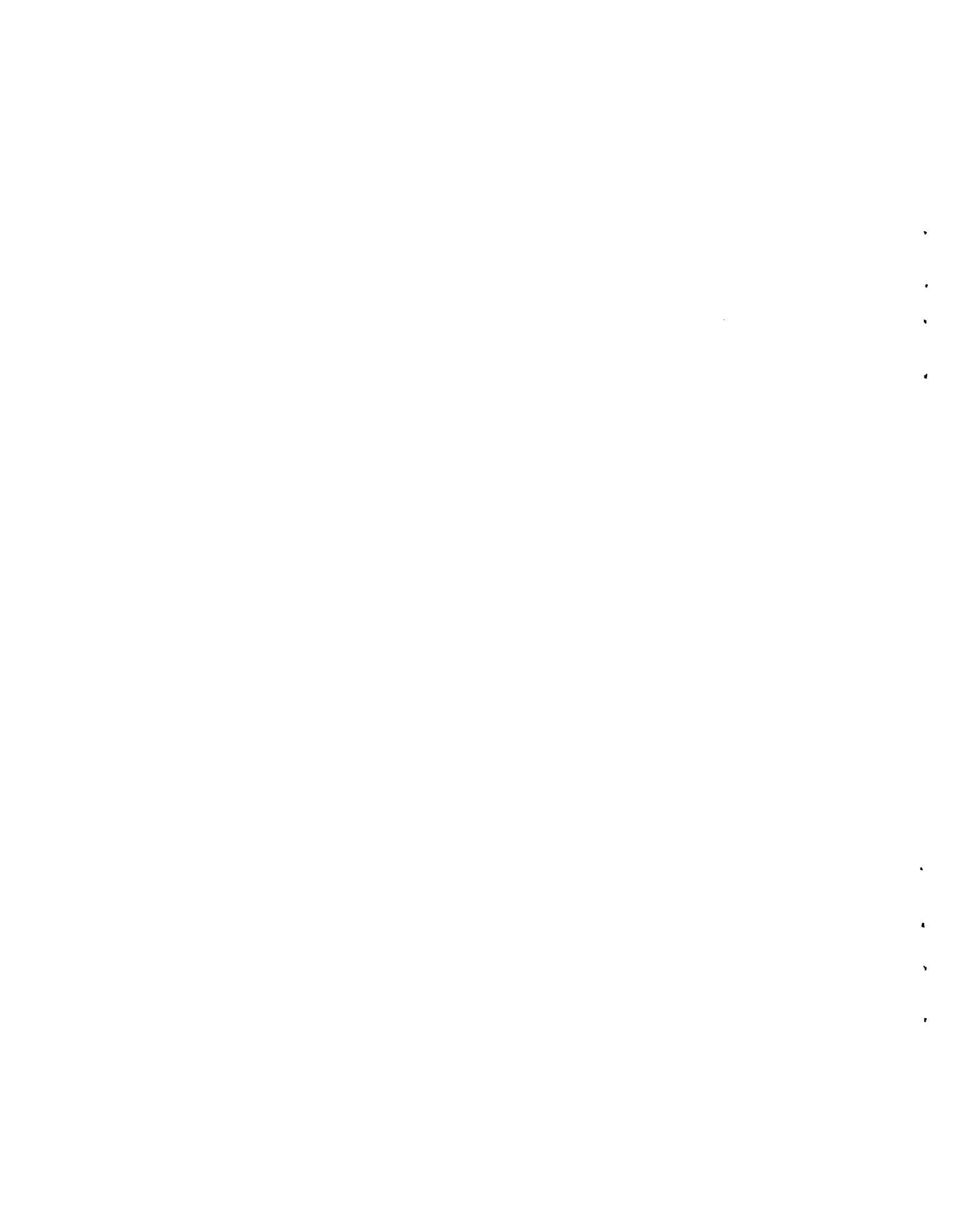
ELECTROLESS NICKEL AND ION-PLATED
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GLASS MIRRORS

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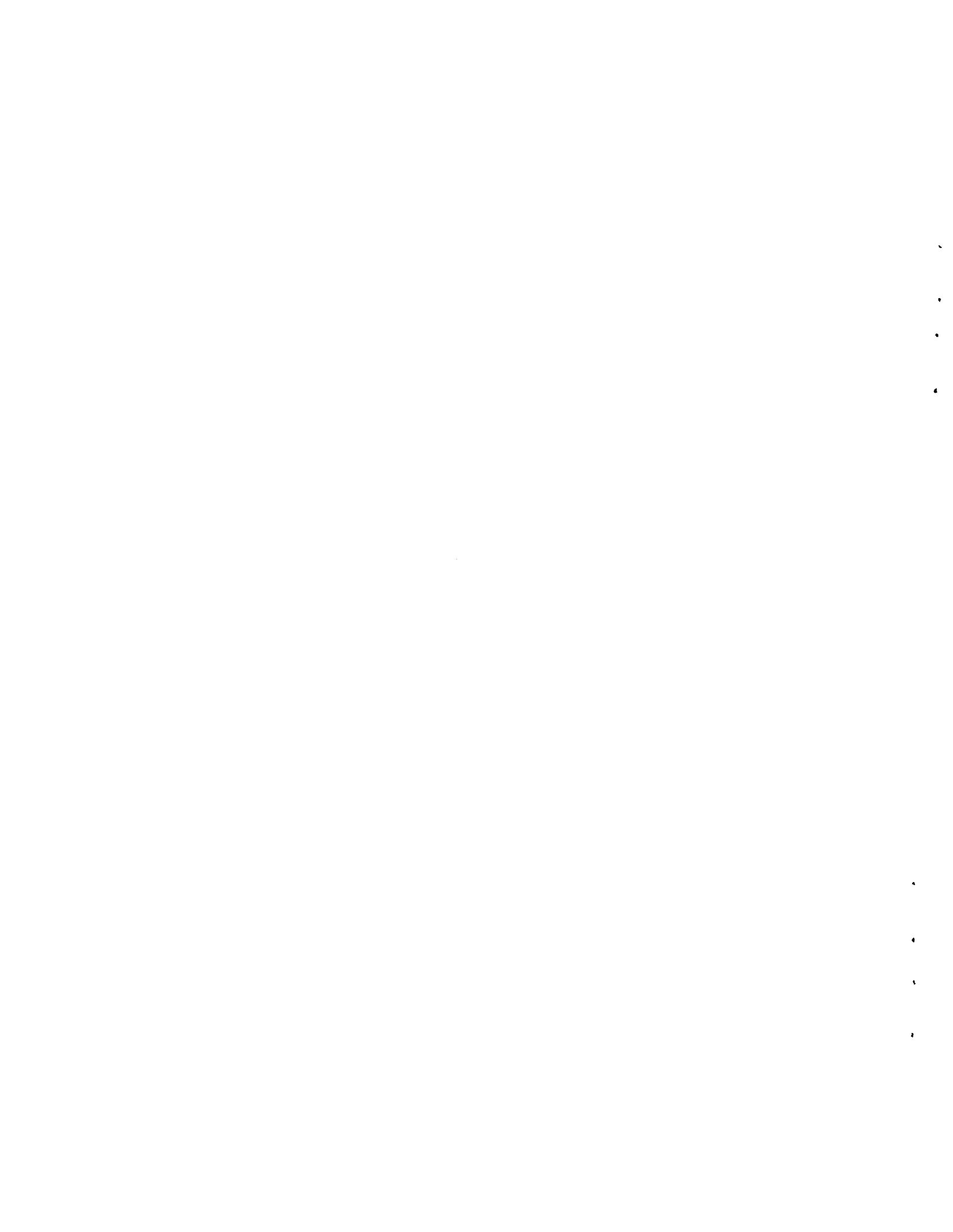
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SUMMARY

A preliminary examination of two methods of protecting second surface silvered glass mirrors from environmental degradation is presented. One method employed silver mirrors overcoated with Al, Ni, 304 stainless steel, Cr, or an Al/Cu alloy prepared by ion-plating. The other method used conventional wet process silver mirrors protected with a thin electroless nickel coating. No attempt was made to optimize the coatings for either method. These experimental mirrors were compared with conventional paint backed silver/copper mirrors after exposure to elevated temperatures and water vapor in order to estimate their relative environmental stability. The electroless nickel mirrors showed consistently more resistance to these stresses than either the conventional or ion-plated mirrors, suggesting that they may provide more durable field service.



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INTRODUCTION

The resistance of conventional paint backed silvered glass mirrors to the environmental stresses commonly found in solar applications has come under increasing scrutiny in the last few years. Degradation of the mirror surface has been found in some field deployed mirrors after less than a year.^(1,2,3) Since mirror lifetimes exceeding twenty years are desired in order to make many solar applications cost competitive, new methods of increasing the life expectancy of these mirrors are being investigated.

Research over the last few years has identified three symptoms that are common to many of the field degraded silvered mirrors. These symptoms are debonding of the silver/glass interface, halide formation on the metallic layers, and agglomeration of the silver layer.⁽¹⁾ Since silver possesses the highest solar weighted reflectivity of the common metals, it is desirable to retain it as the primary reflective material.

Although the glass on which the silver is deposited is "dirty" on the microscopic scale, for these experiments it is assumed that the primary contamination path is from the rear surface of the mirror. This path is through a relatively porous paint barrier in a conventional mirror structure. The research results suggest that a straightforward method of preventing the degradation symptoms from materializing may be to encapsulate the silver layer with a relatively impermeable material. The motivation then exists to replace the paint with an impermeable metallic barrier that is less susceptible to environmental attack.

The current research on improving the mirrors is actually two fold. One thrust deals with understanding and modifying the mirror structure to improve its stability and resistance to environmental stresses. The other thrust is devising testing procedures that simulate the environmental degradation in time periods much shorter than the one to two years required to see major changes in field exposed samples.

Thus, once sample coupons of the experimental mirrors are fabricated, the problem of devising a test procedure to reliably evaluate their performance still remains. The approach taken here involves a statistically designed matrix of test mirrors and "standard" mirrors subjected to various elevated environmental stresses. The motivation for using this approach and the details of implementing the approach now referred to as MATM (Matrix Approach for Testing Mirrors) can be found elsewhere^(2,4). The actual stresses encountered in nature might include temperature, moisture, ultra-violet light, environmental pollutants, mechanical tension or compression forces, etc. Practical considerations limited testing of this set of experimental mirrors to two parameters. The parameters chosen for these experiments are heat and water vapor as a function of time. The evaluations are performed visually and spectrophotometrically. This general approach, although somewhat arbitrary, has shown surprisingly good correlation in ranking the relative life expectancies of numerous generic types of mirror structures.⁽⁴⁾

SAMPLE PREPARATION

The results presented in this paper are for second surface mirrors fabricated in three different ways. The standard commercial paint backed silver mirror is used as a reference to compare the performance of ion-plated mirrors of silver backed with several other metals generally less susceptible to corrosion and standard silver mirrors with an electroless nickel overcoating.

The standard mirrors were prepared according to the usual industry practice⁽⁵⁾ by two different commercial manufacturers. Here silver is deposited to a thickness of 70 nm on soda-lime silicate glass using a wet chemical process. This step is followed by the chemical deposition of approximately 30 nm of copper. The final coating is a gray backing paint similar in composition to an automotive primer. Most commercial mirrors for household and decorative uses are prepared similarly.

The ion-plated mirrors were prepared by Illinois Tool Works. In this system the ions were evaporated into an Ar defined RF plasma from a filament or boat near ground potential. The target substrate was DC biased at 500-700 V. Approximately 100 nm of silver was deposited prior to initiating a codeposition with the overcoating metal for the next 100 nm. Then the overcoating metal alone was deposited. The estimated total thickness of the overcoating layers are: Cr - 400 nm, Ni - 1000 nm, 304 stainless steel - 1000 nm, Al - 1500 nm, and Al(.35)/Cu(.65) alloy-1500 nm.

The ion plated mirrors were made using two different substrate materials: soda-lime silicate float glass and aluminoborosilicate fusion glass. It should be noted that the production of the ion-plated coatings was a one time only effort. No attempts were made to optimize the process or the coating thickness so these mirrors may not represent the best mirrors that can be made using this technique. Nevertheless, they may provide a first order indication of the performance to expect from this type of mirror.

Only one of the five companies attempting to overcoat the standard wet chemistry silver/copper mirror (without paint backing) with a solution-based nickel process was successful. MacDermid Incorporated used their own proprietary chemistry to plate a thin nickel coating on several samples. The plating, done at 85°F for approximately 10 minutes, was initiated using a small dc current. No attempt was made to measure the thickness of the nickel layer or measure the initiation current.

Both the ion plating technique and the electroless nickel process are potentially adaptable to rapid, high volume, low cost processing. Electroless nickel is particularly attractive since it may be compatible with the current wet chemistry mirror processing lines with minimal conversion costs.

TESTING METHODOLOGY

The mirrors were cut into 5 x 5 cm (2 x 2 inch) samples for all of the tests. A minimum of three of each of the samples was run for each applied stress. In all the tests, the qualitative features of the observed degradation were similar for all the samples of a given mirror type.

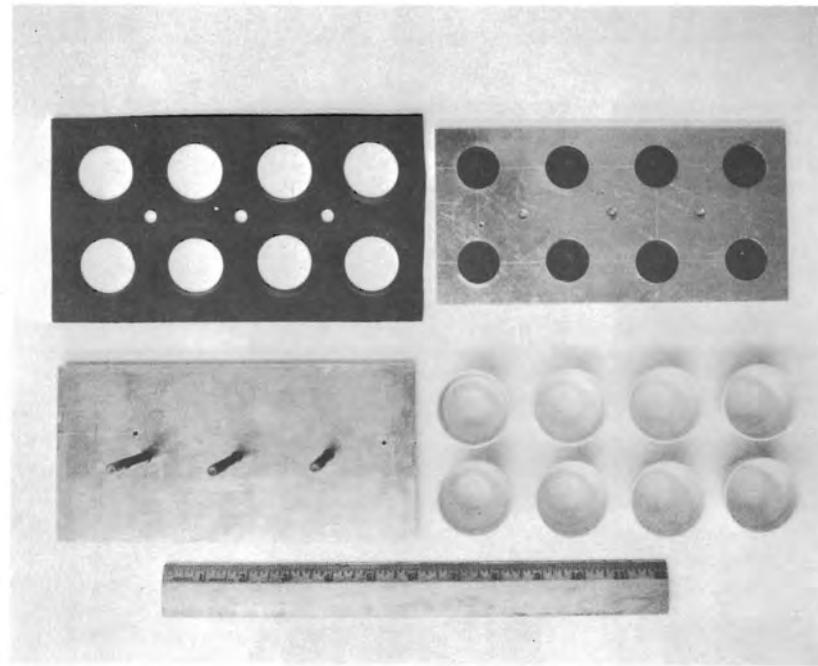
The samples subjected to heat were baked in air at 80°C for specified times up to 1388 hours. The relative ambient humidity was less than 40% during the bake.

The samples exposed to heat and water vapor were placed in the test fixture shown in Figure 1. The mirror coupons were centered over a neoprene gasket which was fitted to a ceramic container. The container was partially filled with deionized water. The coating to be tested was then in direct contact with the water vapor while the glass side of the mirror was kept dry, thus allowing an undegraded view of the interface. The entire fixture was placed in an oven for the predetermined time periods called for in the experiment. The relative humidity in the containers rapidly approached 100%. Condensation was usually observable on the mirror backs, especially near the neoprene seal. All data was consequently recorded at the center of the exposed area.

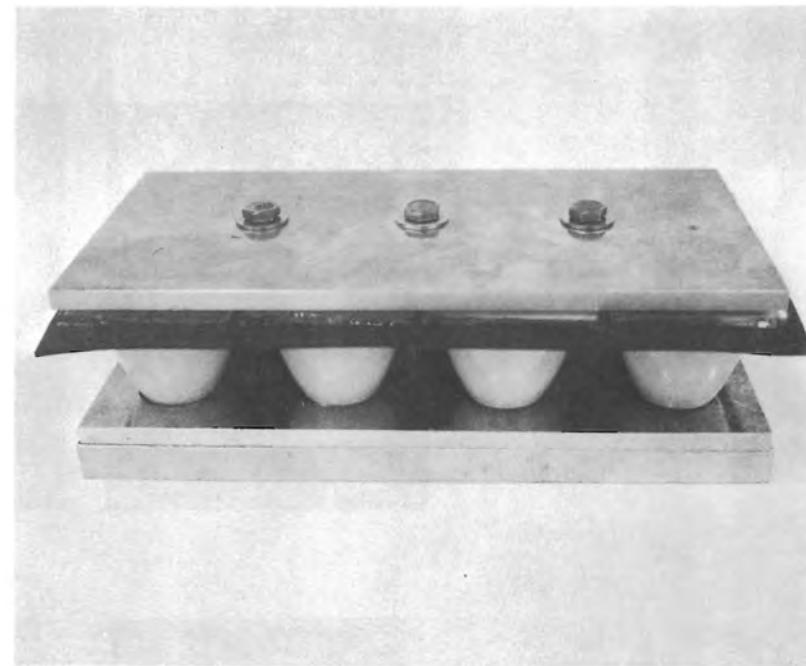
RESULTS

All the 5 x 5 cm (2 x 2 inch) samples were photographed through the glass side at 1X and 100X using standard darkfield techniques. Thus scattering centers on the mirror caused by agglomeration of the silver, debonding at the silver/glass interface, and texturing due to localized surface contamination are clearly visible as the light areas.

The micrographs of the reference mirrors are shown in Figures 2 and 3. Samples from the two different manufacturers are shown for the heat only and heat plus water vapor environments after exposure for 336 hours.



a.



b.

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

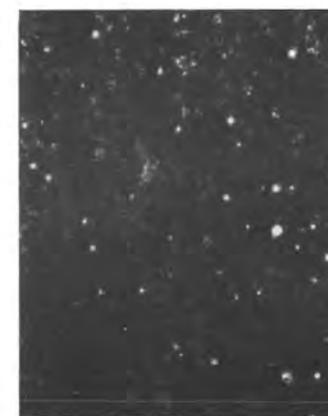
CONTROL

HEAT

HEAT + VAPOR



1X



100X

FIGURE 2. Darkfield Micrographs of Manufacture B Standard Mirrors Stressed for 336 Hours in 80°C Dry and Moist Environments

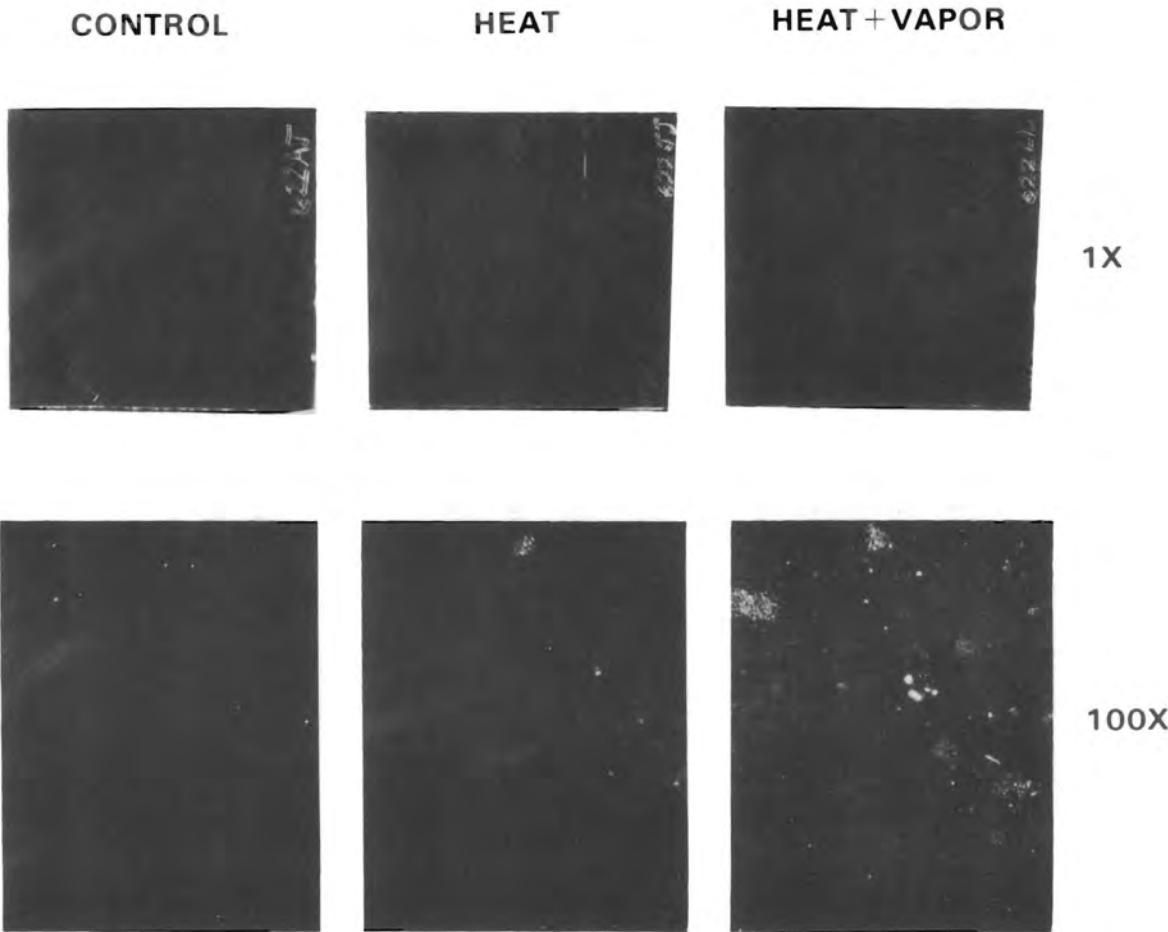


FIGURE 3. Darkfield Micrographs of Manufacture C Standard Mirrors Stressed for 336 Hours in the 80°C Dry and Moist Environments

The degradation is most visible in the 100X micrographs. It is characterized by apparent agglomeration of the silver layer. Both sets of mirrors show more severe degradation in the heat plus water vapor environment than in the heat only environment. The variation between the two sets of mirrors is typical of each manufacturer's samples. The samples from manufacturer C show consistently less degradation than those from B in both environments.

Micrographs of the ion-plated mirrors on soda-lime silicate float glass exposed for 336 hours under conditions identical to the reference set are shown in Figures 4 to 8. Qualitatively, the mirrors on the soda-lime silicate float glass substrate behaved similarly to those on the aluminoborosilicate fusion glass. The fusion glass mirrors generally showed slightly less severe degradation than the float glass mirrors. The mirrors backed with Ni, Cr and Al showed less degradation than the reference mirrors after 336 hours in the heat only environment, but all the ion-plated mirrors were more severely degraded than the reference mirrors after exposure to the heat plus vapor environment. Apparently the metallic overcoatings successfully stabilized the silver film from agglomeration in the dry heat environment. The severe degradation in the moist environment may be due to the suspected porous nature of the ion-plated metallic overcoat.

The modes of failure differed for the different overcoating materials, but were again consistent for the two substrate materials. Cracking and peeling were observed in the Ni and 304 stainless steel overcoated mirrors. In fact, cracking was evident in the unexposed 304 stainless steel samples and it became more severe in both stress environments. The Cr overcoated mirror degraded by means of pinhole formation that may have been accompanied by some agglomeration of the silver. The Al and Al/Cu overcoated mirrors exhibited massive delamination and flaking of the metal layers in both environments.

The results of similar tests performed on the standard silver/copper mirrors overcoated with a chemically deposited nickel coating are shown in Figures 9 and 10. No appreciable degradation was observed after 336 hours,

CONTROL



HEAT

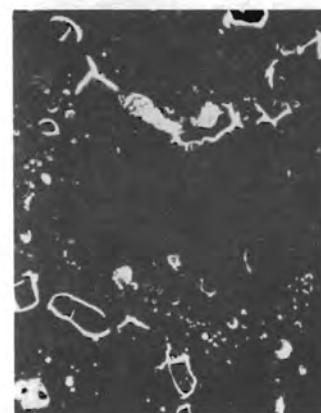


HEAT+VAPOR



1X

6



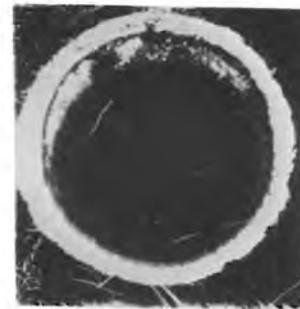
100X

FIGURE 4. Darkfield Micrographs of the Ion Plated Silver, Aluminum Backed Mirrors Stressed for 336 Hours at 80°C in Dry and Moist Environments

CONTROL

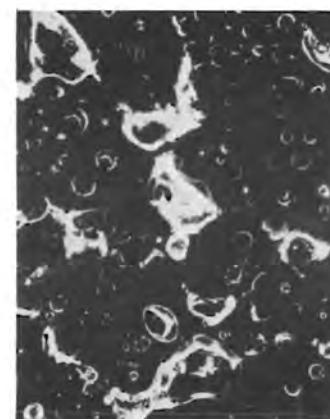
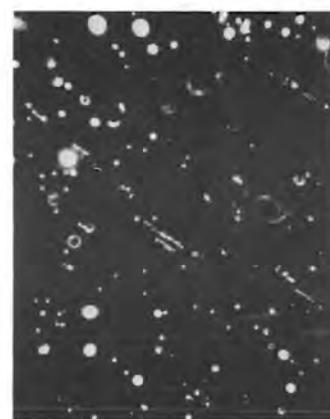
HEAT

HEAT + VAPOR



1X

10



100X

FIGURE 5. Darkfield Micrographs of the Ion Plated Silver, Aluminum (.35)/Copper(.65) Backed Mirrors Stressed for 336 Hours at 80°C in Dry and Moist Environments

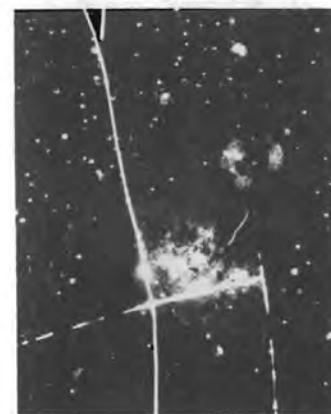
CONTROL

HEAT

HEAT + VAPOR



1X



100X

FIGURE 6. Darkfield Micrographs of the Ion Plated Silver, Chromium Backed Mirrors Stressed for 336 Hours at 80°C in Dry and Moist Environments

CONTROL



HEAT



HEAT + VAPOR



1X

12



100X

FIGURE 7. Darkfield Micrographs of the Ion Plated Silver, 304 Stainless Steel Backed Mirrors Stressed for 336 Hours at 80°C in Dry and Moist Environments

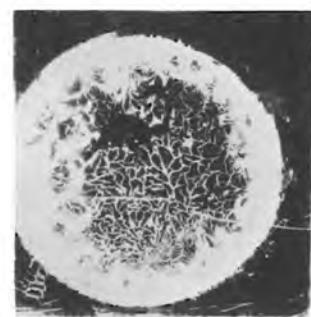
CONTROL



HEAT



HEAT + VAPOR



1X



100X

13

FIGURE 8. Darkfield Micrographs of the Ion Plated Silver, Nickel Backed Mirrors Stressed for 336 Hours at 80°C in Dry and Moist Environments

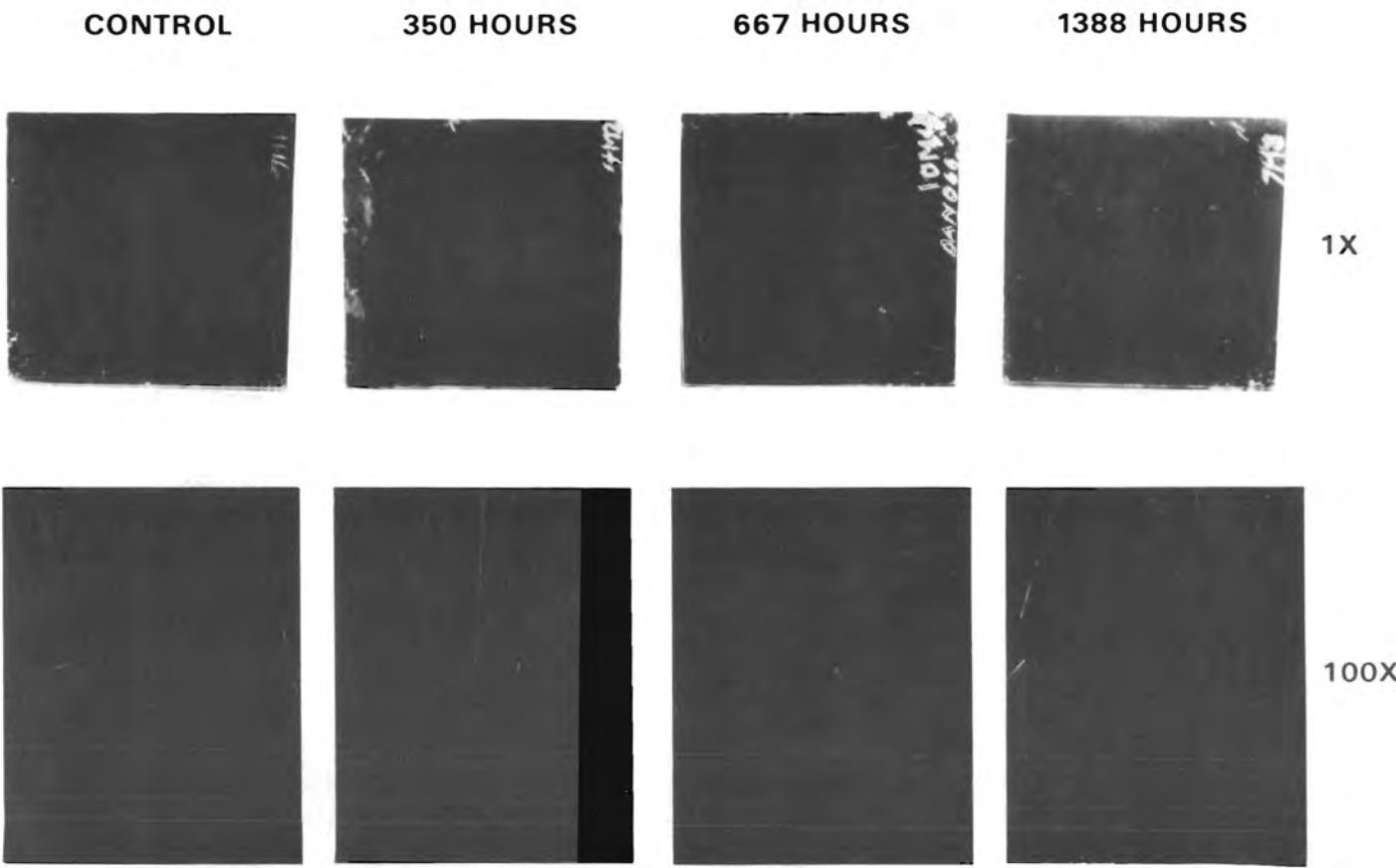


FIGURE 9. Darkfield Micrograph of Standard Silver/Copper, Electroless Nickel Backed Mirrors Stressed at 80°C in a Dry Environment

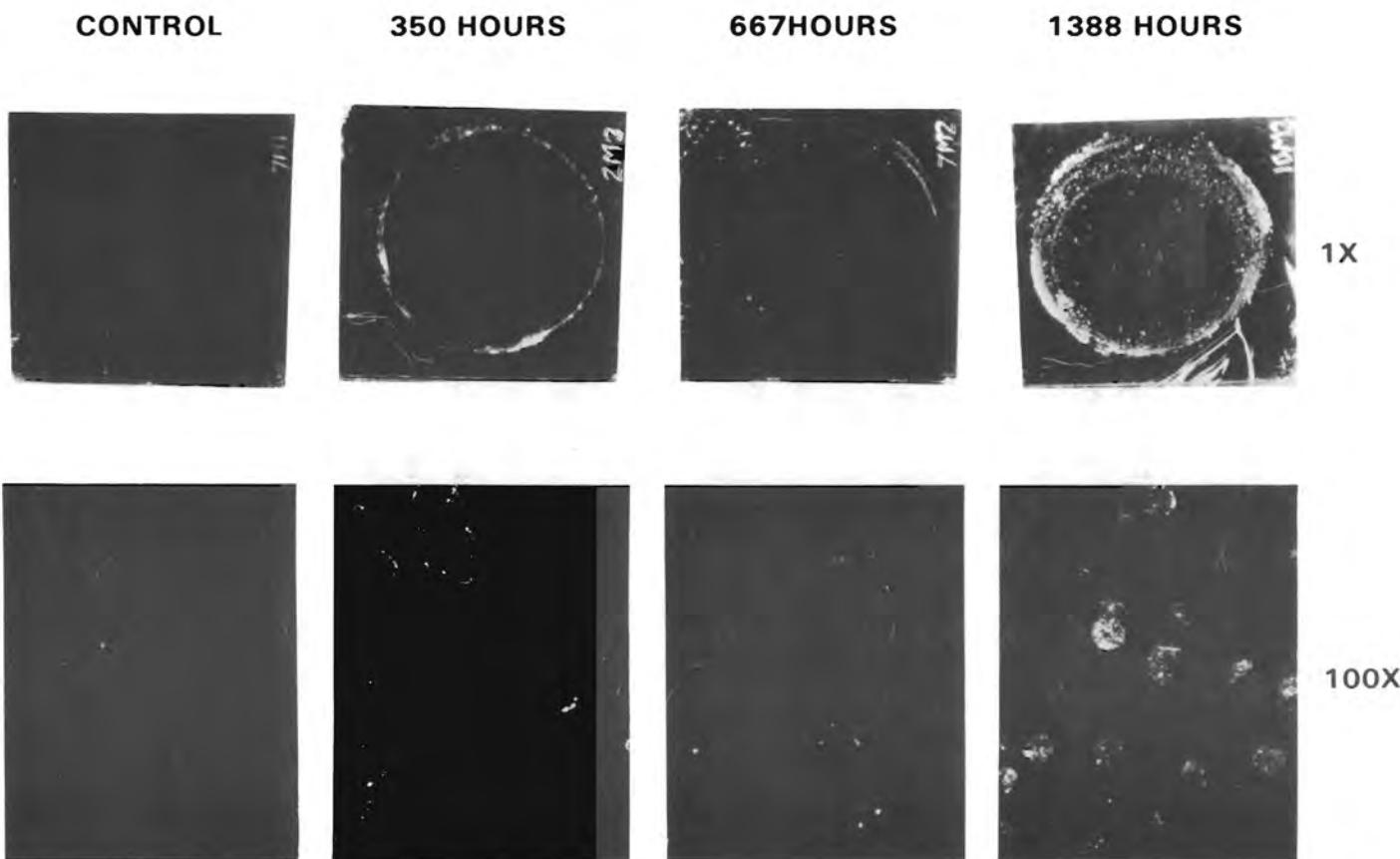


FIGURE 10. Darkfield Micrographs of Standard Silver/Copper,
Electroless Nickel Backed Mirrors Stressed at
80°C in a Moist Environment

so the exposure time was extended to 1388 hours. Note that no visible degradation is evident after 1388 hours in the heat only environment. No appreciable degradation is observable in 667 hours of the heat and water vapor environment. After 1388 hours the mode of failure in the heat plus vapor environment again appears to be agglomeration. These results show dramatic improvement in stability compared to either the reference standard mirrors or the ion-plated mirrors.

It is also interesting to compare the solar weighted hemispherical and diffuse reflectivities of these samples shown in Table 1 for float glass substrates. The measurement error is estimated to be less than $\pm .005$ reflectance units. Reflectance changes were quite small in the dry heat environment for all mirrors on the float glass substrates except the Al and Al/Cu overcoated mirrors. The 304 stainless steel, Ni and Cr mirrors showed losses similar to those of the reference samples. The 20-35% losses exhibited by the Al and Al/Cu mirrors can be attributed in part to increased transmission due to the flaking away of the metal layer. All the ion-plated mirrors had higher reflectance losses than the reference samples in the heat plus vapor environment. Qualitatively, the mirrors on fusion glass substrates performed similarly to the float glass mirrors, with the exception of the Cr overcoated mirrors. The Cr mirrors on fusion glass substrates performed better than both the Cr overcoated mirrors on float glass and the reference mirrors as shown later in the text. The reflectance of the Al and Al/Cu overcoated mirrors on fusion degraded worse than their counterparts on float glass. The reflectance of the 304 stainless steel and Ni overcoated mirrors on fusion glass deteriorated somewhat less in the heat plus vapor environment than those on float glass, but still did worse than the reference samples. The electroless nickel plated mirrors again exhibited superior performance. Both hemispherical and diffuse reflectance losses were negligible in both environments after 1388 hours of exposure time.

The actual spectral hemispherical and diffuse reflectance data for a typical reference standard mirror taken before and after stress testing is shown in Figures 11 and 12. The spectral hemispherical reflectance curves

TABLE 1. Solar Weighted Reflectivity

Mirror Type	Control		Heat Stressed 336 Hours Change In		Heat + Vapor Stressed 336 Hours Change In	
	Hemispherical	Diffuse	Hemispherical	Diffuse	Hemispherical	Diffuse
Standard Mirror Manufacturer #B Ag/Cu-paint	0.918	0.005	-0.006	+0.002	-0.020	+0.003
Standard Mirror Manufacturer #C Ag/Cu - paint	0.918	0.005	+0.001	+0.002	-0.016	+0.002
ITW Ag-Cr	0.899	0.007	+0.006	+0.002	-0.001	+0.004
ITW Ag-SS #304	0.897	0.010	+0.004	0.000	-0.063	+0.186
ITW Ag-Ni	0.905	0.007	+0.006	0.000	-0.106	+0.195
ITW Ag-Al	0.909	0.008	-0.323	-0.001	-0.039	+0.068
ITW Ag-Al/Cu	0.891	0.021	-0.359	+0.014	-0.038	+0.125
MacDermid Ag/Cu - Ni	0.899	0.008	+0.002*	-0.001*	0.000*	+0.005*

*Note: Stressed 1388 hours.

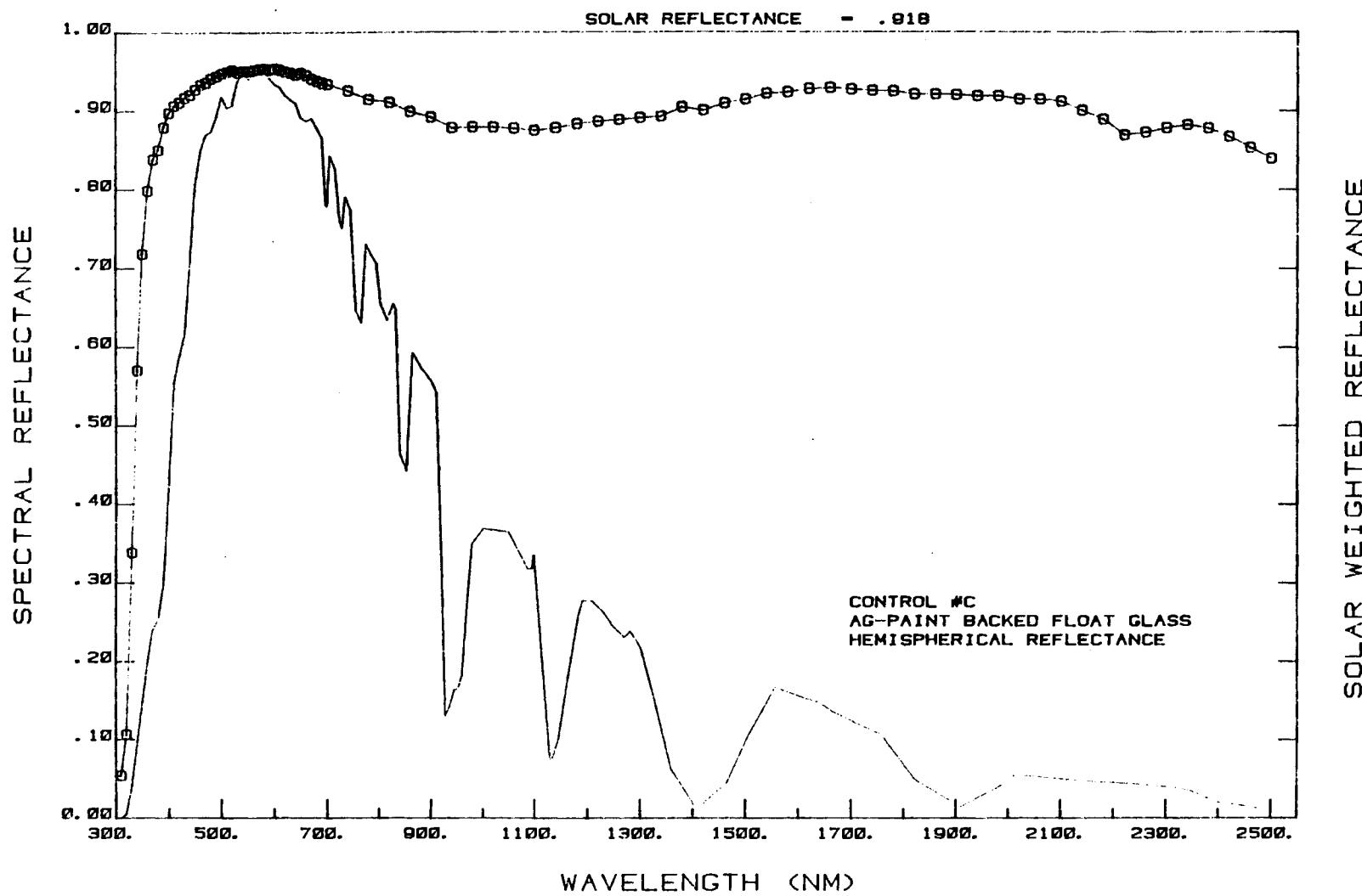


FIGURE 11. Spectral Hemispherical Reflectivity of Manufacture C Mirror prior to Accelerated Environmental Exposure

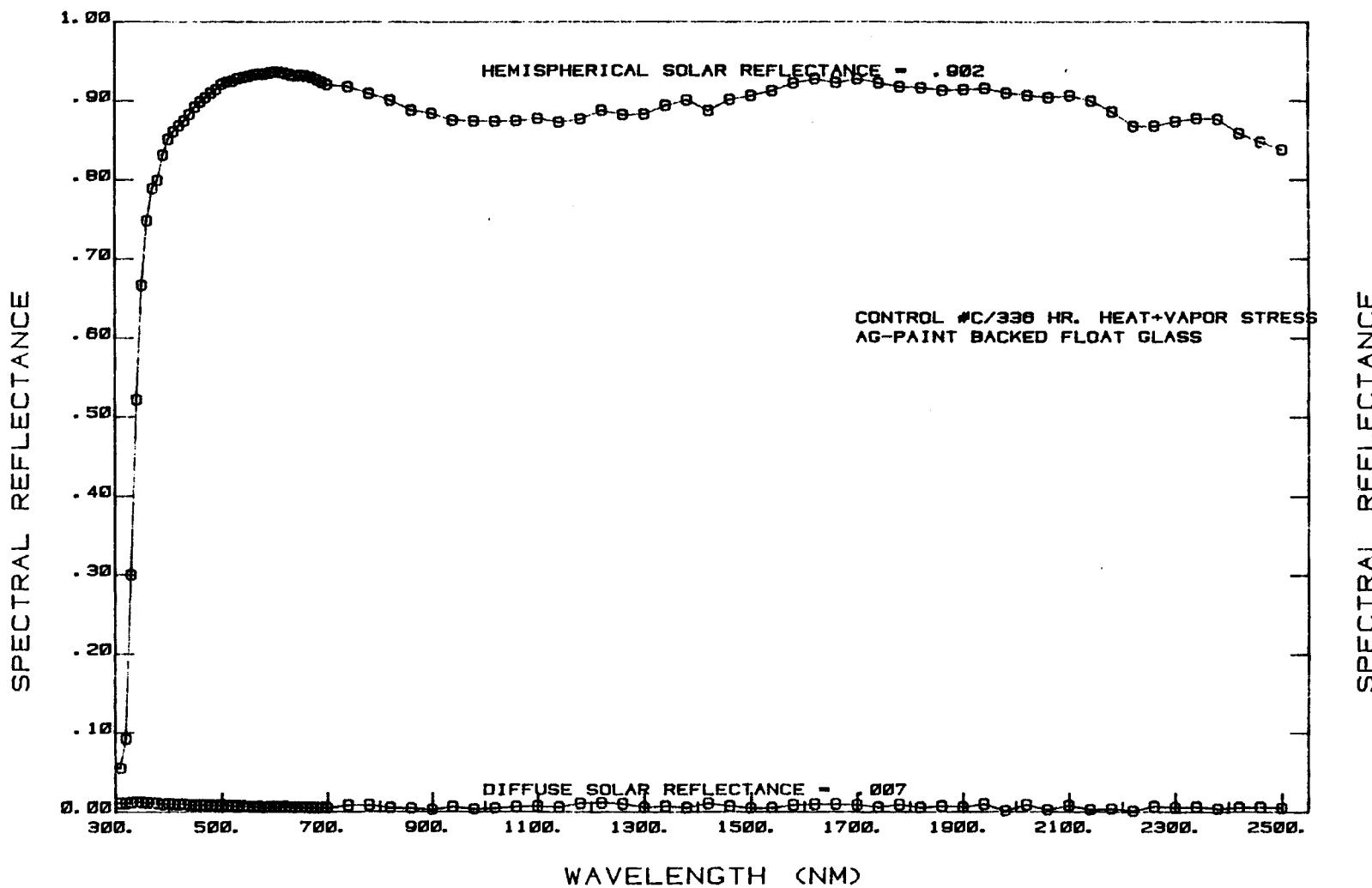


FIGURE 12. Hemispherical and Diffuse Reflectance Curves for Manufacture C Mirror after 336 Hours in an 80°C Heat and Vapor Environment.

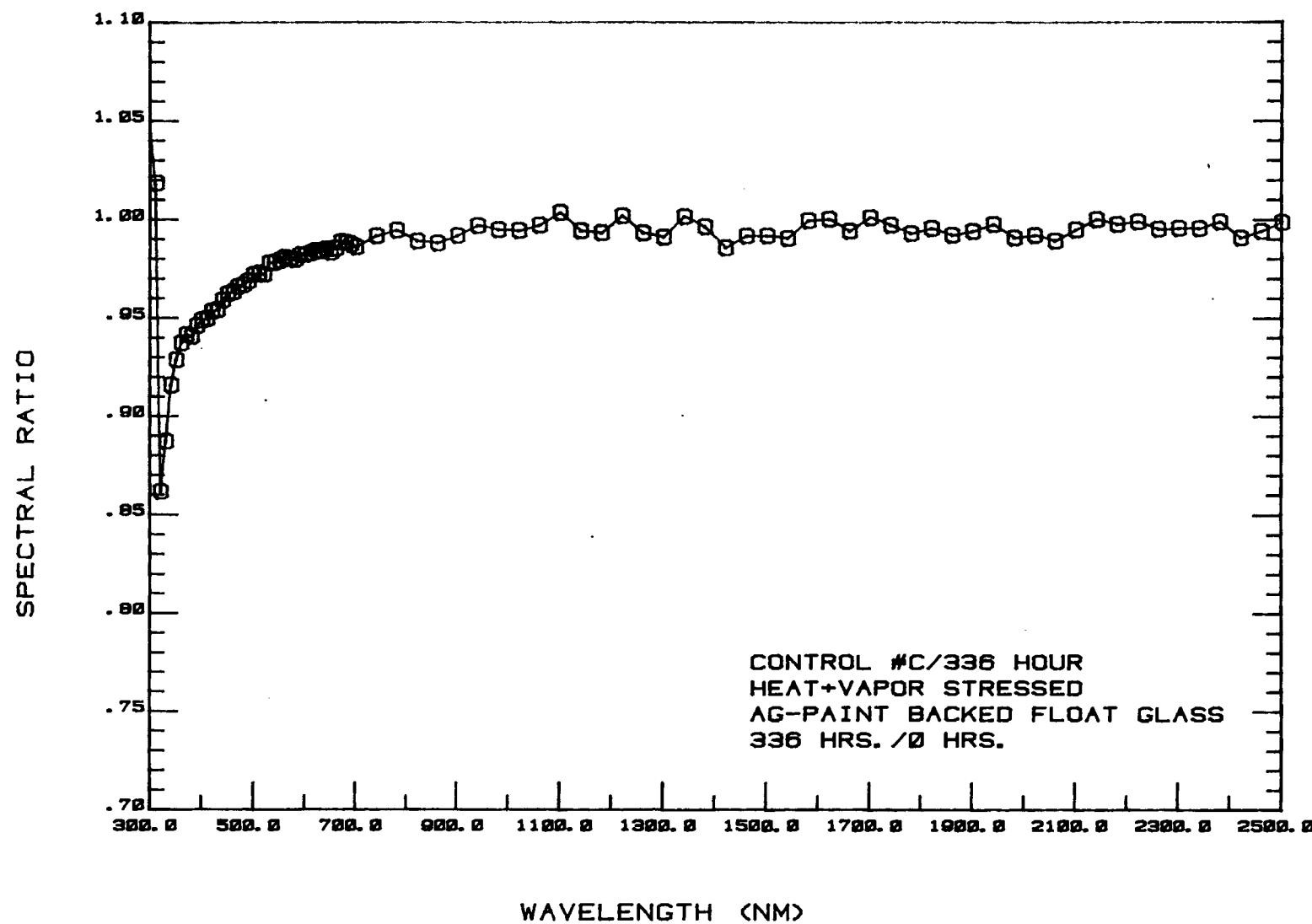


FIGURE 13. After Stress/Before Stress: Reflectivity Ratio Curve for Manufacturer C Mirror.

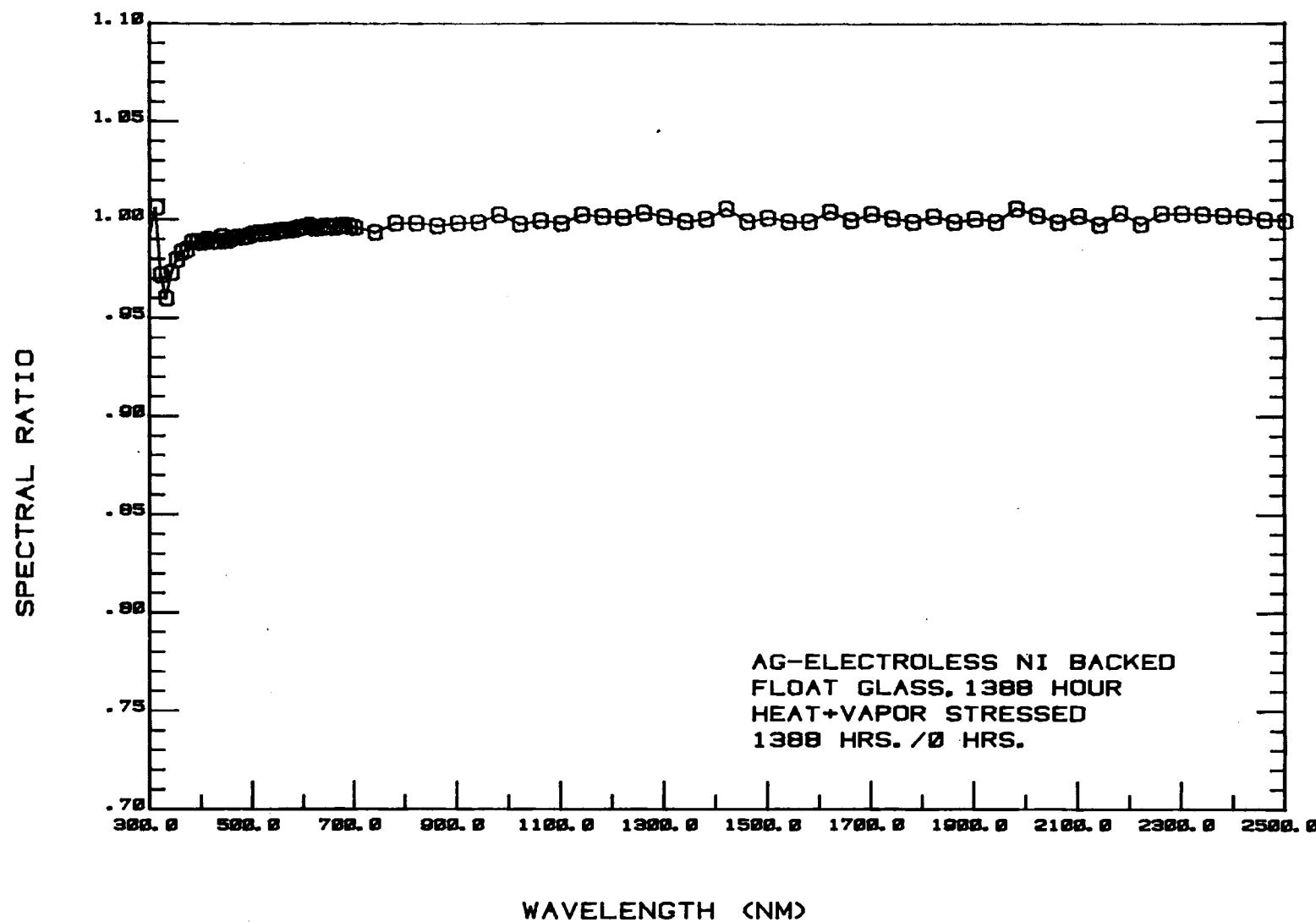


FIGURE 14. After Stress/Before Stress: Reflectivity Ratio
Curve for Electroless Nickel Overcoated Mirror

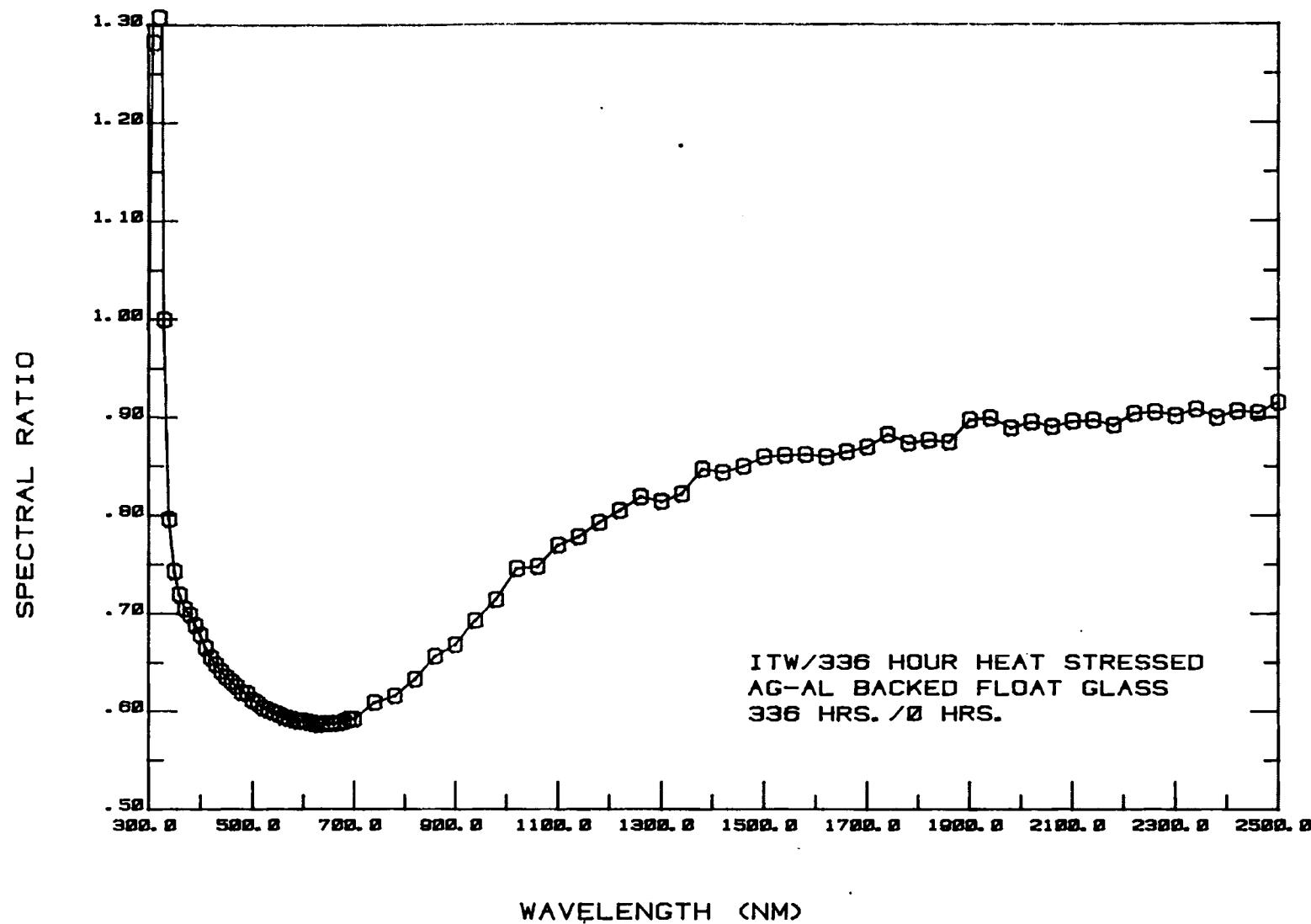


FIGURE 15. After Stress/Before Stress: Reflectivity Ratio Curve for the ITW Aluminum Backed Mirrors

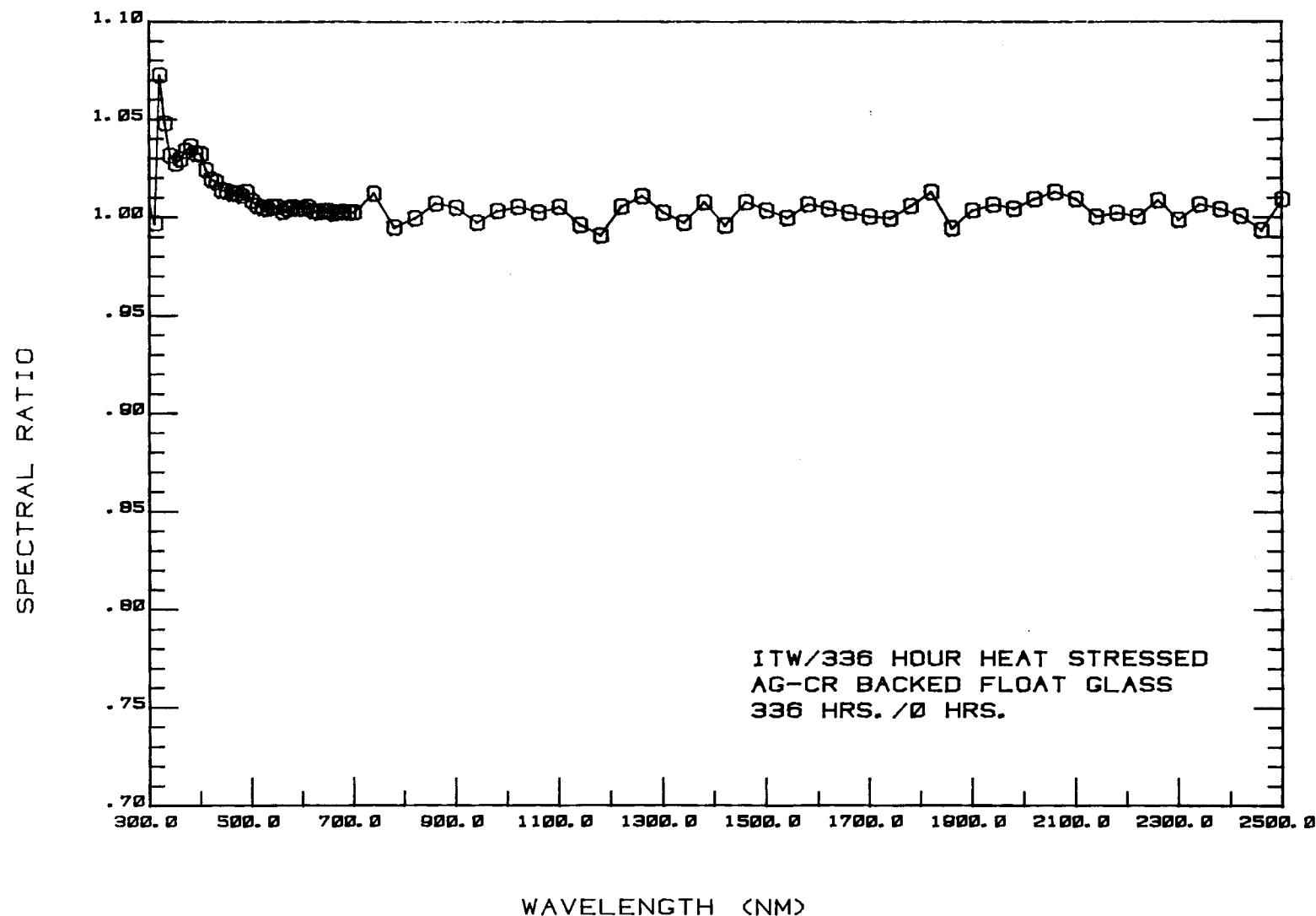


FIGURE 16. After Stress/Before Stress: Reflectivity Ratio
Curve for the ITW Chromium Backed Mirror

for all the other mirror types before stressing look very similar to the reference standard mirror data. The ratio of stressed/unstressed hemispherical reflectances for this mirror is plotted in Figure 13 to more clearly define the induced changes. The most significant changes in reflectivity are found in the UV-visible region (300-750 nm) of the spectra.

In general, the reflectance of most of the degraded mirrors, after exposure in either dry heat or heat plus vapor environments, is characterized by an abrupt drop in reflectance in the 320 - 330 nm region. This absorption may be associated with the silver surface plasmon that is found close to this wavelength region.⁽⁶⁾ Due to the very low reflectance of silver in the 310-330 nm region, small changes in reflectance here will be greatly magnified in the ratio curve, and the absolute magnitude of the initial reflectance drop is small. The reflectivity ratio approaches unity after this initial absorption and, except for the most severely degraded mirrors, the reflectivity ratio remains close to unity through the NIR region. This severity of the degradation of a mirror can be directly related to the reflectance loss in the UV-visible region.

The reflectivity ratio curve for the less severely degraded electroless Ni overcoated mirror is given in Figure 14. Figure 15 shows the ratio curve for the severely degraded aluminum backed ion plated mirror. Some of the ion-plated mirrors exhibited enhanced reflectivity in the UV after exposure to the dry heat environment. The curve for one such mirror (Cr backed) is displayed in Figure 16.

CONCLUSIONS

Based on the above limited tests and data it appears that the life expectancy of a standard wet process silver/copper mirror might be extended considerably by overcoating with a thin electroless nickel coating. Although the set of ion-plated mirrors examined here did not perform as well as the standard paint backed mirrors, a conclusive assessment of the effectiveness of these coatings on mirror durability cannot be made with any degree of certainty without further experimentation to optimize the process parameters.

As with any accelerated testing procedures, the results presented here should be viewed with considerable caution until the mechanics of the degradation phenomena are well understood.



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