

Durability Testing of Antireflection Coatings for Solar Applications

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Durability testing of antireflection coatings for solar applications

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ABSTRACT

Antireflection (AR) coatings can be incorporated into highly transmitting glazings that, depending upon their cost, performance, and durability of optical properties, can be economically viable in solar collectors, agricultural greenhouses, and PV systems. A number of AR-coated glazings have been prepared under the auspices of the International Energy Agency (IEA) Working Group on Durability of Materials for Solar Thermal Collectors. The AR coatings are of two types, including 1) various sol-gels applied to glass and 2) an embossed treatment of sheet acrylic. Typically, for unweathered glazings, a 4–5% increase in solar-weighted transmittance has been achieved. For AR-coated glass, reflectance values as low as 0.5%–0.7% at selected wavelengths (680–720 nm) were obtained. To determine the durability of the hemispherical transmittance, several collaborating countries are testing these materials both outdoors and in accelerated weathering chambers. All materials exposed outdoors are affixed to mini-collector boxes to simulate flat-plate collector conditions. Results for candidate AR coatings weathered at geographically disperse outdoor test sites exhibit changes in spectral transmittance primarily in the high visible range (600–700 nm). Accelerated testing at measured levels of simulated solar irradiance and at different constant levels of temperature and relative humidity have been performed in different countries. Parallel testing with different levels of laboratory-controlled relevant stress factors permits the time-dependent performance of these materials to be compared with measured results from in-service outdoor exposure conditions. Coating adhesion and performance loss resulting from dirt and dust retention are also discussed.

Keywords: Antireflection coatings, durability of optical properties, weathering, accelerated testing

1. INTRODUCTION

AR coatings offer an energy-saving benefit for a variety of buildings and renewable energy applications. Highly transmissive glazings for flat-plate collectors significantly increase optical and collector system efficiency. If constant thermal losses are assumed, a 5% increase in solar transmittance could result in as much as a 10% improvement in energy collection efficiency. The energy gain resulting from increased transmittance was calculated using a “Solar Hot Water Systems” model¹ for a system in the Netherlands with 2.72-m² of collector area. For that location, the simulation predicted an additional energy gain that with current heating efficiencies, energy prices, and expected lifetime, results in additional energy savings of \$10/m². By assuming a return on investment for half the expected collector lifetime, it would be cost beneficial if an AR coating could be applied for less than \$5/m². Such a cost is reasonable compared with coatings for window glazings that are far more complex and that demand much higher homogeneity than for flat-plate applications. Similar analyses for other applications indicate that AR coatings are economically attractive for greenhouse use if their added cost is less than \$7.5/m², and for photovoltaic use if the added cost is less than \$14/m².

Another important consideration in product development and commercialization is the durability of advanced components during in-use service conditions. AR coatings for visible wavelengths are commercially available, but they usually have reduced transmittance in the near infrared; consequently, the solar-weighted transmittance is not increased. AR-coated glazing samples produced by etching glass in fluosilicic acid were exposed outdoors for seven years in Sweden with less than 1% loss of transmittance (after cleaning)². The durability and cleanability of other types of AR coatings is largely uncertain. To address these concerns, a number of new candidate AR coating formulations were identified, samples were prepared, and these were subjected to outdoor and accelerated durability testing. This effort was an international collaboration under the auspices of the IEA Working Group on Materials for Solar Thermal Collectors.

2. SAMPLE PREPARATION

Samples of candidate AR-coated substrates were prepared by Fraunhofer Institutes (ISE, ISC, IWM) in Germany, and Uppsala University (UU) in Sweden. Coatings were applied to two types of substrate materials, low-iron float glass (OptiWhite) and sheet acrylic (Lucite). Uncoated substrates were also prepared for concurrent and comparative testing. A summary of the various constructions is provided in Table 1.

Coating	Substrate	Lab Supplying Sample
None	OptiWhite Low Iron Glass	
Sol-gel	OptiWhite Low Iron Glass	ISE
Sol-gel; 10% H ₂ O, No Thermal Cure	OptiWhite Low Iron Glass	UU
Sol-gel; 10% H ₂ O, Thermal Cure	OptiWhite Low Iron Glass	UU
Sol-gel; 50% H ₂ O, Thermal Cure	OptiWhite Low Iron Glass	UU
None	Lucite Acrylic	
Embossed AR Coating	Lucite Acrylic	ISE

Sol-gels have previously been proposed and discussed for AR coating applications for a number of solar technologies³. Germany prepared AR coatings using sol-gel on glass and an embossed/random surface structure on acrylic sheet substrates⁴. The latter are known as moth-eye AR coatings with periodic structures produced in photo-resist using a UV laser holographic process. Models of one- or two-dimensional periodic structures on these surfaces predict broadband (400–1600 nm) transmittances of 96.2% and 97.0%, respectively. The measured dependent increase in spectral transmittances of materials with AR coatings applied to glass or an acrylic substrate is clearly evident in Figures 1 and 2, respectively. An increase of more than 5% in solar-weighted transmittance is measured for an AR-coated glass, and an improvement greater than 4% is obtained for AR-coated acrylic.

Sol-gel coated glass substrates were prepared by a dip-coating process in Sweden⁵. Initially, both ethanol- and water-based sol-gels were tried. The advantage of water-based formulations is that high-temperature curing processes are avoided, and this provides an option for using heat-sensitive substrate materials such as polymers. Silica particles, which had diameters in the range 60–70 nm, were used to prevent smaller particles from filling in holes, and resulted in a better porosity, lower refractive index, and consequently better AR performance. Reflectance values as low as 0.5%–0.7% at selected wavelengths (680–720 nm) were obtained. Typically, an increase of 5% in solar-weighted transmittance (85%–86% to 90%–91%) was measured for both ethanol-based sol-gel coatings on glass and water based sol-gels on acrylic. A slight loss in transmittance was measured for glass substrate samples that were heat-treated, presumably because of an increase in film density and consequent increase in the refractive index.

Some adhesion problems were observed with the sol-gel coatings when they were applied to acrylic substrates. Surface cleaning (ultrasonic), or surface modification (e.g., glow discharge, chemical etch), or both were investigated in an attempt to improve the adhesion. These processes were unsuccessful in sufficiently improving the adhesion of sol-gel AR coatings deposited onto acrylic sheet substrates. Adhesion of the coatings to the polymer was poor because they cannot be cured at temperatures greater than the glass transition temperature of the acrylic.

AR coatings on glass substrates were prepared by a sol-gel process using carrier formulations both of 50% ethanol/50% water and 90% ethanol/10% water. The latter formulation was applied with and without thermal curing. For these sol-gel coated glass samples, heat-curing just over the softening temperature of glass (550°–570°C) was found to harden the glass (essential in preventing cracking of glass by thermal gradients in collector cover plate applications) without adversely effecting the density (and therefore the antireflection properties) of the SiO_x AR coating. The improved spectral transmittance of these various sol-gel formulations is shown in Figure 3. Both of the 10% water compositions exhibit nearly a 10% increase in transmittance centered around 500 nm. The enhanced transmittance resulting from thermal curing is most pronounced in the near-infrared (NIR) part of the spectrum. For the same 10% water composition, thermal curing results in an additional 1% transmittance at wavelengths greater than 1000 nm. The thermally cured 50% water formulation results in an increase of about 2.5% NIR transmittance, although the maximum in the visible is shifted to 600 nm.

3. ENVIRONMENTAL EXPOSURE TESTING

Samples of AR-coated glazings were subjected to in-service outdoor and accelerated laboratory exposure conditions. Outdoor testing was carried out in Switzerland at the Institut für Solartechnik (SPF), Germany (at ISE in Freiburg), and at three sites in the United States (Golden, CO; Phoenix, AZ; and Miami, FL). A precise and detailed knowledge of the specific environmental stress conditions experienced by weathered samples is needed to allow understanding of site-specific performance losses and to permit service lifetime prediction of candidate AR coatings. Consequently, each operational exposure site is fully equipped with appropriate meteorological and radiometric instrumentation and data-logging capability.

Accelerated exposure testing was also carried out at the temperature (T), and relative humidity (RH), in each chamber as given in Table 2. An Atlas Ci5000 Weather-Ometer[®] was used in the U.S. and custom-designed HS-Simulatoren were used in Germany and Switzerland. The Ci5000 uses a xenon-arc lamp filtered to provide a close match with a terrestrial air mass 1.5 spectrum; the European chambers use an unfiltered metal halide (HMI) lamp source that has an enhanced UV-B ($\lambda = 280\text{--}315$ nm) intensity. The measured irradiance for the light used in the three countries is shown in Figure 4 (compared with a 2X global terrestrial standard spectrum⁶). Note especially that the HS-Simulatoren chambers expose at considerably higher levels of UV-B compared with even a 2X terrestrial solar spectrum. Parallel testing with relevant stress factors at different levels was intended to allow the sensitivity of materials degradation to these factors to be quantified, and allow damage function models to be evaluated. This in turn can be used to compare the time-dependent performance of these materials with measured results from in-service outdoor exposure.

Table 2. Accelerated Weathering Chamber Exposure Conditions		
Country	Temperature (°C)	Relative Humidity (%)
Germany	40	95
Switzerland	80	40
United States	60	80

Samples were exposed outdoors using mini-collector boxes as specified by ASTM testing standards⁷, except that the absorber coatings applied to the interior of the stainless steel boxes were black chrome selective surfaces. Samples exposed outdoors were 15 cm x 15 cm; samples exposed in the accelerated weathering chambers were 7.5 cm x 7.5 cm. To prevent stray light from being incident on the samples (thereby resulting in an unmeasured level of light exposure), a black-chrome selective coated opaque material was placed behind the glazings during exposure to block unrealistic back-scattered light. The selective coating minimizes excessive thermal loading of the glazing samples; an air gap ≥ 2 cm between the glazing samples and the opaque back material was used to allow additional convective heat transfer away from the glazing.

4. OPTICAL CHARACTERIZATION

For flat-plate collector applications, AR coatings are used to increase the hemispherical transmittance of the glazing. Thus, the measure of performance that was chosen to quantify degradation was near normal direct-hemispherical transmittance ($\tau_{2\pi}$) over a specified wavelength range ($\Delta\lambda$) after some time of exposure (t), weighted by a terrestrial air mass 1.5 global solar spectrum^{6,8}. The wavelengths between 600–700 nm were chosen because for AR-coated glazings, this spectral region is particularly sensitive to degradation effects, thereby potentially providing a very rapid quantification of performance loss. The hemispherical transmittance performance parameter is then given by Eq. (1),

$$\tau_{2\pi}(\Delta\lambda = 600 - 700 \text{ nm}, t) = \frac{\int_{\lambda=600 \text{ nm}}^{\lambda=700 \text{ nm}} \tau_{2\pi}(\lambda, t) I(\lambda) d\lambda}{\int_{\lambda=600 \text{ nm}}^{\lambda=700 \text{ nm}} I(\lambda) d\lambda} \quad (1)$$

in which $I(\lambda)$ is the solar spectral irradiance⁶. Broadband performance loss, such as $\tau_{2\pi}$ weighted across the entire solar spectrum ($\Delta\lambda = 250\text{--}2500 \text{ nm}$), would be much less sensitive to narrowband degradation and consequently less useful as a rapid indicator of durability.

All samples were optically characterized prior to exposure ($t=0$) and then periodically as a function of weathering. For samples exposed outdoors, half of the exposed sample surface was measured as received (uncleaned) and then after cleaning; the other half of the exposed sample surface was measured as received (that is, it is never intentionally cleaned, to give an indication of the effect of cumulative soiling). Optical measurements were carried out according to standard test procedures⁸ by the country in which the samples were being exposed. A comparison of the spectral optical measurements of unweathered AR-coated acrylic performed in the three countries is shown in Figure 5; the overall agreement is very good. All data were then analyzed according to Eq. (1).

5. TEST RESULTS

In Figures 6-12, the actual measurements are indicated by the various symbols used; these symbols are connected by lines to allow easy association of common data and to help identify trends. The optical durability of sol-gel coated glass prepared by ISE after exposure outdoors at three sites in the U.S. and in Switzerland and Germany is shown in Figure 6. For the data on samples exposed outdoors, measurements were made as received from field exposure and then after cleaning. This results in two data points at the same exposure time. Generally, the lower value is obtained before cleaning and the higher value after cleaning, although occasionally this pattern is anomalously reversed. For the AR-coated glass, little loss in transmittance between 600–700 nm has been observed. After cleaning, the transmittance of samples exposed in Golden, CO, for more than a year is the same as the initial value. However, a 2% loss in transmittance is experienced by samples exposed in Phoenix, AZ, for the same time. Exposure at the Miami, FL, site is even a harsher environment as shown by the rapid loss of about 4% transmittance (in just one month) that persists after almost a year, even after cleaning.

The AR-coated acrylic material demonstrates very good optical durability at all three sites in the U.S. (Figure 7). After weathering for about a year the transmittance between 600–700 nm can be restored to within 1% of their initial values by cleaning. For this sample, it appears easier to remove dirt and dust deposited during exposure in the U.S. than it is to remove soil accumulated in Europe. Samples exposed in Switzerland and Germany have lost more than 2% in transmittance after weathering for only about six months.

Results are presented in Figures 8–10 for samples (exposed in the U.S. only) having AR coatings made using sol-gel formulations of 10% water/no thermal cure, 10% water/thermal cure, and 50% water/thermal cure. All three formulations exhibit a rapid (0–3 months) loss of 7% to 10% transmittance when exposed in Phoenix, AZ, and Golden, CO. However, upon further weathering, these samples regain most of these losses that result in only net transmittance losses of 1%–3% between 600–700 nm after 250–325 days. The transmittance changes are similar for all three formulations at these two sites. Miami, FL, is the most severe environment for these coatings. After 300 days exposure, both of the 10%-water formulations have lost roughly 4% transmittance, whereas the 50%-water composition has lost more than 6% transmittance, even after cleaning.

In general, weathering of these AR-coated glazings in accelerated exposure chambers produces less degradation than experienced during exposure to in-service real-time conditions. Figures 11 and 12 present results for the AR-coated glass and acrylic materials, respectively. The data (symbols) represent average values for whatever number of sample replicates were available (typically, 3); the error bars are for \pm one standard deviation. These data show that the coated materials tested are fairly insensitive to the simulated solar irradiance, temperature, and moisture to which they were subjected (see Table 2 and Figure 3).

Exposure to, and accumulation of, dust and dirt during outdoor exposures appear to be the most important factors in causing losses in the transmittance of these materials.

6. CONCLUSIONS

The time-dependent changes in hemispherical transmittance of several state-of-the-art AR-coated glazing materials have been assessed for exposure to outdoor and laboratory-controlled environmental conditions. Preliminary results show that the candidate constructions studied are promising and merit further consideration and evaluation. Exposure of samples to five different outdoor sites indicates that the relative severity of the sites in terms of loss in hemispherical transmittance for AR-coated glass is: Miami, FL is the most harsh, followed by Phoenix, AZ, and then Golden, CO; Switzerland and Germany are roughly equivalent and are the least harsh. This ranking is different for AR-coated acrylic where Switzerland and Germany are again similar but are now the most harsh, followed by Phoenix, AZ, Golden, CO, and Miami, FL (least harsh). The AR coatings that were tested do not appear to be particularly susceptible to degradation from exposure to simulated solar irradiance, temperature, or relative humidity to which they have been exposed during durability testing. Soiling seems to be the prime cause of degradation in hemispherical transmittance. For improving the durability in hemispherical transmittance, a fundamental understanding of soiling mechanisms and cleaning processes is needed. This will allow improvements to be made in the durability of these coated materials. In addition, an accelerated testing device needs to be developed that better simulates in-service exposure conditions.

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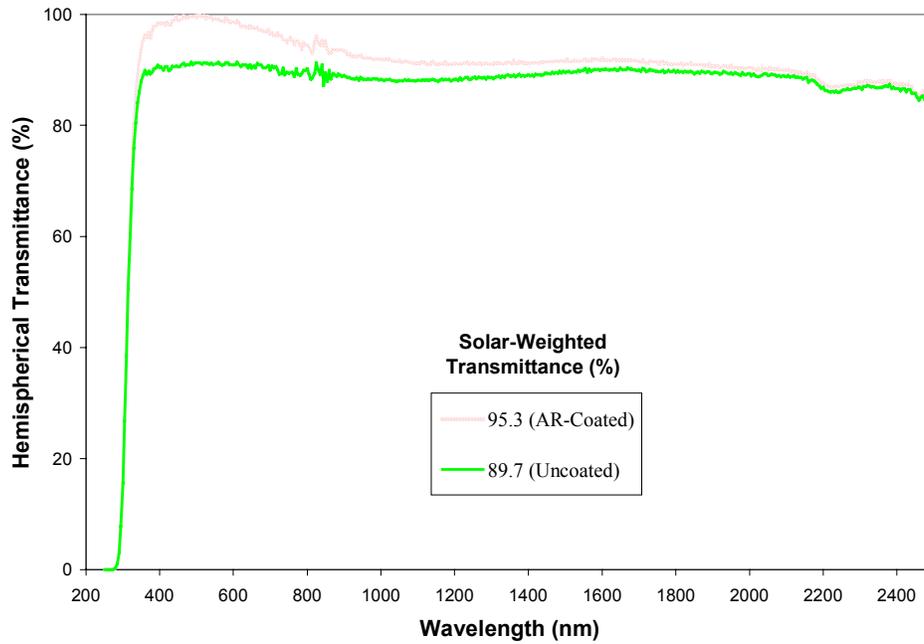


Figure 1. Hemispherical Transmittance from 250 nm to 2500 nm of OptiWhite Glass with and without an AR Coating

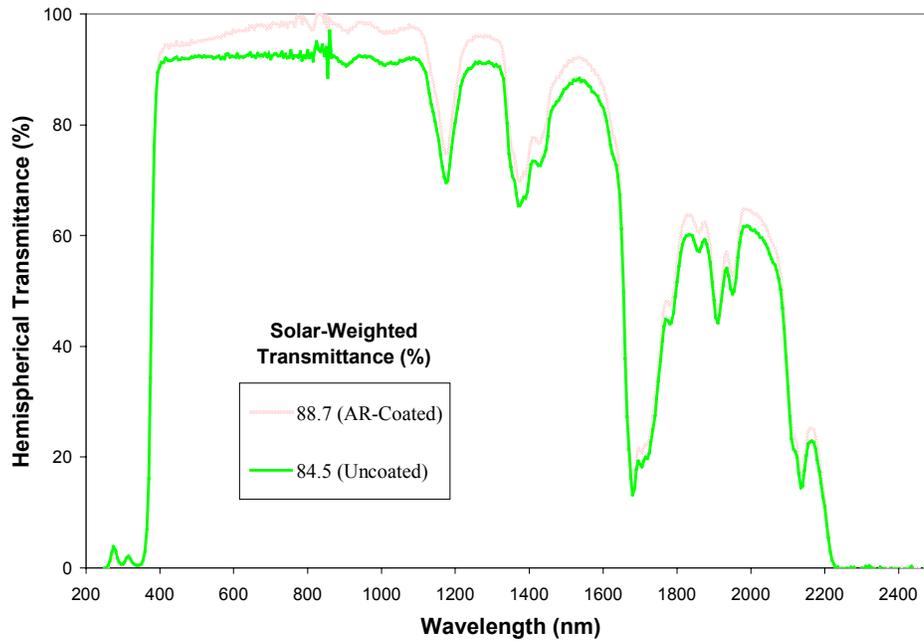


Figure 2. Hemispherical Transmittance from 250 nm to 2500 nm of Lucite Acrylic with and without an AR Coating

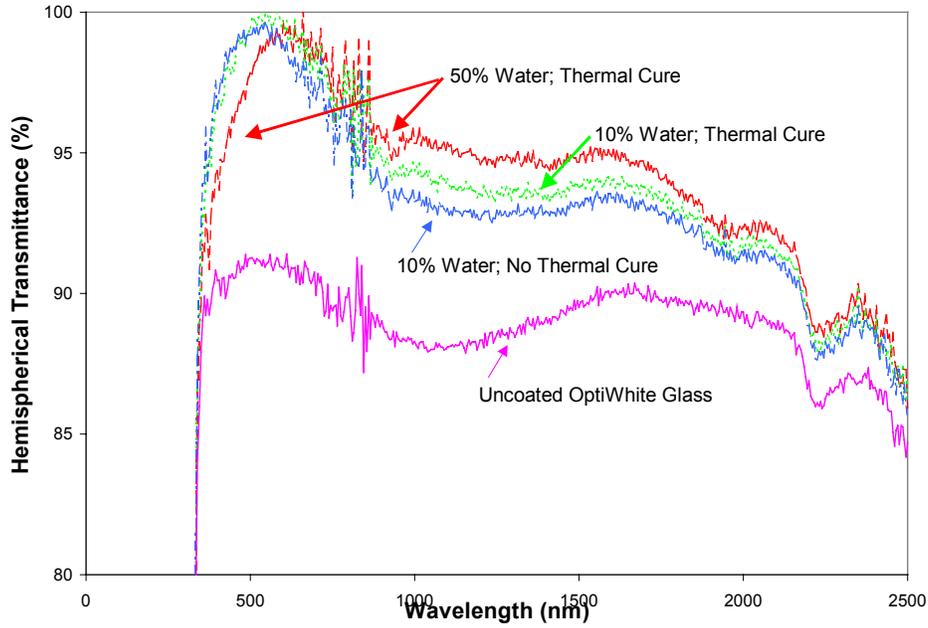


Figure 3. Hemispherical Transmittance from 250 nm to 2500 nm for Various Sol-Gel AR-Coating Formulations Prepared at Uppsala University

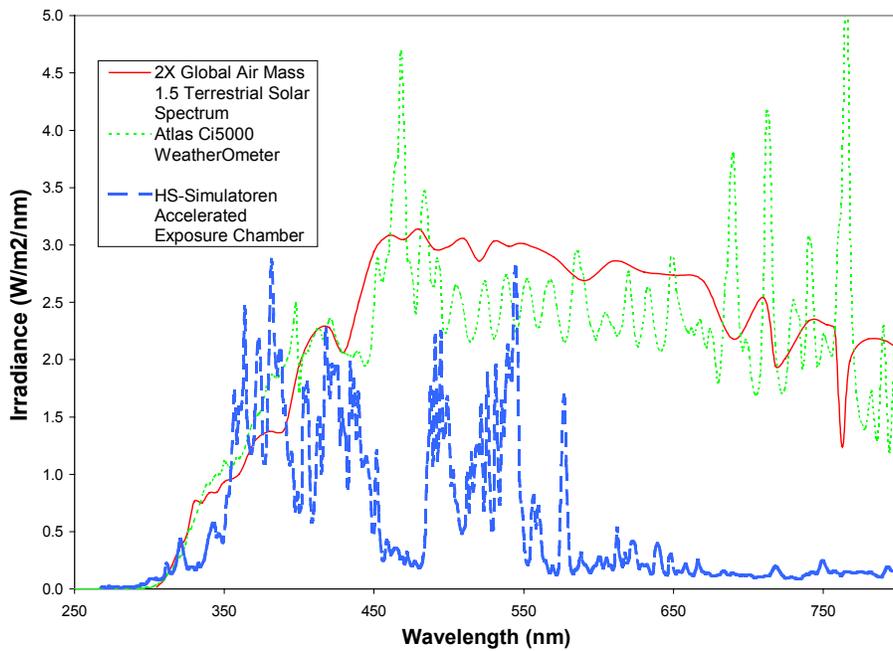


Figure 4. Irradiance of Lamps Used in Accelerated Exposure Chambers Compared with 2X Global Air Mass 1.5 Terrestrial Solar Spectrum

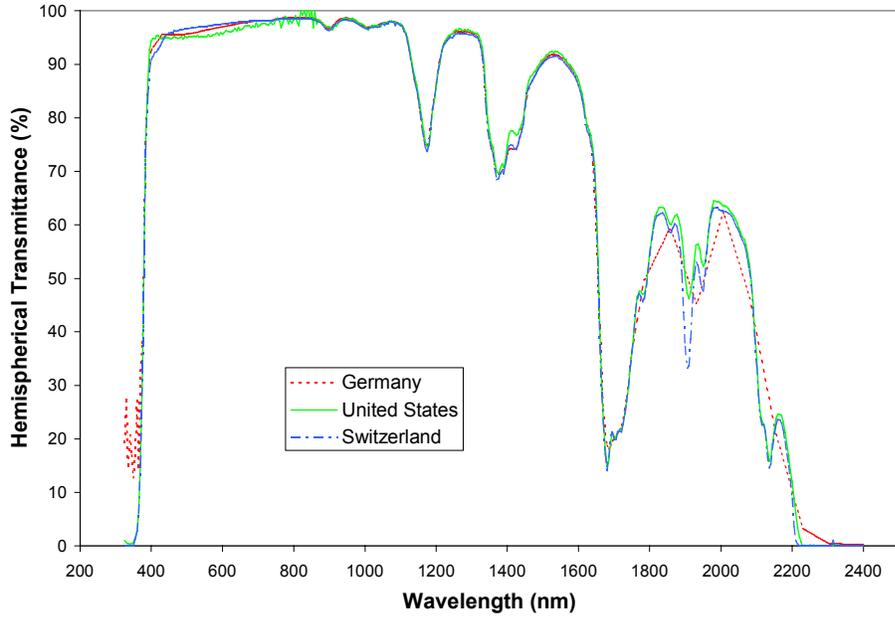


Figure 5. Comparison of Optical Transmittance Measurements Performed in Three Countries

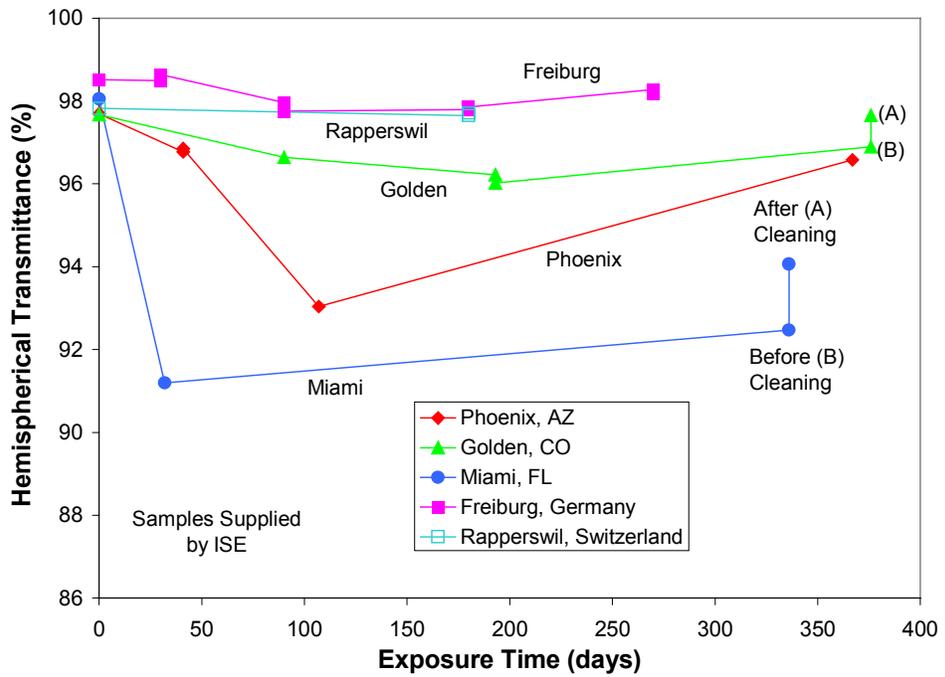


Figure 6. Hemispherical Transmittance between 600–700 nm for AR-Coated Glass as a Function of Time at Five Different Outdoor Exposure Sites

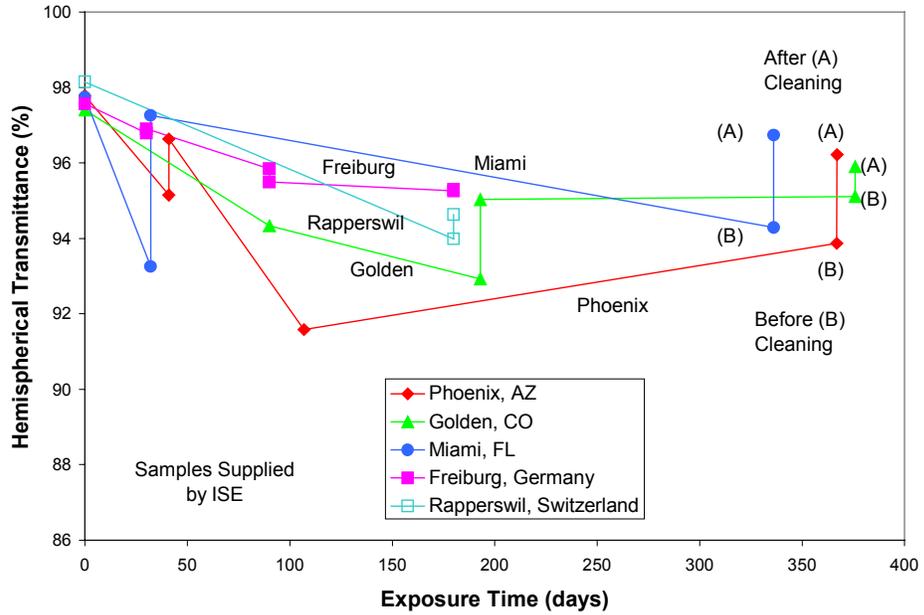


Figure 7. Hemispherical Transmittance between 600–700 nm for AR-Coated Acrylic as a Function of Time at Five Different Outdoor Exposure Sites

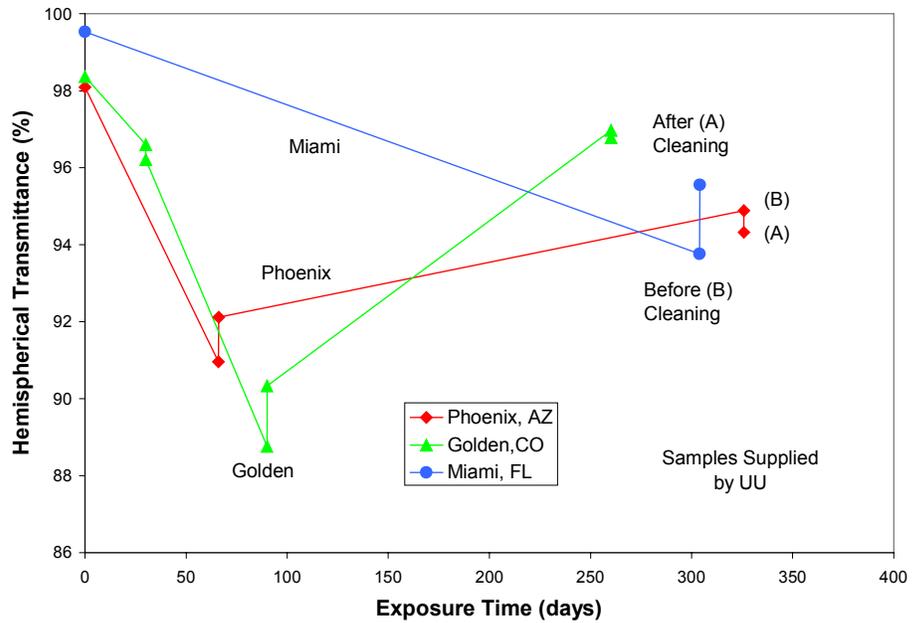


Figure 8. Hemispherical Transmittance between 600–700 nm for AR-Coated (10% Water Sol-Gel; No Thermal Cure) Glass as a Function of Time at Three Different Outdoor Exposure Sites

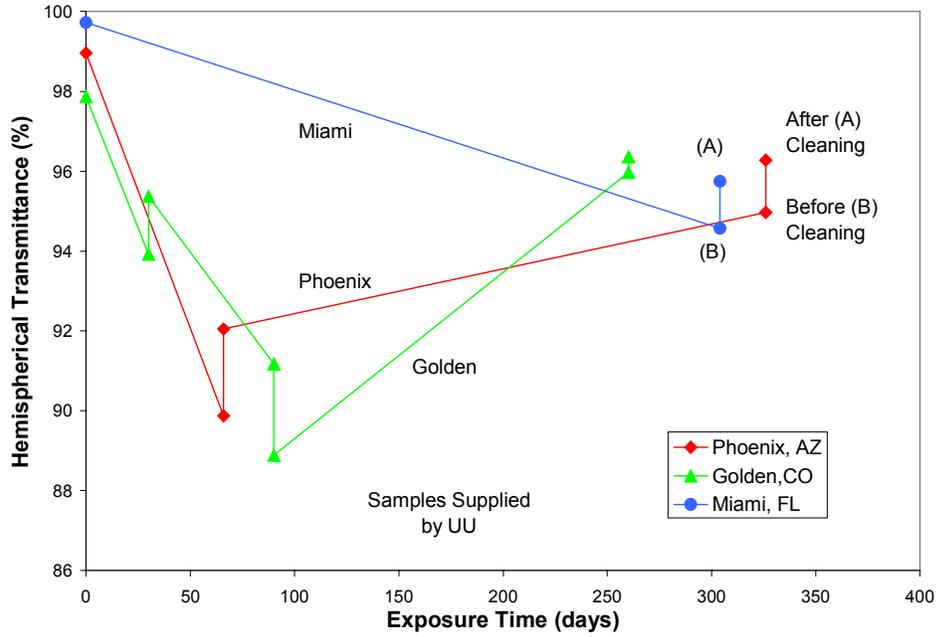


Figure 9. Hemispherical Transmittance between 600–700 nm for AR-Coated (10% Water Sol-Gel; Thermal Cure) Glass as a Function of Time at Three Different Outdoor Exposure Sites

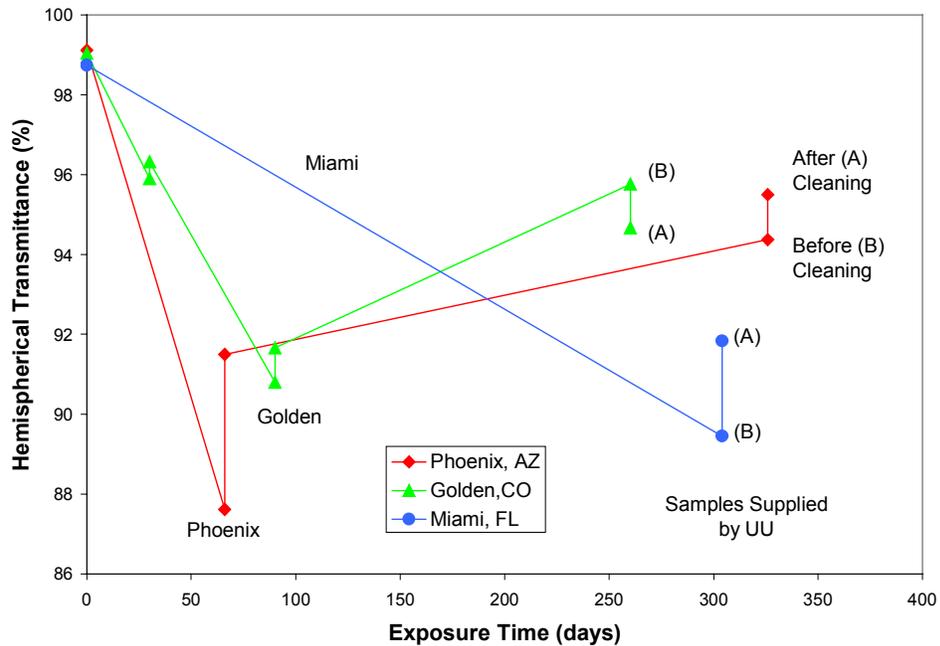


Figure 10. Hemispherical Transmittance between 600–700 nm for AR-Coated (50% Water Sol-Gel; Thermal Cure) Glass as a Function of Time at Three Different Outdoor Exposure Sites

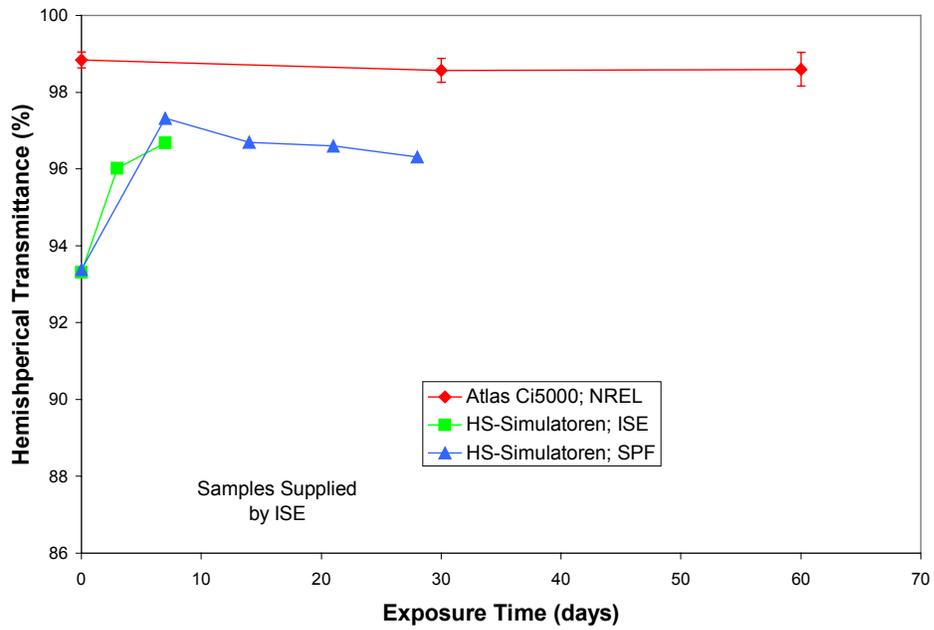


Figure 11. Hemispherical Transmittance between 600–700 nm for AR-Coated Glass as a Function of Accelerated Exposure Time

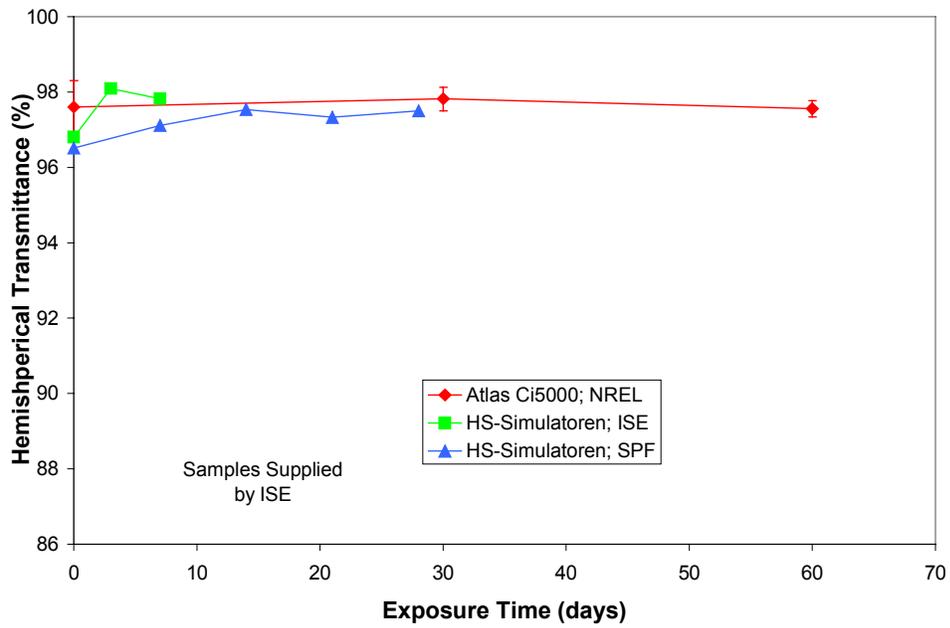


Figure 12. Hemispherical Transmittance between 600–700 nm for AR-Coated Acrylic as a Function of Accelerated Exposure Time

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