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Solarization of Heliostat Glasses

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SOLARIZATION OF HELIOSTAT GLASSES*

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ABSTRACT

We have observed a solar-induced decrease in Fe^{2+} absorption in heliostat glasses from the solar furnace at Odeillo, France. This decrease occurs throughout the sample (not just at the exposed surface) and is of sufficient magnitude to result in an increase of 2.5% in solar transmittance in a period of nine years. Optical and ESR studies did not detect a corresponding increase in Fe^{3+} concentration. The implication of these results on a microscopic model for the observed solarization is discussed. Solar simulation studies produced effects of magnitude and sign similar to those observed in the field exposed samples, and appear to offer an attractive means for screening samples for solarization tendencies.

1. Introduction

The use of second-surface silvered glass mirrors for solar applications has focused considerable attention on the initial and long-term optical properties of the glass superstrate. One prime motivation has been to minimize the absorptive losses in the bulk glass. These losses are known to arise from the presence of iron impurities, primarily Fe^{2+} and Fe^{3+} . The Fe^{3+} absorbs in the ultra-violet (uv) and is of little consequence to solar applications. However, the Fe^{2+} has a broad absorption band centered at 1000 nm, a region in which there is considerable solar irradiance. Low-iron glasses and/or highly oxidized glasses are being considered to minimize absorptive losses arising from the Fe^{2+} band. However, even in these glasses, the residual iron concentration is sufficiently high that a shift in the Fe^{2+} to Fe^{3+} ratio (the "redox" state of the glass) could result in significant changes in the transmittance of the glass superstrate. Therefore, to evaluate the performance of heliostat glass, the initial values and the stability of the Fe^{2+} and Fe^{3+} concentrations should be determined. Solar-induced changes in the optical properties of glass, hereafter referred to as "solarization," have received scant attention in the literature. The very existence of such changes in glasses considered for heliostat applications remains unestablished. In this paper, we report the observation of a solar-induced decrease in Fe^{2+} absorption in (a) soda-lime silicate glasses from mirrors used in the solar furnace at Odeillo, France and (b) solar simulation tests on a variety of glass samples.

2. Experimental Procedures

2.1. *Samples*

We examined a total of seven samples of heliostat glass from the solar facility at Odeillo, France.⁺ Five of these (Samples #6-10) had been in field

⁺ Samples were provided by C. Royere of CNRS, Odeillo, France

use for eleven years. The other two samples (#11, 13) had been exposed to sunlight for only two years prior to being stored in a warehouse. No unexposed samples were available. All samples were 2" by 2" x 0.280", appeared yellow-green when viewed edge-on, and exhibited no discernible differences under visual and microscopic examination. There was no evidence of any weathering. Neutron activation analysis indicated the glasses to be soda-lime-silicates containing approximately 6.3×10^2 wt ppm of Fe and 6.9×10^2 wt ppm of As. The composition of glasses exposed for two and eleven years were nearly identical in all respects (including trace impurities such as the rare earths), confirming the common origin of these samples.

In addition, we subjected a variety of glasses to accelerated solarization tests. Included were samples of the Odeillo glass exposed for two and eleven years, and a number of glasses now being considered for use in heliostats in this country, i.e. float glass with standard (613 wt ppm of Fe) and low (305 wt ppm of Fe) iron concentrations (each 0.120" thick), and an aluminosilicate glass made by the fusion process (0.057" thick pieces of Corning 0317 containing 546 wt ppm Fe).

The compositions of all glasses were determined by neutron activation analysis and are summarized in Table 1. To simplify the table, we have listed only those elements with concentrations in excess of 10 wt ppm. Further, this particular analysis would not have detected Si, Al, Ti, Mg, Mn or V.

Table 1. Glass compositions (wt ppm) determined by neutron activation analysis. Only those elements present in concentration of 10 wt ppm or greater are listed. This procedure was insensitive to the presence of Si, Al, Ti, Mg, Mn and V.

	<u>Odeillo Glass</u>	<u>Standard Float</u>	<u>Low-Iron Float</u>	<u>Corning 0317</u>
Na	1.0×10^5	9.6×10^4	1.1×10^5	9.6×10^4
Ca	6.6×10^4	6.2×10^4	7.0×10^4	2.2×10^3
Fe	6.3×10^2	6.1×10^2	3.1×10^2	5.5×10^2
Zn	—	1.4×10^1	—	—
As	6.9×10^2	—	—	4.6×10^3
Rb	—	—	—	5.0×10^1
Sr	4.4×10^1	7.3×10^1	4.7×10^1	6.0×10^1
Zr	5.7×10^1	9.6×10^1	3.1×10^1	2.7×10^2
Ba	1.6×10^1	5.4×10^1	2.0×10^1	—
Sb	—	—	—	2.2×10^1

2.2. Measurement Techniques

Absorption measurements were performed on a Cary 17-I spectrophotometer. A Cary #1413 specular-reflectance accessory, utilizing a modified Strong V-W configuration, was used for the reflectance measurements. Normally the V-W configuration gives a signal which is proportional to the square of the sample reflectivity. However, when dealing with low reflectivities it is preferable to use a secondary reference mirror in one position in the W-configuration, thereby generating a signal linear in the sample reflectivity. This latter geometry was used in this study. To test the accuracy of this procedure, we measured the spectral reflectance of vitreous silica in the wavelength range from 350 to 2500 nm, and found that the data agreed to within ± 0.0025 with reflectance values calculated from the index of refraction. Observation of the transmitted and reflected beams gave no evidence of a diffusely scattered component. Therefore, measurements of the specular transmittance and specular reflectance are sufficient to characterize the optical properties of the sample.

In a number of cases, it was desirable to establish whether the observed changes in absorption were uniformly distributed throughout the thickness of the sample. To facilitate these measurements, we constructed a beam-collimating stage to profile the transmittance parallel to the sample faces. This assembly consisted of two 0.5 mm slits separated by 7.1 cm, and employed a micrometer-drive translation stage to move the sample through the beam. Tests on a slab of vitreous silica yielded optical densities in excellent agreement with the conventional measurements, and uniform to within 0.001 throughout the sample thickness. However, significantly greater scatter was observed in the heliostat glass samples of interest. This increased scatter was determined to be caused

*Let the intensities of the incident, transmitted, absorbed and reflected beams be given by I_0 , I_t , I_a and I_r respectively. Then the transmittance (τ), absorptance (α), and reflectance (ρ) are defined as I_t/I_0 , I_a/I_0 , and I_r/I_0 respectively. The optical density is given by $\log_{10} I_0/I_t$.

by optical inhomogeneities which either modified the reflectance losses, or displaced the beam incident on the photo-detector. These spurious effects are nearly cancelled by using the difference in optical densities ($\Delta O.D.$) at two wavelengths ($\lambda_1 = 1000 \text{ nm}$ and $\lambda_2 = 1700 \text{ nm}$) as a measure of the Fe^{2+} absorption.

The optical measurements were supplemented with electron spin resonance (ESR) determinations of relative Fe^{3+} concentrations. (Earlier studies [1] have shown that the height of the ESR peak at $g = 30/7$ scales linearly with Fe^{3+} concentration in the range of Fe^{3+} concentration of interest in this study). Measurements were performed at room temperature on a Varian E-112 ESR spectrometer operating at approximately 9.3 GHz. High precision measurements of concentration ($\sim \pm 1\%$) were made possible by preparing identically-shaped samples and using a jig to insure repeatable repositioning of the sample within the cavity.

2.3 Simulations

Accelerated solarization studies were based on the use of an Oriel model 6730 solar simulator with a 1000 W ozone-free lamp, a 2" x 2" output beam assembly, and an air mass two filter. Narrow band interference filters (Oriel) and a radiant flux meter (HP8330A with an HP8334A detector head) were used to characterize the spectral irradiance of the simulator over the wavelength region 300-850 nm. The measured spectral distribution was in good agreement with an air mass 2 solar spectrum over all measured wavelengths and had an overall intensity of ~ 10 suns ($\sim 7.5 \text{ kw/m}^2$). The stated beam uniformity of $\pm 7\%$ permitted all five samples to be simultaneously irradiated. To minimize sample heating, the samples were placed in direct contact with a black-anodized, water-cooled, copper plate. During the study, care was taken to maintain the same sample face toward the simulator, and to interchange sample positions within the beam.

Correlating simulated with real-time exposures requires assumptions about "typical" insolation. The data we use, obtained at Albuquerque, New Mexico, are believed to be representative of the American southwest. The average yearly direct normal insolation at Albuquerque is approximately 2650 kw-hrs/m^2 [2], corresponding to ~350 hours of simulation time. A detailed correlation of accelerated studies with real time data requires a knowledge of the local insolation (direct and diffuse) and of the average orientation of the heliostat with respect to the sun.

3. Experimental Results and Discussion

3.1. *Solar Studies*

The heliostat glass samples exposed to sunlight for two and eleven years at the solar furnace at Odeillo, France, provided an opportunity to study solarization effects occurring during actual field service. Optical and electron spin resonance measurements were undertaken on these samples to determine the magnitude and sign of the solar-induced changes in optical properties, and to explore the mechanisms responsible for the observed changes.

3.1.1. *Transmittance and Reflectance Measurements*

Representative transmittance and reflectance spectra for glasses exposed for two and eleven years are shown in fig 1. The reflectance spectra for the two samples are identical within experimental uncertainty. Consequently, any differences in the transmittance spectra may be assigned to differences in the absorptance of these two glasses. In fig. 1a the infra-red (IR) absorptance is seen to decrease with increasing solar exposure. Little or no change in absorptance was observed at wavelengths less than 60 nm.

A difference spectrum (two year minus eleven year exposure) is shown in fig. 2. The shape and magnitude of this spectrum agree with absorption attributable to Fe^{2+} in this glass. (Absorption data of Fe^{2+} in float glass were obtained by subtracting the spectrum of low-iron float glass from the

spectrum of standard float glass.) The effect of the decrease in Fe^{2+} absorption on the solar-weighted (air mass 2) [3] transmittance is shown in Table 2. The average solar transmittance of samples exposed for eleven years is 2.5% higher than for samples exposed for two years. This change would correspond to a 5% increase in the reflectance of the mirrored glass (assuming no change in the silver).

Table 2. Solar weighted (air mass 2) transmittance of heliostat samples from the solar furnace at Odeillo, France.

SAMPLE #	EXPOSURE	% T (AM2)
6	11 yrs	85.2
7	"	85.2
8	"	85.0
9	"	84.3
10	"	84.6
11	2 yrs	82.0
12	"	82.7

3.1.2. Profiling Measurements

Two different approaches were used to characterize the spatial profile of Fe^{2+} concentration in solarized glasses. To determine if the changes in Fe^{2+} absorption were limited to the near-surface region, approximately 2 μm of material was polished from both the front and back surfaces of samples. No measureable effect on transmittance was observed. Spatial variations on a bulk scale were obtained by using the collimator assembly to profile Fe^{2+} absorption as a function of distance into the sample. These measurements were made parallel to the exposed faces with a spatial resolution of 0.5 mm. The magnitude of Fe^{2+} absorption was obtained from the difference in optical densities at 1000 nm and 1700 nm [i.e. $\Delta\text{O.D.} = \text{O.D.} (1000 \text{ nm}) - \text{O.D.} (1700 \text{ nm})$]. Fig. 3 shows the measured Fe^{2+} profiles for samples exposed to two and eleven years of solar irradiation, respectively. (Each sample also received 1000 hours of exposure to the simulation source.) The sample exposed for eleven years exhibits less Fe^{2+} absorption than the two-year sample throughout the bulk of the specimen. This change in Fe^{2+} absorption varies from the front to the back surface of the (eleven-year) sample. Originally, it was presumed that this spatial variation was due to the attenuation of the incident radiation within the glass. Analysis of Fe^{2+} profiles would then allow the deduction of the spectral region of the incident solar spectrum which is involved in the photo-induced changes. In such a picture, an infinite exposure time would result in the total depletion of solarizable Fe^{2+} sites, and in a spatially uniform (possibly zero) Fe^{2+} absorption. However, accelerated aging studies (see below) suggest that the Fe^{2+} profile after infinite exposure is similar, if not identical, to that of the eleven year exposed samples. Consequently, a complete explanation of the observed Fe^{2+} profile must await a better understanding of the solarization mechanism(s).

3.1.3. Annealing Studies

Portions of samples 9, 11 and 13 were annealed at $\approx 610^\circ\text{C}$ in air at 610°C for 30 minutes. Fig. 4 compares the transmittance of one such sample prior to and after annealing. Annealing is seen to 'reverse' the effect of solarization, i.e., the IR absorptance of the annealed eleven-year samples increases to a value slightly higher than that observed in the two-year-exposed samples. All three annealed samples exhibited similar optical densities at 1000 nm: an O.D. of 0.136, 0.142, and 0.142 were measured on samples 9A, 11A, and 13A, respectively. Thus, in the infra-red (Fe^{2+}) region of the spectrum, the annealed glasses probably (no virgin samples were available) resemble unexposed glasses. However, in the UV region there are annealing-induced changes which do not correspond to solarization effects (fig. 4 vs. fig. 1). The anneal-induced change in shape of the UV absorptance also introduces another complication: is the UV absorptance solely due to the presence of Fe^{3+} , or are there other contributions from impurities and/or the fundamental absorptance edge of the glass?

3.1.4. Fe^{3+} Measurements

The most obvious explanation of the observed photo-induced decrease in Fe^{2+} absorption involves the photo-oxidation of Fe^{2+} : $\text{Fe}^{2+} \xrightarrow{h\nu} \text{Fe}^{3+}$. If such a mechanism applies, a photo-induced decrease in Fe^{2+} concentration should be accompanied by a corresponding increase in Fe^{3+} concentration. Table 3 tabulates the Fe^{2+} , Fe^{3+} , and total iron concentrations in the two and eleven year solar-exposed heliostat glasses. Fe^{2+} concentrations were obtained from the Fe^{2+} absorption peak at 1000 nm ($\epsilon = 28.9 \text{ l/mole-cm}$) [4] and Fe^{3+} concentrations from the total absorptance at 380 nm ($\epsilon = 4.9 \text{ l/mole-cm}$) [5]. Total iron concentrations were determined by neutron activation analysis and are in good agreement with the sum of Fe^{2+} and Fe^{3+} concentrations. The optical data on solar-exposed glasses do not exhibit an increase in Fe^{3+} concentration. Because of possible ambiguities in interpreting the UV absorptance, it was

decided to supplement the optical measurements with electron spin resonance (ESR) measurements of the Fe^{3+} peak at $g = 30/7$. These measurements provide a measure of the relative Fe^{3+} concentrations in the two samples, i.e. $R = \frac{[\text{Fe}^{3+}]_{11 \text{ yrs}}}{[\text{Fe}^{3+}]_{2 \text{ yrs}}}$. Assuming that the above photo-oxidation kinetics apply, and using the Fe^{2+} and total iron concentrations of Table 3, a value of $R = 1.065$ is obtained. Extensive and repeated measurements of the optical absorption at 380 nm and of the $g = 30/7$ ESR peak yield values of $R = 1.00 \pm 0.01$, and 1.00 ± 0.02 respectively. Thus, within experimental uncertainty, two somewhat complementary probes fail to detect any photo-induced change in the Fe^{3+} concentration.

These measurements are difficult to reconcile with a photo-oxidation mechanism. One resolution to this dilemma is to note that the photo-induced Fe^{3+} could retain the local coordination of the Fe^{2+} (octahedral), and hence would not be characterized by the optical and ESR signatures normally associated with Fe^{3+} in silicate glasses. However, neither the optical nor the ESR spectra exhibit any new features ascribable to Fe^{3+} in a novel environment. Another possibility is that the observed photo-induced decrease in Fe^{2+} absorption results from a change in the Fe^{2+} extinction coefficient, rather than a decrease in the Fe^{2+} concentration. This mechanism would involve a photo-induced change in the local environment of the Fe^{2+} (e.g., the ionization of a nearby bond accompanied by atomic relaxations). Such rearrangements are known to affect the extinction coefficient: the Fe^{2+} extinction coefficient varies from ~ 1 to ~ 30 $\ell/\text{mole-cm}$ depending on the details of the local environment [6].

3.2 Solar Simulator Studies

The preceding section has shown that solarization does exist, and that for the particular case studied solarization has resulted in a significant improvement in the optical transmittance of the glass. However, the experiments of White and Silverman [7] suggest that the magnitude and sign

of solar-induced changes depend on the detailed chemistry of the glass, including the nature of minor additives ($\approx 0.1\text{wt}\%$) used as fining and oxidizing agents. Since the nature of solar-induced changes in optical properties cannot be predicted, we have examined the possibility of using accelerated solarization studies for screening potential heliostat glasses. Consequently, solar simulation studies were conducted on Odeillo glass exposed for two and eleven years, standard float glass, low-iron float glass, and Corning 0317 fusion glass. Optical spectra were taken prior to irradiation, and after 250, 500, 750, and 1000 hours of exposure to the solar simulator. The spectra of the irradiated samples were subtracted from those of the unirradiated samples to obtain "difference spectra." Representative difference spectra, after 500 hours of simulation time, are shown in fig. 5. All the samples, except the Odeillo glass exposed for eleven years (not shown), exhibited a photo-induced decrease in optical density for wavelengths greater than 800nm with a spectral shape similar to that of the Fe^{2+} absorption band. The magnitude of these changes after 500 hours of exposure is roughly twice that after 250 hours. However, difference spectra obtained after 750 and 1000 hours show slight, if any, increase over the 500 hour spectra, suggesting that a saturation in photo-induced changes in these glasses has occurred.

At this early stage, we cannot extract a quantitative relationship between exposure to sunlight and exposure to the solar simulator. However, the accelerated solarization of the Odeillo glass which had been exposed to sunlight for two years does produce effects of similar sign and magnitude to those produced by additional 'field exposure.' Quantification of this relationship awaits further studies of the observed saturation in photo-induced changes and the acquisition of data on local insolation and heliostat exposure at Odeillo, France.

Table 3. Determination of iron concentrations (wt% Fe₂O₃)

	Optical Analysis [a]		Neutron Activation Analysis
	[Fe ²⁺]	[Fe ³⁺]	Total iron
Heliostat #13 (exposed for 2 yrs)	0.015	0.086	0.090
Heliostat #6 (exposed for 11 yrs)	0.0094	0.086	0.090

a. Fe²⁺ concentrations were obtained from the Fe²⁺ absorption peak at 1000 nm ($\epsilon = 28.9 \text{ l/mole-cm}$) [4] and Fe³⁺ concentrations from the total absorption at 380 nm ($\epsilon = 4.9 \text{ l/mole-cm}$) [5].

4. Summary

In summary, we have observed a solar-induced decrease in Fe^{2+} absorption in heliostat glasses from the solar furnace at Odeillo, France. This decrease occurs throughout the sample (not just at the exposed surface), and is of sufficient magnitude to result in an increase of 2.5% in solar transmittance in a period of nine years. Simulation studies, employing an Oriel solar simulator, produced effects of similar magnitude and sign in a variety of glasses, and therefore appear to offer an attractive means for screening samples for solarization tendencies.

To aid in the formulation of a microscopic model, a number of additional measurements were performed on the glass samples exposed for two and eleven years. The Fe^{3+} concentration, as measured by conventional optical and ESR signatures, was found to be the same to within 1% in both kinds of samples. Furthermore, neither the optical nor ESR spectra showed any new or unknown features which might be ascribable to "solarized Fe^{2+} ". Profiles of the Fe^{2+} absorption revealed that solarization occurred throughout the sample, with the observed decrease being $\approx 40\%$ larger at the front surface than at the back surface. Simulation studies did not produce any additional changes in the eleven year exposed sample. Unfortunately, these measurements are not sufficient to deduce the actual solarization mechanism. They do, however, provide constraints on existing (e.g. $\text{Fe}^{2+} \xrightarrow{h\nu} \text{Fe}^{3+} + e^-$) and future models. Formulation of a realistic model must await the completion of additional experiments which are currently in progress.

Acknowledgments

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Figure Captions

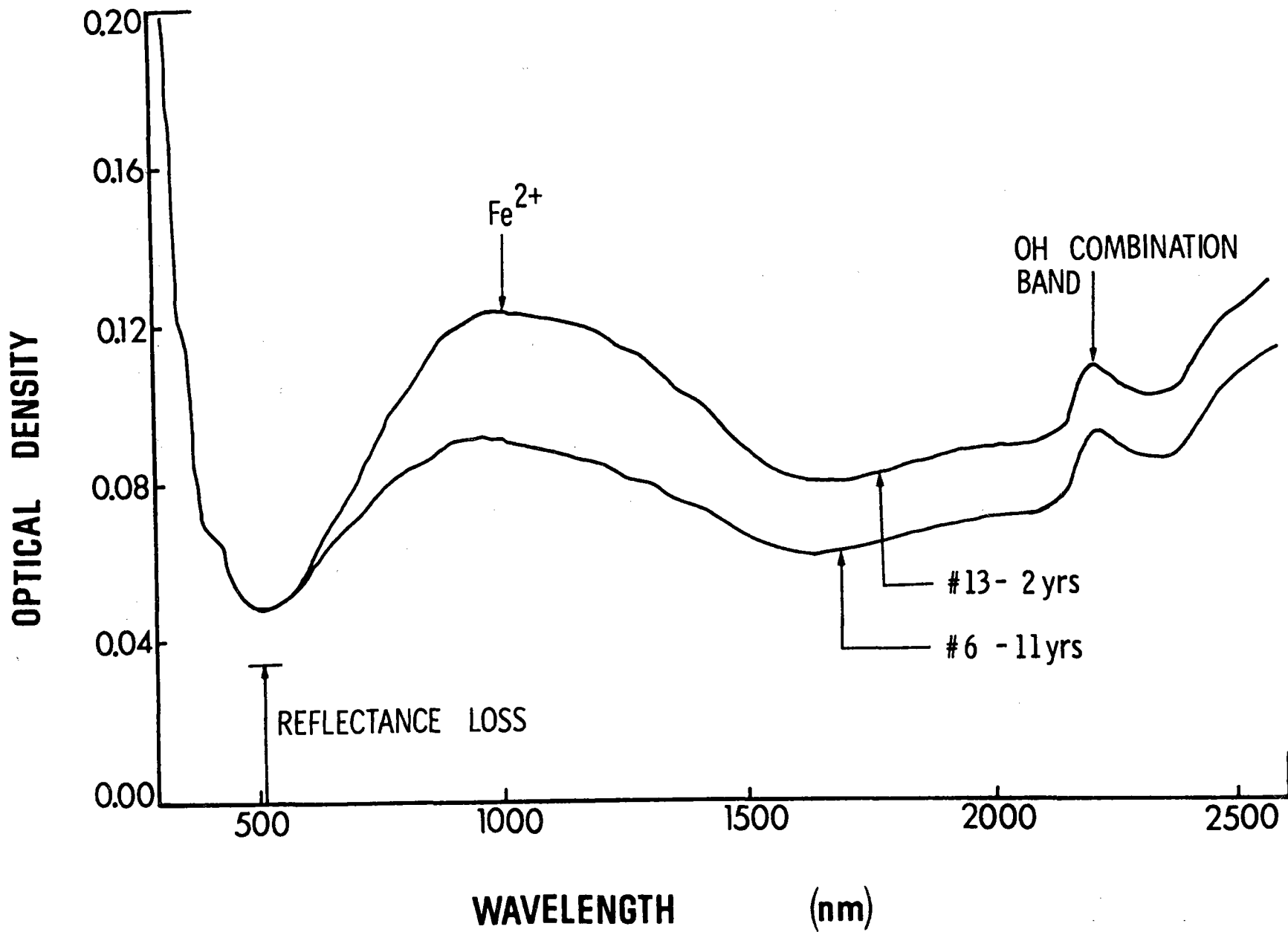
Fig. 1. Representative transmittance (a) and reflectance (b) spectra for the two and eleven year solar-exposed heliostat glass samples from the solar furnace at Odeillo, France.

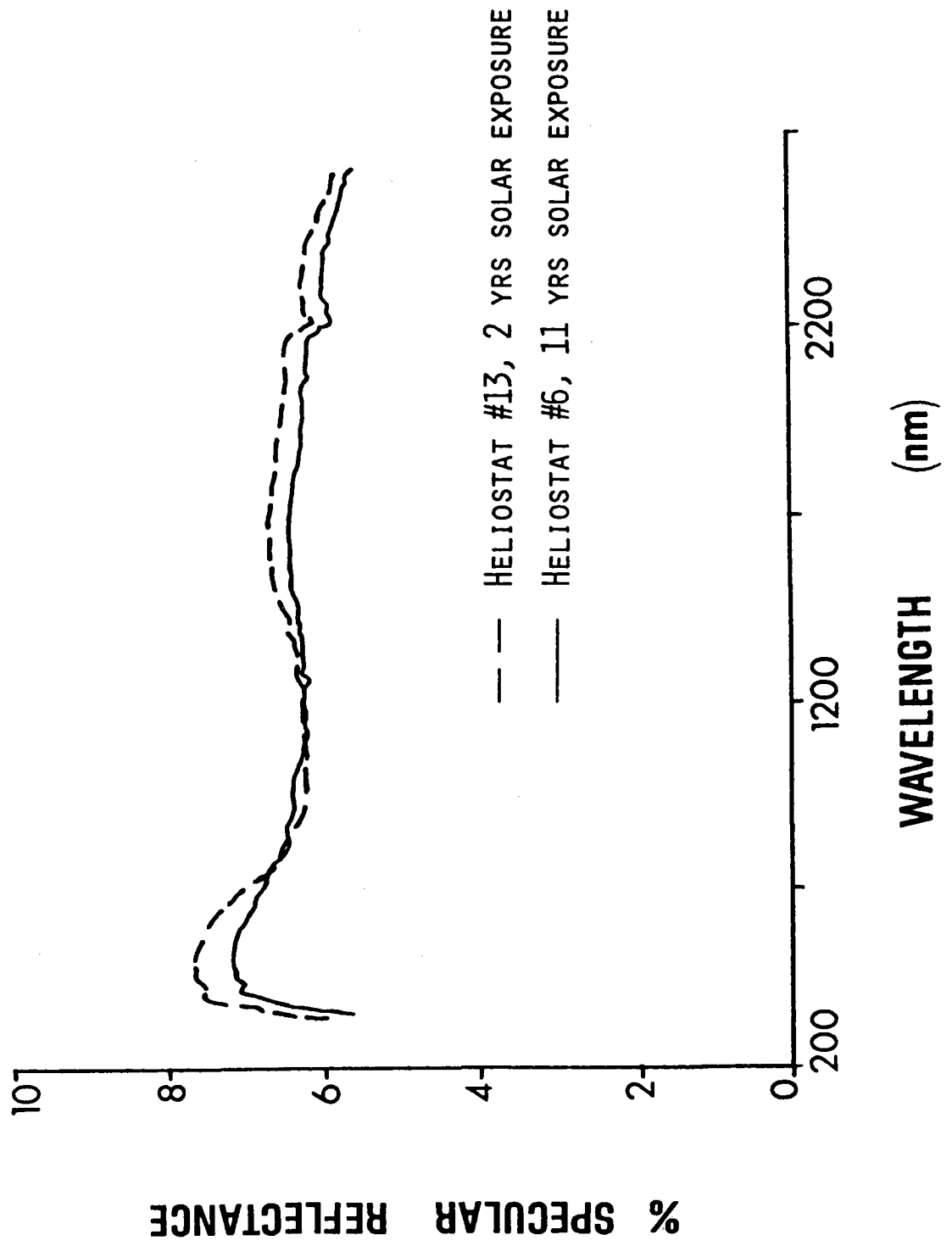
Fig. 2. The difference spectrum of the Odeillo glass (two year - eleven year solar-exposed) compared with the difference spectrum of 0.120" thick standard and low-iron float glass.

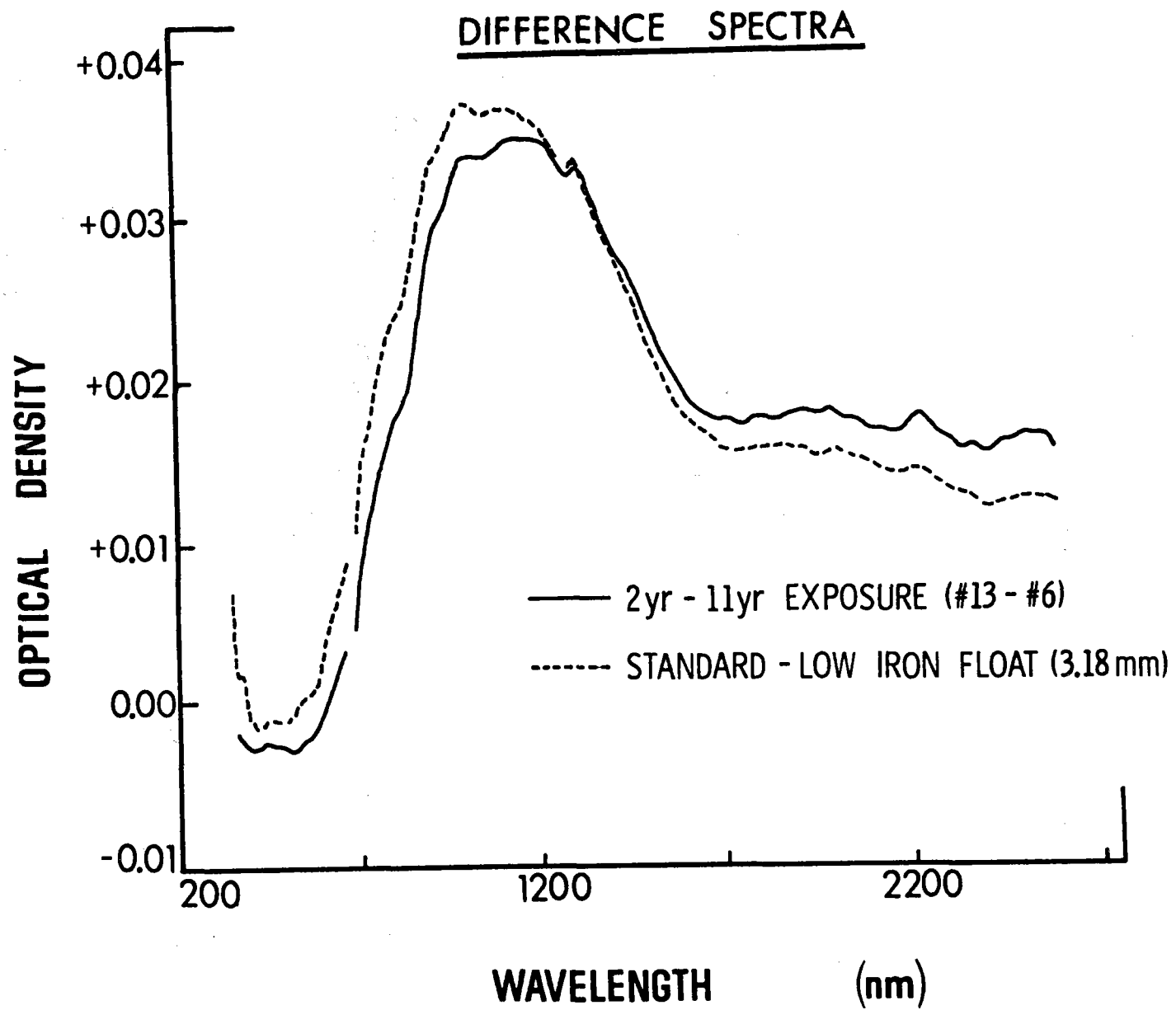
Fig. 3. The measured Fe^{2+} profiles for samples exposed to two and eleven years of solar irradiation (each sample also received 1000 hours of accelerated solarization). These measurements were made parallel to the exposed faces, had a spatial resolution of 0.5mm, and used the difference in optical densities at 1000 nm and 1700 nm, i.e. $\Delta\text{O.D.} = \text{O.D.} (1000 \text{ nm}) - \text{O.D.} (1700 \text{ nm})$, as a measure of the Fe^{2+} absorption. (Solid lines are a guide to the eye).

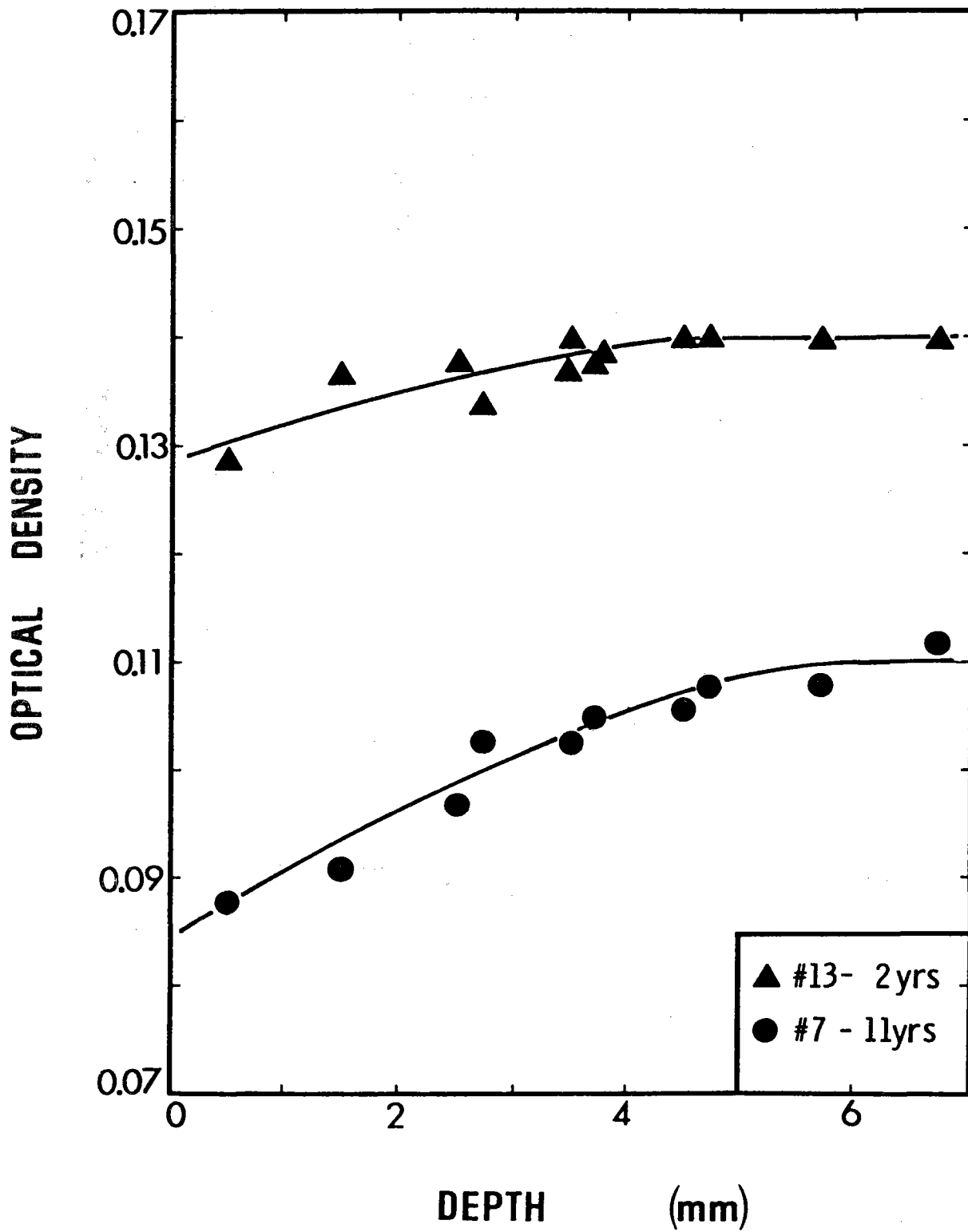
Fig. 4. The effect of annealing on the transmittance of a heliostat glass which had been exposed to eleven years of solar irradiation.

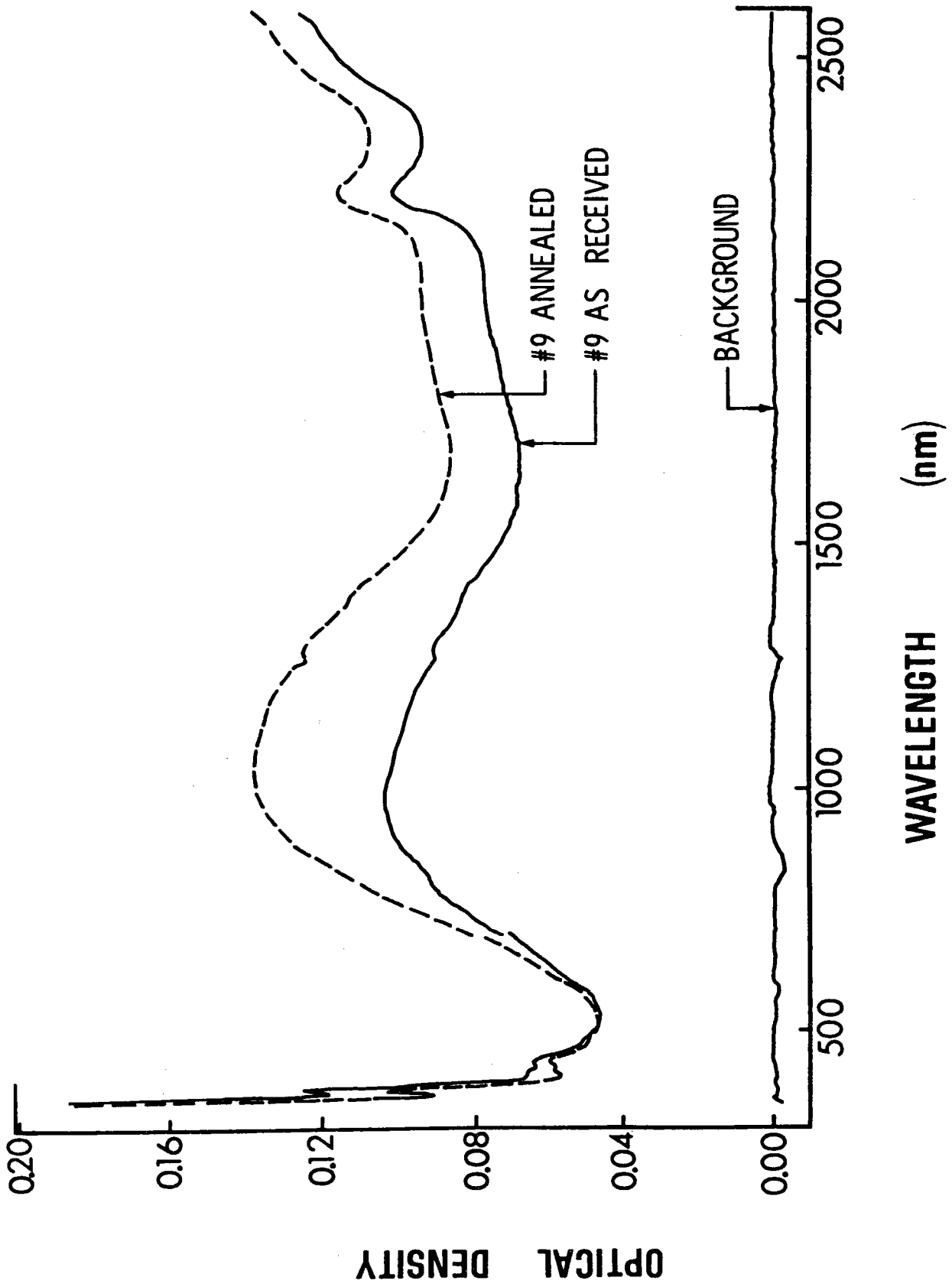
Fig. 5. Difference spectra of a variety of glass samples which had been exposed to 500 hours of accelerated solarization. The difference spectra are obtained by subtracting the spectra of the irradiated samples from those of the unirradiated samples.



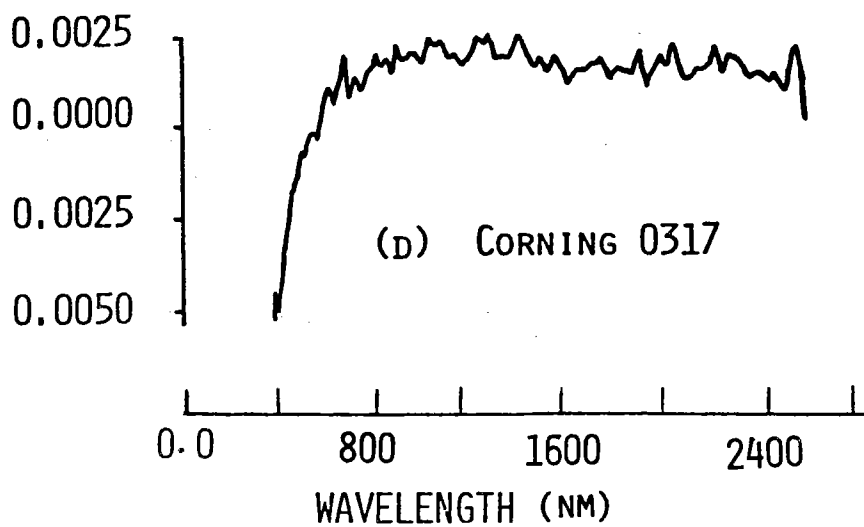
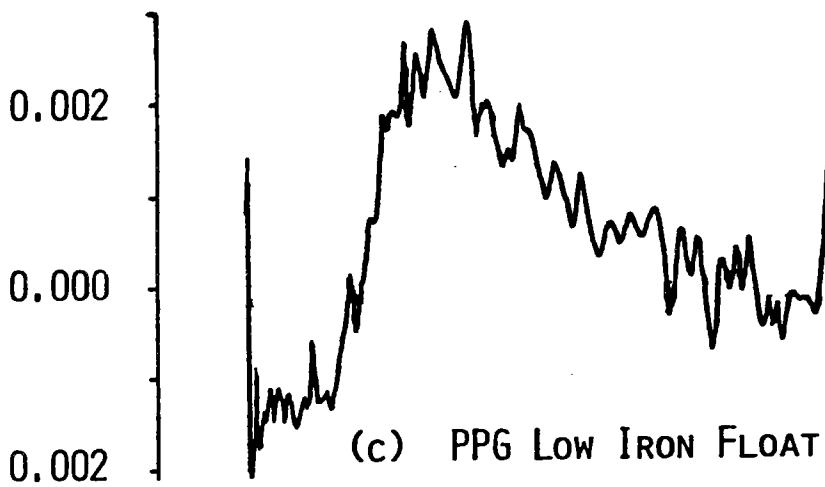
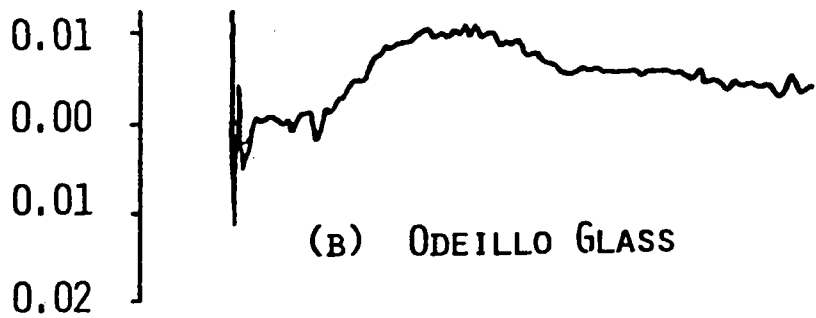
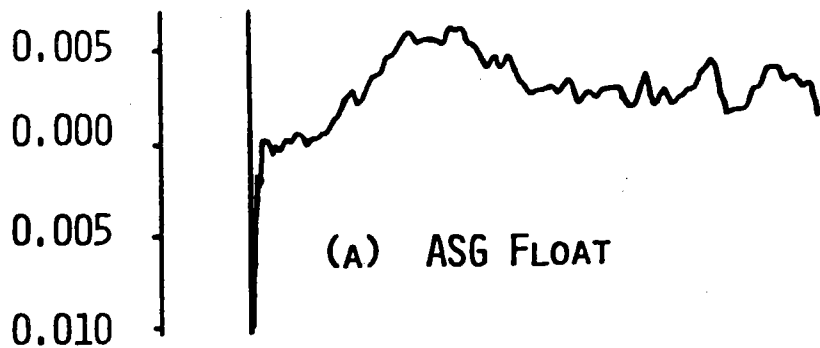








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