

Sol-Gel-Derived AR Coatings for Solar Receivers

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Abstract

This report details a process for applying sol-gel antireflection (AR) coatings to solar receiver envelopes. The process consists of applying a porous film which is subsequently etched to achieve the optimum AR effect. The result is a single-layer interference film with a reflectance minimum at 550 nm. The solar transmittance of coated tubes is typically increased to 0.95 - 0.97, as compared with 0.91 for uncoated tubes. Coated tubes showed no significant decrease in solar transmittance after 16 weeks of operation in a parabolic trough collector system. Recommendations are included for process improvement before industrial scale-up.

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Contents

Introduction	7
Sol-Gel Technology	7
Experimental—Process Optimization	8
Aging Time and Temperature	8
Heat Treatment	9
Etching Time/Temperature	9
Number of Coats/Coating Speed	10
Drying Conditions	11
Full-Scale Processing	11
Solution Preparation	11
Tube Preparation	12
Coating	12
Heat Treatment	13
AR Etch	13
Miscellaneous	14
Results/Discussion	14
Conclusions	15
Future Work	15
Process Improvements	16
References	16
APPENDIX A—Equipment List	17
APPENDIX B—Drawings	19
APPENDIX C—Procedure for F ⁻ Determination	27

Figures

1 Transmittance as a Function of Solution Age	9
2 Reflectance Spectra of Sol-Gel Films During Processing Compared With Uncoated Pyrex	10
3 Transmittance as a Function of Number of Coats Applied	10
4 Schematic of Solution Preparation	11
5 Sol-Gel Preparation Apparatus	12
6 Sol-Gel Coating Apparatus	13
7 Reflectance Spectra of Cycling Experiment Samples	15

Tables

1 Transmittance vs Heat Treatment	9
2 Coating Speed vs Thickness	11
3 Solar Average Transmittance Values	15

Sol-Gel-Derived AR Coatings for Solar Receivers

Introduction

Current design concepts for parabolic trough collectors used in modular line-focus solar systems use a receiver assembly that contains the heat transfer fluid. The receiver consists of a central, black, chrome-finished steel tube surrounded by a glass envelope that functions to minimize thermal losses.¹ At present, the glass envelopes are fabricated of Pyrex® (Corning 7740), which has a solar transmittance of 0.91. The operating efficiency of the collector can be improved by antireflecting the inner and outer surfaces of the envelope, thereby increasing the transmittance of the glass.

Several techniques are currently used to produce antireflection (AR) surfaces. The types of AR surfaces are single-layer and multilayer interference films, and graded refractive index films, each of which has inherent advantages and limitations. Single-layer interference films have reflectance minima that are wavelength specific whereas multilayer interference films exhibit low reflectance over a broad spectral range. Both types of interference films have critical thickness and refractive index requirements. Graded refractive index films, which are typically prepared by etching phase separated glasses, also exhibit low reflectance throughout a broad region of the spectrum. McCollister and Pettit² increased the solar transmittance of Pyrex receiver envelopes to >0.97 by using a phase separation process which resulted in a graded refractive index film. Their process used an extended heat treatment of the tubes near their softening temperature (to phase separate the glass) followed by selective acid etching to form the AR surface.

Brinker and Pettit³ developed an AR film which avoided some of the problems inherent to other AR surface techniques. Their goal was to form a coating, which was not compositionally limited (as are phase separation processes), at a temperature well below the deformation temperature of the glass. An additional objective was to avoid the need to apply a film of precise thickness. Their process used an aged, polymeric sol-gel solution (wt% composition: 71 SiO₂,

18 B₂O₃, 7 Al₂O₃, 4 BaO) which, after application, was heated at 500°C to form a durable, porous glass layer. The thickness and refractive index were then optimized by etching in a manner similar to McCollister and Pettit.² The result was a single-layer, quarter-wave, interference film with a solar transmittance of 0.97, which was not dependent on substrate composition or the original thickness of coating applied.

The purpose of this project was to develop a process, based on Brinker and Pettit's work, for applying sol-gel-derived AR coatings to full-scale (3.0-m long × 6.0-cm diameter) receiver envelopes. Our primary objective was to demonstrate the feasibility of large-scale application of sol-gel technology. Major considerations throughout the project were: (1) the coating should have acceptable AR properties, and (2) the process should be readily adaptable for use in industry.

Sol-Gel Technology

The sol-gel process for making glass uses low-cost, commercially available metal alkoxides as oxide glass precursors. Metal alkoxides (MOR, where M = Si, B, Ti, Al, etc; R = CH₃, C₂H₅, C₃H₇, etc), when mixed with water and a catalyst in an alcohol solution, undergo hydrolysis and condensation reactions to form a polymeric network.⁴ The polymer, which continues to cross-link until a gel is formed, expels its solvent and, upon heating, densifies to form a glass. The polymer shape, pore size, and growth can be controlled by H₂O content, pH, temperature, dilution, and aging to tailor the properties of the sol-gel for a specific application.^{5,6} This process has been used to make bulk glass, fibers, membranes, microspheres,^{7,8} and various types of coatings.

The advantages of sol-gel processing are numerous: potentially greater chemical homogeneity, higher purity, lower processing energy requirements, and

purity, lower processing energy requirements, and preparation of compositions difficult to make using conventional methods. One of the most promising uses of this technology is the application of thin glass coatings onto materials that cannot withstand high-temperature processing. The solution (sol) is deposited onto a substrate by dipping, spinning, or spraying, whereupon the solvent evaporates and gelation occurs. After drying, the porous gel coating is fired to form a dense, transparent glass layer which can function as a protective,⁹ dielectric,¹⁰ or antireflective coating. Several processes which use sol-gel technology for the formation of AR coatings have recently been reported. These include sol-gel coatings which are phase separated and etched to form an AR surface^{11,12} and sol-gel AR coatings for solar^{13,14} and laser applications.¹⁵ Our process, based on Brinker and Pettit's method for AR coatings on Pyrex, is the first large-scale application of sol-gel coating technology in the US.

Experimental—Process Optimization

More complete definition of the critical process parameters was required before optimizing the Brinker/Pettit procedure for large-scale use. The initial parameters of interest were solution aging time and temperature, heat treatment, etching conditions, number of coats, coating speed, and drying conditions. Preliminary coatings were applied to Pyrex panels (0.60-m long \times 6.0-cm wide), and initial processing decisions were based on spectral reflectance data. These measurements were made using a Beckman Model 5270 spectrophotometer over the wavelength range 300 – 2400 nm.

As the process was more clearly defined, the substrate size progressed from panels to 0.60-m (2-ft) sections of actual receiver envelopes, then to 1.5-m (5-ft)-long sections, and finally to full-size, 3.0-m (10-ft)-long receiver envelopes. Because of sample size and geometric limitations of the Beckman spectrophotometer, most decisions during scale-up of the process were based on measurements made using a portable solar reflectometer (Model SSR, Devices and Services Co, Dallas, TX). This instrument allowed solar-averaged transmittance (τ_s) values to be calculated for the curved tube surfaces using the correlation equation

$$\tau_s = 0.987 - \rho_s (D + S)$$

where $\rho_s (D + S)$ is the measured solar reflectance of the surface. Confidence limits of 95% were established which indicated that the reproducibility of a single measurement was ± 0.002 transmittance units (100% transmittance = 1.00 transmittance unit). These measurements are in agreement with transmittance values calculated from the Beckman spectrophotometer data to within ± 0.005 transmittance units. Some processing decisions, particularly those relating to drying and etching parameters, were based on visual observations; e.g., cloudy coatings were discarded, and the color of an acceptable AR coating after etching was dark blue-purple.

The thickness and index of sol-gel-derived films are fixed by solution composition, viscosity, and age; coating speed; and number of coats applied. Films were applied to silicon wafers, and thickness and refractive indices were determined using a Gaertner L119X research ellipsometer equipped with a 6328-Å wavelength He/Ne laser.

A method for monitoring solution stability with long-term use was required before full-scale processing. Viscosity measurements, which can detect continued polymer growth with solution aging, were selected as a method easily adaptable to industrial use. A Wells-Brookfield coneplate viscometer, Model RVT-D with a CP-40 spindle, was used to monitor viscosity as a function of solution age.

Aging Time and Temperature

The process parameter which has the greatest effect on final AR properties is the solution age. Coatings applied from unaged (freshly prepared) solutions exhibit no AR effect regardless of the etching treatment, whereas aged solutions produce coatings with progressively higher solar transmittance values.³ Thus, a minimum amount of polymer growth is apparently needed to achieve the thickness and refractive index required for AR film formation. Brinker and Pettit found that ~ 60 days of aging at room temperature was required to produce films with acceptable AR properties. We have since determined, for this composition, that the "window" for room-temperature aging extends to at least 6 months.

To make the full-scale process more acceptable for industrial use, the aging was accelerated by increasing the solution temperature to 50°C. Our objective was to find a minimum aging time which gave acceptable AR properties and to establish a "window" for 50°C aging. The data in Figure 1 show that the optimum AR effect is achieved after two weeks of aging. To allow for continued polymer growth during room-temperature

coating operations, the minimum aging time at 50°C was set at 6 days, compared with 60 days at room temperature. To minimize excessive aging, the coating solution was stored at 4°C when not in use.

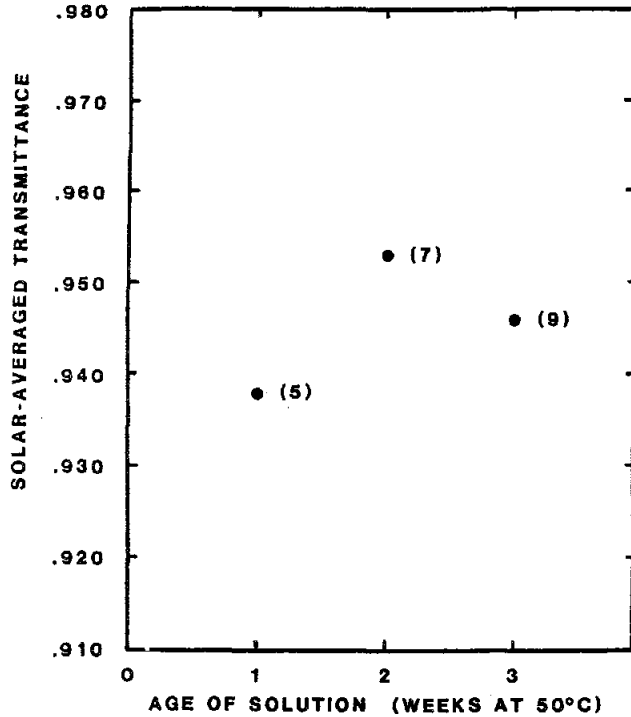


Figure 1. Transmittance as a Function of Solution Age. Single coats of solution aged 1, 2, or 3 wk at 50°C were applied at a coating speed of 12.7 cm/min. Samples were heated at 500°C before etching. The etching time (min) required for maximum solar-averaged transmittance is shown in parentheses.

Heat Treatment

Brinker/Pettit used a 500°C, 1000-min heat treatment to partially densify their AR coatings before etching. The heat treatment schedule for 3.0-m-long receiver envelopes was limited by the equipment available. To process tubes continuously rather than in a batch, a belt furnace was used (Appendix B, Dwg 1). A belt speed of 5 cm/min resulted in a total time of 12 min at 500°C. A comparison of transmittance as a function of etching time for triple-coated samples processed at 500°C for either 12 min or 1000 min is shown in Table 1. The results indicate that maximum transmittance occurs after 15 min of etching regardless of the heat-treatment time. The slightly higher

transmittance values for the 1000-min heat treatment were not considered large enough to justify the great increase in heat-treatment time.

Table 1. Transmittance vs Heat Treatment

Etch time (min)	Solar Transmittance	
	500°C 1000 min	500°C 12 min
0	0.942	0.932
5	0.953	0.958
15	0.970	0.964
20	0.963	0.964
30	0.959	coating removed

Etching Time/Temperature

After application, heated sol-gel coatings must be acid etched to reduce the thickness and refractive index for optimum AR film formation. A final coating, 1127- to 1230-Å thick, with a refractive index of 1.22, must be produced to satisfy the requirements for a quarter-wavelength interference film with a reflectance minimum at 550 – 600 nm.¹⁶ Mechanisms of etching for sol-gel-derived coatings have been previously discussed.⁶ An etchant containing H₂SiF₆/NH₄HF₂ has been used to produce AR films on Pyrex.^{2,3} In the present study, a concentration of 0.26N H₂SiF₆/0.015% NH₄HF₂ resulted in good AR film formation in 3 – 5 min at room temperature.

Figure 2 shows the progression of spectral reflectance changes as a sol-gel film is etched and heated. After initial heat treatment at 500°C, the unetched film exhibits a minimum reflectance of ~ 0.038 at 800 nm. Etching for 5 min decreases both the thickness and refractive index, resulting in a discreet reflectance minimum of only 0.012 at 625 nm. After etching, the film was heated a second time at 500°C in an attempt to remove residual water. The additional heating resulted in continued densification of the coating as evidenced by the shift in minimum reflectance from 625 nm to 550 nm. It was hoped that the increased densification would result in improved coating stability in the field.

Visual monitoring during etching was an effective quality control measure. As etching progressed, the color of the film when dried gradually changed from light blue to the dark blue-purple characteristic of a good AR film. Overetching resulted in a brown coating and eventually in complete film removal.

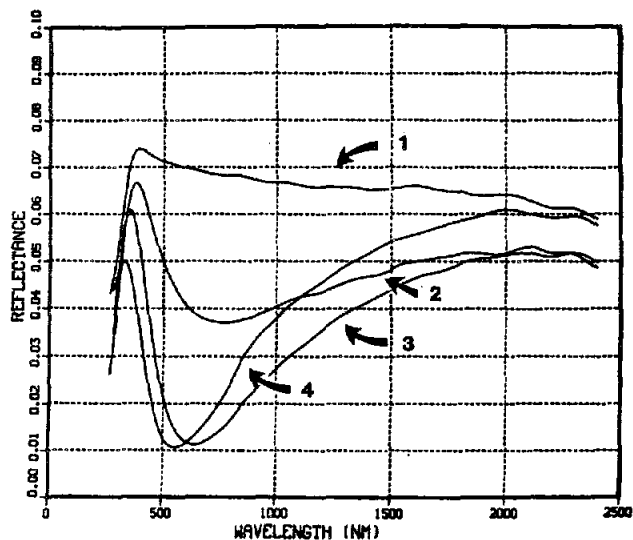


Figure 2. Reflectance Spectra of Sol-Gel Films During Processing Compared With Uncoated Pyrex. Shown are spectra of (1) uncoated Pyrex, (2) unetched film, initial heat 500°C, (3) after 5-min etch, and (4) after 500°C reheat.

Number of Coats/Coating Speed

Initial sol-gel films of this composition required multiple coats for optimum AR effect. Reducing the number of coats needed to produce acceptable AR films would have several advantages, including (1) fewer problems resulting from excessive handling (primarily breakage and surface contamination), and (2) shorter processing times, both resulting in lower cost. A study of coating parameters was initiated to evaluate the trade-offs involved. Figure 3 shows the transmittance of coatings prepared by the application of 1, 2, or 3 coats at a rate of 12.7 cm/min (5 in./min). The application of 2 or 3 coats results in excellent AR film formation after etching. A single coat, which would greatly simplify processing, was far below optimum transmittance, although better results would be expected if the thickness of the single coat was increased.

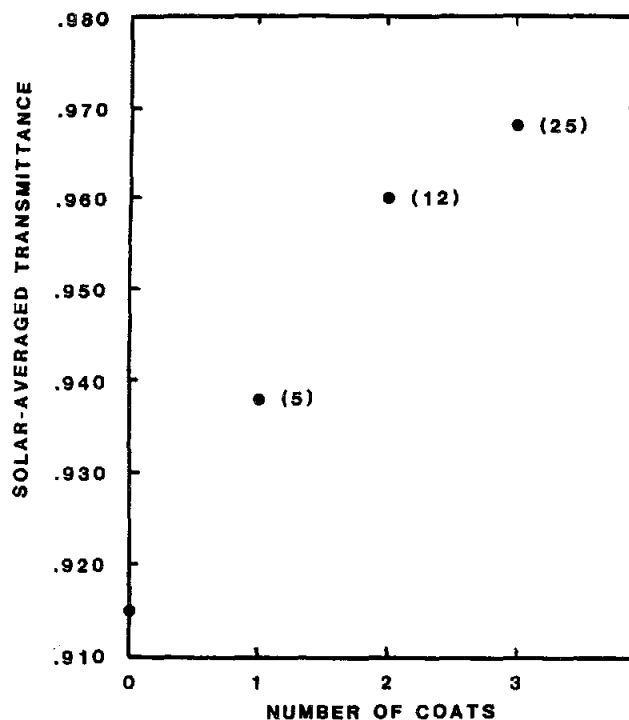


Figure 3. Transmittance as a Function of Number of Coats Applied. 1, 2, or 3 coats of solution aged for 1 wk at 50°C were applied at a coating speed of 12.7 cm/min and heated at 500°C before etching. The etching times required (min) for maximum solar-averaged transmittance are shown in parentheses.

When applying films by dipping, the thickness of the coating is dependent on the viscosity and coating speed; i.e., higher viscosities and/or faster coating speeds result in thicker films. Table 2 shows the results of ellipsometry measurements made along the length of a 7.6-cm sample for a single coat applied at various speeds. As expected, the thickness of the coatings increased as the coating speed increased. The sample coated at 12.7 cm/min was nonuniform, with a thickness <math><1180 \text{ \AA}</math> that is required for a quarter-wavelength film at 550 – 600 nm. The variation in thickness of this sample may be due to drying effects.

The most uniform coating was obtained at a coating speed of 25.4 cm/min (10 in./min). In addition, the thickness of this film was greater than that required for a quarter-wavelength film and would not require excessive etching to achieve optimum thickness. The 38.1 cm/min (15 in./min) results suggest, as has been noted by Sakka,⁷ that there is an upper limit to uniform single-coat thickness. The coating becomes progressively thicker along the length of the sample, possibly indicating "sagging" of the film.

Table 2. Coating Speed vs Thickness

Coating Speed (cm/min)	Thickness (Å)		
	Top	Middle	Bottom
12.7	822 ± 3	823 ± 2	968 ± 1
25.4	1375 ± 2	1357 ± 2	1382 ± 6
38.1	1880 ± 4	1969 ± 2	2149 ± 4

Based on these results, in addition to the aging studies, a single coat of a one-week-old solution applied at 25.4 cm/min was chosen for the large-scale processing. Transmittance values of 0.965 for a test coating applied in this manner indicate that excellent AR properties can be obtained with considerable simplification of the process.

Drying Conditions

As noted in the previous section, samples coated at slow rates may exhibit nonuniformity as a result of differential drying. Increased coating rates resulted in greater uniformity, but as the sample size increased, coatings became increasingly opaque (cloudy), with poor AR properties. A study of experimental variables indicated a correlation between seasonal variations in ambient relative humidity and coating quality. Samples coated in ~20% RH were clear, whereas samples coated at 60% RH were opaque. To standardize drying conditions, subsequent samples were coated and dried in flowing dry nitrogen (N₂), resulting in clear, uniform coatings.

Full-Scale Processing

Solution Preparation

The sol-gel coating solution of the oxide composition (wt%) 71 SiO₂, 18 B₂O₃, 7 Al₂O₃, 4 BaO was used in this process. A schematic representation of the preparation method is shown in Figure 4.

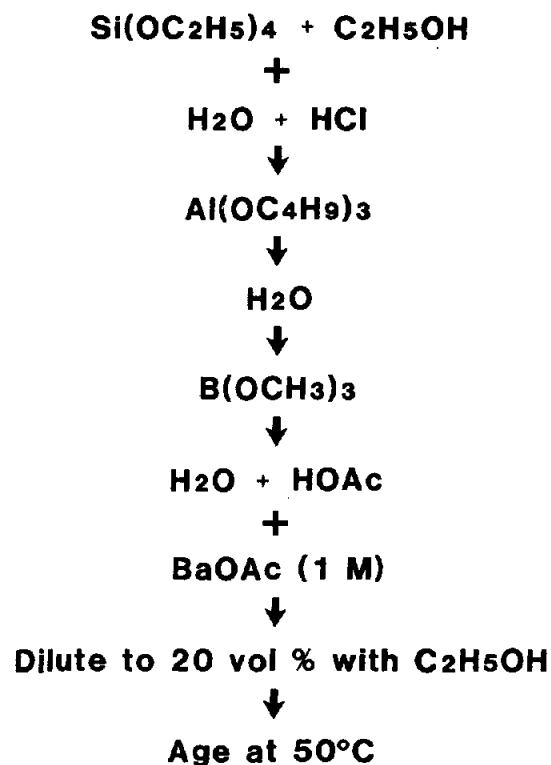


Figure 4. Schematic of Solution Preparation

Theoretical aspects of sol-gel preparation have been previously discussed.¹⁶ Details of the solution preparation are as follows:

1. In a reaction flask equipped with a stirring mechanism and water-cooled condenser (Figure 5) mix:
 - 1220 ml ethanol (absolute)
 - 1220 ml tetraethoxysilane (TEOS)
 - 4 ml 1M HCl
 - 48 ml deionized water
 Stir 1.5 h, 60°C, cool to 40°C.

2. Dissolve 174 g aluminum-sec-butoxide in 180 ml isopropyl alcohol, add to TEOS/alcohol mixture. Stir 10 min, 40°C; add 44 ml deionized water. Stir 10 min, 40°C.
3. Add 269 ml trimethylborate. Stir 1 h, 40°C. Cool to 25°C.
4. At 25°C, add in sequence:
 - 258 ml H₂O
 - 80 ml concentrated glacial acetic acid
 - 258 ml H₂O
 - 108 ml 1M barium acetate
 Stir 15 min, 25°C.
5. Dilute 1 part sol-gel with 4 parts absolute ethanol. Care should be taken to avoid exposure of the undiluted solution to air. The final volume of diluted solution is ~20 L and contains ~2.9 wt% oxides.
6. Age diluted solution at 50°C for a minimum of 6 days to grow polymer to correct size for AR coating.

After aging, the solution should be stored in a freezer approved for flammable materials to minimize overaging.

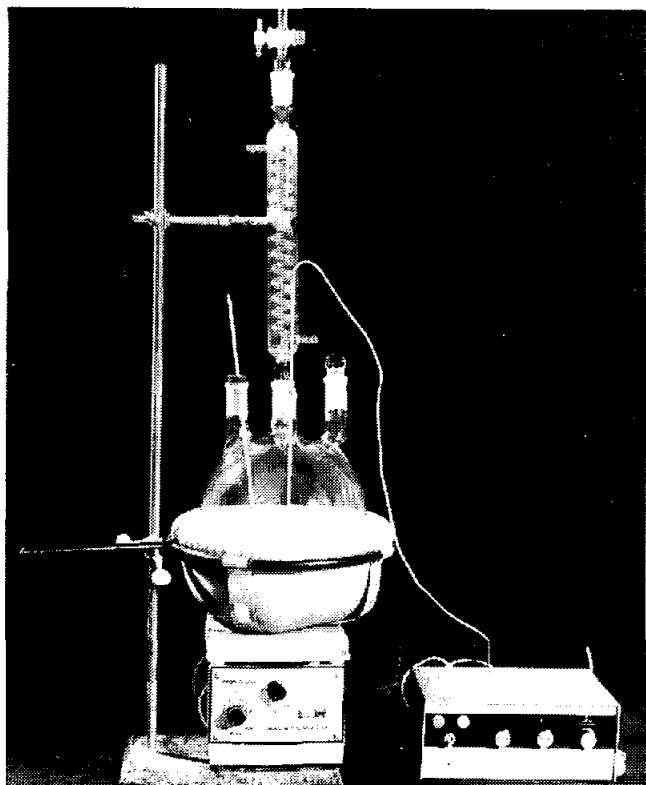


Figure 5. Sol-Gel Preparation Apparatus

Tube Preparation

The Pyrex tubes were received in 3.7-m (12-ft) lengths and were cut to 3.0 m (10 ft) with a diamond saw. Since the tubes are coated vertically, a 6.48- to 6.73-cm flare was fabricated into one end to fit into the top guide and holding fixture (Dwg 2d). The flared tubes were washed in a cleaning solution consisting of 7.1 g Alconox® and 6.4 g trisodium phosphate per liter and rinsed in filtered tap water. The remaining cleaning steps, as well as pre-etching and AR etching, were performed in an acid tank assembly consisting of five individual 4.2-m-long × 15-cm-ID PVC troughs (See Appendix A). The tanks were equipped with immersion heaters and pumps to provide temperatures up to 60°C and to flow rinse water continuously down the tubes.² After a deionized water rinse, the tubes were pre-etched for 5 min in 10% NH₄HF₂ at room temperature. It has been suggested that the pre-etch bath removes a thin surface layer of glass which, as a result of high manufacturing temperatures, may be a slightly different composition than Pyrex.² After pre-etching, the tubes were rinsed in deionized water at 60°C to remove residual fluoride ion (F⁻) and “squeegeed” inside and out to remove particulates (see Dwg 2a,e). The 60°C rinse/squeegee step was repeated a second time and was critical in producing a dry, streak-free surface.

Coating

After cleaning, the tubes were coated immediately to avoid surface recontamination. The critical coating parameters are particulate control in the coating solution, solution temperature during coating, coating speed, and drying conditions. The coating apparatus (Figure 6; Dwg 3 and 4) consists of a 3.3-m-long × 7.6-cm-ID PVC pipe equipped with four inlets at the top to supply dry N₂ gas, which provides a controlled drying atmosphere. The coating solution was pumped from a 20-L glass reservoir into and out of the coating tank and back by an air-drive, positive-displacement pump (see Appendix A). The pump produced constant coating speeds since the pumping speed was relatively insensitive to the height of solution in the tank. An additional advantage of a nonelectric pump is the elimination of the explosion hazard associated with alcohol solvents.

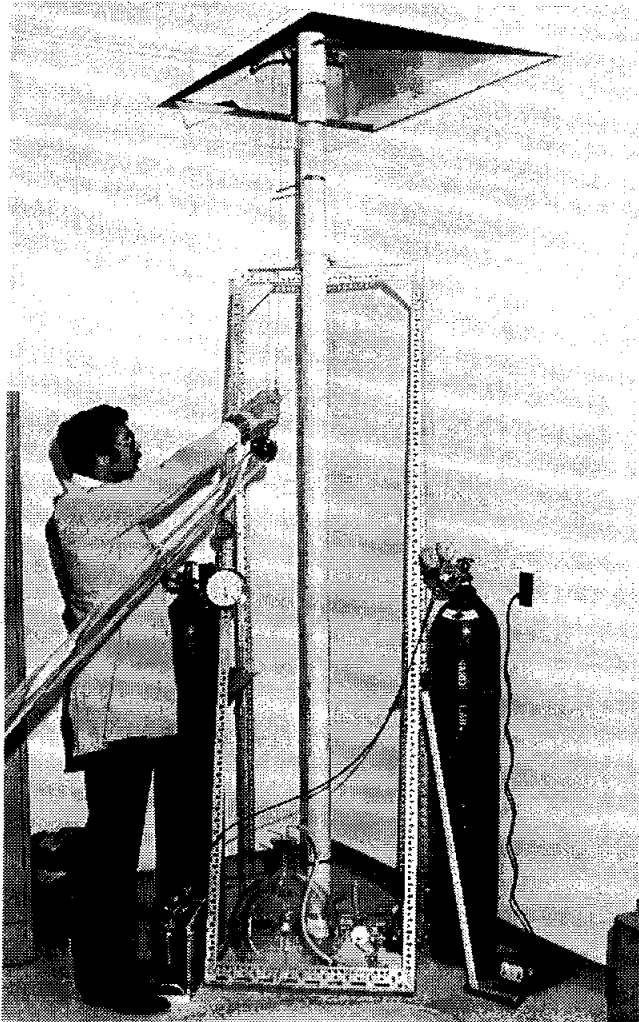


Figure 6. Sol-Gel Coating Apparatus

Each time the tank was filled, particulates were removed from the sol using a 200-mesh nylon filter in an in-line teflon filter holder. Valves at the bottom of the tank isolated the tank from the pump and allowed venting of the tank after coating to aid in drying.

After installation of the centering/holding fixtures (Dwg 2c,d), the tube was attached to a wench cable, raised into a tower (Dwg 4) in the second story of the coating facility, and lowered into the filled coating tank. The top fixture rested on a ledge inside the top of the tank to prevent swaying of the tube during coating. Coating and drying were performed in a dry N_2 atmosphere, supplied at an outlet pressure of 3 psi. A restrictor plate on top of the tank maintained a slight positive pressure of N_2 during coating.

The sol was withdrawn from the tank at a rate of 25.4 cm/min, requiring 12 min to coat the tube. After coating, a vent at the bottom of the tube was opened, a stopper was placed in the top of the tank, and the tube was dried for 15 min in flowing N_2 . Initial results indicated that the inside of the tubes were dry and blue-purple, indicating good AR properties while the outside remained quite wet. This differential drying was the result of an unequal flow of N_2 between the inside and outside of the tube. The only N_2 flow to the outside was through a 0.8-cm space between the tube and the inside wall of the tank, which was even further restricted by the top holding fixture. To promote more uniform drying, a flow restrictor (Dwg 2b) was placed in the top of the tube before coating in order to force more gas along the outside of the tube. Tubes coated with the restrictor in place appeared very uniform in color, indicating equal drying on both the inner and outer surfaces. After drying, the coated tubes were removed to a drying rack (Dwg 5) and dried under infrared lamps at $\sim 70^\circ C$ for 15 min to remove residual solvent.

Because sample tubes which were coated in low temperature solutions ($4 - 20^\circ C$) were nonuniform, with poor AR properties, the temperature of the sol was monitored continuously during coating and maintained at $23 - 25^\circ C$. Solution viscosity was measured each day during coating and ranged from 2 - 3 cP over ~ 3 mo. A single batch of solution was used to coat ~ 30 tubes in this 3-mo period.

Heat Treatment

Tubes were heated in a conveyor furnace modified with entrance and exit ramps as shown in Dwg 1. The ends of the tube were placed on stainless steel carts to avoid contact with the conveyor belt. Total heat-treatment time for each 3.0-m tube was 2 h. At this point, the process could be interrupted for overnight or weekend shut-down. However, as a precautionary measure, the tubes were stored in a low-humidity atmosphere to avoid adverse effects on the unetched coatings.

AR Etch

After heat treatment at $500^\circ C$, the sol-gel film is a partially dense, glass layer with continuous porosity which must be etched to maximize its AR properties.

The critical etching parameters are etchant concentration and temperature, etching time, and temperature of the water rinse. Forty liters of etchant were prepared as follows: 200 ml H_2SiF_6 , 6 g NH_4HF_2 in 39.8 L deionized water. The construction of the acid tanks did not permit storage of the etchant overnight nor was it practical to retrieve it from the acid tanks; thus, the etchant was discarded through a neutralizing tank after each day's use (generally 3 tubes were etched each day).

Each tube was immersed in the room-temperature etchant for the required time and then rinsed in deionized water. Initially, the temperature of the rinse was 60°C to accelerate drying; however, tubes rinsed in this manner were consistently overetched. The elevated temperature of the rinse may have caused accelerated localized etching within the pores before dilution. Subsequent tubes, which were rinsed at room temperature, were not overetched but required forced drying. These tubes were dried vertically in a stream of warmed N_2 flowing down the center of the tube. The N_2 , heated by a gas dryer (Appendix A), promoted evaporation of water from the tubes, leaving a dry surface free of streaks and water spots.

Assessment of the AR properties was made both visually and by measured reflectance. If the etched tube was not blue-purple, with a solar transmittance >0.95 , the etching process was repeated for 1-min intervals to optimize the AR effect. Films sequentially etched in this manner required slightly more total etching to achieve the same increase in transmittance than was required for films etched continuously. When satisfactory AR properties were achieved, the tubes were reheated at 500°C , as discussed earlier.

Miscellaneous

The parabolic trough collectors required tubes 2.95-m (116.125-in.) in length. The holding flare and an adjoining section of tube, totaling 9.8-cm in length, were removed by using a hot wire cutter (Dwg 6) powered by a variable transformer. The tube was scribed with a glass knife, thermally stressed with the hot wire cutter, and quenched with water to propagate a fracture along the scribe mark. The cut end was

beveled slightly with a diamond file to facilitate attachment of the collector hardware. Reflectance measurements were made at 1-ft intervals (9 measurements) along each tube to establish baseline properties. Coated tubes were stored in an airtight box under dry gas ($\sim 20\%$ RH) before installation in the collectors.

Results/Discussion

Twelve receiver envelopes were processed for installation in parabolic trough collectors in one of four Modular Industrial Solar Retrofit (MISR) test systems located at Sandia National Laboratories. After processing, the solar averaged transmittance of the tubes ranged from 0.956 to 0.968 and varied from ± 0.002 to ± 0.006 transmittance units from end to end. Five segments were cut at 2-ft intervals along the length of a representative tube to assess circumferential uniformity. Multiple measurements around each segment indicated a variation of ± 0.003 to ± 0.010 transmittance units. Visual monitoring of AR surface color suggested that there was very little difference between the inner and outer tube surfaces. This observation was extremely difficult to confirm by optical measurements because of the concave inner surface; however, a single measurement on the Beckman spectrophotometer indicated no difference in reflectance between the two surfaces.

After processing, a gradual increase in the reflectance of the sol-gel AR film was noted, indicating a loss in the AR effect. This loss was attributed to adsorption of water onto the high-surface-area, porous surface. Coated samples were exposed to minimum projected solar trough cycling conditions to determine whether the AR loss was reversible. A single cycle consisted of 12 h at 150°C followed by 12 h of high humidity ($>90\%$ RH) at ambient temperature. As shown in Figure 7, an etched and heated sample which was exposed to ambient laboratory conditions for only 14 days exhibited significant AR loss (increase in reflectance). Total AR recovery of the sample occurred after 3 cycles, and the recovery was maintained for at least 14 cycles. It is expected that only a few minutes at 150°C is required for complete recovery; however, this has not been verified.

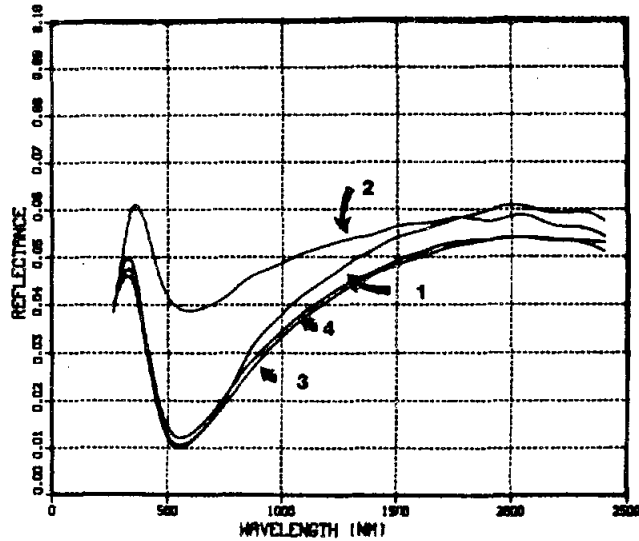


Figure 7. Reflectance Spectra of Cycling Experiment Samples. Spectra of the same sol-gel film are shown (1) after 5 min etch, 500°C heat; (2) after 14 days under ambient conditions; (3) after 3 cycles at 150°C; and (4) after 14 cycles at 150°C.

These results indicate that although the adsorption of water from the atmosphere has an adverse effect on the quality of the AR film, the original AR properties of the film can be restored by exposure to projected solar trough operating conditions. The experimental design was based on conservative estimates of actual collector temperatures. In practice, however, depending on the heat transfer fluid used, operating temperatures may be significantly higher. It was felt that higher temperatures would accelerate the recovery process and have a positive effect on AR coating stability.

Twelve envelopes were installed in the MISR test system in October 1983. After 16 wk of operation in the collectors, reflectance measurements (9 measurements/tube) were made on nine of the tubes and compared with original baseline data. Table 3 shows the transmittance values for these tubes in addition to two tubes that were processed at the same time but stored under ambient laboratory conditions. In most cases, the change in transmittance after 16 wk in the collector was within the replicability for the portable reflectometer (± 0.002). In contrast, the two tubes stored under ambient conditions with no cycling showed a definite decrease in the AR effect. These data confirm the results of the cycling experiment; i.e., the high transmittance values are maintained on tubes that are thermally cycled.

Table 3. Solar Average Transmittance Values

Tube (MISR)	After Processing	After 16 wk	Change
1c	0.963	0.961	-0.002
2c	0.968	0.966	-0.002
3	0.961	0.963	+0.002
4b	0.962	0.966	+0.004
5	0.964	0.960	-0.004
11a	0.966	0.961	-0.005
12a	0.962	0.963	+0.001
14	0.965	0.964	-0.001
18	0.957	0.958	+0.001
13a	0.960	*	
15	0.956	*	
16	0.958	*	
7 (Ambient)	0.962	0.946	-0.016
10 (Ambient)	0.956	0.947	-0.009

* tubes still at MISR

Conclusions

Sol-gel-derived coatings prepared from aged, polymeric solutions were deposited on Pyrex trough collector envelopes, heated to form films with continuous porosity, and etched to maximize the AR properties. The resultant film meets the criteria for a quarter-wavelength, interference AR film with a reflectance minimum at 550 nm. The film has a solar transmittance of 0.95 – 0.97, as compared with 0.91 for uncoated Pyrex, and appears to be quite uniform and reproducible. Coated tubes showed no significant decrease in solar transmittance after 16 wk of operation in a parabolic trough collector system. The process could be easily modified for use in industry.

Future Work

A closely related application of particular interest to the solar industry is the use of refractory sol-gel AR coatings on dish collector apertures. These apertures, which will be fabricated of fused silica, may see final operating temperatures as high as 1200°C. Sol-gel processing is desirable because fused silica cannot be treated by phase separation or neutral solution processing techniques.¹⁵ A result of this project is an

improved understanding of the process variables involved in sol-gel coating techniques that can be directly applied to this and other uses of sol-gel technology.

Process Improvements

- A closer look at heat treatments is warranted: (a) Table 1 suggests that although there is little difference in the maximum transmittance of tubes heated for 12 min or 1000 min, the former may be less durable (note 30-min data), and (b) determine whether indeed post-AR etch heat treatment affects long-term coating stability.
- Develop a method to establish the useful lifetime of the etching solution based on either (a) the quality of AR produced as a function of time, or (b) measurement of F^- concentration and establishment of concentration limits (see Appendix C).
- Improve tank design by using stainless steel tanks for hot water and modify the acid troughs to prevent leakage and inefficient use of acid.
- Redesign the tube-holding fixture to permit elimination of the flare. This would allow the tubes to be recycled.
- Design a jacketed solution reservoir that would allow tighter control of solution temperature during coating and eliminate the need to store the solution in the refrigerator when not in use.
- Optimization of drying conditions would simplify the process and allow tighter control of etching parameters.
- Determine minimal heat-treatment time and temperature for AR recovery after water adsorption.

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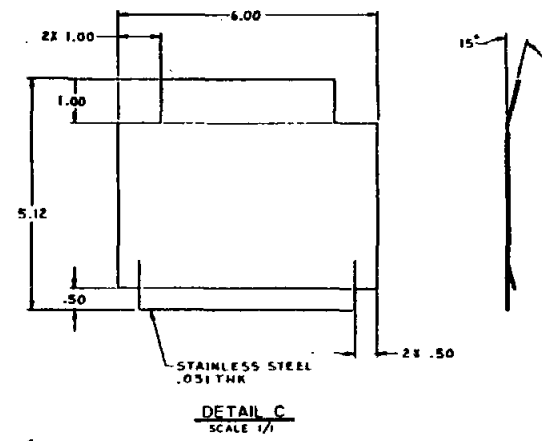
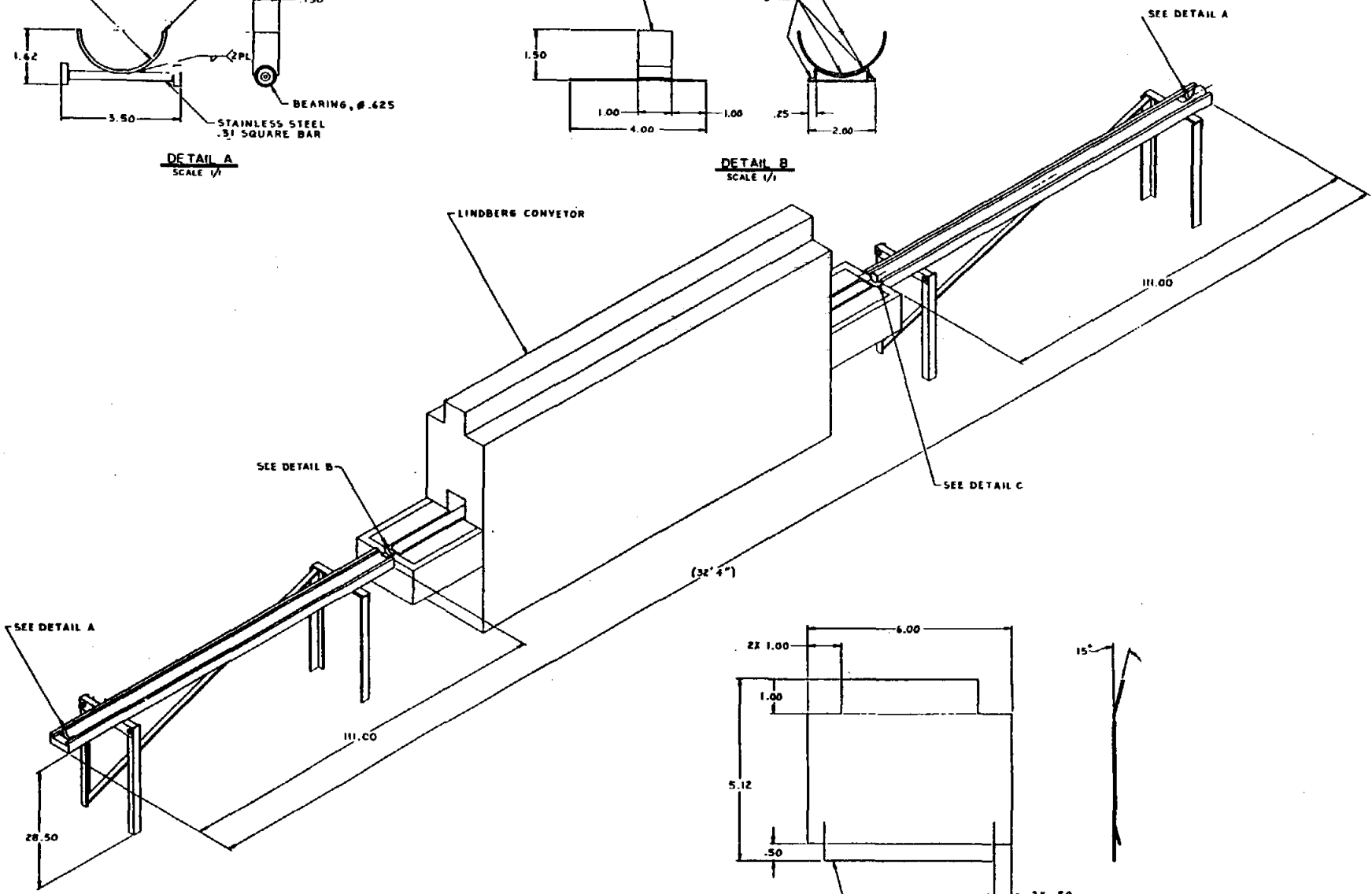
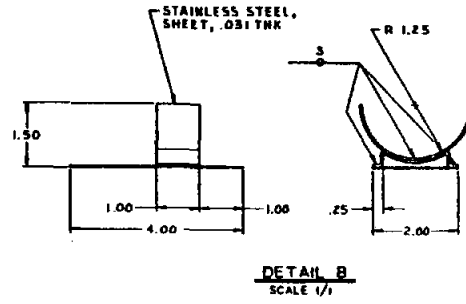
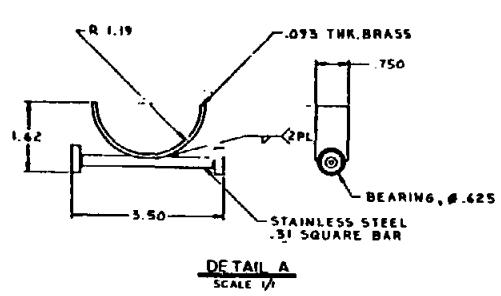
APPENDIX A
Equipment List

Commercial equipment is listed to describe adequately the procedures and processes used. This does not imply endorsement by Sandia National Laboratories nor does it indicate that these items are necessarily the best available for the described purpose.

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2. Explosion proof freezer
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Model 3552
3. Temperature controller
Yellow Springs Instrument Co., Inc.
Scientific Division
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Model 71A
4. Viscometer
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Model RVTD, coneplate digital viscometer
5. Air-drive pump
Gast Manufacturing Corp.
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Model 120-000-110
6. Belt furnace
Lindberg, A unit of General Signal
304 Hart St.
Watertown, WI 53094
Model 47-MT
7. Portable reflectometer
Devices and Services Co.
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Model SSR
8. Acid tanks
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prints available upon request.
9. Serpentine gas/air heater
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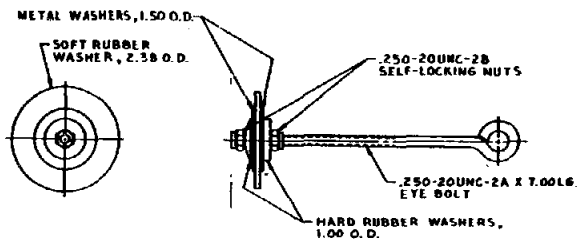
APPENDIX B

Drawings



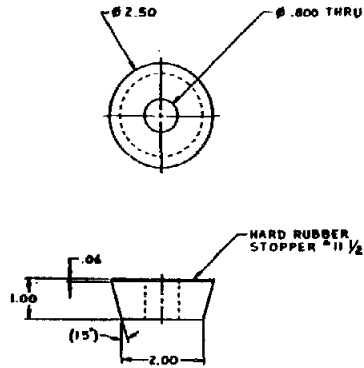
Drawing 1

1A							
UNCLASSIFIED							
F14213 545632							



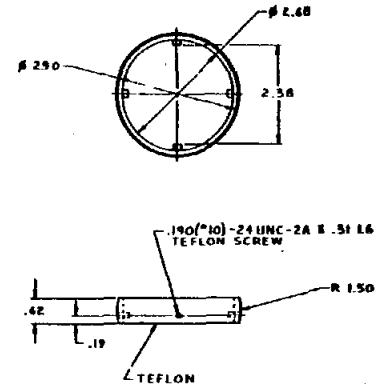
I.D. SQUEEGEE

A



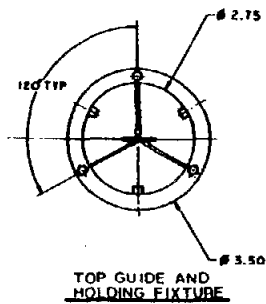
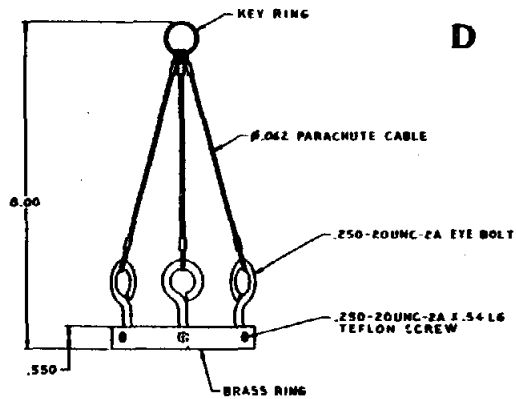
DRYING RESTICTOR

B



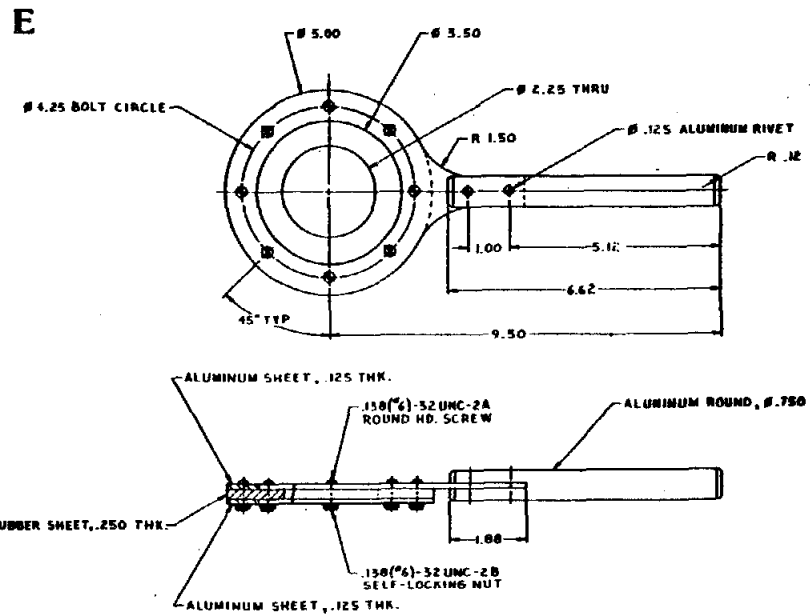
BOTTOM CENTERING FIXTURE

C



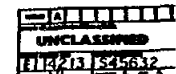
TOP GUIDE AND MOLDING FIXTURE

D



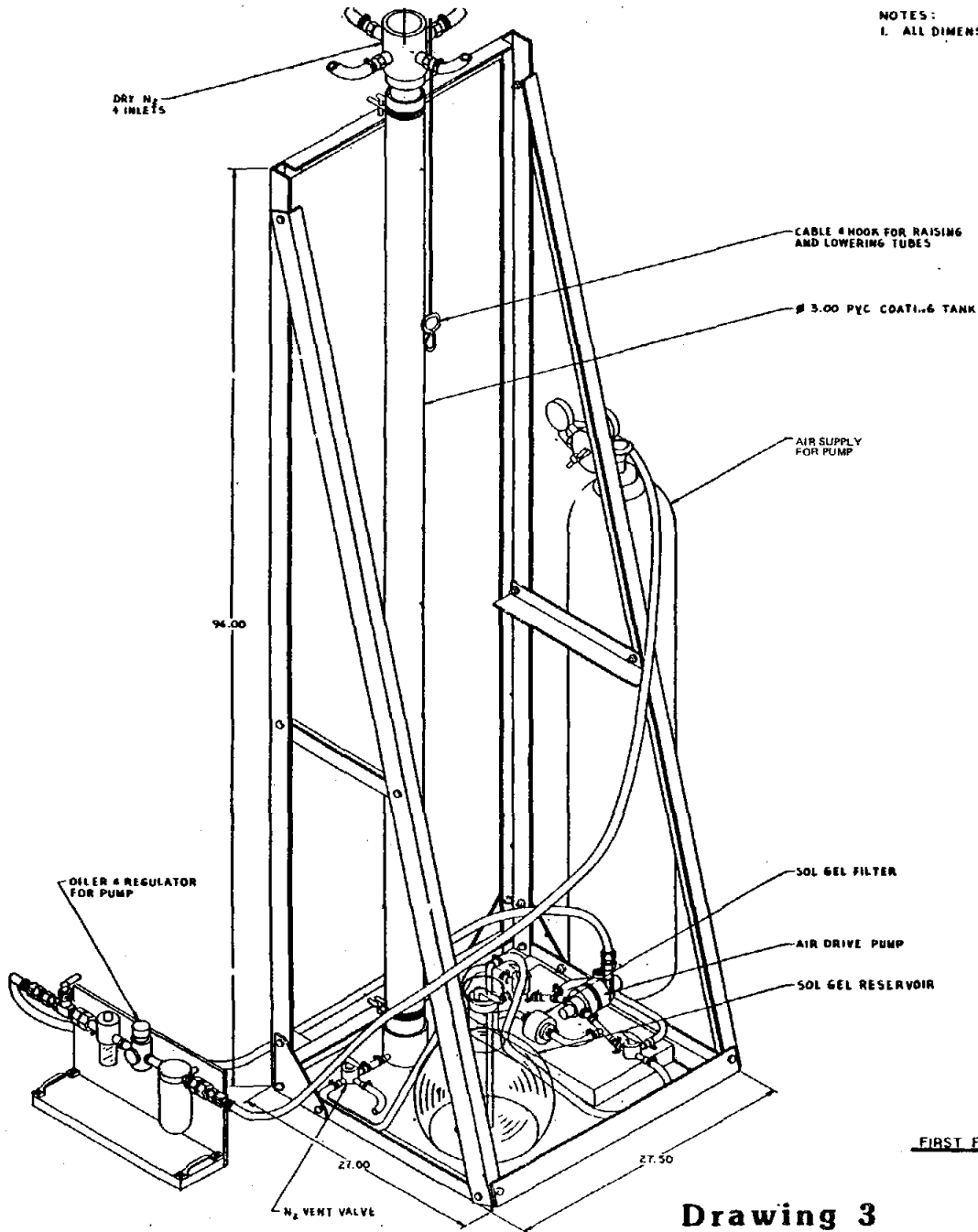
Drawing 2

O.D. SQUEEGEE



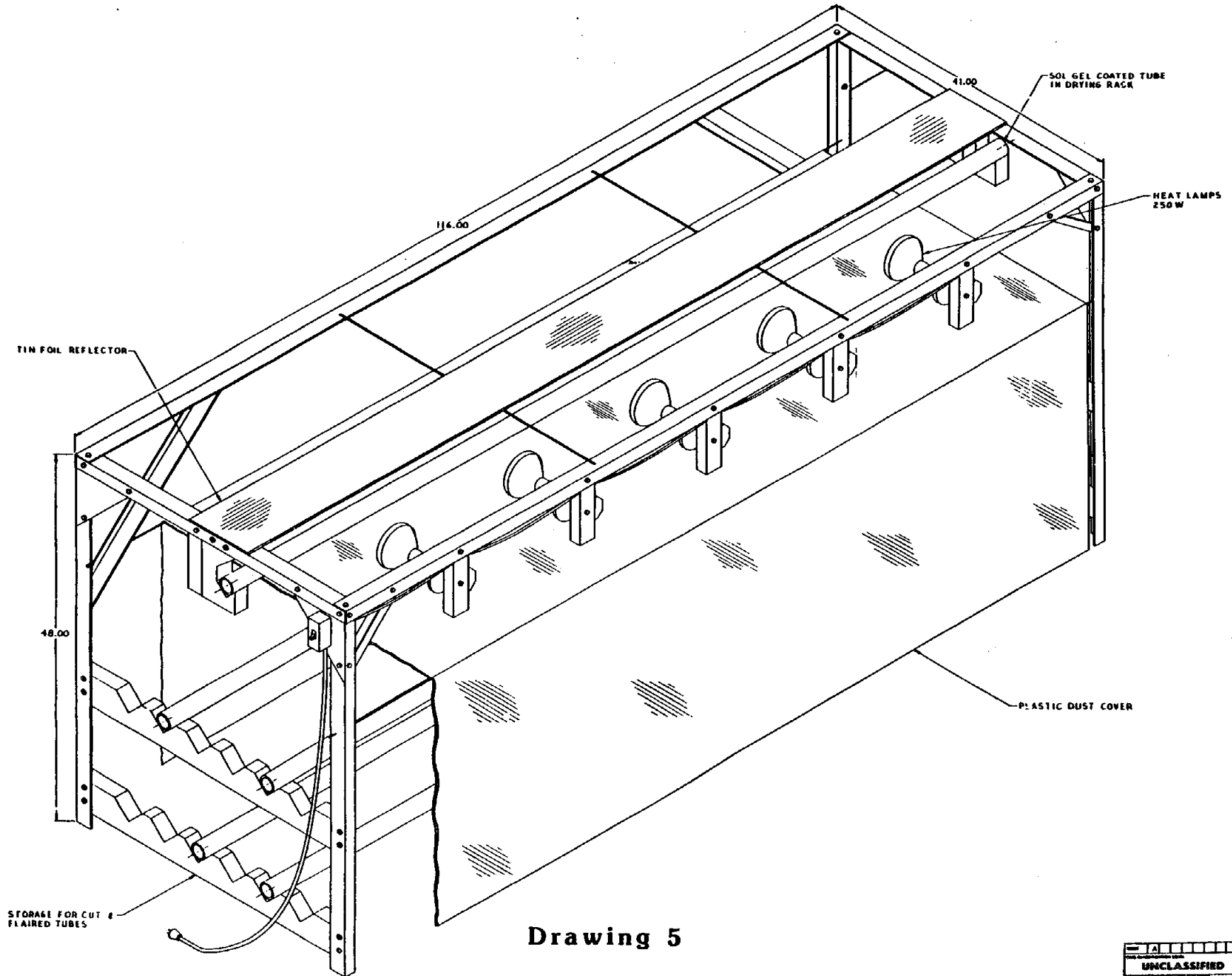
NOTES:
1. ALL DIMENSIONS SHALL BE APPROXIMATE.

545632-000	A	D. NICHOLS, 7651/ M. ARCHULETA, 7651/ S. REED, 7472			
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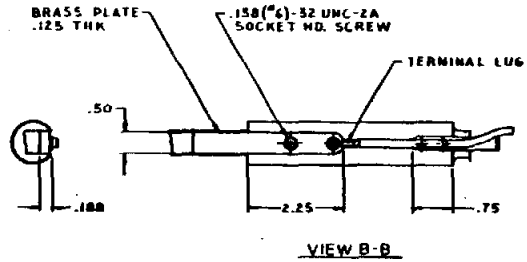
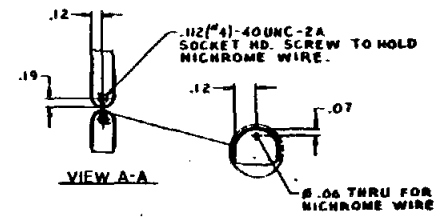
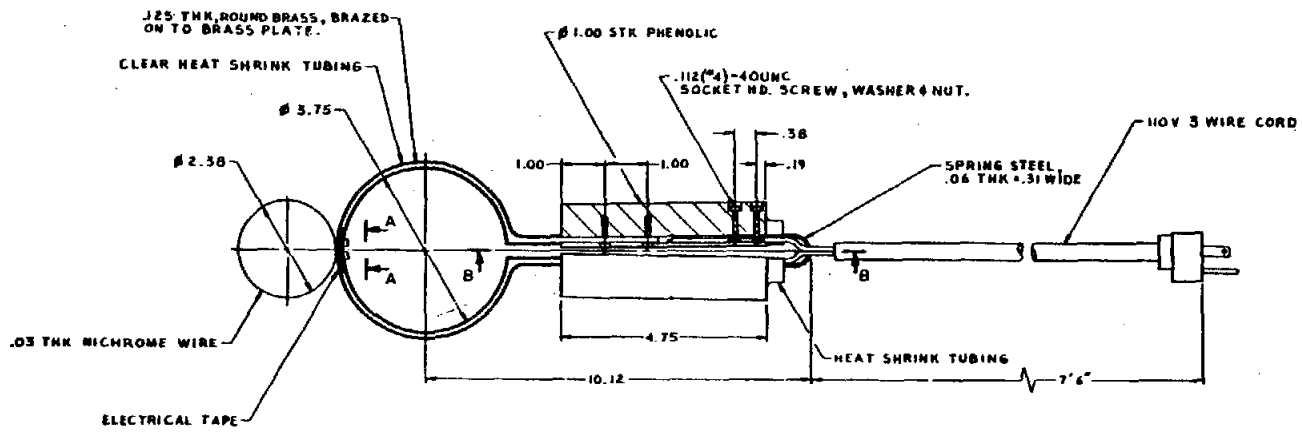
Drawing 3

545632-000	A	D. NICHOLS, 7651/ M. ARCHULETA, 7651/ S. REED, 7472			
		UNCLASSIFIED	SOL GEL COATING APPARATUS		
		UNCLASSIFIED	E114213 545632		



Drawing 5

	A								
UNCLASSIFIED									
F712374 1545632									



GLASS TUBING CUTTER
 NOTE: ALL DIMENSIONS AND CALLOUTS APPLY TO BOTH SIDES OF HANDLE

Drawing 6

APPENDIX C

Procedure for F⁻ Determination

The following procedure was developed to determine the amount of fluoride ion (F^-) present in the AR etching solution. The instrument used was the Orion Research microprocessor ionanalyzer/901. The electrodes used were an Orion double-junction reference electrode (900200) and a fluoride ion electrode (940900). Because fluoride reacts with glass, plastic labware was used throughout the procedure for storing samples and standards.

TISAB II was used to provide a constant ionic background strength, decomplex fluoride, and adjust solution pH. TISAB II is prepared by adding 57 ml glacial acetic acid, 58 g NaCl, and 4 g CDTA (cyclohexylene dinitrilo tetraacetic acid) to 500 ml deionized (DI) water. Place in a water bath for cooling, immerse a calibrated pH electrode into the solution, and slowly add 5M NaOH until the solution pH is between 5.0 and 5.5. Cool to room temperature and dilute to 1 L with DI water.

A commercial solution containing 100 ppm fluoride (Corning 478170) was used as a standard. With the reference and fluoride ion electrodes in the standard solution, the Standard Value switches on the

Orion ionanalyzer were set at 100.0, and the Slope switches were set to values determined by the electrode check. The Sign switch was set at minus and the Mode switch at CONC. Place the electrodes in 100 ml of a solution consisting of 50 ml of the fluoride standard and 50 ml of TISAB II. Press Clear/Read MV and wait for a stable reading. Press Set CONC and rinse electrodes. Place electrodes into a 100-ml solution consisting of 50-ml sample and 50-ml TISAB II. Wait for a stable reading and record the sample concentration. Measurements should be repeated three times and the average concentration reported. The instrument should be recalibrated every 2 h. Changes in temperature should be compensated for by increasing the Slope setting by 1 MV for each 5°C increase in temperature.

The AR etching solution consisted of 25-ml H_2SiF_6 and 0.75-g NH_4HF_2 in 475-ml DI water. Using this procedure, the average fluoride ion concentration in the etching solution was 12 625 ppm.

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