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STUDIES AND TESTING OF ANTIREFLECTIVE (AR) COATINGS FOR SODA-LIME GLASS

Motorola Report No. 2335/1

Final Report

Edward M. Pastirik, Terry G. Sparks and Michael G. Coleman

May 1978

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The JPL Low-Cost Silicon Solar Array Project is sponsored by the U.S. Department of Energy and forms part of the Solar Photovoltaic Conversion Program to initiate a major effort toward the development of low-cost solar arrays. This work was performed for the Jet Propulsion Laboratory, California Institute of Technology by agreement between NASA and DOE.

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1.0 SUMMARY

Experimental results on this feasibility study to establish processes for producing antireflection films on glass are very encouraging. Efforts have been concentrated in three areas: acid etching of glass, plasma etching of glass, and acid development of sodium silicate films on glass. The best transmission to date has been achieved through the acid etching technique, while the most durable films have been produced from development of sodium silicate films. Control of the acid etching technique is presently inadequate for production implementation, and large scale application methods for sodium silicate films need further definition. While films having excellent anti-reflective properties have been fabricated by plasma etching techniques, all have been water soluble, disqualifying the films from a weatherability standpoint.

2.0 INTRODUCTION

1986 goals of the LSSA Project include large scale production of efficient solar array modules, having a twenty year life at a cost of $0.50/peak watt (in 1975 dollars). These goals place stringent requirements on the materials utilized in the modules. Glass covers are a feasible option for 1986 modules and a preferred cover material for modules built in the intermediate term, probably through 1984.

Any encapsulation will introduce both absorption and reflection losses which, in effect, reduce the efficiency of the encapsulated solar cells compared to bare cells. This contractual study addresses the feasibility of appreciably reducing these encapsulation losses for modules using glass covers, maintaining the necessary cost and reliability objectives.

Glass is one of the few materials known to be capable of a life considerably exceeding 20 years. Reflection from the top surface of a glass cover is significant. (Reflection from the bottom surface is equally bad if it is in contact with air; if the bottom glass surface is contacted with an organic encapsulant, reflection from this interface is generally considerably reduced.) Additionally, certain glass compositions have appreciable absorption in the wavelength range useful for solar cells; the best transmittance compositions are low in iron content. Accordingly, specifying low iron content glass will improve transmittance through the encapsulant to the cells, leaving reduction of reflection from the front surface of the glass as the most fruitful area to investigate for reducing optical losses. This program is a feasibility study to investigate and define particularly promising methods for reducing reflections from low iron glass surfaces.

3.0 TECHNICAL DISCUSSION

Three technology areas were proposed for evaluation during this study on forming antireflection coatings on glass: glass surface etching with acid, glass surface etching in plasma, and acid treatment of sodium silicate deposits on glass.

3.1 ACID ETCHED FILMS

Before (and particularly during) World War II, several methods for producing AR films on glass by means of inorganic acid etches were developed in an effort to replace the then-unperfected art of vacuum deposition. The acid etch methods were slowly improved in later years, and a refined technique was published by S. Milton Thompson in the RCA Review, March, 1951. The method described in Thompson's article was used as the basis for our acid etch investigation.

In contrast to other techniques, the acid etch method does not deposit an AR film on glass, but rather produces a film by chemically and optically altering a thin layer of the glass surface itself.

Glass is a solution of a variety of oxide compounds, primarily silicon dioxide, but also metal oxides such as those of calcium, sodium, and magnesium. The object of acid etching is to selectively leach the non-silica components, leaving a skeleton of pure, porous SiO_2.

The etch reported by Thompson consists of a 1.25 molar aqueous solution of fluosilicic acid supersaturated with silica. At certain degrees of super-saturation, the acid selectively dissolves the metal components present in the glass. The skeletonized layer is of low refractive index and constitutes the AR film. By adjusting the temperature and duration of exposure to the acid, the thickness and optical properties of the skeletonized layer can be varied.

3.1.1 EXPERIMENTAL PROCEDURE

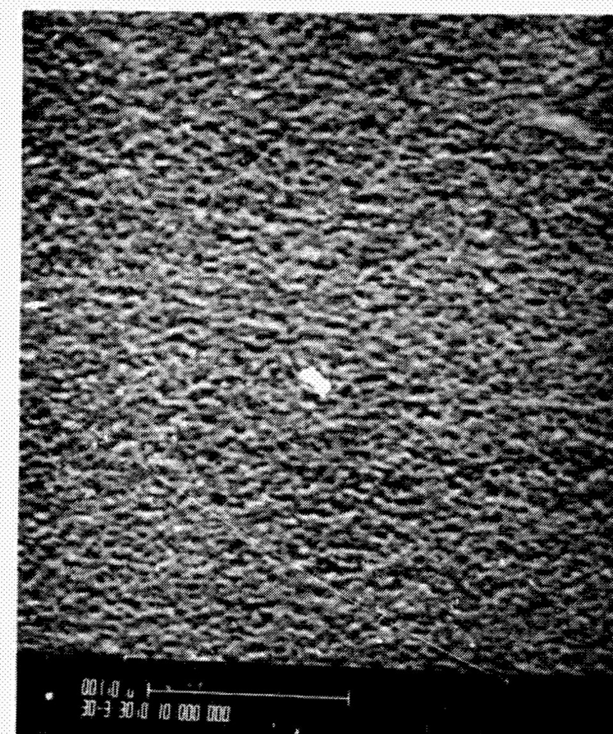
Glass plates were etched on both surfaces in solutions prepared in the laboratory. The solutions were prepared by diluting 1.2 liters of 23% fluosilicic acid to 1.9 liters with deionized water. To this solution, 60 grams of 200-mesh silicon dioxide was added, and stirred at room temperature for 12 hours. After stirring, the solution was filtered through paper and placed in a polyethylene container surrounded by water at 45 ± 1°C. The silicon dioxide saturated acid mixture was supersaturated with silicon dioxide by the addition of an aqueous solution of 4% boric acid. There is no known way of determining the proper degree of supersaturation analytically. By exhaustive trial-and-error, it was found that addition of 11.9 ml of 4% aqueous boric acid solution per liter of silica-saturated flourosilicic acid solution produced a degree of supersaturation conducive to film formation. Etching was performed, then, for 65 minutes.

3.1.2 RESULTS

Although the allowable range of supersaturation for film formation has not been determined with certainty, it is known that the range is extremely narrow. This narrow range, coupled with the fact that supersaturated solutions are unstable and tend to spontaneously revert to their saturated form with time, cause serious problems affecting reproducability of film properties. The skeletonized surfaces have been examined in the scanning electron microscope. Typical examples of micrographs are shown in Figure 1.

Acid etched films can be produced with relative ease, but attainment of superior anti-reflective properties is difficult. At present, untreated glass with a maximum transmission of 92.4% has been filmed to obtain a peak transmission of 99%. This is the best performance obtained. Routinely

30,000X

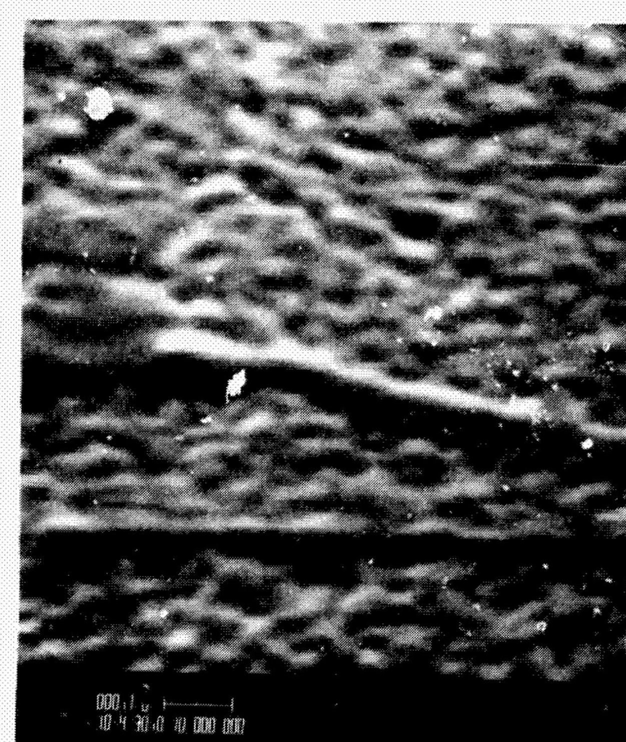


30,000X

FIGURE 1: EXAMPLES OF SKELETONIZED SURFACE
ON ASG LOW IRON GLASS

30,000X

100,000X



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produced films have increased transmission of the same glass to approximately 97%.

The production of filmed glass samples for this feasibility contract has been completed. Due to the difficulties encountered in maintaining the etch solution's chemical composition, the films' optical efficiency varies sli. .ı, from sample to sample. All films, however, produce a peak transmission of at least 97.0%.

3.2 SODIUM SILICATE FILMS

When aqueous solutions of sodium silicate (waterglass) are treated with acid, a gelatinous precipitate results; it is composed of hydrated silicon dioxide and various sodium salts. This reaction can be used to produce AR films on glass, and possibly on certain plastics.

Production of the film is accomplished by applying commercial waterglass, diluted with water, to a glass plate. The plate may be spun to level the waterglass film, then allowing the film to dry, or another application method such as spraying or dipping may be utilized. The plate is next dipped into concentrated sulfuric acid, rinsed, and redried. This process produces a film which is composed of insoluble low-hydrated silicon dioxide and sodium sulfate. Although the sodium sulfate in the film is water-soluble, its removal by exposure to water does not to any great extent affect the mechanical strength of the film. The optical qualities of the film are, in fact, enhanced by the removal of the sulfate, so that intentional washing or weathering may actually improve the AR properties of the film.

It appears that films produced in this way have AR characteristics somewhat poorer than the acid etched films, the difference being a slightly higher index of refraction.

3.2.1 PRODUCTION OF SILICATE FILMS

For the production of silicate films, commercial sodium silicate syrup, manufactured by Humco Laboratories, Texarkara, Texas, was used. The syrup had the following chemical properties:

Density	1.5g/cc
pH	11.3
Dry residue	54.1 wt. %
Na content	5.0 wt. %
Si content	12.0 wt. %
Na_2O/SiO_2	0.26

Production of the silicate films was performed by mounting glass samples on a standard photoresist spinner, flooding the surface of the sample with a solution of sodium silicate syrup in water, and centrifuging the glass sample at 2000 RPM for 30 seconds. The procedure was repeated on the other side of the sample, followed by a 2 minute dip in 96% sulfuric acid.

3.2.2 RESULTS

With solutions containing 13.8 volume per cent sodium silicate syrup, films having allowing 97.5% peak transmission were possible. Reproducibility of optical properties was excellent.

3.3 PLASMA ETCHING OF FILMS

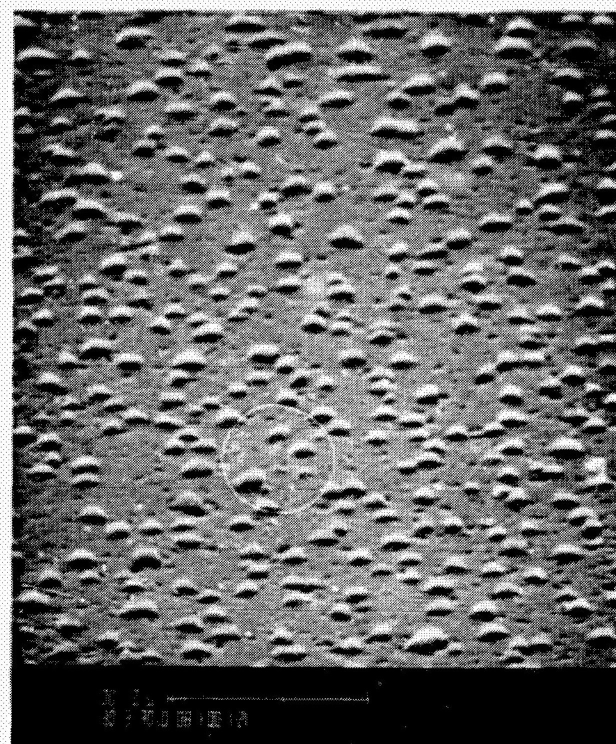
Plasma technology has made a significant impact on the semiconductor industry in recent years by providing highly improved process control at substantially reduced costs for etching, cleaning, and deposition of thin films. Reduced cost is primarily a result of reduced consumption of materials through the elimination of wet chemical steps. This study was an effort to apply plasma technology to produce. on glass, AR films analagous to known acid-etched films, following up on preliminary tests which produced an interference layer on glass etched with a plasma of Freon 14 (CF_4) and oxygen.

The equipment used was a specially modified commercial plasma system. A small parallel plate reactor was mounted inside the chamber of a Tegal Model 411 Plasma Etcher. The 10cm X 10cm glass substrates were placed horizontally on the grounded electrode while the reaction gases were introduced above the glass from holes in the top electrode. At reduced pressure (approximately 500 millitorr) the reaction gases are ionized by an RF field applied between the two plates. Pressure and RF power were variables studied in attempts to produce the desired thickness and uniformity of film. The temperature during etching was near ambient, with slight heating effects from the AR field.

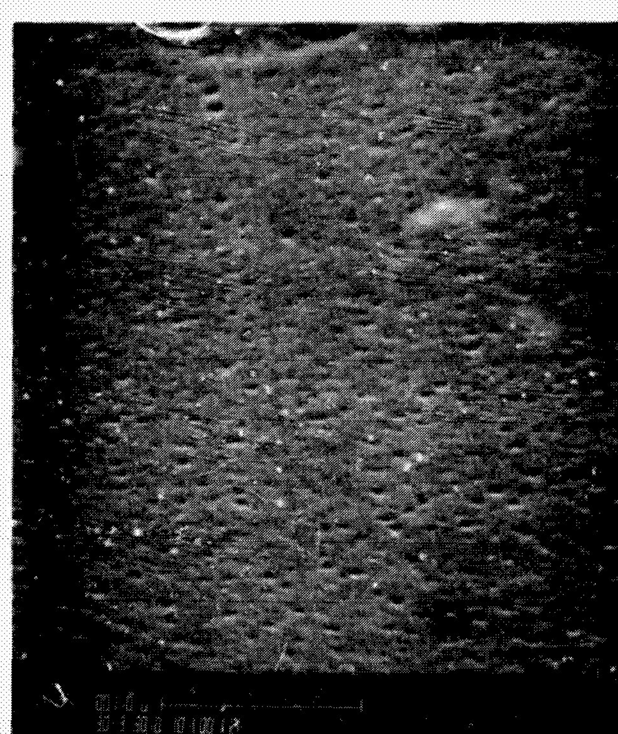
Before plasma treatment, the low-iron soda-lime glass (cut into 10cm X 10cm substrates) was washed in deionized water to remove surface particles. Substrates then received a 5 minute clean in an oxygen plasma to remove any organic residue. A plasma of CF_4 plus 8.5% O_2 was utilized throughout this work. Pressure was vari over the range 0.2 - 0.4 torr. Films having uniform visual a earance and good antireflectance properties were obtained by adjusting pressure in this range so that times of 3 min. to 30 min. were sufficient to produce the films as power was varied from 300 watts to 30 watts.

Films were evaluated in a spectrophotometer for transmittance and were found to have excellent AR properties. Total transmission was increased 5 to 7.5% over most of the 0.4 to 1.0 micron spectrum.

The surface of the plasma etched glass was examined using a scanning electron microscope; micrographs are included in Figure 1. Microprobe



GLASS SURFACE SHOWING ALKALINE FLUORIDE RESIDUE AND SURFACE ETCHING 30,000X



GLASS SURFACE FLUORIDE RESIDUE REMOVED BY WATER

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FIGURE 2: PLASMA ETCHED AR TREATMENT

X-ray analysis revealed the nodules on the surface as probably a calcium/silicon compound. Since the resolution for elements with atomic numbers below 12 is poor for the analysis equipment, it was not possible to positively identify the elements sodium and fluorine. It may however, be suggested that the plasma reaction forms alkaline fluorides or fluosilicates. Some examples, with known indices of refraction, are NaF(1.33), Na_2SiF_4(1.30), CaF(1.43), and $CaSiF_6$. All of these possible reactants are nonvolatile at the plasma process temperature and pressure. Thus a lower-refractive-index, quarter-wavelength layer may be formed (instead of a porous glass structure as in acid etching). The lack of a continuous (i.e., a nodular layer if formed) may be explained by the nonhomogenous microstructure of glass, which can demonstrate large composition variations within the dimensions shown in the micrographs. Also, the nonvolatile nature of the reactants causes the plasma reaction to stop, thus preventing formation of a porous layer.

Durability experiments were performed to evaluate mechanical properties of the films. Theywere soon discovered to be very water soluble, leaving only minor etched surface imperfections. Water solubility of the films virtually eliminates them from further consideration.

Next, an alternative plasma etching system was utilized. A nitrous oxide plasma was introduced to etch the glass, since it is known that nitric acid solutions have been used to leach aklalines from glass surfaces, producing an AR effect. No significant results were obtained using this process.

A recently introduced etching technique using a low pressure plasma with anhydrous HF gas at 180°C to 220°C has been used to selectively etch silicon dioxide in silicon device and integrated circuit applications. The selective effect of this process was thought to have a possible application to AR treatment, so it was tried on glass. Experimental results were similar to those of the original freon plasma process, in that the films were still water soluble.

It was concluded that the nonvolatile nature of some reaction products, and the high water content of the soda-lime glass, prevent the formation of a durable AR treatment by the plasma etching technique.

3.4 PLASMA DEPOSITED FILMS

Another area of application of plasma technology to the semiconductor industry has been the chemical deposition of thin, uniform dielectric films. Although not in the scope of this contract, the plasma deposition area was explored as a matter of completeness. By analogy to the deposited sodium silicate film technique examined in this study, experiments were performed to deposit quarter-wavelength interference dielectric films, using the decomposition of tetraethyloxysilane (TEOS) by an RF field. The film was silicon dioxide deposited on soda-lime glass plates. An interference effect was noted, but the small change in index of refraction did not produce significant reduction in reflection loss. This, combined with process problems and possible cost disadvantages, indicated that further investigation was not merited.

3.5 ACCELERATED DURABILITY TESTING

Durability testing was performed in two ways. Film samples on glass were exposed to ultrasonic vibrations in abrasive-free water as the first test. Subsequent testing was performed by buffing the film samples with a mild abrasive.

Both types of films displayed a degree of resistance to mechanical destruction which could be adequate for withstanding outdoor exposure. The two film types appeared, by both tests, to have similar mechanical strengths, with the sodium silicate films generally displaying superior durability. However, the acid-etched films did exhibit considerably more variability in resistance to ultrasonics than did the silicate films. The cause of this variability may be due to the fact that the acid-etched films used for durability testing were formed from different bath compositions. The acid-etch bath was sufficiently unstable that repeated attempts at sample production were required over a two-week period before enough samples were obtained to allow completion of the durability tests.

3.6 ENHANCED DURABILITY AR FILMS

Two methods which were used in an attempt to enhance AR film durability have produced no encouraging results.

Heat annealing of samples has produced either no readily reproduceable increase in durability or has resulted in partial destruct. . of both silicate and acid-etched films. Although it is possible that a heat anneal treatment could be of definite utility in AR processing if the necessary process parameters are found, further efforts in this area are deemed of questionable value.

Application of silicate coatings to acid-etched film samples has resulted in increased durability, but an antireflective performance that is no better than the silicate films discussed earlier. In view of the fact that samples so treated involve more processing steps than required for silicate films, but are not superior in either durability or optical performance, additional effort in this area seems unjustified.

3.7 CHARACTERIZATION OF TRANSMITTANCE OF AR SAMPLES

Optical characterization of the film samples has been completed. Examples of transmittance curves, taken with a dual beam Cary 17 Spectrophotometer, are shown in Figure 3 for acid-etched films, and in Figure 4 for treated sodium silicate films. The acid etched films have demonstrated a peak transmission at 0.56μm of 99.0% for light incident normal to the surface. The silicate films show a somewhat flatter response, having a rather broad peak centered at about 0.55μm and representing a transmission at that wavelength of 97.5% on the best sample. Both of these results represent striking improvements over the 92.4% transmission obtained for the same type of glass without antireflection coatings.

3.8 LARGE-SCALE COST ESTIMATES

In order to be implemented into a process sequence for making solar modules, any antireflection treatment must prove to be cost-effective. This translates into lowering the cost per watt of power output from the antireflection-treated module compared to the power cost without the treatment, perhaps modified to include life-cycle considerations.

For the case of solar cell modules, the antireflection treatment will be applied only to the outer surface of the glass. The inner surface will, almost certainly, be contacted to a filler material which will have an index of refraction near that of glass. This filler material, such as a silicone compound, will effectively eliminate the back glass optical interface as a reflecting surface, eliminating the need for an antireflection treatment there.

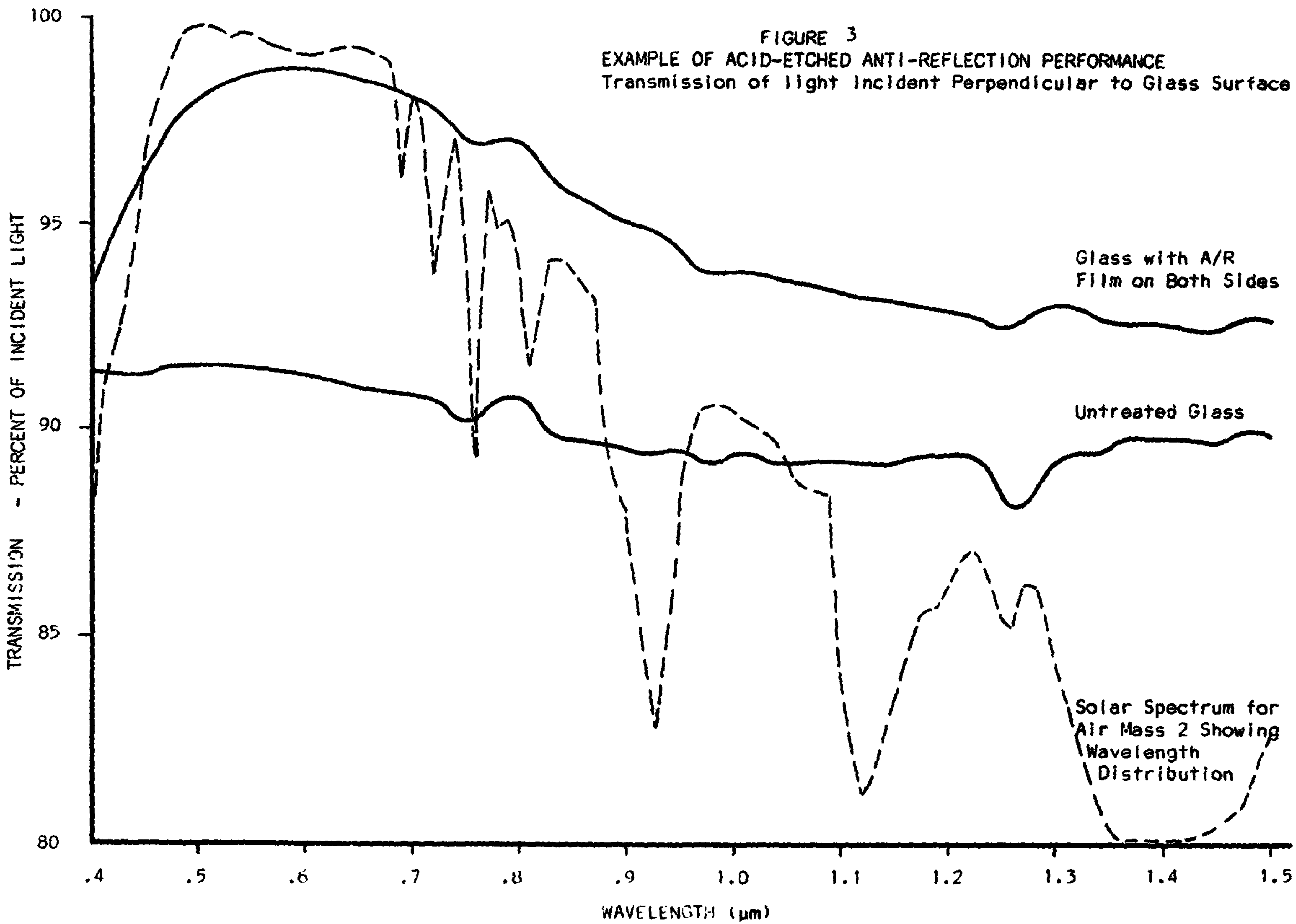
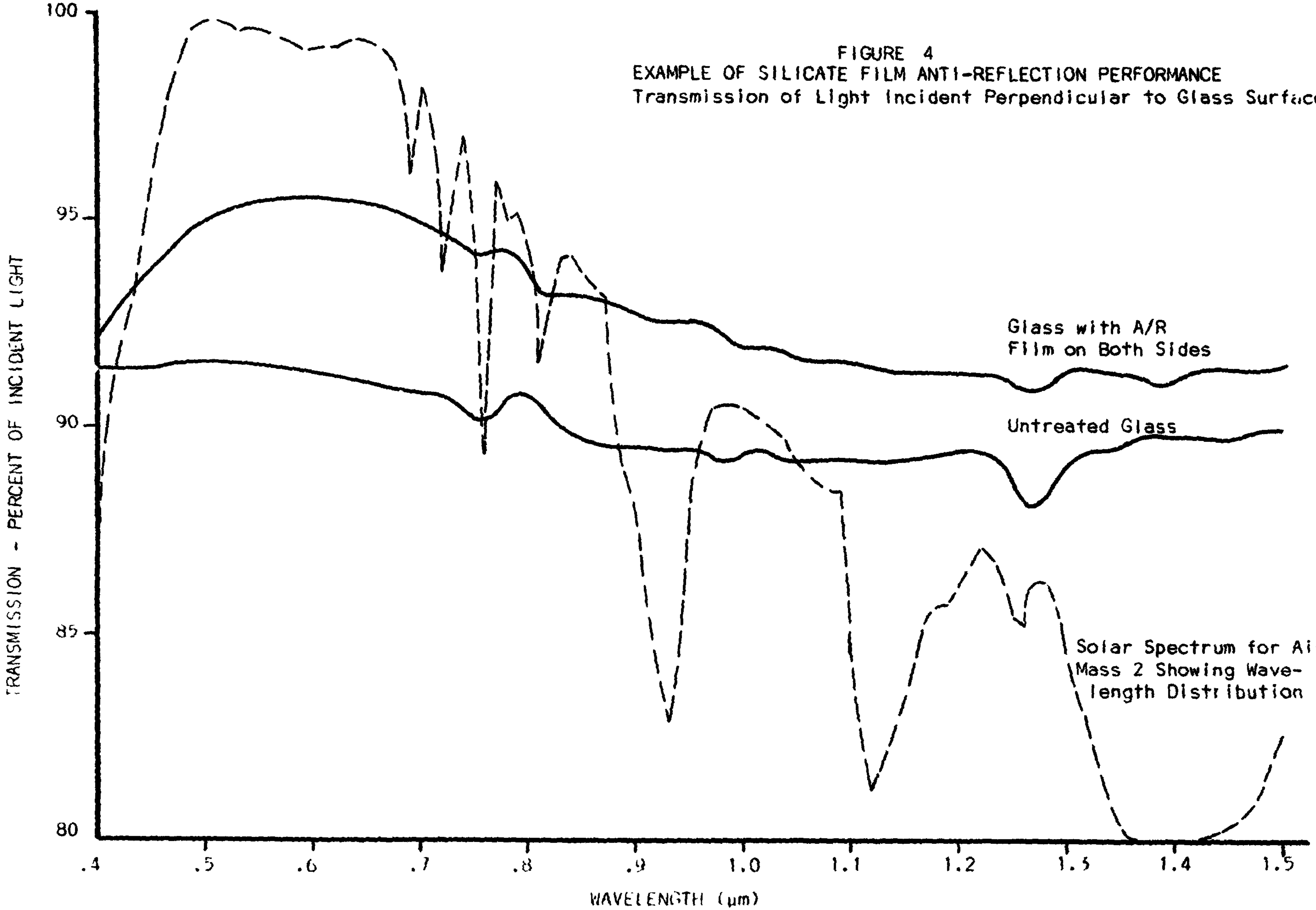


FIGURE 3
EXAMPLE OF ACID-ETCHED ANTI-REFLECTION PERFORMANCE
Transmission of light Incident Perpendicular to Glass Surface

FIGURE 4
EXAMPLE OF SILICATE FILM ANTI-REFLECTION PERFORMANCE
Transmission of Light Incident Perpendicular to Glass Surface



Transmissions of at least 97% of the incident light have been achieved with both the acid etch process and the sodium silicate process. This can be approximated as a 1.5% loss from each surface.

Comparing cover glasses with and without a typically achieved anti-reflection coating, the ratio of output power is

$$\frac{P'_{out}}{P_{out}} = \left(\frac{1 - 0.015}{1 - 0.04}\right) = \frac{0.985}{0.96} \approx 1.026$$

Nominally, then, a 2.6% increase in power can be expected. (If an improved antireflection coating is achieved, the power will be correspondingly increased.

An approximate analytical expression relating output power and optical losses is

$$P_{out} = \eta_M P_{in} (1-L)$$

where

P_{out} = Power output form the module

P_{in} = Incident power

η_M = Module conversion efficiency, not including reflection and absorption losses.

L = Fractional losses from reflection and absorption

For two different loss conditions, L and L', achieved by glass covers without and with an antireflection coating, the ratio of the output powers will be

$$\frac{P'_{out}}{P_{out}} = \frac{1-L'}{1-L}$$

Low iron soda-lime glass has a small absorption co-efficient in the wavelengths of interest. Total transmission losses at normal incidence, from both reflection and absorption, are approximatley 8%. Assuming negligible absorption losses, this can be approximated as a 4% reflection loss from each surface. If solar cell modules can be manufactured at a cost of \$2.00/watt, and the antireflection coating achieve the typical performance discussed here, the treatment may cost no more than about \$0.05/watt. Similarly, at a \$0.50/watt manufacturing cost, the antireflection treatment may cost no more than about \$0.0125/watt. Assume a 70% package fill-factor for round wafers at \$2.00/watt, and a 90% package fill-factor for ribbons at 50¢/watt. For a 14% encapsulated solar cell efficiency, this corresponds to maximum allowable costs of \$4.90/m^2 and \$1.575/m^2 for the \$2.00/watt and \$0.50/watt cases, respectively.

At the present time, technical feasibility of two processes has been established. Uncertainties, exist, as to what the exact, detailed process for either would look like in a manufacturing process sequence. Accordingly, an accurate cost projection is not yet possible for either process. The future manufacturing process for either must be assumed at this time to have a very high throughput and be readily automated. The cost effectiveness of either must, thus, be limited by the cost of the consumed materials. Approximate costs of consumed materials for each process are discussed in the next sections.

3.8.1 ACID-ETCHED FILM

The primary ingredient for the acid-etch process bath is fluosilicic acid. Although this acid has commercial applications, high volume consumption is rare, and as a result, high quantity discount prices are difficult to obtain. For this reason, the cost estimate presented here will assume a worst-case situation in which the acid price is that currently in effect for small volume shipments.

Fluosilicic acid 23% $16.00/gal.

Silicon dioxide 99.7% $11.00/lb

These prices include shipping.

Each gallon of filming solution consists of approximately two-thirds by volume 23% fluosilicic acid, the balance being water, and 0.1 kg high-purity silicon dioxide. Therefore the material cost for the acid etch is:

$$\frac{2}{3}\ \$16.00 + 0.1\,\text{kg} \times \frac{2.205\ \text{lb}}{\text{kg}} \times \frac{\$11.00}{\text{lb}} = \$13.09/\text{gal}.$$

It has been experimentally determined that 1.9 liters of the acid etch is capable of filming both sides of at least 200 10cm square glass sheets (the equivalent of 400 sheets on one side) under optimum conditions. Assuming this bath output may be maintained in a controlled production operation yields the following cost calculation:

$$\frac{\$13.09}{\text{gal}} \times \frac{1\ \text{gal}}{3.875\ell} \times \frac{1.9\ell}{200\ \text{sheets}} \times \frac{1\ \text{sheet}}{200\ \text{cm}^2} \times \frac{10^4\ \text{cm}^2}{\text{m}^2} = \$1.64/\text{m}^2$$

Volume pricing of the fluosilicic acid will, of course, reduce this cost. Further, the purity and/or price of the silica may also be reduced significantly. With these considerations, this process is potentially cost effective at even the $0.50/watt cost.

3.8.2 SODIUM SILICATE PROCESS

The two required reagents for the silicate process are readily available in large quantities and are currently inexpensive. Prices listed below are current and include shipping.

Sodium silicate solution (Baume´ 40) $0.077/lb.
in 50,000 lb. lots

Sulfuric acid (96%) in 3,000 gal. lots $2.69/gal

- The sodium silicate solution, as supplied, has a density of 11.67 lb/gal.
- A successful filming solution consists of 33% by volume of the above sodium silicate solution in water.
- 5 cm^3 of filming solution is found to be sufficient to film 100 cm^2 of glass.

The calculation appears below:

$$\frac{11.67\ \text{lb}}{\text{gal}\ Na_2SiO_3} \times \frac{.33\ell\ Na_2SiO_3\ \text{sol'n}}{1\ \ell\ \text{film sol'n}} \times \frac{0.005\ \ell\ \text{film sol'n}}{100\ cm^2} \times \frac{\$0.077}{\text{lb}\ Na_2SiO_3\ \text{sol'n}} \times \frac{1\ \text{gal}}{3.785\ \ell} \times \frac{10{,}000\ cm^2}{m^2} = \$0.039/m^2$$

To the above sodium silicate cost, the sulfuric acid expense must be added. Experimentally, 0.1 liter of sulfuric acid is sufficient to harden 1 m^2 of film.

$$\frac{0.1\ \ell\ H_2SO_4}{m^2} \times \frac{\$2.69}{\text{gal.}\ H_2SO_4} \times \frac{0.26417\ \text{gal.}}{\ell} = \$0.071/m^2$$

The combined material cost is then:

$0.039/m^2$ for Na_2SiO_3 + $0.071/m^2$ for H_2SO_4 = $0.11/m^2$ for combined materials. This is obviously very cost effective at prices even lower than $0.50/watt if consumed materials costs are a significant part of the total cost.

3.9 ASSESSMENT OF ANTIREFLECTION TREATMENT EFFECTIVENESS AND RECOMMENDATIONS FOR FUTURE DEVELOPMENT

Several methods for forming antireflection films on glass have been studied. Of those methods, two have proven in this study to be feasible for future utilization. The two promising processes are acid etching of the glass surface to form a skeletonized layer, and acid development of an applied sodium silicate layer. Due to the limited slope of this program, complete development of processes has not been possible. As a result, accurate ranking of the two methods cannot be performed with a high degree of confidence.

The acid etched films have exhibited superior optical properties, achieving 99% peak transmission through a glass sheet treated on both sides. The major problem with the acid-etch techniques is control. Future investigations should include correlation of measurable physical properties of the etch bath (e.g., infrared absorption, refractive index, dipole moment, etc.) with etch performance in an effort to arrive at a convenient control technique. Control of this process in an economical fashion must be a prime consideration.

Sodium silicate films have achieved significant improvements in transmission (97.5%) and appear to be superior in durability. The silicate technique is quite well-controlled. Primary importance should be attached to development of an alternative to spinning for adjusting film thickness. Several alternatives, including spraying and dipping, appear possible for this application. Additional efforts should also be expended to promote further improvements in transmission.

4.0 CONCLUSIONS

Evaluations have been performed on a variety of technologies for utilization in forming an antireflection coating on the cover glass of a solar module.

Two process technologies have demonstrated feasibility: acid etching of the glass surface to form a skeletonized surface layer having an index of refraction intermediate to glass and air, and acid development of a sodium silicate layer deposited on the glass surface. Peak transmission losses through treated glass sheets have been reduced to 1% and 2.5% the acid etched and sodium silicate films, respectively, on glass with ated surfaces that has near 7.5%. Both processes require additional definition and development before effective application in a manufacturing environment can be achieved. Development required for the acid etched process centers on achieving improved control of the etchant bath, while alternate methods of film deposition suitable for large sheets of glass are required for the sodium silicate process.

Plasma processes for both etching glass and deposition films have been investigated. No suitable films have been achieved with either technology.

An approximate cost analysis has been performed. Both the acid etched and the sodium silicate films appear potentially cost effective, with the sodium silicate films being cheaper on the basis of consumed material costs.

5.0 RECOMMENDATIONS

Two processes for formation of a cost-effective antireflection coating on glass have shown feasibility. Each requires further development before one can be chosen over the other as a desirable long range technique. It is recommended that this further development of both be performed

6.0 CURRENT PROBLEMS

No specific problems exist.

7.0 WORK PLAN STATUS

The feasibility study is now completed.

8.0 LIST OF ACTION ITEMS

Consideration should be given to advanced development funding of the two feasible process technologies, leading to a definitive choice for incorporation in a manufacturing sequence.