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ANTI-REFLECTION COATINGS APPLIED BY ACID
LEACHING PROCESS

Quarterly Technical Report

1979

By
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Work Performed Under Contract No. NAS-7-100-955387

Motorola Inc. Semiconductor Group
Phoenix, Arizona

MASTER

U.S. Department of Energy

Solar Energy

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ANTI-REFLECTION COATINGS APPLIED BY
ACID LEACHING PROCESS

QUARTERLY TECHNICAL REPORT NO. 2

MOTOROLA REPORT NO. 2370/2

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1 JULY 1979 - 30 SEPTEMBER 1979

JPL CONTRACT NO. 955387

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THE JPL LOW-COST 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.

PROJECT NO. 2370

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1.0 SUMMARY STATEMENT

This report covers the second third of a nine-month program to study the production of anti-reflective coatings for glass solar module covers by means of a silica-saturated fluosilicic acid etch.

The efforts of the program during this second quarterly period have been to evaluate the effects of process variations on the production of coated glass samples, to test the abrasion resistance of coated samples, and to test the chemical durability of samples.

The ultimate goal of this project is improvement of the efficiency of photovoltaic modules through increased transmission of light by the module cover. One method of increasing transmission is by reducing the reflection of light by the module cover, a reflection which accounts for a 4% loss of transmission for each coverglass-air interface.

This study is intended to advance a known antireflection technology to the point where large area glass module covers can be treated to produce uniform, efficient, and durable antireflective films.

The silica-saturated fluosilicic acid etch of Thompson (RCA Review, March, 1951) is the method used to produce the antireflective films for this project. The method produces films by selectively removing metallic ions from a surface layer of soda-lime glass, transforming the layer into a thin film of porous silicon dioxide, which, because of its porous nature, has a refractive index lower than that of the bulk glass. This layer is suitable for drastically reducing the amount of light reflected from a glass surface in contact with air.

3.0 TECHNICAL DISCUSSION

Early effort in this program was directed toward the production of sufficient samples of filmed glass to permit destructive testing of abrasion and chemical resistance. In excess of three hundred 4-inch square filmed glass samples have been prepared and the production phase of this project is now complete.

3.1 PERFORMANCE OF SAMPLES

Although complete data on average AR performance is not yet available, the production effort explained above resulted in at least ten samples which were visually superior to all of the other samples in their antireflection performance. These samples were analyzed by means of an IR-VISIBLE-UV spectrophotometer and a plot of light transmission vs. wavelength of one of the samples is shown in Figure 1. The plot also shows the transmission characteristics of glass with a hardened silicate antireflective film and of the same glass without film for purposes of comparison. Also included is a plot of the solar spectrum for Air Mass 2 showing the wavelength distribution of light and its peak near the peak transmission of the filmed glass sample. As can be seen, the acid etched film represents a marked improvement over plain glass, transmitting 99.8% of incident light at a wavelength of 460 μm vs. a peak 92.6% transmission for unfilmed glass near the same wavelength.

3.2 PRODUCTION OF SAMPLES

Although work on process variation effects on AR performance is not complete, some results are available.

The acid etch method of filming glass depends on the establishment of a 1.25 molar solution of fluosilicic acid supersaturated with silica. The

FIGURE 1: EXAMPLES OF ACID-ETCHED AND SILICATE ANTI-REFLECTION PERFORMANCE.

TRANSMISSION OF LIGHT INCIDENT PERPENDICULAR TO GLASS SURFACE.

supersaturation is achieved by adding 4% aqueous boric acid solution to 1.25 M fluosilicic acid which has been saturated with silica by standard methods. The degree of supersaturation is conveniently expressed in terms of units of "potency". Fluosilicic acid which is saturated with silica is assigned a potency value of zero. Each addition of 0.5 ml of 4% aqueous boric acid solution to one liter of saturated fluosilicic acid causes a potency decrease of one unit. For example, a liter of saturated fluosilicic acid to which 4 ml of 4% boric acid solution is added assumes a potency of -8.0.

The results that have been obtained so far have shown that the ASG Lustraglass^R used exclusively in this research program films at between -19 and -21 potency units. It has been stated by Thompson in his original report that temperature has effects on the filming rate. Our testing shows that even when temperature is held constant, filming rate varies, apparently in response to degree of supersaturation. Moreover, the variation in filming rate is striking, even when the potency variation is small. For example, at 45°C, an AR film that appears deep purple by reflected light is produced in 86 minutes at a potency of -19; while at potency -18.5, 97 minutes are required; and at potency -19.5, 133 minutes are necessary. No reference to this strong potency-dependence has been found in the literature on this method. At present no explanation for the observed change in etch time is available. In future experiments at elevated etching temperatures efforts will be made to see if this effect also occurs at other than 45°C. Repeated trials at 45°C have verified that the observed effect is genuine, although very accurate reproducibility of reduced etch times has been impossible.

3.3 ABRASION TESTING

The abrasion resistance of filmed glass samples was evaluated. Two samples of filmed glass having a transmission of 99.8% at 460 μ m was chosen as the primary

objects of the test. It was felt that abrasion resistance may be dependant on film porosity, and thus, antireflective performance. In order to test this assumption, five samples each of two less efficient antireflective-filmed samples were also tested. All samples were filmed at 45°C.

Abrasion was performed in two ways: without abrasive by a cotton pad, and with the same cotton pad loaded with abrasive powder. The pad was composed of 4 layers of 0.008 inch cotton duck, with a circular area of 13.9 cm². The pad was loaded with weights to yield a pressure of 138 g/cm². After conducting abrasion tests with the pad on samples of filmed glass, the pad was stamped in Buehler No. 40-6475 AB, 3200 mesh abrasive, then tapped to remove adhering clumps of abrasive so that only the abrasive that had been worked into the cloth was available. The abrasive on the pad was renewed for each of the five samples tested. The testing was performed by moving the pad laterally across the filmed glass surface in 2 inch strokes, with visual inspection of the glass after every few strokes. Because of the low number of 99.8% transmission samples available, one sample was tested with the plain cotton pad and one was tested with the pad and abrasive in five places on the same samples. The results are summarized below.

STROKES REQUIRED TO DAMAGE FILMED SAMPLES

TEST FILMS

INCREASING
TRANSMISSION
99.8% TRANSMISSION

ABRASIVE	EFFECT	ACID ETCHED #1	ACID ETCHED #2	ACID ETCHED #3
CLOTH ONLY	First sign of damage	80	30	20
	Moderate damage	200	100	50
CLOTH PLUS ABRASIVE	First sign of damage	20	1	1
	Moderate wear	200	40	20
	Film Removed	>300	200	150

As shown by the results, increasing antireflective effectiveness does adversely affect abrasion resistance. It may be pointed out that in no case was film removal possible without an abrasive powder; only moderate damage could be achieved. Moderate damage was defined as unmistakable damage covering the entire stroked area but not resulting in complete removal of film from any area. With the abrasive powder, damage of all types was inflicted easily on the two most antireflective samples. The least antireflective sample was the most rugged; the ">300" strokes required for film removal caused 95% of the film to be removed from the stroked area, since complete removal appeared impossible. With the other films, removal was complete. Although these results appear discouraging, it may be pointed out that the powdered abrasive test was unusually severe. Even samples of lacquered metal surfaces sustained what would be classified as moderate damage after 30 strokes.

4.0 PROJECTED ACTIVITIES FOR THE NEXT THREE MONTHS

Studies of the effects of potency variations on AR film production will continue with tests extended to include production at 55°C and 65°C in addition to the 45°C temperature that has been in use until now. Staining tests with water and oil soluble stains will be conducted to determine the ease of cleaning the microporous filmed samples. In addition, detergent cleaning tests will be performed to demonstrate the film's endurance of the abrasion associated with periodic cleaning.

5.0 RECOMMENDATIONS

No recommendations are made at this time.

6.0 CURRENT PROBLEMS

No urgent problems have arisen.

7.0 WORK PLAN STATUS

Work is within the schedule limits.

8.0 ACTION ITEMS

Reports are late. Efforts are under way to correct this.

9.0 NEW TECHNOLOGY

No reportable items of new technology have been identified, as yet.

10.0 PROGRAM AND DOCUMENTATION MILESTONES

Activities associated with the total program are shown in the Program and Documentation Milestone Charts Figures 1 and 2 contained in Appendix 1.

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APPENDIX I

PROCESS STEPS	1979 MONTHS								
	APR	MAY	JUN	JUL	AUG	SEPT	OCT	NOV	DEC
Set Up Facility									
Determine Process									
Produce Samples									
Test Abrasion									
Test Chemical Resistance									
Test Process Variations									
Cost Analysis									

FIGURE

DOCUMENTATION MILESTONE CHART	MONTHS								
	APR	MAY	JUN	JUL	AUG	SEPT	OCT	NOV	DEC
Monthly Financial Management Review		▲	▲	▲	▲	▲	△	△	△
Technical Letter Progress Report		▲	▲		▲	▲			
Quarterly Technical Report				▲			▲		
Draft Final Report								△	
Final Report									△
Program Plan	▲								
Baseline Cost Estimate	▲								

FIGURE 2