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EFFECTS OF VARYING OXYGEN PARTIAL PRESSURE ON MOLTEN
SILICON-CERAMIC SUBSTRATE INTERACTIONS

Quarterly Report No. 1

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MASTER

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U.S. Department of Energy

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Solar Energy

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QUARTERLY REPORT NO.1

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October 1979

The JPL Low-Cost Silicon Solar Array Project is funded by DOE and forms part of the DOE Photovoltaic Conversion Program to initiate a major effort toward the development of low-cost solar arrays.

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ABSTRACT

Activity at UMR this quarter has been in three areas:

1) Investigation of the devitrification of fused silica under various oxygen partial pressures, 2) Construction of a thoriatryttria solid solution electrolyte portable oxygen sensor, and 3) Theoretical development and application of a method of calculating the surface free energy of ceramic substrates from silicon sessile drop contact angle and oxygen partial pressure measurements.

1) Fused silica substrates were heated to 1430°C under various oxygen partial pressures to observe the effect of oxygen on the devitrification of fused silica. Some tests were made as silicon sessile drop experiments and some were performed without silicon present. These experiments were a follow-up on previous experiments in which fused silica devitrified in an $\text{H}_2/\text{H}_2\text{O}$ buffer atmosphere at 10^{-19} atm. of oxygen partial pressure. In the present experiments at higher oxygen partial pressures and in atmospheres other than the $\text{H}_2/\text{H}_2\text{O}$ buffer system, some devitrification took place in all cases. The degree of devitrification at the lowest oxygen partial pressures was essentially complete, while atmospheric oxygen ($P_{\text{O}_2} = 0.2$ atm.) produced only a thin surface layer of devitrification. These studies confirmed that silicon sessile drop experiments on fused silica substrates would be impractical in our controlled atmosphere furnace

system which requires long heat-up and cool-down times.

2) Construction of a portable thoria-yttria solid solution electrolyte oxygen cell capable of measuring very low oxygen partial pressures (greater than or equal to 10^{-26} atm. at 1000°C) and designed for use at existing facilities where silicon ribbon and sheet are produced has been constructed and calibration procedures initiated.

3) When the Gibbs adsorption isotherm equation is used with the Young equation, a linear relationship between the cosine of the contact angle and the log of the oxygen partial pressure is obtained. From the slope of this linear relationship, the amount of adsorbed oxygen and the surface free energy for a given substrate may be found. Calculations of the surface free energies for silicon carbide, silicon nitride, and aluminum nitride using our sessile drop contact angle data for these materials have been made. The values thus obtained for the adsorbed coverages of monatomic oxygen are of the same order of magnitude as those found for non-oxide materials in the same temperature range by other investigators using independent methods and the solid surface free energies obtained are not unreasonable.

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INTRODUCTION

The JPL Low-Cost Solar Array project is part of an effort funded by the Department of Energy to develop an economical means of converting solar energy to useful electrical energy. As part of this effort, it is desirable to learn the properties and behavior of those materials that are to be exposed to molten silicon in the process of forming silicon sheets and ribbons. The current study is aimed toward investigating the effects of oxygen on the interaction between molten silicon and various substrate materials by sessile drop studies, and also by measuring the oxygen partial pressure in existing silicon ribbon and sheet production facilities, with the ultimate purpose to correlate the final silicon impurity levels with the partial pressures of the critical gases such as oxygen above the silicon melts.

DEVITRIFICATION STUDIES ON FUSED SILICA

Sessile drop experiments performed by us on three types of high-purity fused silica substrates have previously been reported.¹ At the low oxygen partial pressures used in these experiments, devitrification and deformation of the silica substrates occurred in all cases, which eliminated fused quartz from further sessile drop experiments at that time.

During this quarter, more tests were made on vitreous silica to determine if devitrification could be avoided in our system at some higher oxygen partial pressure than that used previously ($\sim 10^{-17}$ atm.).

Dynasil Fused Silica Sessile Drop

An experiment was carried out in a P_{O_2} atmosphere of 1.5×10^{-12} atm. using the CO/CO₂ buffer gas system to eliminate any devitrification effect of hydrogen. The slow heat-up rate characteristic of our controlled atmosphere furnace contributed to the deformation of the vitreous silica substrate which took place well before the melting temperature of the silicon was reached (Figure 1-A & 1-B). There was so much interaction between the silicon and the buffer gas, which had a relatively high oxygen partial pressure (several orders of magnitude higher than the equilibrium pressure for the formation of SiO₂) that no sessile drop of

¹Eagle-Picher Second Quarterly Progress Report to JPL (May, 1978).

silicon was ever observed, even when the temperature was well above the melting point of silicon. Instead, it appeared that the silicon reacted with the atmosphere to form a coating on the outside of the cube and fine white particles which were carried away in the flowing gas and deposited at the downstream end of the furnace tube (Figure 2-E). By x-ray diffraction, it was determined that the coating on the cube consisted of β -SiC, which appeared as a black coating overcoated with a clear glassy phase assumed to be amorphous silica (Figures 2-A & 2-B). After removal from the furnace, the cube was detached from the substrate to reveal that no elemental silicon remained within the shell. Inspection of the portion of the silica substrate that was in contact with the alumina D-tube (i.e., the part of the silica substrate not exposed to continuous flow of the CO/CO₂ buffer gas) showed some remaining vitreous silica (Figure 2-D). The photographs in Figures 1 and 2 show the progress of the reaction and the condition of the silicon and silica substrate after completion of the experiment.

Dynasil Fused Silica in Air

When the Dynasil fused silica was heated to 1430°C in air, the deformation that occurred at $P_{O_2} = 10^{-12}$ atm. in the sessile drop experiment described above did not take place (Figure 3). Inspection of the sample after cooling shows that devitrification did take place, but this time only on the surface, as seen in Figure 4.

Figure 1 Silicon cube on Dynasil high purity vitreous silica substrate in sessile drop furnace with CO/CO₂ buffer atmosphere. ($P_{O_2} = 1.5 \times 10^{-12}$ atm. at 1430°C)

Figure 1-A: Well below the melting point of silicon. Deformation of the substrate has taken place, indicative of devitrification.

Figure 1-B: 16 minutes later, but still below silicon melting point. A mist of particulate matter can be seen by the silicon cube.

Figure 1-C: 22 minutes, at about 1415°C. Pitting of the silicon cube surface has begun.

Figure 1-D: 53 minutes, above the melting point. Pronounced attack of the silicon surface is now evident and the mist of particles is still visible.

Figure 1-E: 90 minutes, well above the melting temperature. Much build-up of reacted material on the exterior of the cube and particles are still visible in the gas stream.

Figure 1-F: 105 minutes. Particles are no longer visible, indicating that no more elemental silicon is available for reacting.

Figure 2 Dynasil vitreous silica and silicon sample after sessile drop test.

Figure 2-A & 2-B: Two views showing the devitrification of the vitreous silica and the state of the silicon cube. The cube surface is completely coated with black β -SiC and clear amorphous silica.

Figure 2-C: After removing the cube from the substrate, it was found to be hollow, indicating all elemental silicon had been removed, leaving only the reacted coating.

Figure 2-D: The region under the substrate that was in contact with the alumina D-tube is fractured, but still vitreous.

Figure 2-E: A very fine white powder collected at the down-stream end of the sessile drop furnace. It is amorphous to x-rays and assumed to be SiO_2 .

GE Fused Silica in Air

Similar results on thinner samples of vitreous silica were obtained. In order to reduce the amount of surface area in contact with the sample, thus permitting all areas of the sample to equilibrate with the atmosphere uniformly, two fragments of a GE fused silica tube were placed in the furnace as shown in Figure 5, with the upper fragment in point contact with the lower fragment. Again surface devitrification occurred on both fragments indicating that the degree of contact did not strongly affect the devitrification process. In this case, the thickness of the devitrified layer was not as great as in the case of the Dynasil sample (Figure 6).

GE Fused Silica in Helium

A GE Fused Silica sample was heated in a helium atmosphere, which has an oxygen partial pressure between air and the CO/CO₂ buffer used in the Dynasil experiment described above. The degree of devitrification was indistinguishable from that of the sample that was heated in air.

It is concluded that lower oxygen partial pressure atmospheres produce more complete devitrification in fused silica samples upon heating than higher P_{O₂}'s, but with the slow heating and cooling rates in our furnace, there would always be some devitrification of fused silica in any atmosphere used. Fused silica has, therefore, been eliminated from further consideration in the present program.

Figure 3: Dynasil vitreous silica in air at 1430°C. No deformation as in the low PO_2 case.

Figure 4: Dynasil vitreous silica after annealing in air at 1430°C. Devitrification has taken place, but only on the surface.

Figure 5: G.E. fused silica in air at 1430°C. No deformation is evident.

Figure 6: G.E. fused silica after annealing in air at 1430°C showing surface devitrification.

Figure 7-A

Figure 7-B

Figure 7-C

Figure 7-A, 7-B, and 7-C: First steps in assembly of the portable oxygen sensor furnace.

CONSTRUCTION OF A THORIA-YTTRIA
SOLID SOLUTION ELECTROLYTE PORTABLE OXYGEN SENSOR

Our work under a previous JPL contract^Δ has shown that very low partial pressures of oxygen ($\sim 10^{-26}$ atm. at 1000°C) can be measured using a ThO_2 - 7 wt% Y_2O_3 solid electrolyte cell. During this quarter, a portable oxygen detector of this type was constructed. It is intended to be used to measure the oxygen partial pressure in various sheet forming equipment at other JPL contractors' facilities where silicon sheet and ribbon are now being produced.

The following figures show the construction of the O_2 cell furnace which was designed to operate at a temperature of 1000°C . The furnace shell was made of 300 series stainless steel 16 inches long by 7 inches in diameter. The furnace windings consist of two half-round ($3\frac{1}{2}$ " dia. by 12" long) chromium-aluminum-iron alloy resistance heating elements.* They were set into two-inch thick alumina castable end-plates at each end and packed with fibrous silica** insulation (Figure 7). Copper tubing was soldered onto one end of the furnace for a water flow to provide cooling on the end where the thoria tube is located (Figure 8-A). The resistance heating element lead wires extend through the opposite end (Figure 8-B).

^ΔUnder subcontract from Eagle-Picher Industries, Inc.

*Electro Applications, Inc.

**Fiber-Frax H, Carborundum Co.

The thoria - 7 wt% yttria tube has platinum leads attached at the closed end, both inside and outside, with a brushed on half-and-half mixture of two commercial platinum pastes*** thinned with turpentine. The helically wound exterior lead was pasted along the length of the tube, and the free end grounded with a screw to the brass flange. The interior lead was attached with the platinum paste to the closed end of the thoria tube with the free end brought out through the same small alumina tube used to carry away the reference gas used in the O_2 measurements. A two-inch long cylinder of alumina insulating brick helps insulate the high-temperature o-rings which preserve the atmosphere in and around the thoria tube (Figure 9).

The furnace temperature is monitored with a chromel-alumel thermocouple inserted to within $\frac{1}{4}$ -inch of the thoria tube from the opposite end, where the inlet for the test gas is also located.

When the cell is at operating temperature, differences in oxygen concentration from the inner to the outer surface of the thoria tube produce an EMF which is linear with the log of the ratio of the two oxygen partial pressures. Calibration procedures have been initiated and indicate that the cell is operating according to design.

***Engelhard #6082 and Dupont # 9464

Figures 8-A & 8-B: Ends of the oxygen sensor furnace showing copper cooling coils on one end and furnace winding leads on the other end.

Figures 9-A & 9-B: Thoria-Yttria tube and assembly.

Figures 10-A & 10-B: Two views of the completed oxygen sensor.

Figure 11: Schematic diagram of the oxygen sensor.

SURFACE FREE ENERGY CALCULATIONS

Calculation of the solid-vapor surface energy for the CNTD* coated substrates investigated in silicon sessile drop studies under controlled partial pressures of oxygen has been found to be possible.

Theoretical Development

According to the Gibbs adsorption isotherm, the number of adsorbed species, i , per unit area is related to the interfacial free energy and chemical potential of the adsorbed species by:

$$\left(\frac{\partial \gamma}{\partial \mu_i} \right)_T = -\Gamma_i \quad (1)$$

where γ = interfacial free energy,

μ_i = chemical potential of the adsorbed species,

Γ_i = number of adsorbed species per unit area.

For an ideal gas, $d\mu_i = kT \ln P_i$, where P_i is the partial pressure of the species i . For oxygen, we have:

$$\frac{\partial \gamma_{sv}}{\partial (\ln P_{O_2})} = \frac{\partial \gamma_{sv}}{\frac{1}{P_{O_2}} \partial P_{O_2}} = -\frac{1}{2} kT \Gamma_O \quad (2)$$

The $\frac{1}{2}$ appears on the account of the fact that oxygen is adsorbed monatomically, but appears in the gas phase as a diatomic molecule. Hence, Γ_O is the number of atoms of oxygen adsorbed per unit area.

*Chemetal Trademark for their CVD process.

Integrating (2) gives:

$$\begin{aligned}\gamma_{sv} &= \int d(\gamma_{sv}) = \int_{P_{O_2}=1 \text{ atm}}^{P_{O_2}} -\frac{1}{2} kT\Gamma_O d P_{O_2}/P_{O_2} \\ &= -\frac{1}{2} kT\Gamma_O \ln P_{O_2} \Big|_{P_{O_2}=1 \text{ atm}}^{P_{O_2}}\end{aligned}$$

$$\gamma_{sv} = -\frac{1}{2} kT\Gamma_O \ln P_{O_2} \quad (3)$$

The solid-vapor interfacial free energy, γ_{sv} , is related to the contact angle, θ , by the Young Equation:

$$\gamma_{sv} = \gamma_{ls} + \gamma_{lv} \cos \theta \quad (4)$$

where γ_{ls} and γ_{lv} are the liquid-solid and liquid-vapor interfacial free energies, respectively.

We can eliminate γ_{sv} by combining equations (3) and (4), which gives us the result:

$$\cos \theta = - \frac{kT\Gamma_O \ln P_{O_2}}{2 \gamma_{lv}} + \frac{\gamma_{ls}}{\gamma_{lv}} \quad (5)$$

γ_{ls} and γ_{lv} are both assumed to be constant at a fixed temperature. The liquid-solid interface is not exposed to the gaseous environment, and, therefore, γ_{ls} should not be directly affected by changing the P_{O_2} . The vapor pressure of Si and SiO are both much greater than the P_{O_2} in these studies, and, therefore, it is assumed that oxygen is not adsorbed on the liquid-vapor interface making γ_{lv} independent of P_{O_2} . The value of Γ_O can be calculated from the slope of the $\cos \theta$ vs. $\ln P_{O_2}$ curve:

$$\Gamma_O = - \frac{2 \gamma_{lv} \text{ (slope)}}{kT} \quad (6)$$

This value can then be substituted into equation (3) to give a relationship between γ_{sv} and $\ln P_{O_2}$,

$$\gamma_{sv} = + \gamma_{lv} \text{ (slope)} \ln P_{O_2} \quad (7)$$

Surface Energy Calculations for SiC, Si₃N₄, and AlN

Figure 12 shows graphs of contact angle vs. time for silicon sessile drop experiments on each of the three substrates studied in our previous work. These graphs indicate a lower contact angle for lower oxygen partial pressures. From these graphs, we obtained data for plots of $\cos \theta$ vs. $\ln P_{O_2}$ at various times as shown in Figure 13. The slopes of these plots were then used in equations (6) and (7) to calculate the adsorbed oxygen populations, Γ_O , and values for the solid-vapor interfacial free energy, γ_{sv} , for each substrate. These results are shown in Figure 14. The values obtained for Γ_O are the same order-of-magnitude as independently obtained results from the literature² on other non-oxide solids in the same temperature range. Adsorbed oxygen populations are all of the order of 10^{14} atoms/cm². The surface free energy values obtained by this technique are reasonably close to the expected values for these materials.

²E. D. Hondros, *Acta Metallurgica*, 16 1377 (1968).

Figure 12-A: θ vs. t for Silicon Carbide.

Figure 12-B: θ vs. t for Aluminum Nitride

Figure 12-C: θ vs. t for Silicon Nitride

Figure 13-A: Cos Θ vs. Ln P_{O_2} for Silicon Carbide.

Figure 13-B: Cos Θ vs. Ln P_{O_2} for Aluminum Nitride.

Figure 13-C: Cos Θ vs. Ln P_{O_2} for Silicon Nitride.

Figure 14

Calculated values for adsorbed monatomic oxygen population, Γ_0 , for each material investigated using $T = 1700^\circ\text{K}$, $\chi_{\text{Lv}} = 800 \text{ erg/cm}^2$, and the equation $\Gamma_0 = 2m\chi_{\text{Lv}}/kT$ where m is the slope of the graph of $\cos\theta$ vs. $\ln p_{\text{O}_2}$. k = Boltzmann constant

Material	Time	Slope	$\Gamma_0 \times 10^{14}$ atoms/cm ²	$\ln p_{\text{O}_2}$	$\chi_{\text{sv}} = -kT\Gamma_0 \ln p_{\text{O}_2} / 2$ ergs/cm ²	χ_{sl}
SiC	0 min	0.050	3.41	-46.1	1844	} ~1200
	8 hrs	0.052	3.55	"	1920	
	0 min	0.050	3.41	-43.4	1736	
	8 hrs	0.052	3.55	"	1807	
AlN	20 min	0.070	4.77	-46.5	2602	} ~2100
	1 hr	0.067	4.57	"	2493	
	20 min	0.070	4.77	-43.3	2423	
	1 hr	0.067	4.57	"	2321	
Si ₃ N ₄	"0 min"	0.032	2.18	-46.5	1190	~ 700
	20 min	0.026	1.77	"	965	~ 500
	"0 min"	0.032	2.18	-43.3	1107	~ 700
	20 min	0.026	1.77	"	899	~ 500

CONCLUSIONS

When vitreous silica was heated to 1430°C in the UMR sessile drop furnace, devitrification took place. In those cases where the samples were exposed to a very low oxygen partial pressure, the devitrification was nearly complete and there was much deformation, but in higher partial pressures of oxygen, there was only surface devitrification and no observable deformation of the sample at temperature. It has been concluded that there would be some degree of devitrification in every case because of the slow rates of heating and cooling required for the furnace, and hence sessile drop studies on vitreous silica must be ruled out with our present facilities.

The portable oxygen cell has been constructed and, as soon as calibration has been completed, can be used in measurement of oxygen partial pressures in the UMR sessile drop furnace and in the facilities of other JPL silicon sheet Task contractors.

It has been found that the adsorbed oxygen populations and the surface free energies of substrates used in sessile drop experiments can be calculated from oxygen partial pressure and sessile drop contact angle data, giving results of the same order of magnitude for adsorbed oxygen populations as independently measured by other investigators for other non-oxide materials (of the order of 10^{-14} atoms/cm² at

1430°C) and giving solid-vapor surface free energy values which are not unreasonable.