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FUNDAMENTALS OF GLASS-TO-METAL BONDING

VII STUDIES ON WETTABILITY OF PLATINUM BY MOLTEN SODIUM SILICATE

SEVENTH TECHNICAL PROGRESS REPORT

BY

MILTON L. VOLPE
RICHARD M. FULRATH
JOSEPH A. PASK

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Seventh Technical Progress Report

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University of California
Berkeley 4, California
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ABSTRACT

Further studies were made of the effect of the nature of the atmosphere on the wetting of gold and platinum by sodium disilicate at 1000°C. The gases used were oxygen, nitrogen, carbon dioxide, argon, water vapor, carbon monoxide, hydrogen, and several mixtures of some of these gases. Test pressures ranged from 10^{-5} mm. of Hg. to almost one atmosphere. A special experimental setup was constructed for this purpose.

The results showed that the wetting of gold was unaffected by the atmosphere and that gold was thus inert. The platinum-sodium silicate contact angle, on the other hand, was dependent on the nature and the pressure of the gas. The results were interpreted on the basis of selective adsorption of the gases at the various interfaces and of absorption of oxygen and hydrogen by platinum.

INTRODUCTION

This paper describes further studies of the basic phenomena involved in glass-to-metal wetting and bonding, which are underway in this laboratory. Earlier results have been reported in parts I and III of this series.^{1,2}

The previous studies represented a broad survey of the wettability of metals by sodium silicate glasses including studies of the effect of glass composition, metal type, and atmosphere. It was found that the wettability of platinum by sodium disilicate was greatly sensitive to small changes in gas pressure. Gold, on the other hand, behaved more like an inert metal in the sense that it did not show this sensitivity. The presently reported studies were undertaken to extend those observations. The objective was to determine more exactly the effect of the nature and pressure of the gas phase upon wetting in a relatively non-reacting liquid glass-solid metal system. Again, the systems under study were sodium disilicate-gold and sodium disilicate-platinum. The degree of wetting was expressed, as before, by the angle of contact made by a drop of glass placed on the metal surface, and measured inside the glass on a plane perpendicular to the interface.

The relationship between the contact angle and the various interfacial tensions is given by the well-known Young's equation^{3,4}:

$$\gamma_{SG} = \gamma_{SL} + \gamma_{LG} \cos \Theta$$

The symbols represent: γ_{LG} - surface tension at the liquid-gas interface, γ_{SG} - surface tension at the solid-gas interface, γ_{SL} - interfacial tension at the solid-liquid interface, and Θ - contact angle. A derivation and detailed discussion of this equation has been given in previous papers,^{1,2,4}

Young's equation was used to interpret the experimental data. In particular, it helped in determining which interfacial tensions were affected by the gases that caused a change in contact angle. In the case of systems in which gross chemical reactions do not occur, such effects are due to the accumulation of gases at the interfaces. From the relationship between interfacial tension and free energy,^{3,5} such an accumulation can only result in the lowering of the surface tension or energy. Therefore, a decrease in cosine (increase in contact angle) could only be brought about by preferential accumulation at the solid-gas surface. On the other hand, an increase of cosine would indicate a decrease in the liquid-solid interfacial tension provided that the gas did not affect the liquid-gas interfacial energy.

EXPERIMENTAL PROCEDURE

Apparatus

To permit the determination of the effect of gases of known composition at low pressures, an essentially gas-tight apparatus was necessary with a minimal possibility of contamination by vapors from the walls, etc. This objective was accomplished by the construction of the apparatus shown in Figure 1, the essential parts of which were fused silica and glass. The fused silica tube was joined to the pyrex part of the apparatus by means of a ground glass joint which was sealed with a low vapor pressure, high melting wax. It was immersed in a furnace heated by electrical resistance elements. Test temperatures within the tube were usually at 1000°C. The furnace construction and its temperature control system, within an overall variation of 5° at 1000°C, were described previously.²

The furnace tank could be evacuated to a pressure of less than 10^{-5} mm. of Hg. The glass apparatus was evacuated through connections to the tank and thus could also be brought down to approximately the same pressure; it could also be isolated from the tank by large bore high vacuum stopcocks. An advantage of this arrangement was the elimination of gas diffusion through the fused silica tube by maintaining a minimal pressure gradient between the inside and outside of the tube.

After appropriate outgassing and isolation of the tube, the leak rate was found to be less than 10^{-3} mm. of Hg. in 10-1/2 hours at room temperature. At the operating temperature a slight pressure increase was observed especially when platinum was present in the test system, but this increase became negligible when the vapor trap closest to the tube (A) was cooled by liquid nitrogen.

Measurement of Pressure.

A directly heated thermocouple type pressure gage was used for measuring

pressures of gas additions to the glass system. It had an effective range of 10^{-3} to one millimeter of Hg. and a precision of approximately $\pm 10\%$. Readings were not corrected for the type of gas whose pressure was being measured.

For a continuous record of pressure changes, the thermocouple pressure gage was connected to a recording potentiometer. The potentiometer was calibrated against the pressure gage scale.

Attempts were made to measure pressures below 10^{-3} mm. of Hg. with a cold cathode ionization gage. This gage was found to effectively "clean up" small amounts of gases in the isolated system and was therefore unusable at very low pressures under static conditions. However, such a gage was attached to the furnace tank surrounding the fused silica tube where "clean up" was not a factor because of continued pumping. It indicated that the lowest attainable pressure was somewhat less than 10^{-5} mm. of Hg.

Measurement of Contact Angles.

Contact angle measurements were made by sighting the drop, illuminated from behind, through a telescope equipped with a filar micrometer eyepiece. Angles were measured directly using a graduated protractor connected to the eyepiece. In a few cases the accuracy of the method was checked by calculating the contact angle from values of the height and base width of the drop assuming it to be part of a sphere. In addition, to test for sphericity the drop parameters were used to calculate its volume. This value was compared with the volume calculated from the weight and density of the drop. The calculated angles were always slightly larger than those directly measured, but the difference was within the experimental error of measurement. The estimated precision of the reported values is $\pm 5^\circ$.

Materials

The gases used were oxygen, nitrogen, argon, carbon monoxide, hydrogen, carbon dioxide, and water vapor. All of these, except the latter two, were

further treated by passing through a trap immersed in liquid nitrogen to remove condensible impurities during the course of filling the storage bulbs shown in Figure 1. Water vapor and carbon dioxide were fractionally distilled into the bulbs. The storage bulbs after repeated flushings with the particular gas were filled nearly to atmospheric pressure.

Sodium disilicate glass was synthesized from C.P. sodium carbonate and quartz sand with an analysis of 99.98% SiO_2 . The proper mixture was fused at 1500°C in a platinum container. Fining was accelerated by rotating the crucible in an inclined position. The glass was then vacuum degassed at a temperature of 1200°C and stored in an evacuated desiccator. Heatings of 10 mg samples at 1000°C in vacuum over a period of 24 hours indicated average weight losses of 0.06 to 0.10 percent per hour, presumably of soda.

Platinum and gold were of the highest purity commercially available. Prior to each use the metal specimens were washed in hydrofluoric acid, rinsed in distilled water, lapped with #600 paper, washed in distilled water and dried. Other cleaning procedures that were tried on platinum were: thermal etching at 1300°C , heating in oxygen at 1300°C , etching in aqua regia, and polishing with levigated alumina. The method of treatment showed no significant effects.

Experimental Technique

A description of the experimental procedures follows with references to parts of the apparatus shown in Figure 1. A 10 mg. glass piece was first attached to the metal surface of 2 sq. cm. area by heating for a few seconds in a bunsen flame. The glass-metal sample was then placed on a flat silica plate which was fused to a silica rod connected to a Pyrex glass cap through a graded seal. The platinum -10% rhodium thermocouple was used to measure

and control the temperature. It was brought in through vacuum-tight seals in the Pyrex glass cap and was adjusted to touch the metal test plate. This entire assembly was lowered into the furnace silica tube and sealed through ground glass joints with high vacuum grease or low vapor pressure high melting wax depending upon the temperatures developed at the joints.

The entire system was pumped down to a pressure of 10^{-5} mm. Hg. with stopcocks (1) and (2) open to provide the maximum pumping speed. The furnace was then gradually heated to the operating temperature and pumping continued for about ten hours or, with platinum specimens, until the contact angle decreased to 20° . Gases were then added to the system from the storage bulbs with stopcocks (1) and (2) closed and with trap (A) cooled in liquid nitrogen, except for carbon dioxide and water vapor. When controlled low pressures were desired, additions were first made with stopcock (3) also closed. The pressure in (C) was then adjusted to a predetermined value by manipulation of stopcock (2). The gas was expanded into the silica tube by opening stopcock (3). Pressure and angle readings were made periodically for two hours or until no change was observed.

RESULTS AND DISCUSSION

Results in Vacuo

When sufficient pumping time was allowed for the removal of surface and bulk contaminants, the contact angle of glass on platinum was always found to decrease from an initially high value to 15-25°. This angle was always obtained at the lowest attainable pressures and is in agreement with the measurements made in the all-metal furnace used for the earlier experiments.²

On the other hand, no amount of pumping decreased the contact angle below 60° for glass on gold. This result is also in agreement with the previous findings.

Results in Presence of Added Gases

Sodium Disilicate on Gold

The contact angle of glass on gold at 1000°C. was found to be unaffected by either oxygen, carbon dioxide, water vapor or hydrogen at pressures ranging from 10^{-3} mm. of Hg. to nearly one atmosphere. Specific values are listed in Table I. For each of these gases, the angle remained at the "in vacuo" value of about 60° in test runs that lasted as long as 6 to 10 hours. Thus, it can be inferred that these gases did not influence any of the interfaces in the gold-glass system; that they were all equally adsorbed would seem highly improbable.

In addition to lack of evidence for surface adsorption no evidence of bulk absorption was obtained. In an isolated system no change in pressure with time was noted.

Behavior of Platinum

Platinum was found not to be inert to all gases at 1000°C. in an isolated system. With a low pressure of hydrogen or oxygen and with a vapor trap cooled by liquid nitrogen, a gradual decrease in pressure was obtained which was deduced to be primarily due to the solution of these gases in the platinum. In one series

with oxygen as the added gas, the vapors in the trap were collected and analyzed with a mass spectrograph. The analysis⁶ showed the composition to be approximately one-third carbon dioxide and two-thirds water vapor. The origin of these gases has not been determined. Possible sources were the reaction of oxygen with hydrogen and carbon dissolved in the platinum, or with stopcock grease. As a matter of fact evidence of condensable gases was present in all experiments with platinum; thus, a liquid nitrogen cooled trap was maintained in the system except for the experiments with water vapor or carbon dioxide.

The drop in pressure with time that occurred only with oxygen and hydrogen was considerably greater than that expected from the above reactions indicating that these gases were dissolved in platinum. Experimental evidence is presented in Figure 2 for several systems that were properly trapped: with nitrogen the system maintained essentially a constant pressure with time; with oxygen and no platinum, the pressure within the system decreased slightly probably due to reaction with the stopcock grease; and with oxygen and platinum, the pressure dropped appreciably.

This pressure decrease could not be explained on the basis of adsorption alone because a layer thickness of 500 to 1000 times the normally expected thickness of only a few angstroms would be required. A buildup of an oxide is also untenable because no stable bulk compounds of platinum should exist at 1000°C.⁷ Absorption, thus, must occur. Upon introduction of successive amounts of oxygen or hydrogen, the sorption rate decreased, but the equilibrium saturation was not completely reached within the experimental times. Similar findings have been reported in the literature.⁸ Because of this solubility, it was impossible to obtain data below 10^{-3} mm. of Hg. relating contact angle to pressure in atmospheres of hydrogen and oxygen. Even at higher pressures, the values recorded for these gases must be considered to be only approximate averages.

Sodium Disilicate on Platinum

The effect of oxygen, nitrogen, argon, carbon dioxide, water vapor, carbon monoxide and hydrogen on the contact angle of this system was studied at a temperature of 1000°C . The pressures covered a wide range of values but most tests were in the range from 10^{-3} to one millimeter of Hg. Some of the results are listed in Table II. Nitrogen, carbon dioxide and argon with increasing pressure caused a slight increase in the contact angle; oxygen, a slight decrease; and water vapor, carbon monoxide and hydrogen, an appreciable increase.

The relatively small increase noted with the first group could be due to an accumulation of a small partial pressure of water vapor at the higher total pressure because of suspected cold trap inefficiency. In oxygen the angle decreased steadily with increasing pressure reaching a value of 6° at an atmosphere of oxygen or air. In water vapor appreciable change in the contact angle occurred in the pressure range of 1 to 5×10^{-4} mm. of Hg. In carbon monoxide the maximum contact angle was reached in the range of 1 mm. of Hg. In hydrogen, the maximum angle was attained at pressures near 10^{-2} mm. of Hg; at higher pressures, the angle appeared to decrease slowly. In all cases the maximum attainable angle appeared to be in the range of 60 to 70° .

Table III shows the effect of adding nitrogen and oxygen to a system containing water vapor. The addition of nitrogen even up to a pressure of one mm. of Hg. did not affect the contact angle; a small amount of oxygen decreased the angle to values obtained with that amount of oxygen alone. Thus, it appears that oxygen easily displaces water vapor adsorbed on the surface of the platinum regardless of relative amounts of the gases. Carbon monoxide was also displaced by oxygen, probably by the formation of carbon dioxide.

Earlier studies without the use of a silica tube² showed an increase in contact angle, from 15° to 60° , with an increase of pressure, from 10^{-5} mm. Hg. up to about 10^{-2} mm. of Hg. The increase was achieved by isolating the system.

On the basis of the present data this rise in contact angle can now be attributed to the adsorption of accumulated water vapor or some other adsorbable compound vapor in the absence of oxygen. The furnace that was used contained hot tungsten and molybdenum, both of which are good "getters" and should have removed the oxygen leaving nitrogen and vapors.

Accurate measurement of gas pressures was difficult. This situation was particularly so in the case of water vapor and carbon dioxide because of the formation of these gases during the experiments. Such a condition was critical considering the fact that the region of sensitive variation of contact angle with pressure occurred at very low water vapor pressures. Measurements with hydrogen and oxygen were also difficult because of the solution of these gases in the platinum.

Another complication that affected equally all measurements should be mentioned. In all cases the contact angle changes that were reversible became more sluggish with increase of test time. For example, a gas condition that would cause a forty degree change in contact angle in an hour would, after a test period of about twenty hours, cause only a twenty degree change in the same time. Eventually, the drop became essentially immobile. The magnitude of this effect seemed to depend upon the glass sample and the nature of the experiments. The sluggish behavior appeared to be caused by some slow change in the glass rather than in the platinum and could possibly be due to the loss of soda. This phenomenon is still under investigation.

CONCLUSIONS

The results demonstrate that the effect of the gas phase upon the wetting of a surface active or non-inert metal by molten glass is not only significant but also quite specific. The magnitude of the effect was found to depend upon the nature of the metal and also upon the composition and pressure of the gas.

Gold acted as an inert metal as judged by the constancy of the contact angle (approximately 60°) with sodium disilicate glass with all gases at all pressures. This behavior indicated that none of the gases were adsorbed at the various interfaces. Platinum, however, behaved as a non-inert metal. At the test temperature of 1000°C it showed both specific adsorption and absorption but no evidence of formation of bulk compounds. Hydrogen and oxygen were found to be readily absorbed.

Young's equation can be applied to the wetting results for the interpretation of the effect of a gas phase upon the various interfaces. Carbon dioxide, nitrogen, and argon did not affect the platinum-glass system at low pressures; the contact angle of $15\text{--}20^\circ$ was similar to that obtained in vacuum. Thus, it appears that these gases were probably not adsorbed on platinum. Additions of water and carbon monoxide caused an increase of the contact angle to about 60° , indicating that adsorption occurred primarily on the surface of the metal.

Oxygen caused a steady decrease in contact angle with increase of pressure suggesting that it has a greater effect on the glass-metal interfacial tension than on the surface tension of the metal. This particular observation is significant because experiments indicated that platinum has a considerably greater affinity for oxygen than water vapor which caused an appreciable increase in contact angle. The strong evidence for absorption of oxygen by platinum suggests that the possible path for movement of oxygen to the glass-metal interface is through the metal.

The large increase in contact angle with hydrogen indicates that it primarily reduces the surface tension of platinum. However, hydrogen may also migrate to the glass-metal interface because of existing evidence of its absorption by platinum. Although its effect on the interfacial tension is relatively minor, a positive effect is suggested by the small decrease in contact angle with higher pressures of hydrogen.

The studies to date have indicated that generalizations can be made on the basis of Young's equation. Any gas that only adsorbs on the metal surface, thus lowering its surface energy, will cause an increase in contact angle. If the gas is also absorbed, it can diffuse to the metal-glass interface where it may also lower the interfacial energy. The effect on the contact angle will be determined by the balance of forces.

Such specific knowledge of the effect of these gases upon the various interfaces now makes it possible to do some speculating or hypothesizing in regard to their influence on adherence between glass and metal. This adherence is determined to a large extent by the "work of adherence," defined as the energy expended in cleanly separating the glass from the solid.³ Only changes in the glass-metal interfacial tension, as for example brought about by gas adsorption at the interface, would be expected to affect the strength. Oxygen and possibly hydrogen are such gases. The room temperature strength of a glass-to-metal seal would be complicated by the existence of strains due to a mismatch of thermal expansions and possibly other unknown factors.

Experiments designed to test this hypothesis by measurement of the strength of seals prepared under various conditions are now being pursued. In addition, studies will be extended to other systems such as sodium disilicate-iron.

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TABLE IEffect of Atmosphere on Contact AngleNa₂O. 2Si O₂ on Gold(Pressures in 10⁻³ mm. of Hg.)

<u>Gas</u>	<u>10</u>	<u>100</u>	<u>1000</u>
O ₂	65°	65°	64°
CO ₂	61°	60°	--
H ₂ O	66°	65°	65°
H ₂	64°	63°	66°

Contact Angle in Vacuum (10⁻⁵ mm. of Hg.):
 63° ± 2°

TABLE IIEffect of Atmosphere on Contact AngleNa₂O. 2Si O₂ on Platinum(Pressures in 10⁻³ mm. of Hg.)

<u>Gas</u>	<u>10</u>	<u>100</u>	<u>1000</u>
O ₂	15°	13°	10°
N ₂	22°	21°	32°
CO ₂	20°	29°	--
A	17°	20°	37°
H ₂ O	58°	63°	--
CO	40°	55°	66°
H ₂	56°	51°	49°

Contact Angle in Vacuum (10⁻⁵ mm. of Hg.):
 22° ± 3°

TABLE IIIEffect of Gas Mixtures on Contact AngleNa₂O. 2Si O₂ on Platinum

% H ₂ O	<u>100%</u>	<u>60%</u>	<u>20%</u>	<u>0%</u>
Gas Mix- ture				
H ₂ O + O ₂	60°	17°	15°	13°
H ₂ O + N ₂	60°	60°	60°	20-30°

Initial water vapor pressure: 5×10^{-2} mm. of Hg.

FIGURE 1 — DIAGRAM OF APPARATUS

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FIGURE 2 - SORPTION OF GASES BY PLATINUM
(Temp. = 1,000°C. - Trap Cooled By Liquid Nitrogen)

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