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Author
Tso, Stephen Ta-Chuen

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THE WETTING BEHAVIOR OF \( \text{B}_2\text{O}_3 \) CONTAINING GLASS ON GOLD

Stephen Ta-chuen Tso
(M. S. thesis)

December 1976

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THE WETTING BEHAVIOR OF
$\text{B}_2\text{O}_3$ CONTAINING GLASS ON GOLD

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THE WETTING BEHAVIOR OF B₂O₃ CONTAINING GLASS ON GOLD

Stephen Ta-chuen Tso

Materials and Molecular Research Division, Lawrence Berkeley Laboratory and Department of Materials Science and Engineering, University of California, Berkeley, California 94720

ABSTRACT

The effect of chemisorbed contaminants on the wetting behavior of sodium borate glass and gold was studied by the sessile drop technique. The results showed that the structure of the gold surface was constant and that its surface energy did not change in the pressure range from 10⁻⁸ atm to 10⁻⁵ atm at 900°C. Water vapor changed the composition of the glass and greatly enhanced the evaporation of glass, but adherence did not develop. Adherence developed in an oxygen atmosphere >15µ mHg (>2 x 10⁻⁵ atm) due to a decrease of the interfacial energy caused by saturation of the gold/glass interface with the oxide of the substrate.
I. INTRODUCTION

In a thick film system, glass is used to bind gold powder to a glass or ceramic substrate. Because of the need for bonding of gold to glass for such applications as well as others, the gold-glass system is an important system to study. Moreover, it is expected that a noble metal like gold would provide a simple model system to study the glass-metal wetting behavior.

Previous work in this laboratory\textsuperscript{1,2} investigated the wetting behavior of platinum and glass. Sessile drop experiments were conducted to study this behavior. It was found\textsuperscript{2} that the contact angle decreased in oxygen. The contact angle also decreased with the removal of carbonaceous contaminants. It was proposed that carbon chemisorption on the Pt surface reduced the surface energy, and that removal of chemisorbed carbon by oxidation increased the Pt surface energy. This increase in Pt surface energy accounted for the decrease of contact angle.

In the present study, the glass-gold system was investigated in some detail. The possibility of carbon contamination on the Au surface was of particular interest. In contrast to the Pt case, however, there was no evidence of carbon chemisorption on the gold surface. The gold surface was found to be inert to the carbonaceous vapor. Different atmospheres were introduced to reveal their effects on the wetting behavior of gold and glass. Explanations and comparisons of the results were made.
II. THEORY AND LITERATURE SURVEY

1. Adsorbates on Gold Surfaces

Recent research studies have shown that although gold has no stable bulk compounds at elevated temperature, there are some stable compounds of gold at the surface. It was proposed that the readily changing valency of surface atoms is the reason for the stability of surface compounds.3

A clean gold surface can be obtained by prolonged heating at a high temperature in a high vacuum or by argon bombardment followed by annealing at a lower temperature. Such a clean surface, once obtained, is extremely difficult to maintain. It was shown3 that oxide formation became rapid above 500°C at a pressure of 10⁻⁶ torr and that the resulting gold oxide was bound strongly. Heating to 800°C for 12 hours in vacuum (6 x 10⁻⁶ torr) produced no reduction in the oxygen peak height in Auger Electrospectroscopy Analysis. On the other hand, adsorbed hydrogen atoms will recombine to form hydrogen molecules and leave the Au surface in the temperature range from 77°K to 340°K.4 Chemisorption of hydrocarbon was reported to be difficult on Au surfaces5 due to the large activation energy of the dissociative chemisorption. On water vapor-pretreated films,6 at least part of the H₂O monolayer is adsorbed irreversibly. The H₂O molecules can be displaced by excessive H₂S. However, H₂S can not displace the chemisorbed oxygen. Interaction between the chemisorbed H and Co on polycrystalline gold surface was reported.7 The preadsorbed CO increased the rate of adsorption of atomic hydrogen.
2. Oxygen Chemisorption on Gold Surfaces

Usually, the easiest way to remove impurities on the gold surface is to heat the gold in oxygen at elevated temperature. This method was partially successful in removing impurities by oxidation but produced a stable oxygen-covered surface. It was observed that the probability of an oxygen atom, incident upon the gold surface, being reflected into the gas phase will decrease rapidly above 350°C. This result is consistent with the postulate that the metal-O bonding involved the mixing of the O 2p orbitals with the s state of the metal. No reaction occurred with ground state O. The oxygen molecules were observed to react only with hot gold, suggesting that oxygen molecules dissociated on chemisorption and then formed gold oxide. The rate of oxygen adsorption falls exponentially with surface coverage. As the temperature rises the amount of adsorption increases.

At high temperatures the growth of a thick oxygen film has been observed on gold by optical methods. More than one form of adsorbed O was present. Domange proposed that the structure of the oxide layer was similar to that for two dimensional sulfide layers on gold. He suggested that sulfur formed 3 structures on the (110) face before the saturation structure. In the first stage the S is not organized, but then forms a C(4 x 2) structure. These two stages constitute a precursor state, after which S is adsorbed bidimensionally. Another analogy is the Cl adsorption on the gold surface. Three types of Cl species were observed on the surfaces as the surface reaction proceeded. These were (1) Cl adsorbed at the metal surface, (2) the ion of the surface metal chlorides and (3) Cl₂ or Cl adsorbed on the chlorides.
3. Effect of Chemisorbed Oxygen on Au Surface Properties

It was reported\(^{15}\) that a large reduction of Au-Au friction was caused by chemisorbed O. However, in contrast to Au-Au friction, Si\(_2\)O\(_2\)-Au friction was enhanced by chemisorbed O. This high static friction was attributed to the electrochemical formation of bonds or bridges which require the presence of an adsorbed O layer. Allessandrini\(^{16}\) also confirmed that the reaction between Au film and thermally oxidized Si was strongly influenced by the partial pressure of O\(_2\). When samples were held at an elevated temperature in a dynamic vacuum, an apparent decomposition of the oxide film with subsequent migration of Au along the exposed Si surface was observed.

Moore and Thorton,\(^{17}\) using Au\(^{198}\) in the sessile drop experiment found indications that gold diffused into the silica lattice at high oxygen pressures. During vacuum heating no diffusion occurred and there were no indications of reaction or adhesion between the gold and the silica. The observed bonding at high oxygen pressures was evident primarily at the outer rim of the gold drop. In addition, the bonding strength was weaker in air than in oxygen (at the same oxygen partial pressure).

4. Theory of Mechanism of Adherence\(^1\)

The layer of oxygen at the metal surface is held by covalent ionic bonds created by partial transfer of electrons from the surface metal atoms to the oxygen atoms. If the superposition of a glass on this surface does not cause the oxygen and the associated metal ions to be dissolved, these surface oxygen atoms will intercoordinate with the surface oxygen atoms of the glass. This occurrence will result in
increased screening of the silicon ions and lower the internal energy of the glass at the interface. This conditions produces a strong chemical bond.

If the oxide is completely dissolved from the metal surface before the particular glass composition is saturated with that oxide, the surface of the glass comes into direct contact with the metal atoms. The mobility of the molten glass will cause its surface at the interface to rearrange itself to a minimum energy state consisting essentially of a surface of charge-satisfied oxygens and well-screened silicon atoms. Since the oxygens of the glass are already bonded, no electron transfer from the metal to the glass can occur. Such an interface results in a weak Van der Walls bond.

If the glass composition is saturated before the available oxide is completely dissolved from the metal surface, strong bonding will occur. As before, intercoordination then occurs between the oxygens of the adsorbed oxide and the oxygens at the glass surface.
III. EXPERIMENTAL PROCEDURES

1. Preparation of Specimens

Gold of 99.95% purity was obtained from the Lawrence Livermore Laboratory. The foil was 20 mils thick and cut into 1/2" x 1/2" squares. The gold plates were polished through a set of dry polishing papers and given a final polish on a lap wheel with 0.3 micron alpha-alumina powder. All the plates were cleaned with an ultrasonic cleaner, degreased in acetone, alcohol and distilled water; then stored in a dessicator before use. Heat treatment in any atmosphere was possible and was done if necessary.

The glass was obtained from Dow Chemical Company. Its composition was 94% B₂O₃ and 6% Na₂O. Since the glass usually contained a great deal of dissolved gas, it was vacuum degassed. The glass bars, as received, were broken into pieces, put into an alumina crucible, and heated in vacuum (1 x 10⁻⁵ torr) at 900°C until the glass ceased to bubble. The average time for the degassing was one day. Only the central portion of the degassed glass that was not in direct contact with alumina crucible was used for the sessile drop experiments. Such degassed glass was stored in a dessicator until ready to be used.

In the process of degassing, some of the vapor condensed on the cold part of the furnace. In order to understand the compositional change of the glass during the process of degassing, both degassed and as-received glass were analyzed. The results were:

<table>
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<th>Degassed</th>
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<tr>
<td>B</td>
<td>Principal constituent in each.</td>
<td>6. %</td>
</tr>
<tr>
<td>Na</td>
<td>6. (?)</td>
<td>3. (%)</td>
</tr>
<tr>
<td>K</td>
<td>1. (%)</td>
<td>1. (%)</td>
</tr>
<tr>
<td>Al</td>
<td>0.01</td>
<td>0.3</td>
</tr>
</tbody>
</table>
As Received Degassed

Si  .005  .006
Mg  .002  .003
Fe  .015  <.01
Ca  .006  .004
Cu  <.001  --

Apparently, sodium oxide was boiled off the glass while aluminum ion diffused into the glass from the crucible in the process of vacuum degassing.

In order to determine the stability of degassed glass, the weight of a drop of glass was measured and recorded successively after heating for 1, 4, 20 and 40 hours. The surface area of the glass drop was also measured. The evaporation rate was calculated from the weight change and the surface area.

2. Wetting Test Procedures

Two furnaces were used during this research. The first furnace contained a tantalum tube resistance heating element as shown in Fig. 1. The total pressure in this furnace was about $1 \times 10^{-5}$ torr. An alumina dee was placed inside the tantalum tube to support the specimens. A Pt - Pt/Rhodium thermocouple and a temperature controller were used to measure and control the furnace temperature. The specimen was placed in the central portion where the temperature was isothermal and monitored.

The second furnace is a one-inch diameter fused silica tube furnace as shown in Fig. 2. A portion of the silica tube was in the interior of a clam shell Kanthal resistance wire furnace. The advantage of this arrangement was that the atmosphere inside the tube furnace was completely separated
FIG 1.

Schematic of contact angle furnace I.
Fig. 2

Schematic of fused-silica tube furnace.
from the heating element; atmosphere control was thus possible. Temperature was measured and controlled by a Platinel #1 thermocouple at the center of the furnace. The tube was fitted with an optically flat end for viewing. The contact angle can be measured in the same way as described before.

Contact angles in both cases were measured by sighting with a telescope fitted with a protractor and movable cross hairs. The contact angle was measured with an estimated accuracy of ±2°. All measurements were made at 900°C.

Sufficient time was taken in all of the experiments to assure that equilibrium contact angles had been achieved. Six hours was the average time for each run. Sometimes the heating lasted much longer to determine if the equilibrium contact angle had really been reached.

3. Atmosphere Control

The fused silica tube was fitted with a quarter inch tubulation for gas input. The pressure in the furnace was measured by an ion gauge (<10⁻³ torr) or by a thermocouple gauge (>10⁻³ torr). A leak valve and a flow meter were used to control the flow of the gas introduced into the tube furnace.

The nitrogen gas used was obtained from Lawrence Berkeley Laboratory; its reported purity was 99.999%. The oxygen gas used was also obtained from LBL and was 99.6% pure. The major impurities were argon (4000 ppm max.), nitrogen (500 ppm max.), air (25 ppm max.) and carbon dioxide (1.5 ppm). The water vapor in the experiments was obtained by evaporation of distilled water from a sealed container connected to the furnace tube; the other end of the furnace tube was connected
to the vacuum pump. During the course of the experiments a liquid nitrogen cold trap was activated as required.

4. Adherence

Adherence was checked qualitatively. After each experiment, the gold foil was bent with the glass drop on it. If there is no adherence, the whole glass drop pops off leaving a very smooth interface. If there is adherence, the glass drop breaks up into slivers as shown in Fig. 3.
Adherence of glass to gold indicated by appearance of fracture in the thin glass layer introduced by bending the metal piece (magnification ~ 128X).
IV. RESULTS AND DISCUSSION

1. Stability of Au Surfaces

Sessile drop experiments were conducted with sodium borate glass on gold. The gold foil was treated in three different ways: (1) as received, (2) heat-treated in air at 900°C for more than 8 hours, (3) heat-treated in vacuum (10^{-8} atm) at 900°C immersed in carbon powder for 24 hours. These treatments of the gold foil were conducted to investigate whether the wetting behavior of glass on gold was affected by possible carbon contamination of the gold surface. The heat treatment in air remove the carbon contamination from the gold surface by oxidation. The heat treatment in vacuum with the gold immersed in carbon powder was expected to produce a carbon contaminated surface if such contamination was possible.

Sessile drop experiments were performed in vacuum. The observed contact angles varied at the beginning for the differently-treated gold pieces but all converged to 46° after 3 hours. This contact angle of 46° remained unchanged for another 10 hours indicating that the gold surface had reached a constant structure irrespective of treatment which remained unchanged. It is believed to be the final equilibrium contact angle since it was reproducible in the pressure range of 10^{-8} atm to 10^{-5} atm.

The sensitivity of the sessile drop experiment to gold surface contamination can be illustrated as follows. The surface energy of the sodium borate glass is approximately 80 ergs/cm² at 900°C. The gold surface energy is 1300 to 1700 ergs/cm². Using Young's equation (1)

\[ \gamma_{sv} - \gamma_{sl} = \gamma_{lv} \cos \theta \]  

(1)
and assuming that the interfacial energy $\gamma_{sl}$ and the glass surface energy $\gamma_{lv}$ remain constant, a simple calculation shows that even a minor change in the gold surface energy of 12 ergs/cm$^2$, which is less than one hundredth of the original value, will cause a change of contact angle from 46° to 60°. The unique equilibrium contact angle thus indicated that the carbon treatment resulted in no specific adsorption of carbon on the gold surface. Considering the possibility that all of the gold surfaces might have been saturated with carbon at very low carbonaceous gas partial pressure due to carbonaceous vapor backstreaming from the oil diffusion pump or mechanical pump, an experiment was performed with an activated liquid nitrogen cold trap; using a gold specimen that was heat treated in air, a contact angle of 46° was observed. Therefore, the gold surface was inert to carbon adsorption from carbonaceous vapor. Such behavior, however, could be explained by postulating the development of a chemisorbed oxygen layer in all cases, as described by Chesters and Somorjai, which would account for the steady state contact angle. Experiments with and without a cold trap using platinum indicated carbon adsorption.

2. Effect of Ambient Atmospheres

Three different kinds of atmosphere were introduced into the furnace to reveal their effects on the wetting behavior. They were water vapor, nitrogen and oxygen.

a) Water Vapor. At a pressure of 500μmHg the contact angle varied in the range of 39° to 60°. Bubbles were produced at the gold-glass interface, and the glass drop vaporized and disappeared in less than 2 hours. In order to make a comparison of the rate of evaporation
in oxygen and in water vapor, comparative rates were measured. The evaporation rate in oxygen was \(2.8 \times 10^{-7}/\text{sec-cm}^2\) and in water vapor it was about \(1.8 \times 10^{-4}/\text{sec-cm}^2\). It is believed that the water vapor was absorbed by the glass, and the change in its composition highly enhanced the evaporation rate of glass. The bubbles observed at the interface suggested that it was a boiling phenomenon. The gold surface provided nucleation sites for these bubbles.

b) Nitrogen. At a pressure of 1 atm the wetting behavior of sodium borate glass on Au was found to be the same as that in the vacuum. Steady state contact angles of 46° were obtained. Nitrogen neither induced any reaction nor changed the surface energy of the gold or glass.

c) Oxygen. At pressures greater than 15 \(\mu\text{mHg}\) the contact angle was 6°; at lower pressures, 46°. The observed relation between the steady state contact angle and the partial pressure of oxygen is shown in Fig. 4. The procedure consisted of first obtaining the steady state contact angle of 46° in vacuum and then introducing the oxygen into the furnace. A kinetic effect was observed; the higher the oxygen partial pressure, the shorter the time it took for the contact angle to decrease from 46° to 6°. At 15 \(\mu\text{mHg}\) the angle remained unchanged for about 20 minutes before it started to decrease to 6°. It is presumed that this kinetic effect is associated with the growth of an oxide layer of some unknown but steady state thickness. It was also observed that in the oxygen atmosphere the glass drop tended to move in one direction until it reached the edge of the gold substrate. This phenomenon was not observed in vacuum, nitrogen or water vapor. As the glass drop moved, a film of glass was left behind the drop, which was so thin that it could not be
Contact angle vs. oxygen pressure at 900°C for soda borate glass on gold.
seen with the telescope. It became apparent when the gold pieces were bent to check the adherence because it fractured into slivers which indicated the presence of adherence. Adherence was also observed at the rim of the glass drop. With continued heating, the adherence deteriorated. It was also observed that the thin layer of glass vaporized after 4 hours of heating in oxygen.

3. Mechanism of Adherence

No adherence was observed in vacuum, nitrogen or water vapor. Adherence was only observed in oxygen. This result is consistent with the theory proposed by Pask and Fulrath which says that adherence will occur when the glass at the interface is saturated with the metal oxide and a continuous electronical structure is formed across the interface.

The fact that in oxygen a film of glass was left behind the glass drop suggested that its composition was different from the bulk glass. It is postulated that the film tended to be saturated with gold oxide at the interface, because an oxide layer is formed on gold in air and good adherence was observed. Deterioration of adherence with prolonged heating was attributed to diffusion of gold oxide into the glass resulting in loss of saturation at the interface. The rim of the glass drop, however, still retained its adherence. This condition can be accounted for by retention of saturation due to diffusion of oxygen along the interface from the gold/glass/air periphery line.

4. Interfacial Energy Considerations

a. Oxygen. The decrease of contact angle to 6° with the introduction of oxygen can be interpreted with the use of Young's equation (Eq.1). It has been reported that oxygen has little effect
on the surface energy of glass; \( \gamma_{lv} \) thus remains unchanged in oxygen. Therefore, a decrease of contact angle indicates that the value for \( (\gamma_{sv} - \gamma_{sl}) \) must have increased. Formation of a chemisorbed oxygen layer on the gold surface could only decrease \( \gamma_{sv} \). So \( \gamma_{sl} \) must decrease sufficiently to account for the decrease of the contact angle. This decrease in \( \gamma_{sl} \) above 15 \( \mu \text{m Hg} \) can be attributed to oxygen diffusing into the glass/gold interface with the formation of a chemical bond. The movement of the drop which occurred only in oxygen can also be explained on the basis that the glass has a driving force to dissolve the oxide layer and any slight unbalance of wetting driving forces on the periphery of the drop could cause the drop to move. If we assume that \( \gamma_{sv} \) remains unchanged over the pressure range, i.e., the Au surface became saturated at the low partial pressure of oxygen, the decrease of contact angle from 46° to 6° corresponds to a decrease of interfacial energy by 24 ergs/cm².

b. Water Vapor

Water vapor has been shown to decrease the surface energy of glass which should decrease the contact angle. However, an increase of contact angle was observed which indicated an overall decrease in the driving force for wetting. The lack of adherence in specimens tested in water vapor suggests that \( \gamma_{sl} \) remains essentially the same as in all cases examined at low ambient pressures. The higher contact angles then can only be accounted for on the basis of a reduction of \( \gamma_{sv} \).

Support of this analysis is provided by Roland who reported adsorption of \( \text{H}_2\text{O} \) on gold. Moreover, it is reported that when heating gold in \( \text{H}_2\text{O} \) vapor, the oxygen peak in AES analysis was observed. Since hydrogen atom can not be detected by AES, the observed oxygen peak also suggests the adsorption of \( \text{H}_2\text{O} \) on gold.
V. CONCLUSIONS

Under the low pressure ambient conditions of the experimental setup the driving force for wetting of gold by sodium borate glass ($\gamma_{lv} = 80$ ergs/cm$^2$) at 900°C is a reduction of 56 ergs/cm$^2$ of the surface energy of the gold by the glass. This value is based on the observed contact angle of 46°.

It was also determined that sessile drop experiments in an oxygen atmosphere >15μmHg resulted in a decrease of the gold/glass interfacial energy. Development of adherence, in turn, was observed only in oxygen. These results are consistent with previous findings. The decrease in interfacial energy is associated with saturation of the interface with the oxide of the substrate resulting in chemical bonding between gold and glass.

Interpretation of the experiments indicated that the Au surface energy was unchanged in the pressure range of $10^{-8}$ atm to $10^{-5}$ atm at 900°C. This also indicates that the structure of the gold surface remains constant. Auger analysis of these surfaces would be most interesting. Possibilities are the presence of a clean surface or a surface with a chemisorbed oxygen layer. At pressure >2 x $10^{-5}$ atm (15μmHg) of oxygen, then, a steady state oxide layer with a lower $\gamma_{sv}$ would be expected. Also, the nature of the Au surface in water vapor could be different.

Water vapor appeared to be absorbed by the glass as indicated by a higher vaporization rate of the glass with boiling occurring at the glass/metal interface. However, no reaction with gold or development of adherence occurred, as observed for platinum and sodium borate.
ACKNOWLEDGMENT

I would like to express my sincere thanks to Professor J. A. Pask for his continuous guidance and instruction on this work.

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