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CORROSION STUDIES IN MOLTEN ALKALI CARBONATES: PART I. SILVER METAL

by

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that metals such as Pt and Ag show attack, both in static experiments¹ and in electrochemical oxygen evolution studies². Results from recent potentiostatic corrosion studies³ for a series of metals confirm these observations for corrosion in fused carbonates. The present communication reports the results of an investigation designed to gain further insight on the chemical and thermodynamic aspects of silver corrosion in carbonate melts.

EXPERIMENTAL

Materials: As described in the preceding papers from this Laboratory^{1,2}, the alkali carbonates (Reagent Grade chemicals) were dried to constant weight under CO₂ atmosphere at 600°C and stored over P₂O₅ until required for use. For the present corrosion studies the ternary eutectic mixture⁴ Li₂CO₃: Na₂CO₃: K₂CO₃: 4:3:3 (mole ratio) m.p. 397°C, was used for all the measurements.

The foils of Ag were prepared from Ag sheet metal, spectroscopic grade purity, and used without further pre-treatment.

Apparatus: The arrangement of the corrosion specimen, gas inlet and thermocouple tubes, and electrodes in the high temperature assembly are shown in Figure 1. In line with the limited observations^{1,3}, a crucible of a gold-palladium alloy (80%-20%) was selected for containing the molten carbonates since this alloy (apparently as resistant to attack as Au) has a somewhat greater tensile strength than Au at high temperatures. Both the corrosion specimen and the crucible had contact wires (of the same metals as specimen and crucible, respectively) so that electrochemical dissolution rates as well as static (equilibrium-type) measurements could be made.

Measurements and Results: About 60-70 g. of the ternary molten carbonate mixture were used in each measurement. The atmosphere in the furnace chamber and above the melt was purged with a continuous but slow stream of dry CO_2 from the gas inlet tube (Figure 1).

The behaviour of Ag metal, suspended freely but not as part of a closed electrical circuit, was investigated for the temperature range 650-900°C. A series of experiments in which an anodic current was passed through the specimen were also undertaken to investigate the faradaic corrosion of Ag in the same temperature range and melt environment. The data and results for these two series of experiments are in Tables 1 and 2 respectively. The corrosion specimens after each experiment were treated with hot water to remove adhering carbonates and soluble products, and the weight changes were noted by direct weighing with a precision analytical balance. Where an anodic current was imposed on the specimen both the "apparent" current density and total time of electrolyses were observed. The measurements were not made potentiostatically, and it is sufficient to note from the polarization curves for Ag, reported previously³, that in this range of current densities the anodic overpotentials are very nearly zero (relative to the Pt- O_2 reference electrode).

In all of these measurements no direct stirring of the melts to hasten the dissolution processes was used but agitation, due to convection effects, is not ruled out. The solidified carbonate melts, after the experiments, were examined for ionic silver salts, but analytically detectable amounts of Ag^+ ions were not found. The silver is found only as the reduced metal in these solidified melts.

A separate series of experiments were carried out in which AgCl , Ag_2SO_4 , Ag_2CO_3 , and Ag_2O were added, each separately, to the ternary molten carbonate mixture at $600^\circ\text{--}700^\circ\text{C}$. The thermal instability of Ag_2O and Ag_2CO_3 is well known but both AgCl and Ag_2SO_4 are stable at 600°C in the pure state, and have been used as Ag^+ ion solutes in inorganic melts such as fused nitrates. As in the corrosion experiments, the presence of Ag^+ ions could not be analytically detected in this series of tests.

THERMODYNAMIC CONSIDERATIONS

In principle the types of mass transfer for the corrosion of metals in molten salts may be classed⁵ as "faradaic" and "non-faradaic". The former embraces those corrosion processes in which there is no true solubility of the metal in the molten salts but rather where the act of corrosion requires the oxidation of M to M^+ ; the latter embraces the processes which do not require an oxidation phenomenon or equivalent electrochemical path but rest on a finite solubility of the metal, presumably in the atomic or molecular non-oxidized state. In contrast to aqueous systems, dissolution of metals in the non-oxidized state has been reported⁶ in high temperature fused salt systems. While metals in general seem to be only appreciably soluble in their own molten salts, the direct dissolution or "non-faradaic" solubility, even if quite small, may be significant in mass transfer processes involving thermal gradients.

In the present system, a limited but finite dissolution of Ag in molten carbonates is noted (Table 1), and the question is whether this solubility is faradaic (due to the presence of some oxidant in trace amounts, e.g. impurities), or whether it is a "non-faradaic" process. The

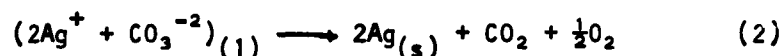
fact that the dissolution can be electrochemically enhanced (Table 2) is support for the view that in the former systems the dissolution is by transfer of Ag^+ ions at the metal-molten salt interface. It is of interest to examine this problem thermodynamically.

It is recognized that^{1,2} the dissociation:

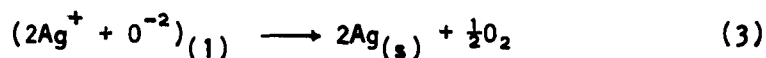


is finite in molten carbonates at all temperatures above the melting point. Finite CO_2 equilibrium dissociation pressures for the molten carbonates (e.g. Li_2CO_3 , 735°C, 11.6 mm; Na_2CO_3 , 885°C, 15 mm) imply significant mass-law controlled CO_2 , and O^{-2} activities in these melts. Both the CO_3^{-2} and O^{-2} ions can reduce silver; the thermal decomposition of Ag_2CO_3 and Ag_2O respectively are examples of these process, i.e.,

(a) Ag_2CO_3 :



and (b) Ag_2O :



It can be readily shown that the temperature dependence of the thermodynamic free energy change for these chemical processes is expressed by :

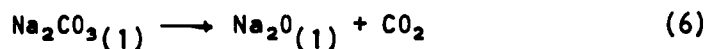
$$\Delta G'_T = \Delta G^\circ_{298}(T/298) - \Delta H^\circ_{298} \left[(T-298)/298 \right] + \int_{298}^T \Delta C_p dT - T \int_{298}^T \frac{\Delta C_p dT}{T} \quad (4)$$

where ΔG°_{298} and ΔH°_{298} and ΔC_p refer to the most stable (i.e. standard) state the constituents have at 25°C, respectively, and the symbol Δ has the

significance generally used for chemical reactions (i.e. Σ (products) - Σ (reactants) for the respective thermodynamic properties). For each substance the process of fusion must be taken into account, as required if the melting point is less than the temperature of concern, or if it has been assumed that the constituent is in a hypothetical liquid state. This contribution is expressed by the equations:

$$\Delta G_T^f = (T_m - T) \Delta S_{T_m}^f + \int_T^{T_m} \Delta C_p dt - T \int_T^{T_m} \frac{\Delta C_p}{T} dT \quad (5)$$

where $\Delta C_p' = C_{p(s)} - C_{p(l)}$. Such increments must be added to ΔG_T^f for each product and subtracted for each reactant in the chemical process being considered. The temperature dependence of ΔG_T^f thus gained for the above two processes, and the Na_2CO_3 dissociation⁷:



are given in Table 3. The numerical values for ΔG_{298}° , ΔH_{298}° , C_p were taken from standard tabulations^{8,9} and the recent entropy and heat capacity data for molten carbonates in this Laboratory¹⁰. The temperature and entropy of fusion for Na_2O were estimated as $1600^\circ \pm 400^\circ\text{K}$ and 7.5 ± 1.5 e.u. respectively; these properties, similarly, were unknown for Ag_2CO_3 and Ag_2O and the estimated values, $1400^\circ \pm 500^\circ\text{K}$, 7.5 ± 1.5 e.u. and $1100^\circ \pm 200^\circ\text{K}$, 7.5 ± 1 e.u., respectively, were gained by comparison with the data known for related carbonates and oxides.

From the ΔG_T relations (Table 3), values for equilibrium constants are readily gained. The values thus found at 900° and 1200°K respectively are:

(a) Ag_2CO_3 dissociation:

$$\frac{p_{\text{CO}_2} p_{\text{O}_2}^{1/2}}{a_{\text{Ag}_2\text{CO}_3}} = K_1; \quad K = 10^{5.7 \pm 0.5} \quad 10^{6.5 \pm 0.4}$$

900°K 1200°K

and

$$\frac{p_{\text{O}_2}^{1/2}}{a_{\text{Ag}_2\text{O}}} = K_2; \quad K = 10^{2.4 \pm 1.3} \quad 10^{2.0 \pm 1.0}$$

900°K 1200°K

The activities for silver carbonate and silver oxide can be expressed by the Temkin¹¹ ionic fractions, assuming these solutes mix ideally with the alkali carbonates. Thus it follows that:

$$a_{\text{Ag}_2\text{CO}_3} = \frac{N_{\text{Ag}^+}^2 N_{\text{CO}_3^{2-}}}{N_{\text{Ag}^+}^2 N_{\text{CO}_3^{2-}}} \quad (9)$$

and

$$a_{\text{Ag}_2\text{O}} = \frac{N_{\text{Ag}^+}^2 N_{\text{O}^{2-}}}{N_{\text{Ag}^+}^2 N_{\text{O}^{2-}}} \quad (10)$$

For the pure alkali carbonates in the molten state, $N_{\text{CO}_3^{2-}}$ is for all practical purposes equal to unity. The activity of the oxygen ion species, and the ionic fraction, can be calculated from a knowledge of ΔG_T for the dissociation expressed as (6) for Na_2CO_3 . The values thus found are:

$$\frac{a_{\text{Na}_2\text{O}} p_{\text{CO}_2}}{a_{\text{Na}_2\text{CO}_3}} = K_3; \quad K = 10^{-12.3 \pm 1.1} \quad 10^{-7.6 \pm 0.8}$$

900°K 1200°K

If N_{CO_2} is taken as unity, it follows that N_{O_2} is given quite simply by:

$$N_{\text{O}_2} = \frac{K_3}{p_{\text{CO}_2}} \quad (12)$$

in molten carbonates. Combining (10) and (12), the activity of Ag_2O may be expressed as:

$$a_{\text{Ag}_2\text{O}} = \frac{N_{\text{Ag}^+}^2}{K_3/p_{\text{CO}_2}} \quad (13)$$

It also follows that the Ag^+ concentration in molten sodium carbonate would be given by the expressions:

(a) from (7) and (9)

$$N_{\text{Ag}^+} = \frac{\left(\frac{p_{\text{CO}_2} p_{\text{O}_2}^{\frac{1}{2}}}{K_1} \right)^{\frac{1}{2}}}{K_3} \quad (14)$$

or

(b) from (8) and (13)

$$N_{\text{Ag}^+} = \frac{\left(\frac{p_{\text{CO}_2} p_{\text{O}_2}^{\frac{1}{2}}}{K_2 K_3} \right)^{\frac{1}{2}}}{K_3} \quad (15)$$

As an illustration, if $p_{\text{CO}_2} + p_{\text{O}_2} = 1 \text{ atm.}$, and $p_{\text{O}_2} = 0.01 \text{ atm.}$, it is predicted for the Ag_2CO_3 dissociation (2) in molten carbonates that the Ag^+ ion activities would be respectively:

$$900^\circ\text{K}, \quad N_{\text{Ag}^+} = 10^{-3.4 \pm 0.3}$$

and

$$1200^\circ\text{K}, \quad N_{\text{Ag}^+} = 10^{-3.8 \pm 0.2}$$

For the Ag_2O dissociation processes (3) in molten carbonates, similarly it is found that N_{Ag^+} is greater than unity, i.e., the concentration of O^{2-} ions will be too small to reduce silver.

Relative to the ternary Li, Na, K/ CO_3 eutectic mixture, the calculation of the O^{2-} ion concentration is similarly possible, but the limits of error are still greater since the thermodynamic properties of Li_2O , K_2O and K_2CO_3 are unknown and also must be approximated by estimation methods. The results of such a calculation show clearly that the O^{2-} ion concentration predicted above for Na_2CO_3 is, as well, a good approximation for the ternary melt. It is sufficient to note for the present purpose that even if the value for $N_{\text{O}^{2-}}$ in the ternary mixture were considerably greater than in pure Na_2CO_3 , (by as much as a factor of 10^5), the Ag^+ ion concentration would still be determined by the CO_3^{2-} ion concentration.

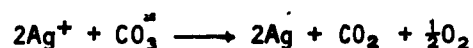
DISCUSSION

A small but finite silver corrosion under apparently "non-faradaic" or no-electrical-load conditions is clearly demonstrated by the present measurements (Table 1). A result of the thermodynamic method is that the saturation solubility for Ag^+ ions in molten carbonates can be predicted. Thus from the preceding considerations it is seen that when the silver ionic fraction reaches a value between 10^{-3} and 10^{-4} , the carbonate melts are Ag^+ ion saturated in the $600^\circ\text{--}900^\circ\text{C}$ temperature range. This corresponds to 10-100 mg Ag per 60 g of the ternary eutectic carbonate mixture. Comparison with the corrosion results of the "non-faradaic" experiments (Table 1) shows that the observed solubilities all fall within the concentration range predicted thermodynamically. The assumption of

metallic solution of Ag (as non-oxidized Ag) in molten carbonates seems no longer necessary, but this process is nevertheless not ruled out. Additional experimental studies are required to resolve the mechanism of solution unambiguously. The view is advanced that this "non-faradaic" corrosion of Ag is actually due to the presence of an oxidizing agent such as oxygen in trace amounts. The spread in the Ag weight loss results (last column, Table 1) is random and as such is understood if the "oxidant" were present as an impurity of variable trace concentrations. Quantitative knowledge of solubility of oxygen in molten carbonates seems important and remains to be investigated. A finite and appreciable solubility seems not unreasonable if it is recognized oxygen $\text{CO}_2 - \text{CO}_3^{2-}$ solvation interactions possibly exist in molten carbonates much as the rare gas-water molecule interactions discussed elsewhere for aqueous solutions¹². The thermal decomposition of molten carbonates⁷, e.g. $\text{Na}_2\text{CO}_3 \longrightarrow \text{Na}_2\text{O} + \text{CO}_2$ (liquid phase) and $\text{Na}_2\text{O} \longrightarrow 2\text{Na} + \frac{1}{2}\text{O}_2$ (vapor phase), may also contribute the oxidant impurity at sufficiently high temperatures. Further discussion of this is best deferred until outstanding problems such as the above gas solubility studies, have been investigated.

When Ag is electrochemically oxidized, e.g. by an anodic current, the dissolution rate becomes a question of diffusion and convection. Should these transport processes be rapid enough to avoid saturation in the electrode environment, a current efficiency for electrochemical Ag dissolution of 100% would be predicted. From an inspection of the "faradaic" corrosion results (Table 2), it is apparent that in the

electrolyses with current densities of $1-2 \text{ ma cm}^{-2}$ this condition is approximated. For the electrolyses where current efficiencies less than 100% were observed (Table 2) it is apparent that additional electrode reactions such as the carbonate ion oxidation and oxygen evolution as noted elsewhere² are competing processes. Migration of ionic silver in these electrolyses to the gold-palladium crucible was observed (as deposits of metallic silver alloyed with the gold-palladium cathode); silver is also found dispersed in the solidified melts as reduced silver metal. The latter may be attributed to a process such as:



when the thermodynamically predicted saturation solubility for Ag^+ in molten carbonate has been attained. Stated in other words, once the electrochemical oxidation potential of the carbonate ions has been attained, reduction of ionic to metallic silver is to be expected in these systems.

ACKNOWLEDGMENTS

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Table 1

Corrosion of Ag in the Molten Li,Na,K/CO₃
Eutectic Mixture Under CO₂ Atmosphere

<u>Corrosion Conditions</u>			<u>Silver Specimen</u>
<u>t(°C)</u>	<u>Time (hr)</u>	<u>Li,Na,K/CO₃(g)</u>	<u>-Δw(mg)[†]</u>
670°	17.8	60	20.1
690	2.5	60	0.7
840	5.0	60	13.7
870	15.5	60	21.5
900	17.5	60	38.6

†

The minus sign indicates a weight loss for the Ag metal foil after the immersion in the molten carbonate for the period of time and temperature noted for the experiment.

Table 2

Anodic Corrosion of Ag in the Molten Li,Na,K/CO₂
Eutectic Mixture Under CO₂ Atmospheres

<u>Corrosion Conditions</u>			<u>Silver Specimen</u>	
<u>t(°C)</u>	<u>time (hr)</u>	<u>i^a(ma cm⁻²)</u>	<u>observed[*]</u>	<u>predicted^{**}</u>
			<u>-Δw(mg)</u>	
680	1.60	0.3-30	28.8	----
680	3.83	1.5	28.6	30.9
680	0.66	15	40.9	53.7
900	4.66	1	38.6	37.6
900	1.50	10	91.7	120.7
900	0.43	50	101.0	134.2
900	0.43	50	104.2	134.2

^a Current density estimated from the "apparent" surface area of the Ag specimens. The surface areas of the latter were all in the range 1-2 cm²

^{*} Observed weight loss was gained by direct weighing.

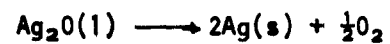
^{**} Predicted weight loss is based on 100% current efficiency for anodic dissolution of Ag as Ag⁺

Table 3

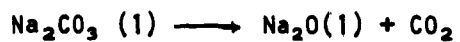
Temperature Dependence of the Thermodynamic Free
Energy Change for Certain Thermal Dissociation Reactions



$$\Delta G = 17500(\pm 2000) - 21.08T + 8.84 \times 10^{-3} T^2 \\ + 0.77 \times 10^5 T^{-1} - 4.76 T \ln T$$



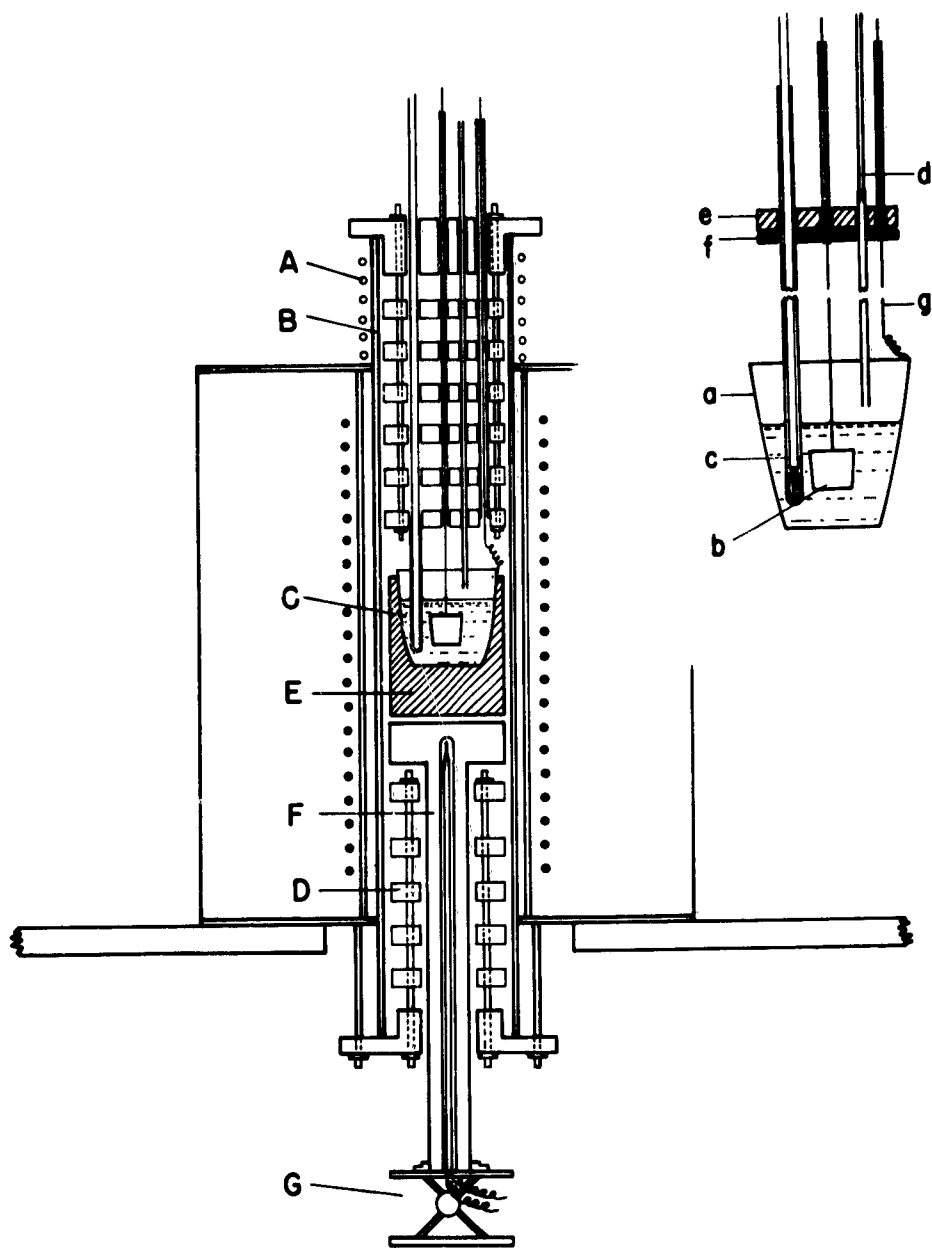
$$\Delta G_2 = -3300 (\pm 5500) + 5.73 T + 5.45 \times 10^{-3} T^2 \\ - 0.26 \times 10^5 T^{-1} - 2.63 T \ln T$$



$$\Delta G_3 = 87900 (\pm 4500) - 96.60 T + 1.60 \times 10^{-3} T^2 \\ + 1.03 \times 10^5 T^{-1} + 7.90 T \ln T$$

FIGURE CAPTION SHEETHigh Temperature Molten Salt Corrosion Assembly (500-1000°C)

A- water cooled Cu spiral; B- ceramic core; C- corrosion test melt and specimen; D- ceramic heat baffles; E- alundum crucible cradle; F- thermocouple; G- vertical rise variable lift. Insert: a- Au-Pd alloy crucible; b- corrosion specimen; c- thermocouple (Au-Pd sheathed); d- gas inlet (Au-Pd); e,f- ceramic and Au-heat baffles; g- contact wire for the crucible for electrochemical studies.



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