CO Gas Production by Molten Salt Electrolysis from CO2 Gas

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CO2 gas is decomposed to CO and C by the molten salt electrolysis using CaCl2–CaO and solid state electrolyte, zirconia, as the anode. Partially CO2 gas dissolves to form CO3²⁻ and it is electrochemically decomposed to carbon. The other portion of CO2 gas bubbles reacts with metallic Ca electrochemically deposited near the cathode, and forms C or CO gas. By increasing the flow rate of CO2 gas to the reactor, a high concentration of CO gas is generated. By increasing the concentration of CO2 gas in the initial gas, a large amount of CO gas was produced in the exhaust gas, and its rate approached to 3.32×10⁻⁸ m³/s in our experimental setup. These experimental evidences reflect the electrochemical decomposition of CO3²⁻ in the molten salt.

KEY WORDS: carbon dioxide; carbon monoxide; molten salt electrolysis; zirconia.

1. Introduction

Many attempts to reduce CO2 gas are widely examined to promote the countermeasures of global warming. In Japan, a large amount of CO2 gas is emitted from the steel making industries, and the exhaust gas from blast furnaces and converters particularly affects the emission of CO2 gas.1) If the emitted CO2 gas can be efficiently decomposed to carbon and oxygen, or to CO gas and oxygen, these products such as carbon can be used as the reducing agent of iron ore. Oxygen will be also used as blowing gas into the converters. CO gas has been used as the effective fuel in the steel making plants. This means that all the products after CO2 decomposition can be consumed in the steel plants as a precious raw material or energy resources, as shown in Fig. 1.

The authors have applied the molten salt electrolysis that has been developed as the method of reduction of metallic oxides,2,3) and tried to decompose CO2 gas as one of gaseous oxides.4–6) This method is interesting because it can decompose CO2 gas directly to carbon and oxygen at high temperatures, which are also available as the exhaust heat in the steel plant.4–6) The molten salt was also used as the media to dissolve CO2 gas and to electrolyze CO3²⁻ to carbon precipitates. For example, Yin et al. successfully converted CO2 gas in molten Li2CO3–Na2CO3–K2CO3 to carbon deposition on the cathode.7) Novoselova et al. electrochemically synthesized carbon nanotubes from CO2 in molten salts.8) The dissolution of CO2 gas into the molten salt and the electrolysis of CO3²⁻ anion were basically accepted as the reasonable mechanism.7–13)

\[ \text{CO}_2 \text{(gas)} + \text{O}^2^- \Rightarrow \text{CO}_3^{2-} \] ........................ (1)

\[ \text{CO}_3^{2-} + 4e^- \Rightarrow \text{C} + 3\text{O}^2^- \] ........................ (2)

\[ E^0 = -1.02 \text{ V} \]

This reaction looks strange because the CO3²⁻ anion reacts with electron at the cathode, not at the anode. CO gas formation can not be explained in two reactions, and the additional thermochemical reaction such as Boudouard reaction among C, CO and CO2, should occur if CO gas coexists.

In the previous studies from the authors’ group,4–6) CaCl2–CaO and LiCl–Li2O are used as molten salt for the decomposition of CO2 gas and the production of carbon. Because these salts can dissolve both the reductants, Ca14,15) and Li14,15) and the by-products, CaO17–19) and Li2O20) many oxides could be reduced to metallic state at the cathode2,3,17) Their reactions were analyzed as the thermo-chemical reduction by calcium and lithium deposited electrochemi-

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cally by the electro-deposition from CaO and Li2O, respectively.4–6) This mechanism is a little different from the previous reports3–13) at the point that the electrolysis of CO32– was not considered, but that metallic reductant works as the agent for thermochemical reaction.4–6)

Their reported reaction mechanisms can be briefly summarized as follows, by taking the example in the CaCl2–CaO bath. CaO dissolves firstly in the melt,17–19) and it ionizes in the molten CaCl2 above its melting point, 1115 K. When an electrical voltage is applied between two electrodes in the molten salt, metallic Ca is produced on the cathode as in Eq. (4). This Ca precipitate dissolves near the cathode due to solubility of Ca in CaCl2 melt,24,25) and reacts as the reductant with CO2 gas as in Eq. (6). At this time, the by-product CaO is simultaneously produced as well as carbon, and CaO is again ionized in the melt in Eq. (3). Therefore, these reactions can be circulated. In short, CO2 gas is decomposed continuously without any new addition of CaO.

\[
\begin{align*}
\text{CaO} & \Rightarrow \text{Ca}^{2+} + \text{O}^{2-} \quad \text{.......... (3)} \\
\text{Ca}^{2+} + 2\text{e}^- & \Rightarrow \text{Ca} \quad \text{.......... (4)} \\
\text{E}^0 & = -2.65 \text{ V} \\
\text{Ca} \text{ (on cathode)} & \Rightarrow \text{Ca (in CaCl}_2\text{)} \quad \text{.......... (5)} \\
2\text{Ca (in CaCl}_2\text{)} + \text{CO}_2\text{ (gas)} & \Rightarrow \text{CaO + C} \quad \text{.......... (6)}
\end{align*}
\]

Conventionally a carbon anode has been often used in the molten salt electrolysis, however, it is consumed and oxidized to CO2 gas due to the anodic reaction with O2– anion. In order to resolve this problem, the ZrO2 solid electrolyte that is a good oxygen ion conductor21,22) is applied as the anode instead of carbon electrode.4–6,23)

\[
\begin{align*}
\text{O}^{2-} \text{ (in CaCl}_2\text{)} & \Rightarrow \text{O}^{2-} \text{ (in ZrO}_2\text{)} \quad \text{.......... (7)} \\
2\text{O}^{2-} \text{ (in ZrO}_2\text{)} & \Rightarrow \text{O}_2\text{ (gas)} + 4\text{e}^- \quad \text{.......... (8)}
\end{align*}
\]

This new electrode is the anode which can remove oxygen from molten salt. This idea enables to prevent the produced carbon from reoxidizing because oxygen is emitted out of the reaction system. Both reactions at the cathode and anode worked efficiently in the CaO–CaCl2 bath.

However, as mentioned above, another mechanism was reported that CO2 gas once dissolves in the molten salt as carbonate ion and that it is decomposed on the cathode.3–13) Because the both mechanisms are possible depending on the applied potential, the authors infer that carbon production by CO2 gas decomposition can be caused by combining these two mechanisms. The preferable mechanism may be selected by the applied electrochemical and thermodynamical conditions.

Carbon production was the major concern in previous studies. For example, Otake et al.4–6) reported the carbon production from CO2 gas with CaO–CaCl2 and Li2O–LiCl at 1 173 K and 923 K, respectively. They showed the existence of carbon nano-fibre as well as spherical particles of amorphous carbon. The formation of carbon fibre in the molten salt was interesting. The precious carbon fibre could be formed in the liquid, not in open environment such as gaseous atmosphere. The two morphologies of carbon implied, however, that at least two mechanisms were working in CO2 decomposition. The contamination of iron in the carbon fibre may suggest the electro-deposition of CO32– on the steel cathode. Gupta et al.24) reported that Sn-filled multi-walled carbon nanotubes and carbon nanoparticles were synthesized by molten salt electrolysis, when SnCl2 was used as a part of electrolyte.

Only a few examples were reported that CO gas was formed as by-product in those electrolysis, however, it has not been studied systematically about the conditions where CO gas was dominantly formed. If the mechanism of CO gas formation is revealed, it will be possible to produce selectively CO gas starting from CO2 gas.

A strong reducing atmosphere is thermodynamically needed to remove oxygen from CO2 gas, and a specially careful handling is required to produce carbon powder from CO2 gas because the produced carbon is ultrafine powder frangible even in open air. On the other hand, such a strong reducibility for carbon precipitation may be not required for CO gas production. Additionally CO gas can be continuously taken out from the reaction vessel because it is gaseous. A high concentration of CO mixed with the unreacted CO2 gas can be used as fuel in the steel industries.

The purpose of this study is to investigate the conditions of CO gas production and to produce high concentration of CO gas by CO2 gas decomposition by using the electrolysis in molten CaCl2–CaO.

2. Experimental

The experimental procedures are referred to briefly here because the previous papers reported the details.4–6)

**Figure 2** shows the schematic appearance of the furnace and reactor used in this study. A MgO crucible (99.5% in purity, 90 mm in diameter, 200 mm in height) was set with CaCl2–CaO mixed powder (approximately 600 g) which
had been precisely weighed so that the concentration of CaO is 2.0 mol%. The salt was dehydrated at 873 K in vacuum. After one night, it was continuously heated to 1173 K in an argon gas atmosphere, and CO2–Ar mixed gas was then blown through the cathodic stainless steel pipe (6 mm in outer diameter) into the molten salt at a desired gas flow rate and a desired gas concentration. A constant voltage of 3.10 V was applied between two electrodes at 1173 K. The concentration of CO2 gas and CO gas emitted out of the system during electrolysis was measured with an infrared spectrophotometry. Because the response of spectrophotometry was varied by the gas flow rate, it was calibrated before and after each measurement using the standard gas.

3. Results and Discussion

3.1. Current and Gas Analysis during Electrolysis

Figure 3 shows the time dependency of CO2 and CO gas concentrations in the exhaust gas and that of current density during the electrolysis. The measured current in the circuit was divided by the anodic surface area and shown as current density in Fig. 3.

Just after the voltage was applied, the electrolysis time was started to count. It is common that the large current density was measured at the initial stage of the electrolysis, but that it decreased as the electrolysis time proceeds. The current approached to a stable at the later stage. The decrease of CO2 concentration in the exhaust gas was confirmed at the initial stage of the electrolysis. However CO2 gas concentration gradually increased at the later stage. On the other hand, CO gas concentration increased at the initial stage, and it decreased at the later. Due to the dead volume in the reacting vessel, the analyzed concentration is delayed from the real reaction time at the molten salt, but the slow increase and decrease of CO2 and CO gas, respectively, at the later stage show the change of reaction mechanism during the electrolysis.

The amounts of the decomposed CO2 gas and the produced CO gas were calculated from the area hatched in Fig. 3. Resultantly from Fig. 3(c), CO2 gas was decomposed at $3.32 \times 10^{-8}$ m$^3$ per second, and about half of them were served for the conversion to CO gas. And current efficiency was calculated. It was defined as the ratio of the electric charge consumed for the CO gas production to the all of electric charge consumed for this electrolysis. In this condition, current efficiency was 36.9%. It was low because electric charge was consumed to produce carbon as well as CO gas. Therefore, suppressing the carbon production increased the current efficiency.

3.2. Feeding Rate of CO2 Gas during Electrolysis

CO2 gas can be decomposed partially by calciothermic reduction and partially by the decomposition of carbonate ions, as shown in Eqs. (6) and (2), respectively. If the larger amount of CO2 gas is supplied, namely if CO2 gas is blown at the higher rate, CO2 gas will be converted to CO gas preferentially.

$$\text{Ca} + \text{CO}_2 \rightarrow \text{CaO} + \text{CO} \quad \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots 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vol%CO₂-Ar was used. The gas flow rate from the cathodic nozzle was $0.517 \times 10^{-6}$, $1.67 \times 10^{-6}$ and $2.58 \times 10^{-6}$ m³/s at Figs. 3(a), 3(b) and 3(c), respectively. The rates at (b) and (c) were greater than that at (a) by about 3 times and 5 times, respectively. Little CO gas was produced when the gas flow rate was small, but by increasing the flow rate, significantly large amount of CO gas was produced.

The amount of decomposed CO₂ gas and the amount of produced CO gas, $v$, were also calculated from the concentration $C(t)$ as a function of time, $t$, and a constant gas flow rate, $Q$.

$$v = Q \int_0^t C(t) \, dt$$  \hfill (12)

Figure 4 shows the relationship of the amounts of decomposed CO₂ gas and produced CO gas as a function of the gas flow rate. The amount of decomposed CO₂ gas reached the maximum when the gas flow rate was $1.67 \times 10^{-6}$ m³/s. The amount of produced CO gas reached the maximum when the gas flow rate was $2.58 \times 10^{-6}$ m³/s. This indicates that CO gas favorably generates when the flow rate becomes larger. The amount of produced CO gas drastically increased in the range of gas flow rate of $0.5 \times 10^{-6}$ and $1.7 \times 10^{-6}$ m³/s.

Several reasons will be discussed: The first reason is as follows. As the gas flow rate is larger, the stirring of the molten salt becomes more intense due to gas bubbles, and the electrochemically produced calcium at the cathode disperses out easily. The thermochemical activity then becomes lower, and CO gas is preferentially produced.

The second reason is as below; Because the amount of CO₂ gas blown per unit time increases, the CO₂ gas can not sufficiently contact with calcium, and the perfect reduction from CO₂ gas to carbon does not complete, but the conversion to CO gas became favorable.

Thirdly, the contribution of carbonate ions can be considered apart from calcium. As the gas flow rate increases, the amount of carbonate ions also increases. This is because the dissolution of CO₂ gas to the molten salt becomes more significant, as the larger amount of unreacted CO₂ gas remains. In cathodic reactions with carbonate ion, 4 electrons are used for carbon production, but only 2 electrons are used for CO gas formation.

$$\text{CO}_2^- + 2e^- \rightarrow \text{CO} + \text{2O}^2^-$$ \hfill (13)

Therefore, CO gas production occurs more easily, when the larger amount of carbonate ions are decomposed with the constant electric charge.

### 3.3. Effects of Gas Concentration

Figures 5(a) and 5(b) show the results when the gas mixture of 6 vol%CO₂ and 16 vol%CO₂ are used, respectively. CO₂ gas was decomposed in both cases, and the CO gas evolution was detected in Fig. 5(b), while the formation of CO gas was not seen in Fig. 5(a).

Figure 6 shows the ratio of the amount of produced CO to that of decomposed CO₂. When the initial CO₂ gas concentration was higher, the ratio increased. This means that the rate of CO gas production increased with increasing in the CO₂ gas concentration. Larger portion than 90% of the supplied CO₂ gas was converted to CO gas in the high concentration range between 14 and 20%. This shows that CO gas was more favorably produced as the concentration of CO₂ gas was higher.

As CO₂ gas concentration is higher, the molar fraction of carbonate ion may increase in molten salt, and it affects the production rate of CO gas. This correlation may be explained easily by the carbonate route in reaction mechanism as shown in Eqs. (1) and (2). However, as the high carbonate ion concentration is an evidence that the supplied CO₂ is not converted to carbon but to CO gas and carbonate.
the effective conversion to CO gas may be explained by cal-
ciothermic reaction, too. This means that too much CO2 gas
bubbles existed per the electrodeposited Ca, and a large por-
tion of CO2 gas dissolved as carbonate ions. However, the
reacted portion was converted to CO gas due to dean Ca
concentration.

4. Conclusion

As a result of the constant-voltage electrolysis using mol-
ten salts consisting of CaCl2–CaO and solid state electrolyte,
zirconia, as the anode, the production of CO gas was
enhanced by increasing the gas flow rate. The higher conver-
sion to CO gas from CO2 gas was obtained at the higher con-
centration of CO2 in the initial gas mixture. 3.32×10−8 m3/s
of CO gas was produced in our experimental conditions.

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Fig. 6. Ratio of amount of formed CO gas to that of decomposed
CO2 gas as function of initial gas concentration of CO2.