The effects of oxygen pressure on the corrosion of iron embedded in mortar with various Cl\textsuperscript{−} contents were examined by using a novel hyperbaric-oxygen accelerated corrosion test (HOACT). Exposure and electrochemical tests of a pure iron sample embedded in mortar with a cover thickness of 5 mm were performed in HOACT condition with pressurized 100% oxygen gas and with relative humidity of 95%. For comparison, these tests were also performed in ambient air with the same relative humidity. Oxygen reduction current density of iron embedded in mortar increased with an increase in oxygen pressure, suggesting that the corrosion of the embedded iron is expected to be accelerated with oxygen gas pressure assuming an oxygen reduction limited corrosion current density. Iron corrosion in mortar without Cl\textsuperscript{−} was minimal regardless of oxygen gas pressure. In contrast, the corrosion of iron in mortar with Cl\textsuperscript{−} was accelerated with increasing the oxygen gas pressure up to a certain pressure as expected, and the thickness of the rust formed on the iron surface increased with Cl\textsuperscript{−} concentration under each oxygen gas pressure, indicating that Cl\textsuperscript{−} is necessary to initiate corrosion which is enhanced in the pressurized oxygen gas. However, when the oxygen pressure exceeded a certain level, the corrosion was suppressed. Electrochemical impedance spectroscopy measurement using a sensor consisting of a pair of carbon steel electrodes in mortar showed that the charge transfer resistance of the carbon steel under excessively high oxygen gas pressure became high in comparison to that at lower oxygen pressure, indicating the suppression of corrosion initiation is due to formation of a protective passive film under the excess supply of oxygen. Consequently, the pressurized oxygen gas accelerates the corrosion of iron in mortar with Cl\textsuperscript{−}; however, excess oxygen suppresses corrosion. The optimum condition to efficiently accelerate corrosion of iron in mortar was evaluated.

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expected further acceleration of corrosion and the effects of oxygen gas pressure and Cl⁻ concentration in mortar on corrosion of the iron in mortar were examined.

**Experimental**

**Samples.**—A commercially pure iron coupon was used for measurements of potentiodynamic polarization curves and for HOACT under various oxygen gas pressures. An iron sheet with a thickness of 1 mm (99.5%, The Nilaco Corporation) was shaped into coupons with a size of 7 x 7 mm². Hereafter, the coupon is denoted by ‘Fe sample.’ After both sides of the Fe samples were ground with #800 grid SiC paper (Struers) and rinsed ultrasonically in distilled water and in acetone for 5 min, respectively, a polyvinyl chloride stick was attached to the back side of the Fe sample (Fig. 1). The back side and the edges of the Fe sample were sealed with epoxy resin to prevent them from being exposed to mortar. A coated conducting wire was soldered to the back side of the Fe sample when it is used for polarization tests. Subsequently, the Fe sample was embedded in mortar with cover thickness of 5 mm. The specimen of Fe sample embedded in mortar is denoted by ‘Fe in mortar’, hereafter.

A corrosion sensor shown in Fig. 2 was used for electrochemical impedance spectroscopy (EIS) measurements. The corrosion sensor consists of a pair of ring and rod electrodes of SD345 carbon steel which is used for reinforcing steel bars in Japan. The ring and rod electrodes are fixed on an epoxy resin base with a narrow gap of 100 μm between the electrodes. Since the two electrodes are placed concentrically, there is no anisotropy in the electrochemical measurements. The exposed surface areas of the two electrodes are the same. The corrosion sensor was embedded in mortar with cover thickness of 5 mm. Although SD 345 carbon steel used for the EIS measurement is different from pure iron used for the potentiodynamic polarization test and for the HOACT, it is assumed that both materials show similar corrosion behavior.

Mortar was produced by mixing water, ordinary Portland cement and fine aggregates. The weight composition of water: cement: fine aggregates was 0.6:1:3. In the mortar for the corrosion test in ambient and in HOACT, a NaCl aqueous solution was used as mixing water to enhance corrosion. Chloride concentrations in the mixing water were 0, 0.62 and 2.06 M. On the other hand, distilled water was used as the mixing water for the mortar used for the potentiodynamic polarization and EIS measurements. The mortar embedding Fe sample and the corrosion sensor were cured over 28 days in a chamber in which the relative humidity (RH) was kept at 95% or higher using saturated K₂SO₄ solution in a beaker placed in the chamber.

**Potentiodynamic polarization tests.**—Potentiodynamic polarization tests were carried out for Fe in mortar with a scan rate of 20 mV/min to cathodic direction at room temperature. Since the purpose of the test is to evaluate the dependence of oxygen reduction reaction of Fe in mortar on the supplied oxygen gas pressure, a saturated Ca(OH)₂ solution was adopted as the electrolyte which simulates the pore solution in mortar. Here, it is assumed that additive NaCl in mortar, which was used to accelerate depassivation in HOACT, does not affect oxygen diffusion through mortar significantly.

The oxygen flux through mortar was expected to vary depending on the oxygen gas pressure. Mercury-mercury oxide (Hg/HgO) electrode (0.098 V vs. SHE at 25°C) and a Pt wire were used as reference and counter electrodes, respectively. Fe in mortar was immersed in the electrolyte in a glass vessel which was placed in a high pressure container designed for HOACT, and the internal gas pressure in the container was varied using a regulator connected to a pressurized oxygen gas cylinder (Fig. 3). Ambient air was also introduced into the container to perform the test under the partial oxygen gas pressure of 0.02 MPa. The hyperbaric oxygen gas pressure of 0.4, 0.6, 1.1, and 2.1 MPa are 20, 30, 55 and 105 times as high as the partial pressure of oxygen in ambient air. The potentiodynamic polarization tests were conducted 24 h after supplying oxygen gas or ambient air to stabilize the open-circuit potential of Fe in mortar.

**Hyperbaric-oxygen accelerated corrosion test (HOACT).**—In the tests, Fe in mortar was exposed to pressurized oxygen gas or ambient air with relative humidity over 95%RH in the 4.5-L-size HOACT container. Fe in mortar was placed in the container, and the pressurized oxygen gas of 0.1, 0.4, 0.6, 1.1, and 2.1 MPa or ambient air (0.02 MPa of oxygen gas partial pressure) was introduced in the container (Fig. 4). The relative humidity in the container was kept over 95% by using 500 mL of a saturated K₂SO₄ solution. After HOACT for 14 d, the Fe sample was extracted from mortar, and the surface of the Fe sample was observed using one-shot 3D profilometer (VR-3000, KEYENCE). Back scattered electron images of cross sections of the Fe sample were taken using a scanning electron microscope (Quanta FEG, FEI) to gauge the thickness of the formed rust layer. The
extracted Fe samples after HOACT were stored in a desiccator at room temperature, when the Fe coupons were not analyzed immediately after the extraction.

**Electrochemical impedance spectroscopy (EIS) measurement.**—EIS measurement was performed to examine the effect of oxygen gas pressure on the time-dependent change of protectiveness of passive film formed on a carbon steel conventionally used for steel rebar. The corrosion sensor in mortar described before (Fig. 2) was placed in the HOACT container with the pressurized oxygen gas of 0.6 and 2.1 MPa or with ambient air (0.02 MPa of oxygen partial pressure). The impedance of the corrosion sensor was measured every 24 h for 14 d. Potential perturbation was applied between the ring and rod electrodes with an amplitude of 10 mV at frequencies from 100 kHz to 10 mHz. The charge transfer resistance, $R_{ct}$, which corresponds to the protectiveness of passive film, was calculated from the diameter of the capacitive loop of obtained Nyquist plots.

**Results and Discussion**

**Oxygen reduction reaction on iron in mortar.**—Figure 5 shows the cathodic polarization curves of Fe in mortar in ambient air and in pressurized oxygen gas at 0.4, 0.6, 1.1 and 2.1 MPa. Cathodic current originating from oxygen reduction reaction gradually increased with a decrease in potential. To evaluate the corrosion rate of Fe in mortar in the pressured oxygen gas, the oxygen reduction current density (ORCD) at the OCP was calculated by extrapolating the Tafel slope to the OCP. The obtained ORCD at the OCP was plotted as a function of oxygen pressure in Fig. 6. The ORCD increased proportionally to the oxygen gas pressure.

According to Henry’s law, the dissolved oxygen concentration in the pore solution in mortar increases proportionally with increase in supplied oxygen gas pressure (Eq. 1), because the temperature is constant at room temperature in this study.

$$ C = \frac{p}{KR_T} $$

where $C$ (mol m$^{-3}$) is the concentration of dissolved oxygen in mortar just beneath the surface, $p$ (Pa) the (partial) pressure of oxygen gas, $K$ the constant, $R$ the gas constant (J K$^{-1}$ mol$^{-1}$), and $T$ the temperature (K).

Because the ORCD continuously increased and did not indicate a plateau, it is not able to obtain the diffusion limited current density in this study. However, the diffusion limited current density is expected to increase with an increase in the oxygen gas pressure. The relationship between diffusion limited current density, diffusion coefficient and thickness of the diffusion layer is shown in following equations,

$$ i_L = \frac{zFDC}{\delta} $$

$$ \frac{i_L}{zF} = J = \frac{DC}{\delta} $$

where $i_L$ (A m$^{-2}$) is the diffusion-limited ORCD, $J$ (mol m$^{-2}$s$^{-1}$) the oxygen flux, $z$ the number of moles of electrons per mole of oxygen molecule in the oxygen reduction reaction that is 4 in this case, $F$ (C mol$^{-1}$) is the Faraday constant (96485 C mol$^{-1}$), $D$ (m$^2$ s$^{-1}$) the diffusion coefficient of dissolved oxygen through mortar, and $\delta$ (m) the thickness of the diffusion layer.

The proportional relationship between ORCD and oxygen gas pressure obtained in this study revealed that the oxygen flux through mortar can be controlled by changing the oxygen gas pressure because the $\delta$ and $D$ were presumed to be constant for the following reasons. The entire mortar cover of 5 mm can be regarded as the thickness of the diffusion layer, $\delta$, because the oxygen diffusion layer formed by the oxygen consumption on Fe surface grew over 10 mm in mortar cover thicker than 10 mm. The diffusion coefficient, $D$, of molecules in aqueous solutions does not generally depend on the outside gas pressure, and micropores in mortar were filled with Ca(OH)$_2$ solution.

The diffusion coefficient, $D$, of dissolved oxygen thus considered to be independent on the oxygen gas pressure. Hence, the proportional
The relationship between ORCD and oxygen gas pressure indicated that the oxygen flux, \( J \), was in proportion to the concentration of dissolved oxygen, i.e., the applied oxygen gas pressure. The corrosion of Fe in mortar was, therefore, expected to be accelerated proportionally to the oxygen gas pressure of HOACT as far as the corrosion is limited by oxygen reduction reaction.

**Accelerated corrosion of Fe in mortar in pressurized oxygen gas.**—Figure 7 shows the surface images of Fe samples after HOACT of 14 d at various oxygen gas pressures and with varied Cl\(^-\) content in mortar. Fig. 8 shows the percentage of corrosion area on the surface of Fe sample in Fig. 7. The percentage of corrosion area to the whole sample surface was determined by measuring the rusted area.

![Figure 7](image)

![Figure 8](image)

Corrosion of Fe in mortar was minimal in ambient air regardless of the content of Cl\(^-\) because the low oxygen reduction currently density under low oxygen gas pressure resulted in the suppression of iron dissolution in spite of the addition of aggressive Cl\(^-\). At a certain range of oxygen gas pressure, rust was formed in the presence of Cl\(^-\), whereas rust formation was not obvious without Cl\(^-\) addition regardless of oxygen gas pressure. With low Cl\(^-\) content in mortar, which was produced using 0.62 M NaCl solution as a mixing water, rust was formed at the edge of the Fe sample at 0.1 MPa of oxygen and rusted area decreased at 0.4 MPa. With high Cl\(^-\) content in mortar, which was produced using 2.06 M NaCl solution as a mixing water, rust was formed slightly at the vicinity of the edges of the Fe sample in ambient air while rusted area was obviously wide at 0.1, 0.4, 0.6 and 1.1 MPa. The rusted area with high Cl\(^-\) content increased remarkably with oxygen gas pressure up to 0.6 MPa. However, the rusted area decreased at above 0.6 MPa and no apparent rust was observed at 2.1 MPa, though the measured ORCD shown in Fig. 6 increased proportionally to the applied oxygen pressure suggesting that corrosion limited by oxygen reduction reaction is supposed to be enhanced with oxygen pressure. These results indicate that enhancement of oxygen supply at appropriate pressure is necessary to enable corrosion acceleration of Fe in mortar in addition to Cl\(^-\) addition for breakdown of passive film but excess oxygen supply results in suppression of corrosion.

**Discussion**

Fig. 9 shows the representative SEM images of cross sections of the Fe samples observed using BSE mode. From the cross section images, the thickness of the rust was measured. Mean thickness of the rust layer was obtained from measured thicknesses of 5 views randomly taken on each specimen. The mean thicknesses of rust layers for the varied conditions are summarized in Fig. 10. The rust formed on some Fe samples in pressurized oxygen gas were several-micro-meter thick. The rust of the Fe in mortar containing Cl\(^-\) grew with oxygen gas pressure until thickness reached to the maximum. On the other hand, the rust thinned when the oxygen gas pressure was increased excessively. This tendency was similar to the change in corrosion area shown in Fig. 8. The thickest rust layer was formed in 0.6 MPa oxygen gas pressure.
gas with high Cl\(^{-}\) content in mortar. Up to 0.6 MPa, the rust grew almost proportionally to the oxygen gas pressure in mortar with high Cl\(^{-}\) content. With the low Cl\(^{-}\) content in mortar, the maximum rust thickness was obtained at lower oxygen pressure.

It is indicated that corrosion was initiated due to the passive film breakdown caused by Cl\(^{-}\) and the corrosion progress was accelerated by the enhancement of oxygen reduction reaction in proportion to the oxygen gas pressure in a proper oxygen pressure range. Thus, corrosion of Fe in mortar containing Cl\(^{-}\) was successively enhanced in the pressurized humid oxygen gas at >95%RH utilized in the present study as well as the HOACT of Fe in mortal immersed in a NaCl solution in our previous study.\(^{16}\)

As was explained above, corrosion of Fe in mortar with high Cl\(^{-}\) content was suppressed in 1.1 and 2.1 MPa of oxygen gas, despite of the sufficient addition of Cl\(^{-}\) in mortar for depassivation. This result indicates that HOACT has an upper limit oxygen gas pressure to efficiently enhance the corrosion of Fe in mortar. To accomplish a more accelerated corrosion test in high pressure oxygen gas, it is necessary to understand the mechanism of corrosion suppression under excessively high oxygen pressure. Therefore, the effect of oxygen gas pressure on the corrosion of Fe in mortar was examined by means of electrochemical impedance spectroscopy as will be described in the next section.

**Effect of excessively high oxygen gas pressure on passivation of Fe in mortar.**—Electrochemical impedance measurements were carried out at various oxygen gas pressures using a corrosion sensor (Fig. 2) embedded in mortar to understand the corrosion suppression at excessively high oxygen gas pressure. The mortar covering the sensor was without Cl\(^{-}\) because Cl\(^{-}\) disturbed the evaluation of the protectiveness of the passive film resulting in low reproducibility especially under relatively low oxygen gas pressure. Figure 11 shows Nyquist plots of the impedance spectra obtained at 0 d, 7 d and 14 d of the duration of HOACT at 2.1 MPa of oxygen gas. A part of a large capacitive semicircle was observed in the low frequency range (Fig. 11a) and a part of a small capacitive semicircle was observed in the high frequency range (Fig. 11b). The diameter of the large capacitive semicircle increased with time. From these parts of capacitive semicircles, an equivalent circuit for the impedance spectra on the sensor in mortar is proposed (Fig. 12). The parameters for the equivalent circuit are the solution resistance (\(R_{\text{sol}}\)), the resistance (\(R_{\text{mor}}\)) and capacitance (\(C_{\text{mor}}\)) attributable to mortar, charge transfer resistance (\(R_{\text{ct}}\)) and electric double layer capacitance (\(C_{\text{dl}}\)). McCarter et. al and Song indicated that a capacitive loop appears in EIS measurement because there are discontinuous conductive paths in concrete.\(^{19,20}\) On the other hand, Someya et. al reported that the capacitive loop in high frequency is due to the ions in concrete.\(^{31}\) In any case, the capacitive loop in high frequency is influenced by the structure and composition of mortar. Therefore, the resistance and capacitance of mortar is described as \(R_{\text{mor}}\) and \(C_{\text{mor}}\). It is assumed that the locus in the low frequency range was related to \(R_{\text{ct}}\) and \(C_{\text{dl}}\) and the locus in the high frequency range

![Figure 9. Cross section images of Fe sample surface retrieved from mortar after 14 days of HOACT at various oxygen gas pressures.](image)

![Figure 10. Thickness of rust formed on Fe in mortar for 14 days of HOACT at various oxygen gas pressures.](image)
Effect of pressurized oxygen gas and chloride ions on the corrosion acceleration of Fe in mortar.—When enough amount of Cl\(^-\) presents in mortar with high pH, depassivation takes place and the subsequent iron oxidation reaction (anodic reaction) is enhanced according to the enhancement of the coupled oxygen reduction reaction (cathodic reaction) in hyperbaric oxygen environment up to about 0.6 MPa. At excessively high oxygen gas pressure above this pressure, however, passivation is enhanced and no obvious corrosion occurred.

Low Cl\(^-\) content in mortar did not induce depassivation in HOACT with oxygen of 0.6 MPa or higher and no obvious corrosion occurred without additive Cl\(^-\). It is revealed that breakdown of passive film requires the addition of sufficient content of Cl\(^-\) when the high pH of pore solution in mortar is maintained. In contrast, depassivation presumably occurs even with low Cl\(^-\) content when the mortar is neutralized with CO\(_2\) and the relative concentration ratio of [Cl\(^-\)]/[OH\(^-\)] exceeds 0.6, which is the threshold for chloride corrosion. Furthermore, the neutralization of mortar may enhance depassivation with Cl\(^-\) even at exceedingly high oxygen gas pressure. If it is the case, further acceleration of corrosion of Fe in mortar can be expected by applying hyperbaric oxygen gas higher than 0.6 MPa.

The rust formed in 0.6 MPa oxygen gas with humidity of >95%RH for 14 d was ca. 3 μm in average thickness as shown in Fig. 10, while the rust of Fe in mortar with the same cover thickness formed by 30 d of HOACT in a NaCl solution at 0.6 MPa of oxygen was ca. 3.7 μm in thickness in our previous study. In the previous paper, the oxygen gas pressure of 0.5 MPa was the gauge pressure and the absolute pressure was 0.6 MPa. Note that the oxygen pressure given in this paper is absolute pressure. As a result, the corrosion rate of Fe in mortar in the humid oxygen gas of 0.6 MPa is about 1.6 times as high as that in the NaCl solution under oxygen gas of the same pressure. This comparison indicates that the corrosion of Fe in mortar in humid oxygen gas adopted in the present study is accelerated more effectively than that in a NaCl solution exposed to pressurized oxygen gas. This is presumably because the oxygen diffusion in mortar in the humid oxygen gas is faster than that in a NaCl solution.

Corrosion acceleration of iron in concrete with HOACT.—We have reported that the oxygen reduction reaction on a Fe sample in mortar was higher than that on a Fe sample embedded in cement paste. This difference is attributed to the dissimilar characteristics of the micro-pores in cement paste and that in mortar containing fine aggregates and to the higher transportation coefficient in mortar. Coarse aggregates in concrete generally form larger micro-pores than those in mortar and in cement paste, implying that the diffusion of oxygen and resulted oxygen reduction reaction on iron in concrete is higher than that in mortar and in cement paste. Hence, we can expect the applicability of HOACT for corrosion acceleration of steel rebar in concrete specimen with relatively thick cover simulating actual concrete structures, and most probably HOACT allows us to reproduce cracking of the concrete specimen with steel rebar. However, as it has been discussed above, excessively high oxygen pressure results in
suppression of corrosion. The study to optimize the test condition to effectively accelerate corrosion of rebar in concrete is ongoing.

Conclusions

The effect of oxygen gas pressure and Cl\textsuperscript{−} concentration in mortar on the corrosion of iron in mortar was examined to adequately maximize the acceleration of iron corrosion by means of the novel hyperbaric-oxygen accelerated corrosion test. The following conclusions were derived.

1. The oxygen reduction current density of Fe in mortar increased proportionally with an increase in oxygen gas pressure in the pressure range from ambient pressure to 2.1 MPa in this study.

2. Corrosion acceleration of Fe in mortar, produced using 0.62 M and 2.06 M Cl\textsuperscript{−} solution as mixing water, increased with an increase in oxygen gas pressure up to 0.1 and 0.6 MPa, respectively. However, corrosion was suppressed by further increase of oxygen gas pressure. The maximum corrosion acceleration rate was achieved with the high Cl\textsuperscript{−} content in mortar and in 0.6 MPa oxygen gas.

3. In mortar without Cl\textsuperscript{−}, no obvious rust was formed on iron samples regardless of oxygen gas pressure, indicating that Cl\textsuperscript{−} is necessary to initiate corrosion of iron in mortar.

4. The EIS results showed that the $R_{ct}$ of carbon steel in mortar without Cl\textsuperscript{−} increased with time and the increase of $R_{ct}$ in 2.1 MPa oxygen gas was obviously higher than that in 0.6 MPa oxygen gas and that in ambient air. These results suggest that the passivation of iron in mortar is enhanced in excessively high pressurized oxygen gas.

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