Development of flow-accelerated corrosion prediction method

(1) Acquisition of basic experimental data including low temperature condition

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Abstract

A series of study is presented to develop a prediction method for pipe wall thinning in power plants in order to improve the maintenance management for piping system. As the first report, experiments for flow-accelerated corrosion (FAC) of carbon steel specimens were conducted and basic data were obtained, focusing on relatively low temperature condition. FAC rate is seen to decrease by lowering temperature and the trend curve tends to remain to keep considerable level in lower temperature around 50 °C. Obtained data would mean that FAC susceptibility of pipeline in condensate demineralizer downstream and deaerator upstream in PWR plants is comparable. In terms of pH, a large drop in FAC rate is seen around pH 9.0 as in previous studies, and its ratio of pH 9.2 to pH 7.0 is approximately 1/10. This ratio fairly agrees with iron solubility in each pH condition at 150 °C while it is smaller in lower temperature condition, which may require additional effect related to pH and temperature to be considered to evaluate the FAC rate. The effect of iron contents on FAC rate is confirmed by referring to saturation solubility of iron. It is suggested that FAC may occur even when the iron concentration in the bulk water is in saturation level, which should be considered in the modelling process for FAC prediction.

Key words : Pipe wall thinning, Flow-accelerated corrosion, Thinning rate, Carbon steel, Low temperature

1. Introduction

For the stable operation and maintaining integrity of power plants, pipe wall thinning is one of the important issues to be managed. Especially, Flow-Accelerated Corrosion (FAC) requires thorough attention with continuous care for its potential of catastrophic ruptures of large diameter pipes in main piping systems, as seen in Surry (U.S.NRC, 1986) and Mihama (NISA, 2004). In Japan, technical management rules for pipe wall thinning have been established based on thinning rate and residual lifetime evaluation relying on pipe wall thickness measurement results, for the use in boiling water reactors (BWR), pressurized water reactors (PWR), and fossil plants (JSME 2006a, 2006b, 2006c).

With wall thickness measurement data, thinning trend at each component can be evaluated, individually. In addition, when more than three measurement data obtained in different outages are available, thinning rate would be calculated with least-square method and the management can be conducted with a certain accuracy and proper conservativeness. However, this may result in enormous number of measurements for each outage in power plants, and in partial, there are some elements evaluated excessively conservative that may be forced to replace much earlier than their essential residual lifetime. On the contrary, there are problems for components whose thickness is hard to be quantified with ordinary measurement tools or in lack of reliable measurement data.

For future improvement of the pipe wall thinning management in Japanese power plants, introduction of prediction method and code for thinning rate and residual lifetime evaluation is expected, while in other countries, several codes or software are already utilized in their plant piping management (Chexal, 1999, Zander, 2003, Persoz, 2006, Tomarov, 2002). Applying such code assured with valid accuracy and reliability, most of the abovementioned problems would be

solved or more relaxed than before.

The authors have been conducting researches to develop pipe wall thinning prediction method for the use in domestic power plants. At first, fundamental data from original experiments and related numerical simulations are obtained, followed by the equation modelling to predict the thinning rate based on thinning phenomena mechanism. After verifying with experimental and power plant data, the prediction model is finally installed into an in-house prediction software developed for the management in domestic power plants, followed by further improvement and renewals of the model and the software. A series of paper will be presented for each step in this developing process. In this paper, basic experiments for FAC are conducted and FAC rate data is obtained which would be used for verification of the prediction model in the next step.

Many experiments for FAC have been done in the past since the 1970's (Keller, 1974, Berge, 1980, Bignold, 1980 etc.), and the thinning trend by FAC has been revealed against essential parameters, such as temperature, pH, dissolved oxygen, chromium content, fluid velocity, and so on. Through these studies and experiences, susceptible condition for FAC is understood for each parameters and the knowledge is applied practically to power plant operation to prevent or to reduce pipe wall thinning by FAC. In terms of temperature, it is well known that the profile of FAC rate shows a peak around 130 to 150 °C, and decrease towards lower or higher temperature region from the peak.

However, recently, it is pointed out that FAC may considerably occur in relatively low temperature around 50 °C, especially in deoxygenated and neutral water condition for both PWR and BWR plants (Crockett and Horowitz, 2009). In addition, actual FAC rate data with even lower temperature condition of 35 °C in BWR condensate line system is shown (Yoneda 2009). Since most of the experimental studies in the past mainly focused on the susceptible temperature condition for FAC (Abe, 2010, Lister, 2011, Utanohara, 2013 etc.), test data obtained with low temperature is scarcely found in publications. Hence, in this study, FAC experiments are conducted including temperature condition lower than 150 °C and down to 50 °C. The obtained data are related and organized with essential parameters and the trend is analyzed comparing to previous studies.

2. Experiment

2.1 Test loop

FAC experiments were conducted with an apparatus named "PRINTEMPS" loop, and its schematic is shown in **Fig. 1**. The apparatus is mainly consisted of low-pressure and low-temperature (LP/LT) loop, and high-pressure and high-temperature (HP/HT) loop. In LP/LT loop, the loop water condition is measured and controlled to maintain water chemical condition for the test. In HP/HT loop, the loop water condition is raised up to the actual plant pipe condition to enhance FAC at the test section. Pure deaerated water is created in the reservoir tank by nitrogen gas bubble purge, and chemical-mixed water is fixed at the chemical tank, where ammonia is mainly contained with certain concentration for PWR condensate condition. The water from three tanks is injected to the HP/HT loop by the injection pump with adjusted mixture. In HP/HT loop, the loop water is driven and circulated by the pump and heated by the dual heaters, and flows through 3 parallel test sections. Each section is equipped with automatic flow rate control valve, which enables to conduct test with three different velocity conditions in one run. A partial of loop water is returned from the HP/HT loop through the heat exchanger and the cooler. After settling at LP/LT condition, water chemical conditions are measured. Measurement items are pH, dissolved oxygen (DO), Redox potential (ORP), and conductivity. The loop water is collected to the drain tank after all the measurements, polished in ion exchange resin, and sent back to the reservoir tank again. All piping and components are made of stainless steel or Teflon tubing, except for the test specimen in the test section.

2.2 Test section

The test section is a vertical rectangular flow duct with a cross-section of 20 mm times 10 mm and a length of 400 mm, as shown in **Fig. 2**. The test specimen is a carbon steel round disk with 40 mm in diameter and 5 mm in thickness, as shown in **Fig. 3**, and it is fitted in the height of 250 mm from the test section inlet. To enhance FAC on the specimen surface, turbulence promoter can be installed in the specimen upstream to create flow contraction. In this situation, test specimen with a cut in the upstream side is used to fit in with the turbulence promoter to the test section. The contraction height of the turbulence promoter (h) can be changed up to 3 mm i.e. contraction area ratio of 0.7. In front of the test section, observation window made of Pyrex glass is set to visualize the specimen surface and to enable optical measurement of the thinning amount.

Fig. 1 Schematic of the experimental apparatus "PRINTEMPS"

Fig. 2 Schematic of the test section
(Left: Decomposed view, Right: Dimensions around test specimen)

Fig. 3 Photos of test specimen

2.3 Test condition

The test loop condition of the experiment is listed in **Table 1**. The main parameter is pH, temperature, velocity and contraction height. Ammonia is injected for alkaline pH control, and no additives for neutral pH. Half of the tests is focused on low temperature condition from 50 to 150 °C with neutral pH, and the other half for pH effect with constant temperature at 150 °C. For each test specimen (TS), mean velocity (V) and contraction height conditions are set individually. DO is kept below 1.0 ppb with continuous nitrogen gas purge into reservoir tank. ORP is not set as an aimed value, however, the loop water is maintained in a reducing condition. Duration time of the tests is not set constant since the decision for terminating each run depends on the confirmation of FAC trend of the specimens which differs between test loop condition or some irregular events which may force loop shut-down. As a result, most of the tests are conducted

around 3 to 4 weeks.

Test specimens for the test are all manufactured from the same carbon steel plate. Its chemical composition is shown in **Table 2**. Chromium content, the most important material parameter for FAC, for the specimen is 0.001 wt% which is very low level in terms of FAC inhibition according to previous studies (Horowitz 2003, Trevin 2005).

Table 1 Matrix of the test loop condition

Test run	Temperature [°C]	pH _{25°C} [-]	Velocity (V) [m/s], Contraction height (h)[mm]			DO [ppb]	ORP [mV]	Duration [hours]
			TS-1	TS-2	TS-3			
#01	50	7.0	4.5m/s, 3mm	4.5m/s, 0mm	10m/s, 0mm	<1.0	-200 ~ -500	690
#02	80	7.0	4.5m/s, 3mm	4.5m/s, 0mm	(N/A)			500
#03	100	7.0	4.5m/s, 3mm	4.5m/s, 0mm	10m/s, 0mm			530
#04	120	7.0	4.5m/s, 3mm	4.5m/s, 0mm	(N/A)			380
#05	150	7.0	4.5m/s, 3mm	4.5m/s, 0mm	10m/s, 0mm			540
#06	150	8.8	4.5m/s, 3mm	4.5m/s, 0mm	10m/s, 0mm			240
#07	150	8.9	4.5m/s, 3mm	3.0m/s, 3mm	1.5m/s, 3mm			600
#08	150	9.0	4.5m/s, 3mm	4.5m/s, 2mm	4.5m/s, 1mm			820
#09	150	9.2	4.5m/s, 3mm	4.5m/s, 0mm	10m/s, 0mm			600
#10	150	9.4	4.5m/s, 3mm	3.0m/s, 3mm	1.5m/s, 3mm			580
#11	150	9.8	4.5m/s, 3mm	3.0m/s, 3mm	6.0m/s, 3mm			450
#12	50	9.2	4.5m/s, 3mm	4.5m/s, 0mm	(N/A)			630

Table 2 Chemical composition of the test specimen material (wt%)

Carbon steel JIS STPT480						
C	Si	Mn	P	S	Cr	Mo
0.25	0.24	0.80	0.017	0.010	0.001	<0.01

2.4 Thinning measurements

Thinning amount of the test specimen is measured with two methods in this experiment; in-situ measurement and post-test measurement, either of them applying optical LASER technique.

For the in-situ measurement, displacement sensors (KEYENCE LK-G150) is used. In front of each test section, the sensors are mounted on a 3-axis automatic stage assembly (SIGMA KOKI SGSP20-35) and the test specimen surface is scanned along a programmed routine lines periodically through the observation window. Scanning area is limited within a small area on the specimen center, 4 mm in width and 10 mm in height. Periodic scanning is conducted with 12 hours interval, and each scanning includes 5 repeats to improve measurement accuracy by calculating the average values for the area.

For the post-test measurement, 3D geometry measurement system (KEYENCE KS-1100) is used. The whole specimen surface is scanned optically after the test, and compared with the initial status scanned before the test.

3. Results and discussions

3.1 Surface observation

After a certain duration time, the test specimen is taken out from the test section. Some photo samples of post-test specimen are shown in **Fig. 4**, all with the same velocity and flow contraction conditions. Compared to the pre-test status shown in **Fig.3**, among the specimen surface, the area exposed to water in the flow duct is seen to be corroded by FAC. For the tests in susceptible condition such as neutral pH or temperature at 150 °C, considerable effect is observed on the exposed surface. For the test in alkaline pH with low temperature condition, thinning is not comparably enhanced as the grinded line of manufacturing is still remained.

Fig. 4 Photos of post-test specimen surface (conditions with $V=4.5\text{m/s}$, $h=3\text{mm}$)

3.2 FAC rate

3.2.1 In-situ measurement results

From the in-situ measurements with laser displacement sensor, thinning propagation and FAC rate are evaluated. An example of scanning data set is shown in **Fig. 5**. As described in chapter 2.4, small limited area in the specimen center is scanned periodically and difference in depth is seen between the data obtained with intervals of about 10 days. Average values for these 2-dimensional scanned data are calculated to evaluate FAC rate. Some examples are shown in the time-series plots in **Fig. 6**. As a trend for each run, nearly constant thinning amount increase can be seen with time, and the FAC rate is estimated by linear approximation. Values of FAC rate are comparable with those estimated by post-test scanning shown afterwards in **Fig. 9(a)**, however, considerable scattering is seen for each plot. Consequently, this in-situ measurement results are referred only for relatively large FAC rate cases.

Fig. 5 Example of in-situ scanning measurement data (Run #05 TS-3)

Fig. 6 Thinning trend obtained by in-situ scanning measurement (pH7.0)

3.2.2 Post-test scanning results

Post-test scanning is conducted for all test specimens to evaluate thinning amount compared with the initial state. An example of the scanned data for the whole surface of a specimen is shown in Fig. 7. Considerable thinning can be seen in the central region exposed to the flow path while the regions aside remained with initial thickness. With flow contraction, maximum thinning region is seen in some distance from the upstream edge, and the thinning amount is gradually decreased towards the downstream.

To compare the results in terms of major parameters, thinning data profile on the vertical center line is extracted for each test condition and converted to FAC rate by simply dividing by duration time. In Fig. 8, effects of temperature, pH, velocity, and contraction height are shown, respectively. As well-known from previous studies, FAC rate increase can be seen with temperature rise from low temperature condition to 150 °C, with pH decrease from alkaline to neutral condition, and with velocity increase. For the test conditions with flow contraction height of 3 mm, shown in Fig. 8 (a), (b) and (c), the profiles are almost resembled as they have a peak around 10 mm from the upstream edge. This is due to the turbulence and recirculation flow created in the contraction downstream, as seen in downstream of orifice installed in pipelines (Bignold, 1980, NISA, 2004, Makino, 2006, Utanohara, 2012 etc.). When the contraction height is lowered, the peak position is shifted towards the upstream edge as shown in Fig. 8(d).

Fig. 7 Example of post-test scanning for the whole specimen surface (Run #05 TS-1)

Fig. 8 FAC rate profiles in streamwise direction

Fig. 9 Normalized FAC rate profiles for h=3mm contraction cases

In the following section, averaged FAC rate values are compared. The average values are calculated based on the certain section from the streamwise profile plotted in Fig. 8, i.e. region of 5 to 20 mm from upstream edge. This region was selected since, for 3mm contraction height case, it represents the common profile including the peak at 10 to 15 mm from upstream edge for most of the cases as shown in Fig. 9, and for no contraction case, it may exclude small disturbance

effect of specimen upstream edge.

3.2.3 Average data in terms of major parameters

Present FAC rate data in terms of temperature is shown in **Fig. 10(a)**. FAC rate is seen to decrease from 150 °C to lower condition, and the ratio of the FAC rate at 50 °C to 150 °C is approximately 1/7 to 1/6. However, the trend curve is not approaching towards negligible rate in low temperature condition, and instead remains to keep considerable level which may require attention. For instance, FAC rate for 50 °C with pH 7.0 is nearly the same or rather larger than that of 150 °C with pH 9.2. This would mean that in PWR plant pipeline, FAC susceptibility in condensate demineralizer downstream with temperature around 30 to 50 °C and neutral pH at before pH agent injection point, where wall thickness inspection was not recommended or required in the past (EPRI, 1999, Yamakami, 2008), and deaerator upstream with temperature around 140 to 150 °C and alkaline pH about 9.2 in normal AVT (All Volatile Treatment) condition, known to have large potential of catastrophic pipe rupture (NISA, 2004), is comparable.

To confirm the availability of FAC rate data in low temperature region obtained in the past, and to compare qualitative trend in terms of temperature, present data is plotted with those of previous studies (Bignold, 1980, Heitmann, 1982, Tsuruta, 2006, Fujiwara, 2010) in **Fig. 10(b)**. As mentioned in the introduction, not much data is obtained so far and no previous data was found to extend down to 50 °C besides the present. At least, FAC rate decrease from temperature about 120 °C down to about 80 °C seems to be a common qualitative trend for all of the data. And for temperature region below 80 °C, some data keep decreasing while the others, including the present, do not decrease. Further extension of data acquisition in low temperature region is required to confirm overall trend.

Fig. 10 Average FAC rate trend in terms of temperature

Present FAC rate data in terms of pH is shown in **Fig. 11(a)**, and a large drop is seen around pH 9.0. In **Fig. 11(b)**, data are converted to relative values based on neutral pH data, and plotted with data from previous studies (Abe, 2010, Kastner, 1990). Most of the data are seen to show almost similar behavior in the region from neutral pH to alkaline pH of about 9.5, and the ratio of the FAC rate at pH 9.2 to pH 7.0 is approximately 1/10.

Fig. 11 Average FAC rate trend in terms of pH

According to previous studies, FAC rate is strongly related to iron solubility, and in some simple FAC model proposed, it is shown that FAC rate is proportional to iron solubility. Iron solubility trends for pH 7.0 and pH 9.2 are shown in **Fig. 12** in terms of temperature (Fujiwara, 2011) calculated with hydrolysis constants of ferrous ions derived in previous study (Tremaine, 1980). When the temperature is 150 °C, the ratio of solubility between the two pH conditions is approximately 1/10 while it is 1/30 at 50 °C. Hence, FAC rate difference between neutral pH and alkaline pH in the temperature at 150 °C may be explained with the solubility, and for lower temperature condition, additional effect related to pH should be considered to evaluate the FAC rate.

Fig. 12 Comparison of iron solubility between pH 7.0 and 9.2

Present FAC rate data in terms of velocity is shown in **Fig. 13**. FAC rate is seen to increase monotonic with velocity, and it is proportional to the power of mean velocity with exponent from 1.0 to 1.5, for cases with considerable FAC rate. This exponent for velocity dependency varies between previous data (Bignold, 1980, Heitmann, 1982) which may be influenced by the turbulence condition enhanced due to experimental geometry.

Fig. 13 Average FAC rate trend in terms of velocity

3.3 Oxide film composition by XRD

To identify oxide film composition formed on test specimen surface, analysis with X-ray diffraction (XRD) spectroscopy is conducted for representative specimens. Fe_3O_4 (magnetite), $\alpha\text{-Fe}_2\text{O}_3$ (hematite) and $\gamma\text{-FeOOH}$ (Lepidocrocite) are set as the target composition of the oxide film. The results are shown in **Fig. 14**. In test condition with temperature of 150 °C, magnetite is found in the oxide film, especially in high pH condition. For the low temperature condition of 50 °C, very slight indication of magnetite is found in neutral pH condition, while none of the targeted compounds are detected in alkaline pH condition. There may be very thin oxide film formed below the detection level with the utilized equipment, which require to re-analyze in the future. As a whole, magnetite is only found in the oxide film on present test specimen surface which is one of the conditions that proves the occurrence of FAC.

Fig. 14 XRD analysis results for test specimen surface

3.4 Iron concentration in the loop

When FAC is simply modelled considering as a diffusion process of dissolved iron from oxide film surface to the bulk water by fluid induced mass transfer, Fick's law will be implemented. In this case, the driving force of diffusion will be the dissolved iron concentration difference between the oxide film surface and the bulk water. In this sense, when iron concentration in the bulk water is in saturation level, there would be no driving force which means no occurrence of FAC. This may also be adapted to a sophisticated FAC model such as Sanchez-Caldera's (1988).

The test loop used in the present study is not a once-through loop, and released iron originated from test specimen would increase and accumulate to a certain amount within the test duration time. However, the iron concentration will be balanced at a maximum value which is naturally determined as the ratio of dissolution rate from test specimens M to the flow rate of the injection pump q . Total iron concentration in the upstream of the test section at a certain time $C(t)$ can be

expressed, by adding the concentration in former time period and iron supply from the test specimens, which would be the concentration in the test section downstream, and subtract the extracted portion to the LP/LT loop, as Eq. (1).

$$C(t) = \left[C(t-1) + \frac{M}{Q} \right] \left(1 - \frac{q}{Q} \right) = \frac{M}{Q} \sum_{i=1}^t \left(1 - \frac{q}{Q} \right)^i \quad (1)$$

Q is the flow rate of the circulation pump in the HP/HT loop. Dissolution rate M is calculated by summarizing iron release from the exposed area of three test specimen surface by assuming constant FAC rate based on experimental results. With sufficient duration time, total iron concentration in the loop will eventually saturate as in Eq. (2).

$$C(t) \rightarrow \frac{M}{Q} \left[1 / \left\{ 1 - \left(1 - \frac{q}{Q} \right) \right\} \right] = \frac{M}{q} \quad (2)$$

This calculation is based on the assumption that total iron is dissolved, and in reality, some portion should be in non-dissolved form. In that sense, the term “total iron contents” is applied instead of total iron concentration. It is also assumed that there is no dissolved iron supplied from the LP/LT loop, based on the measured results of conductivity in the pure water tank showing 0.06 to 0.08 $\mu\text{S}/\text{cm}$, which is nearly in the pure water level and proving that most of the impurities are removed when passing through the ion exchange resin and filters.

In **Fig. 15**, the assumable maximum value of total iron contents for each test run is compared with saturation solubility of iron in each test run's temperature and pH condition. This figure shows that in about half of the test runs, total iron contents is nearly equal to or larger than saturation solubility. This would mean that the dissolved iron concentration of the loop water is nearly in saturation level, and iron compounds should also exist as flowing particles or deposits in the loop. However, as shown in **Fig. 6**, at least in neutral pH condition, thinning rate is confirmed to be constant during the test. For alkaline pH condition, although the thinning rate is not confirmed from in-situ measurement, FAC rate data are shown to present similar qualitative behavior with previous studies, which may be assumed to be valid. Hence, it is suggested that FAC may occur even when the iron concentration in the bulk water is in saturation level, which should be considered in the modelling process for FAC prediction.

Although the reason for this results cannot be clearly explained, it may be hypothesized qualitatively by considering condition difference between the bulk and the specimen surface. At the specimen surface, hydrogen will be produced through oxidation process of iron in water, and hydrogen concentration would be relatively higher than that in the bulk. In general, iron solubility is in proportion to the one-third power of partial pressure of hydrogen (Sweeton, 1970, Tremaine, 1980). Accordingly, iron solubility at the specimen surface would be relatively higher than that in the bulk. In Fig. 14, saturation solubility is evaluated based on the bulk condition, however, actual solubility at the specimen surface may be higher which may relatively lower total iron contents than saturation level.

4 Conclusions

Experiments for FAC of carbon steel specimens were conducted and basic data were obtained. Especially, data in relatively low temperature condition down to 50 °C were acquired, which is scarcely found in previous studies although FAC susceptibility in low temperature pipelines are pointed out with care in plant management, recently.

In terms of temperature, FAC rate is seen to decrease by lowering temperature and the ratio of FAC rate at 50 °C to 150 °C is approximately 1/7 to 1/6, and the trend curve tends to remains to keep considerable level in lower temperature. FAC rate for 50 °C with pH 7.0 is nearly the same or rather larger than that of 150 °C with pH 9.2, which would mean that FAC susceptibility of pipeline in condensate demineralizer downstream and deaerator upstream in PWR plants is comparable.

In terms of pH, a large drop in FAC rate is seen around pH 9.0 as in previous studies, and its ratio at pH 9.2 to pH 7.0 is approximately 1/10. This is compared to iron solubility for its correlation with FAC rate, and when the temperature is 150 °C, the ratio of solubility between the two pH conditions is approximately 1/10 while it is 1/30 at 50 °C. Hence, FAC rate difference between neutral pH and alkaline pH in condition with 150 °C may be explained with the solubility, and for lower temperature condition, additional effect related to pH should be considered to evaluate the FAC rate.

Total iron contents in the test loop is compared with saturation solubility of iron to confirm its effect on FAC rate, and in about half of the test runs, total iron contents is nearly equal to or larger than saturation solubility, although FAC rate remained constant within the test duration. It is suggested that FAC may occur even when the iron concentration in the bulk water is in saturation level, which should be considered in the modelling process for FAC prediction.

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