

REDUCING TUBE BUNDLE DEPOSITION WITH ALTERNATIVE AMINES

C.W. Turner^{*}, S.J. Klimas^{*}, and P.L. Frattini[#]

ABSTRACT

Particle deposition rates have been measured in a high-temperature loop for magnetite and hematite depositing onto Inconel-600 under flow-boiling conditions with pH controlled using one of the following amines: morpholine, ammonia, ethanolamine, or dimethylamine. Hematite particles deposited at rates an order of magnitude greater than those measured for magnetite, although the hematite deposition rate dropped when the loop was operated under reducing conditions. The magnetite deposition rate was influenced by the amine used to control the pH, with the relative rate decreasing in the following series: morpholine (1) : ethanolamine (0.72) : ammonia (0.51) : dimethylamine (0.25). These trends in deposition rate are discussed in terms of the surface chemistry of the corrosion products. Deposition rates for both magnetite and hematite increased significantly once the mixture quality exceeded about 0.3, which may be related to a change in the heat transfer mechanism from nucleate boiling to two-phase forced-convection through a thin film.

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INTRODUCTION

Corrosion products that are produced in the balance-of-plant are transported to the steam generator (SG) where they deposit on the tube bundle, tube-sheet, and the tube support structure. The adverse effects of deposit accumulation on the performance once-through and recirculating SGs is well documented (1,2). For example, deposition on the tube bundle can reduce the thermal efficiency of the SG. The build-up of deposit on the tube support structure tends to restrict the flow paths, which can lead to excessive pressure drop across the tube support structure and, ultimately, to operational problems. Finally, deposits that form on the tubes and within the tube/tube-support and tube/tube-sheet crevices can become sites for localized corrosion and subsequent tube failure.

Although much work has been done to optimize the selection of pH-control reagents, i.e., amines, to minimize the generation and transport of corrosion products from the balance-of-plant to the SG (3,4), very little was known about how different amines might influence the deposition rates of corrosion products once they have reached the steam generator. Thus, a program co-funded by Atomic Energy of Canada Limited and the Electric Power Research Institute was initiated to determine the effect of the choice of pH-control reagent on the deposition rates of corrosion products under typical SG operating conditions (5). We report here the affect of the choice of pH-control reagent on the deposition rates of magnetite and hematite under flow-boiling conditions.

EXPERIMENTAL PROCEDURES AND ANALYSIS

Procedures

Details of the experimental methods and analyses are reported elsewhere (5). The deposition experiments reported here were performed under flow-boiling conditions in the H-3 high-temperature loop located at the Chalk River Laboratories (CRL). A schematic of the loop is shown in Figure 1. The main components of the loop are chemical addition tanks and sampling lines to control loop chemistry, a positive displacement pump and flow control valve to regulate the flow rate and loop pressure, respectively, an interchanger to preheat the water upstream of the test section, and a heated test section where deposition rates are measured under the thermohydraulic conditions of interest. The nominal test conditions used for the deposition experiments are shown in Table 1.

All tests were performed using Inconel 600 for the heated test section. Each test section was rinsed sequentially with hexane and methanol, and then pre-conditioned in the loop under experimental conditions for 48 hours. The experiments were performed using one of the following 5 pH-control

reagents: morpholine, ammonia, ethanolamine, dimethylamine, and potassium hydroxide. The high and low-temperature pH and calculated concentrations of free base are listed in Table 2. The high-temperature pH was calculated for conditions in the test section at a steam quality of zero.

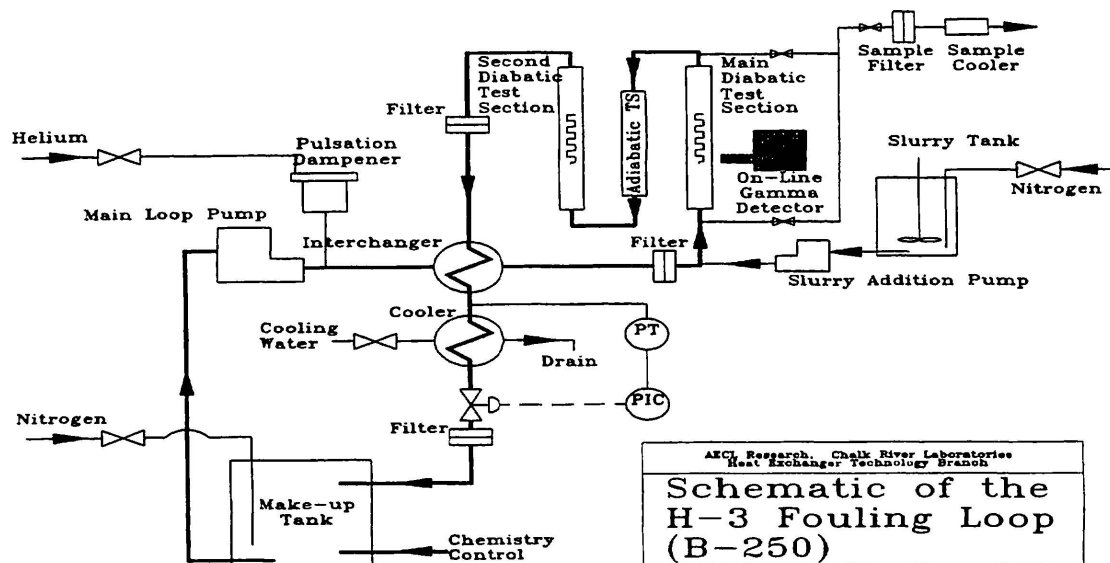


Figure 1: Schematic of the loop used for the deposition experiments.

During each experiment, a suspension of radioactive corrosion product, i.e., magnetite or hematite, was continuously injected from the suspension tank into the loop at a location approximately 2 m upstream of the heated test section. A positive displacement chemical addition pump was used for this purpose, and the injected stream was diluted in the main loop circuit by a factor of approximately 200. The deposition of the colloidal particles onto the test section was monitored by a high-efficiency on-line γ -ray detector (high-purity germanium). Particles that did not deposit were removed by a system of filters downstream of the test section before the water was returned to the loop main tank. The magnetite used in this investigation was prepared by aging a fresh precipitate of $\text{Fe}(\text{OH})_2$ for 2 h under mildly oxidizing conditions at 90°C and $\text{pH} \approx 9$ (6). The phase purity of the final product was confirmed by x-ray diffraction. A commercial reagent-grade product was used for the hematite deposition tests. For each test, a portion of the corrosion product was activated in the NRU reactor at CRL to produce the radioisotope ^{59}Fe , which then served as a radiotracer to monitor deposition on the test section. The activated corrosion product was dispersed in water in an ultrasonic bath and equilibrated with base in the suspension tank at the target pH prior to injection into the loop.

The concentration of corrosion product in the loop was determined by hot filtration using a silver membrane filter with $0.2 \mu\text{m}$ pore size. Sampling either the inlet or outlet of the test section gave essentially identical results, which is consistent with the magnitude of the deposition rates reported below. The filtrate was analysed for pH, dissolved oxygen, and hydrazine. Samples of the activated suspension were taken from the suspension tank and dissolved to determine the specific activity of the corrosion product.

After the completion of each experiment the test section was cut into 30 mm sections so that the radioactivity on selected sections could be measured using an off-line γ -ray detector.

Table 1: Nominal test conditions used for the deposition experiments.

Thermalhydraulic Conditions	
Pressure	5.6 MPa (absolute)
Mass Flux	300 kg/m ² s
Heat Flux	250 kW/m ²
Steam Quality	-0.15 to 0.25*
Chemistry Conditions	
pH _T	6.2
Dissolved oxygen	< 10 μ g/kg
hydrazine	10 - 100 μ g/kg
corrosion product (loop)	1 mg/kg

*: Steam quality extended to 0.52 towards the end of the test program.

Deposit activity was converted to deposit loading using the specific activity of the corrosion product, and the deposition rate calculated from deposit loading by assuming a linear rate of build-up on the test section. This assumption was confirmed for each test using the on-line γ -ray detector. Deposit morphology was determined using Scanning Electron Microscopy (SEM).

Table 2: Conditions used to achieve a high-temperature pH of 6.2 in the test section at zero steam quality.

pH control reagent	pH _{270 °C}	pH _{25 °C}	Concentration (mg/kg)
Morpholine	6.2	9.28	11.8
Ethanolamine	6.2	9.57	5.0
Ammonia	6.2	9.67	3.0
Dimethylamine	6.2	9.18	0.70
Potassium hydroxide	6.2	9.02	0.50

Data Analysis

The deposition rate determined by the simultaneous deposition and re-entrainment of particles is given by:

$$\frac{dm}{dt} = K_{2\phi}\rho_{2\phi}C(t) - \lambda m(t). \quad (1)$$

The re-entrainment rate, $\lambda m(t)$, was negligible under the experimental conditions of this investigation and, therefore, it could be omitted from Equation 1. Thus, the deposition rate was normalized with respect to concentration of corrosion product to calculate the normalized deposition rate constant:

$$K_{2\phi} \rho_{2\phi} = \frac{dm}{dt} \frac{1}{C(t)}, \quad (2)$$

The rate constant, $K_{2\phi} \rho_{2\phi}$, does not change significantly with steam quality, hence is more practical to use than $K_{2\phi}$ (m/s).

The deposition rate constant under fully developed nucleate boiling conditions, $K_{2\phi}$, consists of 2 components: the forced-convective and the boiling deposition rate constants:

$$K_{2\phi} = K_{FC} + K_b. \quad (3)$$

At zero steam quality, it has been shown (7,8) that, to a very good approximation:

$$K_{2\phi} \cong K_b. \quad (4)$$

The boiling component, K_b , has been postulated to be proportional to the rate of vaporization of the liquid (9). Consequently, the proportionality constant, known as the boiling deposition coefficient, a_b , can be evaluated using the following equation:

$$a_b = K_b \rho_{2\phi} \frac{H_{f-g}(1-X)}{q''}. \quad (5)$$

This is permissible for X close to zero because the value of the forced-convective deposition rate constant, K_{FC} , is very low in comparison to the boiling deposition rate constant, K_b , at both subcooled conditions and at low mixture qualities. The deposition coefficient a_b is, in effect, a deposition rate constant normalized with respect to heat flux and pressure (the value of the heat of vaporization, H_{f-g} , is pressure-dependent). Its magnitude represents the fraction of suspended crud deposited per kg of liquid evaporated.

The local thermodynamic steam quality in Equation 5 is calculated from the expression:

$$X(l) = \frac{H_{in} + \frac{q}{F} \frac{l}{L} - H_{SAT}}{H_{f-g}}. \quad (6)$$

RESULTS

Magnetite Deposition onto Inconel-600

Figure 2 shows an example of the buildup of the decay-corrected activity of ^{59}Fe on the heated test section during the course of an experiment, as recorded by the on-line γ -ray detector for $X \approx 0$.

The data in Figure 2 are for the deposition of magnetite onto Inconel 600 under morpholine pH control (experiment D35), but the results are typical for the other experiments in this investigation. There is a stepwise increase in activity upon commencement of the injection of active suspension into the loop at $t = 0$. Thereafter, the activity measured by the on-line γ -ray detector increases linearly with time, signifying a constant rate of buildup of deposit on the test section. After the injection pump is switched off at $t = 10$ h, the deposit activity slowly decreases as particles are “scrubbed” from the surface by the flowing steam/water mixture. It is clear from Figure 2 that the rate of removal is significantly lower than the rate of deposition, thus justifying the use of Equation 2 for the calculation of the deposition rate constant.

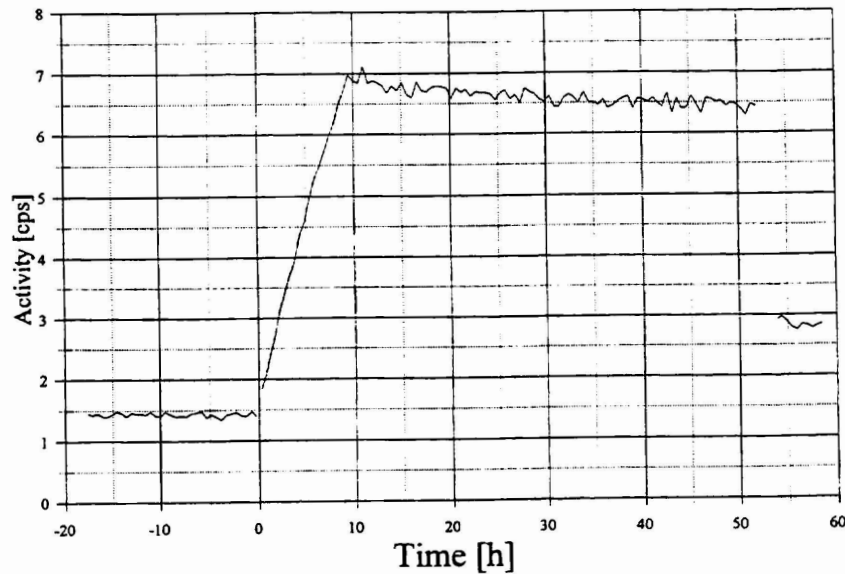


Figure 2: Example of the buildup of activity on the test section during the typical experiment, as measured by the on-line γ -detector (Experiment D35)

Figure 3 shows typical examples of the trend in wall superheat and normalized deposition rate constant, $\rho_{2\phi}K_{2\phi}$, as a function of mixture quality along the length of the test section for magnetite depositing onto Inconel 600 with pH controlled by morpholine (a), ethanolamine (b), ammonia (c), and dimethylamine (d). The wall superheat averaged 7.4°C in the tests. A local maximum in the deposition rate was normally observed between $X = -0.1$ and 0.0 , in the vicinity of the transition from sub-cooled to saturated nucleate boiling heat transfer. In some cases the deposition rate dropped significantly with increasing mixture quality, as illustrated in Figure 3 (b), while in other cases the deposition rate was almost independent of mixture quality in the range $X = 0$ to $X = 0.25$, as shown in Figure 3 (a). Another trend illustrated in Figure 3 is that the deposition rate of magnetite under flow-boiling conditions is dependent upon the volatile amine used for pH-control, with the highest rates measured under morpholine pH-control and the lowest rates observed when pH was adjusted using dimethylamine.

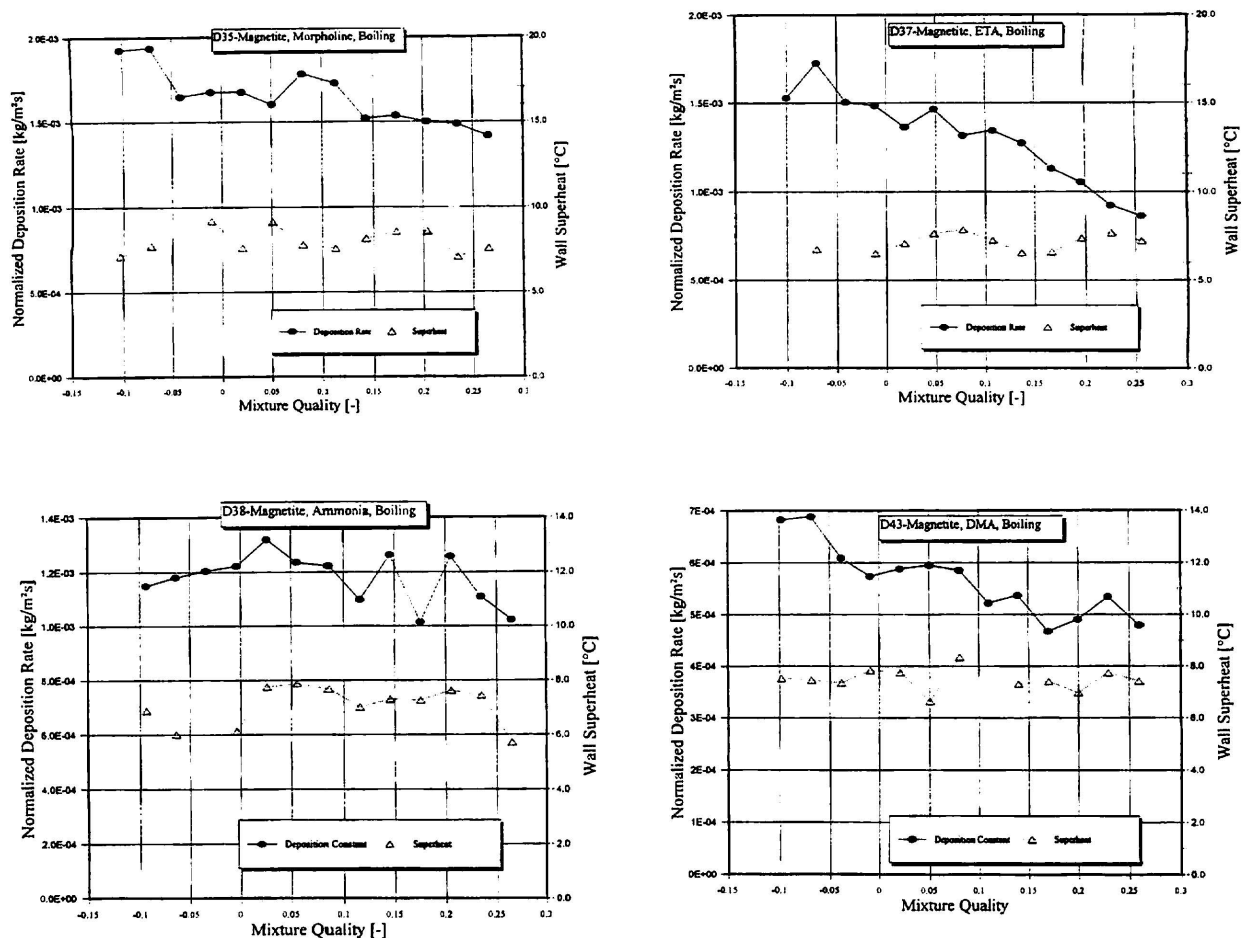


Figure 3: Typical examples of the normalized deposition rate constant and wall superheat versus mixture quality for magnetite deposition onto Inconel-600 for pH-control with morpholine, ammonia, ethanolamine, and dimethylamine.

Figure 4 shows a comparison of the normalized deposition rate constants, averaged for mixture qualities from 0 to 0.25, for magnetite particles depositing under flow-boiling conditions with the four different amines used in this investigation. Each entry in the Figure is itself an average of the results from at least two, and sometimes as many as seven, separate experiments. A complete list of the tests performed and results from individual tests is given in Reference 5. Also shown in the Figure are average results for the deposition of hematite particles under flow-boiling conditions, which are discussed in the next section.

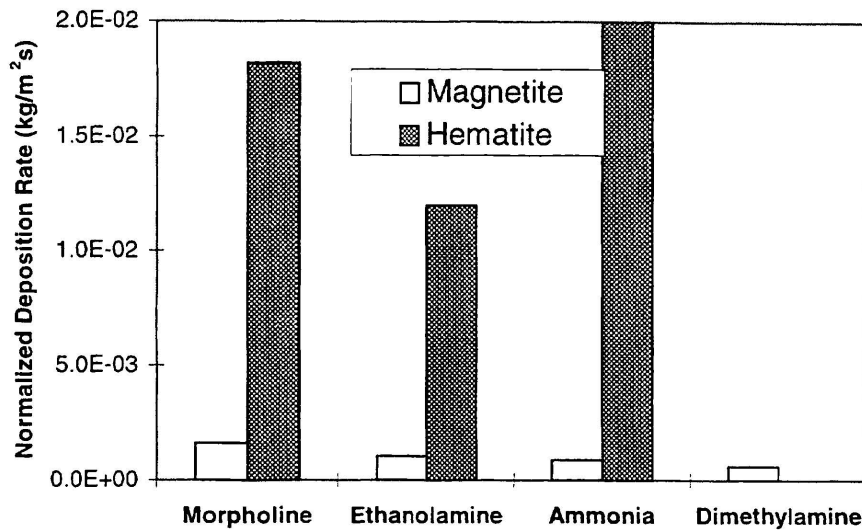


Figure 4: Impact of amine used for pH control on the normalized deposition rate constants, $\rho_{2\phi} K_{2\phi}$, averaged for $X = 0 - 0.25$ for magnetite and hematite deposition under flow-boiling conditions.

Hematite Deposition onto Inconel-600

Examples of the trend in wall superheat and the normalized deposition rate constant, $\rho_{2\phi} K_{2\phi}$, with mixture quality for the deposition of hematite particles onto the surface of Inconel-600 under flow boiling conditions are shown in Figure 5. The wall superheats and the general features of the trend in deposition rate with mixture quality for hematite deposition are similar to those noted previously for magnetite deposition under the same conditions. One significant difference, however, is that the hematite deposition rates are about an order of magnitude higher than the rates measured for magnetite depositing under similar operating conditions. The hematite results were less consistent, however, from one test to another with the same amine, with some rates being significantly lower than others. The differences were eventually traced to the concentration of dissolved oxygen in the loop water. All tests were done with hydrazine concentrations ranging from 50 to 150 $\mu\text{g/kg}$, while dissolved oxygen concentrations ranged from 0 to 25 $\mu\text{g/kg}$. In 4 out of 5 cases, the tests with low hematite deposition rates were the ones in which the dissolved oxygen concentration was zero. (See, for example, Figure 5(d)). For this reason, the deposition rates for hematite shown in Figure 4 are averaged over only those tests which had a residual oxygen concentration $\geq 5 \mu\text{g/kg}$. No result is shown for DMA because neither of the two DMA tests satisfied this criterion.

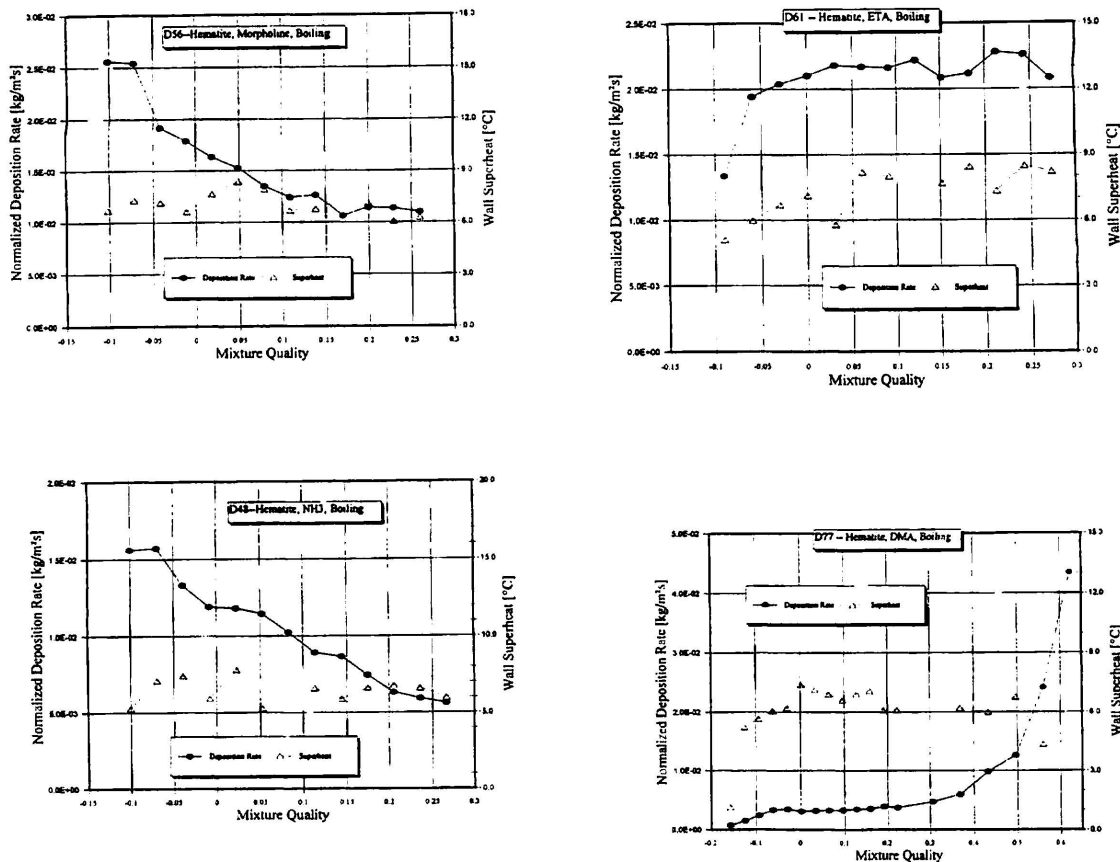


Figure 5: Normalized deposition rate constant and wall superheat versus mixture quality for hematite deposition onto Inconel-600 for pH-controlled with morpholine, ammonia, ethanolamine, and dimethylamine.

Deposition at High Steam Quality

Towards the end of this test program, modifications were made which extended the steam quality at the outlet of the test section from 0.25 to just over 0.60. Figure 5 (d) shows a typical example of the impact that operating at high steam quality has on the particle deposition rate. Although the results shown are for hematite depositing under dimethylamine pH-control, similar results were observed for both magnetite and hematite regardless of the amine used to control pH. The steam quality at which the deposition rate began to increase significantly varied somewhat from test to test. In two case elevated deposition rates were observed at mixture qualities as low as 0.15, but in general the behaviour was as shown in Figure 5 (d); a gradual increase in rate between $X = 0.3$ and $X = 0.5$, followed by an abrupt increase in rate for $X > 0.5$.

DISCUSSION

The work presented here has demonstrated that:

- The deposition rate of hematite is significantly higher than for magnetite under flow-boiling conditions
- The deposition rate of magnetite is dependent upon the nature of the amine used for pH-control
- The deposition rate of hematite is sensitive to the dissolved oxygen concentration when the water contains 50 to 150 $\mu\text{g/kg}$ hydrazine
- The particle deposition rate under flow-boiling conditions increases significantly for mixture qualities in excess of 0.3.

The difference between the deposition rates of magnetite and hematite is most likely related to differences in the sign of the surface charges of these corrosion products at $\text{pH}_{270} = 6.2$. It has been shown previously by room temperature loop deposition tests performed as a function of pH that the particle deposition rate is highest when the particle and substrate are oppositely charged, and that the deposition rate is reduced significantly when the pH is adjusted to a region where the particle and substrate have the same sign of charge (10,11). The reduction in the deposition rate in the latter case is attributed to the force of repulsion that develops between similarly-charged surfaces (12,13).

The forces that affect the particle deposition rate are the same ones that determine whether or not a colloidal suspension of particles is stable against agglomeration. Factors that reduce the magnitude of the force of surface repulsion act to both decrease the stability of a colloidal suspension and to increase the rate of particle deposition. Conversely, increasing the magnitude of the repulsive force tends to increase the stability of a colloidal suspension and to decrease the rate of particle deposition. The fact that the deposition rate of magnetite particles onto Inconel 600 is lower than the rate for hematite suggests that the surfaces of magnetite and Inconel 600 are similarly charged at $\text{pH}_{270} = 6.2$, whereas the surfaces of hematite and Inconel 600 appear to be oppositely charged.

The sign of the charge on the surface of a metal oxide at any given pH is determined by the PZC, or point of zero charge, of the oxide. The surface will be negative when the pH is greater than the PZC and positive when the pH is less than the PZC. Shoonen (14) has calculated the PZC for a number of metal oxides for temperatures up to 350°C, and predicts that the PZCs for magnetite and hematite at 270°C are at pH 4.6 and 6.7, respectively. Thus, for the tests under flow boiling conditions, i.e., $\text{pH}_{270} = 6.2$, the magnetite particles are predicted to be negatively charged and the hematite particles are predicted to be positive. From the relative deposition rates of magnetite and hematite, it would appear that the surface of Inconel 600 is also negatively charged under the experimental conditions. This is corroborated by streaming current measurements performed at room temperature, which found a PZC of 4.2 for Inconel 600 that had been autoclaved for 24 h in water adjusted to pH 9 with morpholine (15).

The tests showed that the average particle deposition rate for magnetite under flow-boiling conditions is dependent upon the amine used for pH-control, and decreases in the following order (normalized to the rate with morpholine):

morpholine (1) : ethanolamine (0.72) : ammonia (0.51) : dimethylamine (0.25).

Additional tests showed that the lowest deposition rates were measured when a non-volatile, inorganic base, such as potassium hydroxide, was used to control the pH (5).

The equilibrium between a base and its conjugate acid is given by:



It is postulated that adsorption of the conjugate acid, HA^+ , onto the surface of the negatively charged magnetite particle increases the particle deposition rate by making the surface of the magnetite particle less negative. The upper limiting case is given by deposition of a positively charged particle, i.e., hematite, which is observed to have a deposition rate that is one order of magnitude greater than that for magnetite. The lower limiting case is given by deposition of magnetite where adsorption of the positively charged conjugate acid is zero. This corresponds to the maximum negative charge possible on magnetite for a given pH_T , and was likely achieved to a good approximation in the tests with pH controlled using potassium hydroxide.

The dependence of the deposition rate of hematite on trace concentrations of dissolved oxygen in the loop is consistent with other evidence that surface charge plays a strong role in determining the particle deposition rate under flow-boiling conditions. In those tests which had residual hydrazine and zero dissolved oxygen in the loop, the surface of the hematite particles would have been thermodynamically unstable with respect to reduction to magnetite. Since the deposition rate of magnetite is significantly lower than that for hematite, any tendency for the surface of the corrosion product to convert from hematite to magnetite would be expected to result in a reduction in the deposition rate, as observed. The kinetics for the reduction of hematite to magnetite are known to be slow, and it is not argued here that a significant fraction of the hematite particle has been reduced to magnetite. The measured deposition rates imply, however, that only the surface layer needs to be reduced to affect the particle deposition behaviour, and that reduction of the surface layer is relatively fast.

It is not clear why the particle deposition rate increases significantly for mixture quality in excess of ≈ 0.3 . According to the Hewitt and Roberts flow pattern map for vertical flow (16) there is a transition from churn flow to annular flow at $X \approx 0.17$, but there is no apparent change in deposition behaviour at this mixture quality. It has been noted previously (5), however, that trends in the heat transfer coefficient with thermodynamic quality are a good indicator of the trend in particle deposition rate, i.e., both heat transfer and particle deposition rates increase in a similar fashion as the mode of heat transfer changes from single-phase forced-convection to sub-cooled nucleate boiling to saturated nucleate boiling. There is a transition in the annular flow regime from saturated nucleate boiling to two-phase forced-convection through a liquid film which is accompanied by a cessation of nucleate boiling and an increase in the heat transfer coefficient. The impact of this change in the mode of heat transfer on the expected particle deposition rate is under investigation.

The results of this investigation suggest that both high steam qualities and oxidizing conditions in the feedtrain may lead to higher deposition rates in the SG. In addition, the choice of amine used for pH control in the balance of plant may affect the deposition rate of magnetite in the SG, depending on the extent to which the amine adsorbs onto the surface of magnetite corrosion products.

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NOMENCLATURE

a = coefficient	-	λ = removal rate constant	(1/s)
l = distance along test section	(m)	ρ = density	(kg/m ³)
m = deposit mass	(kg/m ²)	<i>Subscripts</i>	
q = applied power	(kW)		
q'' = heat flux	(kW/m ²)		
t = time	(s)		
C = concentration	(kg/kg)	b = boiling	
F = flow rate	(kg/s)	fc = forced-convection	
H = enthalpy	(kJ/kg)	f-g = vaporization	
K = deposition rate constant	(m/s)	in = inlet	
L = test section length	(m)	sat = saturation	
X = mixture quality	-	2 ϕ = two-phase	

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DISCUSSION

Authors: C.W. Turner, S.J. Klimas, P.L. Frattini, AECL and EPRI

Paper: Reducing Tube Bundle Deposition with Alternative Amines

Questioner: M. Rootham, Westinghouse

Question/Comment:

You have considered the effect of base strengths and amine concentration on the rate of deposition. The stability of the amine/metal complex should also be evaluated (or considered). (The stability of the complex formed on the iron oxide surface may determine whether the particle sticks to another surface.)

Response:

Our continuing investigation into the effect of amines on particle deposition under flow-boiling conditions includes measurements of the adsorption and desorption of amines on the surface or corrosion products as a function of temperature. By correlating what is happening at the surface of the corrosion product with particle deposition rates, we hope to get a better understanding of why different amines give different deposition rates.

Questioner: J. Gorman, Dominion Engineering

Question/Comment:

Could you comment on your particle sizes, how representative they are of actual particles in plants, and how particle sizes could affect results?

Response:

Sampling of corrosion products in the feedtrain shows particle sizes ranging from sub-micron to cover 10 microns in size. However, examination of tube deposits shows that they are composed primarily of particles in the micron and sub-micron range; the larger particles evidently do not contribute significantly to the tube-bundle deposit. The particle size used in our loop tests was 0.15 microns for hematite and 0.25 microns for magnetite, which puts them at the low end of the size range observed on tube deposits from the stations.

Questioner: D. Lister, University of New Brunswick

Question/Comment:

The experiments you just showed us involved initially clean test sections. What would be the effects of starting with test sections that were already fouled and that could, therefore, have different modes of heat transfer?

Response:

We know from work with clean tubes that the tube surface morphology is important both for heat transfer and for particle deposition rate. We tried some preliminary tests with pre-fouled tubes where the tube deposit was produced artificially. We don't think the deposit morphology was representative of tube deposits in the SG, so we are working on ways of making the morphology more representative before proceeding with more deposition tests with pre-fouled tubes.

Questioner: P. Berge, EDF

Question/Comment:

Are you aware of plant data confirming your data on the difference in sludge deposits with the different amines? I do not think that we observed marked differences, in EDF plants, with either ammonia or morpholine.

Response:

We do not, at present, have a plant data base to compare our measurements with. The amount of deposit on the tube bundle depends on the plant operating history; e.g., number of start-ups, crud burst during start-up, etc., and all of this would have to be taken into account when comparing tube bundle deposits from one plant to another. It would be very valuable to compare tube deposit data from either new plants or plants that have been recently chemically cleaned that are operating with different amines for pH control.

Questioner: R. Staehle, University of Minnesota

Question/Comment:

Do you see an effect of potential on the formation of deposits? Here, by potential, I mean the combined effect of hydrogen (or lack of it - as it raises the potential) and hydrazine (as it lowers potential) in the homogenous solution in the homogenous redox sense. The same implication would apply to the heterogeneous tube electrode.

Response:

The pH was fixed and the hydrazine concentration maintained between 50 and 100 µg/kg for all the tests in the series, so the electrochemical potential of I-600 likely did not change from one test to the other. It was not a parameter that was measured or varied systematically during the investigation.

