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Hideout in Steam Generator Tube Deposits

**Impuretés dans les dépôts des tubes de générateur
de vapeur**

P.V. Balakrishnan, K.J. Franklin, C.W. Turner

AECL

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by

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**IMPURETÉS DANS LES DÉPÔTS DES TUBES DE GÉNÉRATEUR DE VAPEUR**

par

P.V. Balakrishnan¹, K.J. Franklin² et C.W. Turner²**RÉSUMÉ**

On a effectué des études sur les impuretés des dépôts des tubes de générateur de vapeur en utilisant des tubes revêtus de magnétite. On a suivi les impuretés des solutions de chlorure de sodium à 279 °C en utilisant une sonde de conductivité à haute température en ligne, ainsi qu'en effectuant une analyse chimique des échantillons de solutions provenant de l'autoclave dans lequel on a effectué les études. On n'a observé des impuretés importantes qu'à un flux thermique supérieur à 200 kW/m², correspondant à une chute de température supérieure à 2 °C sur les dépôts. Le facteur de concentration résultant des impuretés a augmenté fortement de façon non linéaire avec le flux thermique (variant autant que la puissance quatre du flux thermique). La diminution du facteur de concentration apparente avec une épaisseur de dépôt croissante sous-entendait que les interstices dans le dépôt étaient occupés par un mélange de vapeur et d'eau, ce qui est en accord avec la conclusion des mesures de conductivité thermique des dépôts d'une étude distincte. Des analyses des dépôts après les essais d'impuretés n'ont montré aucune évidence d'espèces de soluté dans ces impuretés, probablement du fait que les concentrations sont très proches des limites de détection et également en raison du fait qu'elles s'échappaient du dépôt à la fin des essais. Cette étude a montré que les impuretés dans les dépôts peuvent concentrer les solutés dans la masse de l'eau des réacteurs de vapeur par un facteur allant jusqu'à 2×10^3 . Dans certains essais, des signes de corrosion ont été évidents sous les dépôts, avec un certain enrichissement de chrome à la surface du tube. L'enrichissement de chrome dénote généralement un milieu acide, mais la mobilité nécessaire du chrome pour qu'il soit incorporé dans le dépôt épais de magnétite peut indiquer la corrosion sous un milieu alcalin. Un milieu alcalin pourrait résulter d'une accumulation préférentielle de sodium dans la solution du dépôt lors du processus de dépôt d'impuretés.

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ABSTRACT

Hideout in deposits on steam generator tubes was studied using tubes coated with magnetite. Hideout from sodium chloride solutions at 279°C was followed using an on-line high-temperature conductivity probe, as well as by chemical analysis of solution samples from the autoclave in which the studies were done. Significant hideout was observed only at a heat flux greater than 200 kW/m², corresponding to a temperature drop greater than 2°C across the deposits. The concentration factor resulting from the hideout increased highly non-linearly with the heat flux (varying as high as the fourth power of the heat flux). The decrease in the apparent concentration factor with increasing deposit thickness suggested that the pores in the deposit were occupied by a mixture of steam and water, which is consistent with the conclusion from the thermal conductivity measurements on deposits in a separate study. Analyses of the deposits after the hideout tests showed no evidence of any hidden-out solute species, probably due to the concentrations being very near the detection limits and to their escape from the deposit as the tests were being ended. This study showed that hideout in deposits may concentrate solutes in the steam generator bulk water by a factor as high as 2×10^3 . Corrosion was evident under the deposit in some tests, with some chromium enrichment on the surface of the tube. Chromium enrichment usually indicates an acidic environment, but the mobility required of chromium to become incorporated into the thick magnetite deposit may indicate corrosion under an alkaline environment. An alkaline environment could result from preferential accumulation of sodium in the solution in the deposit during the hideout process.

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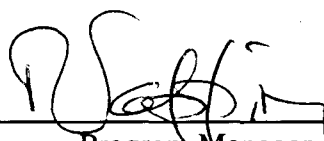
VALUE AND IMPLICATIONS

This work demonstrates that concentration of impurities, up to a factor 10^3 to 10^4 over that in the bulk solution at heat fluxes $>200 \text{ kW/m}^2$, can occur under or in deposits found on steam generator tubes. The data also suggest that corrosion takes place as a consequence, possibly by accumulation of sodium ions to generate alkaline crevices.

This work shows that hideout in tube deposits should be taken into account when determining hideout in crevices and sludge piles by hideout return studies.



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1. INTRODUCTION

Corrosion of condensate and feedwater systems and ingress of condenser cooling water inject solid matter into steam generators during their operation. The solids collect at the bottom of the steam generators as sludge piles and on the heat-transfer tubes as deposits. Deposits formed during the boiling heat-transfer process tend to be porous. Such deposits offer significant resistance to mixing of the fluid contained in the pores with the bulk fluid and thus lead to the retention of the bulk water solutes within the deposits[1,2]. This process is called "hideout". The process reverses on removing the heat flux and terminating the boiling (i.e., when the steam generator operation is interrupted), to release the solutes contained in the deposits to the bulk water. This is called "hideout return".

Chemical reactions occurring in the concentrating liquid could produce corrosive environments at these sites[3]. Various forms of localized corrosion may ensue; e.g., stress corrosion cracking, intergranular attack, pitting and wall thinning of the tube material, and corrosion of the support structure leading to denting or deformation of the tubes. It has become common practice at nuclear power stations to study the hideout return from steam generators as stations are being shut down, to obtain the inventory of chemicals hidden out in crevices and to clear the crevices of the inventory during that process. The chemistry within the crevices during station operation is then derived from the inventory, to assess possible corrosion risks and to devise a suitable chemistry control strategy (e.g., "molar ratio" control). If significant hideout takes place in tube deposits, appreciable errors can occur in the crevice inventory estimates especially when sludge piles are shallow. To assess this situation, as well as to seek mechanisms for tube free-span corrosion observed at some once-through steam generators (OTSG), hideout studies were conducted at Chalk River Laboratories using tube samples from an OTSG. These studies suggested that bulk water solutes can concentrate by factors as high as 10^4 in the deposits.[4] This report describes the results of a more systematic study made in our laboratories using steam generator tubing with simulated deposit.

2. EXPERIMENTAL

2.1 Test Equipment

The tests were performed in a 1-L stainless steel autoclave (Figure 1). The tube sample under test was mounted on the thermowell in the autoclave head. The length of the tube sample was such that its top would be above the water level in the autoclave during the test. The bottom of the tube was plugged gas-tight with a stainless steel plug. An electric cartridge heater placed inside the thermowell provided the heat flux required for the hideout studies. A stainless steel coil heat exchanger located inside the autoclave removed the heat supplied by the cartridge heater during the tests. A conductivity probe monitored the change in ionic solute concentration in the autoclave water during the tests. The conductivity probe consisted of two narrow cylindrical rings of platinized platinum foil placed around an electrically insulating cylinder (Teflon) with platinum wire leads spot-welded to the rings. The leads came out of the autoclave head through

a Conax seal. The autoclave was equipped with a sampling line for extracting liquid samples during the tests and to drain the autoclave when needed.

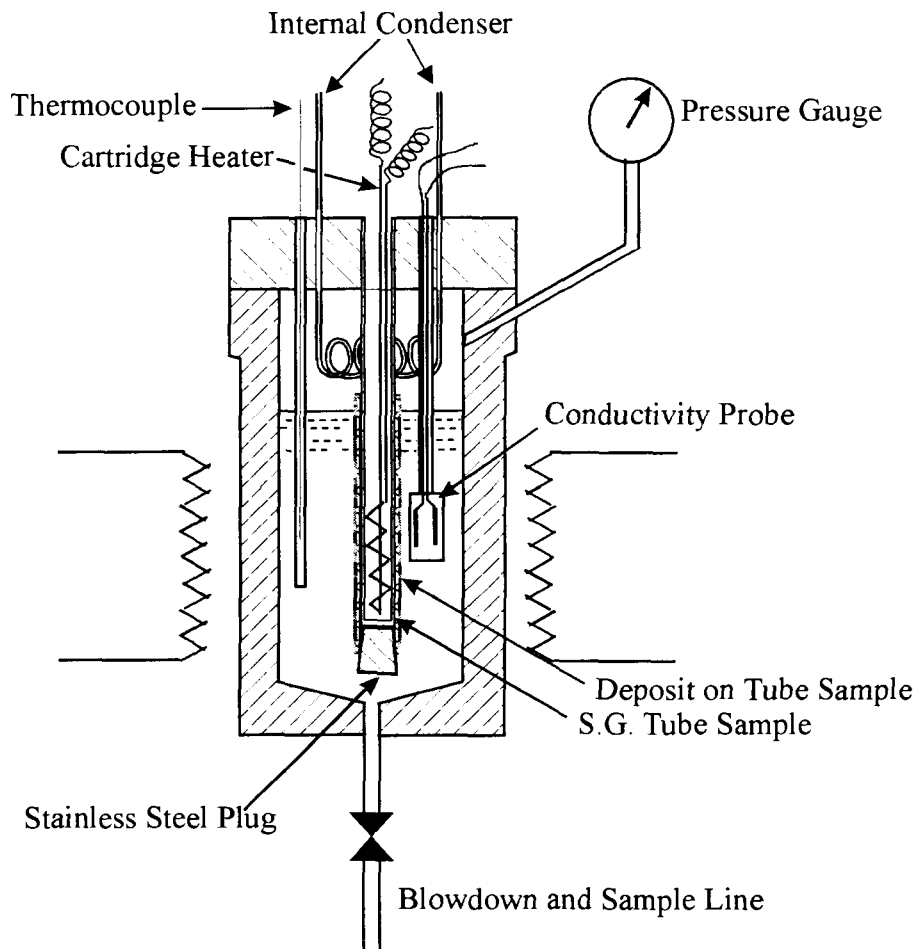


Figure 1. Autoclave Set-up for Studying Hideout in Deposits.

A Jenway PCM3 conductivity meter was used to measure the conductance. The recorder output of the conductivity meter was connected through a Keithley 7001 switch to a Keithley 617 electrometer and thence to a computer. Calibrated Type K thermocouples were used to measure the temperature of the autoclave, both for controlling and for recording. The output of the recording thermocouple was connected to an Omega analog-to-digital converter, TAC80B-K, which was in turn connected to the computer through the Keithley 7001 switch.

2.2 Preparation of Tube Samples

Tube samples for the tests were prepared by coating Alloy 600 tubes (0.51 inch O.D.) with magnetite. The procedure used is described in the following subsections.

2.2.1 Tube Preparation

The Alloy 600 tubes were cleaned by sanding them under a stream of water using silicon carbide paper. They were then dried with a paper towel, rinsed with methanol to remove residual water and dried in an oven at 80°C. The tubes were then etched in a solution consisting of 10 mL of concentrated nitric acid, 20 mL of concentrated hydrochloric acid and 30 to 40 mL of glycerol. The etching was performed by immersing the tubes in the acid solution for 10 min with constant agitation. Following the etching, the tubes were transferred to a beaker of water and allowed to sit for several minutes. They were subsequently rinsed with running water, followed by a methanol rinse and oven drying. During the experiments, it was noted that the surface roughness of the tube following the cleaning and etching operations could vary from sample to sample.

2.2.2 Preparation of Magnetite Colloid

The magnetite colloid used to coat the Alloy 600 tubes was prepared by precipitation from ferrous sulphate solution by titration with potassium hydroxide in the presence of potassium nitrate. Solutions were thoroughly degassed to minimize reactions with dissolved oxygen that result in the formation of hematite (Fe_2O_3) and goethite ($\text{FeO}(\text{OH})$). Once the desired pH was reached, the colloidal suspension was rinsed with degassed, distilled water until the pH was approximately 7 and the conductivity of the rinse solution dropped to about 20 $\mu\text{S}/\text{cm}$. Both the mean particle size and the particle-size distribution are affected by the stirring rate, the rate of potassium hydroxide addition, the final pH of the solution and the temperature. Vigorous stirring, rapid addition of potassium hydroxide and excess hydroxide concentration all result in the formation of smaller particles. Rapid addition of potassium hydroxide results in the formation of colloidal particles of more uniform size. If the hydroxide and the ferrous ions are used in stoichiometric proportion, the colloidal particles will be spherical in shape. Particles from solutions containing excess hydroxide tend to become more cubic in shape. By “aging” the colloidal suspension for 2 h at 90°C, the average diameter of the particle can be increased from 0.01 μm up to a maximum of about 0.25 μm .

To produce the best quality of coating, the concentration of the colloid should be in the range of 90 to 100 mg Fe_3O_4 per g colloidal suspension. This concentration was generally obtained by allowing the suspension to settle overnight and then using a pipet to remove any supernatant liquid. The concentration was determined by transferring aliquots of the well-agitated suspension to beakers and weighing the sample before and after oven drying.

2.2.3 Coating of the Samples

Immediately before coating a tube sample, the colloidal suspension was placed in a bottle and agitated on a wrist-shaker for 1 h. The dipping instrument, consisting of a variable-speed motor attached to a worm drive, was calibrated for a pull rate of 1 cm/s. The thoroughly mixed colloidal suspension was loaded into a graduated cylinder. The sample tube to be coated was immersed in the suspension for several seconds and then withdrawn at a uniform rate. The thickness of the deposit varied with the viscosity of the colloidal suspension. The coated tube

was allowed to dry in air for several minutes before being transferred to a drying oven at 55°C, where it was allowed to dry overnight.

2.2.4 Sintering Coated Tubes

Samples were sintered at temperatures ranging from 615°C to 800°C by mounting the coated tube in a holder designed to fit inside a quartz liner of a tube furnace. Both ends of the quartz liner were filled with turnings of zirconium metal to getter traces of oxygen. The turnings were thoroughly degreased by soaking in hexane and then rinsing with methanol and allowing to dry in air. Ethylene glycol was used in a trap at the outlet of a process tube to eliminate backflow of oxygen. The assembly was purged with argon for 30 min at a flow rate of 2 L/min. For a 6-L process tube, these conditions allowed for ten volume changes. After purging, the flowrate was decreased to 0.5 L/min and the furnace was set to the desired temperature. The sample was sintered at temperature for 1 h while continuing the flow of argon gas. After 1 h, the heater was turned off and the sample allowed to cool slowly to room temperature under the flow of argon. Slow cooling rates were used to minimize the potential for spalling, since the magnetite and underlying metal have different thermal expansion coefficients.

2.2.5 Deposit Characterization

Extraneous impurities (e.g., sodium, chromium) in the magnetite used for coating the tube were determined by dissolving the magnetite in acid and analyzing by inductively coupled plasma (ICP) atomic emission spectroscopy (AES). Typical analysis results are shown in Table 1.

Table 1. Impurities in Typical Batches of Magnetite Colloid as Determined by ICP-AES.

Magnetite Batch No	*Na (wt.%)	*Cr (wt.%)
B961204	<0.06	0.002
B961107	0.012	<0.004
B961115	0.018	<0.004

* Detection limits vary with the amount of sample available for analysis

The thickness of the deposit (coating) was determined by laser profilometry or by scanning electron microscopy (SEM) of the polished cross-section of the tube samples. The deposit loading, which is the mass of material per unit area of substrate, was determined by weighing a sample of the tube before and after the coating was removed by dissolution. The deposit was removed by pre-treatment with an alkaline permanganate solution (80 g NaOH + 24 g KMnO₄ in 800 mL of water at 85°C for 1 h) followed by dissolution using a modified Clark's solution consisting of 500 mL of 12 mol/L HCl, 15-20 g Sb₂O₃ and 25 g SnCl₂ made up to 1 L with water. The latter solution was used at room temperature.

Deposit morphology was studied using an optical microscope or a scanning electron microscope. Macroscopic features of the deposit were determined visually or by using an optical microscope.

Inspection of the deposit on the scale of 1 to 1000 microns was done using SEM (see Figures 2 and 3, Section 3).

2.3 Hideout Test Procedure

Hideout tests were performed using a solution of sodium chloride containing chloride at a concentration of 5 mg/kg. The conductivity probe was calibrated using 0.001 mol/kg KCl solution (specific conductivity = 146 $\mu\text{S}/\text{cm}$) in the autoclave. Two different conductivity probes were used during the course of the study. The cell constant (the factor for conversion of the conductance readings to conductivity) for the first probe (used in Runs #1 to #7) was 5.8. The cell constant for the second probe (used for Runs #8 onwards) was 2.0. After the calibration of the conductivity probe the autoclave was drained and thoroughly washed with deionized water, and 500 mL of the test solution was placed in it. The autoclave head, with the magnetite-coated steam generator tube sample mounted on the thermowell, was then placed on the autoclave and the autoclave was sealed. The leak-tightness of the autoclave was confirmed by pressurizing it with nitrogen gas to 6.2 MPa (900 psi) and observing the pressure gauge to detect any decrease in the pressure for a period of at least 20 min. The computer program for data acquisition to collect the temperature and conductivity data was then started. The autoclave was then heated to the test temperature of 279°C. After a suitable period to ensure steady conditions, the heat flux on the cartridge heater was turned on. The temperature inside the tube ("primary temperature"), as monitored by a thermocouple placed in the tight space between the cartridge heater and the tube, was 304°C for a heat flux of 150 kW/m^2 . The progress and status of hideout was monitored using the conductance readings. Once a steady state was indicated at a given heat flux, the run was either terminated or the heat flux was set at a new value and the run continued. When high heat fluxes were applied (e.g., >200 kW/m^2), cooling water to the internal cooling coil was turned on for heat removal. When terminating a run, in most cases the heat flux was just turned off. However, in some cases the autoclave was drained while the heat flux was still on to retain any solutes hidden out in the deposit, so the deposit could be analyzed for the solutes.

Water samples were collected from the autoclave at the start of the runs, periodically during the runs while the heat flux was on and at the end of the runs. The samples were analyzed for sodium using ICP and for chloride using ion chromatography (IC). The deposit was analyzed by ICP, neutron activation or SEM using the energy dispersive x-ray (EDX) system.

3. RESULTS

3.1 Deposit Properties

The deposit, produced by coating tubes with magnetite as described in the previous section, is referred to in this report as deposit or oxide deposit, to distinguish it from the oxide film produced by corrosion of the tube material. The corrosion-produced oxide film is referred to in this report as corrosion film or oxide film.

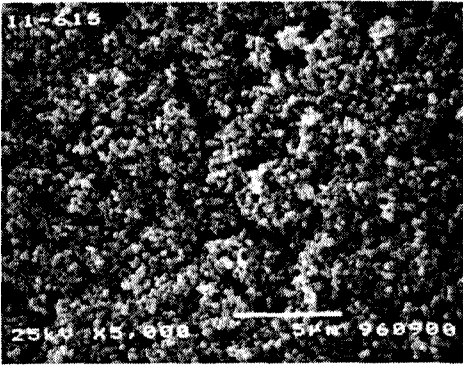
3.1.1 Deposit Morphology

The effect of sintering temperature on the morphology of the magnetite coating is shown in Figure 2. Sintering at 615°C or lower had a minimal effect on the colloidal magnetite particles. Coatings prepared under these conditions were very “soft” in that they were easily rubbed off. At 695°C, the colloidal particles began to coalesce and the coating became harder. As shown in Figure 2, this trend continued through 750°C and 796°C; a very hard coating was formed at 796°C. The sintering temperature of the tube furnace appeared to be a more important parameter than the length of time the sample was held at that temperature. Coatings sintered at higher temperatures seemed to exhibit lower porosity and tended to form cracks on a macroscopic scale.

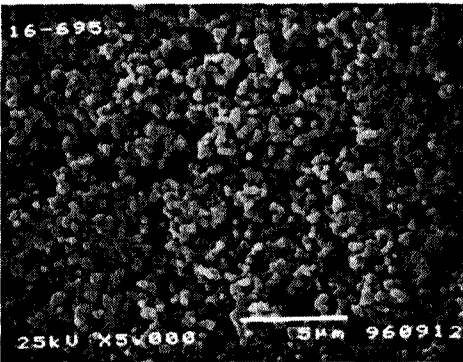
The preferred method for preparing coatings for most experiments is to first lay down a thin layer which is sintered at 800°C to form a hard, adherent inner coating (10-15 µm thick). Subsequent coatings, typically 15 µm thick each, can be added to this base coat. Figure 3 shows a cross-sectional view of a sample consisting of three coatings prepared according to this procedure. The first layer, sintered at 800°C, shows the agglomeration of magnetite particles that is typical of this temperature. Although the second and third layers were both sintered at 700°C, the second layer exhibits a somewhat higher degree of agglomeration, presumably as a result of being sintered twice. Based on the evidence in this cross-sectional view, the porosity of the coating decreases with increasing sintering temperature.

Table 2. Characterization of Tube Deposits.

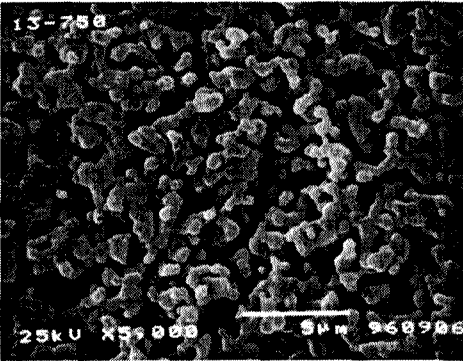
Run #	Date	Magnetite Batch	Coat	Sinter Temp.(°C)	Thickness (µm)	Loading (kg/m ²)	Porosity (%)
4	95.12.12	(J. Uhle)	> 1	?	43	0.16	28
8	96.01.22	B951124	1-4	800	63	0.16	52
11	96.02.26	B951124	1-3	800	34	0.15	15
15	96.04.10	B9603045	1	700?	23	0.05	58
16	96.04.22	B951219 B9604045	1,2 3	800 700	51	0.12	56
24	96.07.25	B960524	1-5	800	42	0.15	29
25	96.08.12	B960528	1 2	700 800	40	0.08	62
26	96.08.19	B960528	1 2,3	800 700	52	0.16	40
27	97.02.27	B960531	1 2-4	800 700	19	0.16	?
28	97.03.18	B960531	1 2,3	800 700	16	0.06	26
29 29B	97.04.01 97.04.08	B960501	1 2-5	800 700	72	0.23	38
31	97.04.21	B960501	1 2-4	800 700	69	0.22	40
33	97.07.14	B970110	1 2,3	800 700	31	0.10	39



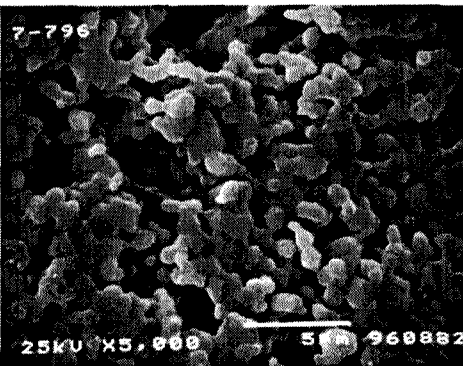
2A. Fe₃O₄ sintered at 615°C for 1 h.



2B. Fe₃O₄ sintered at 695°C for 1 h.

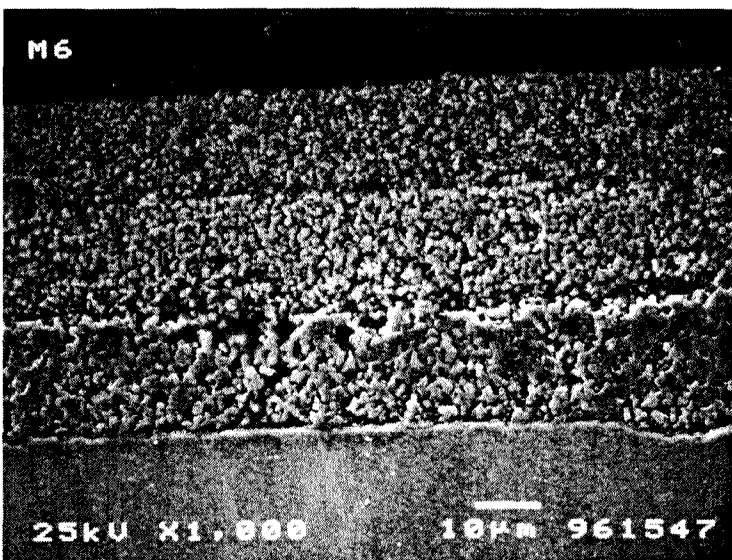


2C. Fe₃O₄ sintered at 750°C for 2 h.



2D. Fe₃O₄ sintered at 796°C for 1 h.

Figure 2. Effect of Sintering Temperature on Magnetite Coatings.



- Mounting Resin.
- 3rd Coat sintered at 700°C.
- 2nd Coat sintered at 700°C.
- 1st Coat sintered at 800°C.
- I-600 Tube.

Figure 3. Cross-Sectional View of Magnetite Coating on I-600 Tube.

Because of the sintering of the coating, which was typically done at a temperature of 700°C to 800°C for about 1 h, it is very likely that the Alloy 600 tube material used in these tests was in a state of partial sensitization.

The effect of sintering temperature and number of coatings on the loading and porosity of the deposit is shown in Table 2. The bulk porosity was obtained as the ratio of the measured density to the theoretical density. The lowest porosities were obtained for samples 3 and 24, which were sintered at 800°C. The porosity of sample 8 sintered at the same temperature seems to be anomalously high. The tabulated results also show that the thickness of a deposited layer can vary from batch to batch of magnetite colloid (e.g., samples 11 and 16). This variation probably correlates with the viscosity of the colloidal suspension at the time of coating. Finally, there is generally an inverse relationship between deposit loading and porosity.

3.1.2 Physical Characteristics

The deposit thickness, loading and porosity for 13 representative tubes are listed in Table 2. The deposit thickness and deposit loading generally increase with the number of coats applied but the scatter in the data (e.g., 16-52 µm and 0.06 to 0.16 kg/m² for 3 coats) indicates that variations in other parameters such as the viscosity of the colloidal magnetite play a significant role in the nature of the deposit that is formed. The upper bound for deposit thickness per coating operation is 18 µm. The deposit porosity ranged from a low of 15% (Run #11) to a high of 62% (Run #25). None of the parameters is strongly correlated with the sintering temperature, although the SEM photographs shown in Figure 2 appear to show that higher temperatures result in greater density in the coating, as marked by increasing particle coalescence.

3.2 Hideout Tests

A total of 33 runs were made. Table A1 in Appendix 1 lists the runs, showing the significant parameters. Three of the 33 runs used tubes with stainless steel wire gauze (200 mesh) wrapped around it to simulate porous deposits. These runs were made to demonstrate the suitability of the experimental set-up to detect and monitor hideout. Twelve runs were made with tubes having no wire gauze wrapping or magnetite coating. These were blank runs, where no hideout was expected; they were meant to verify that no crevices or other such sites, where hideout can occur, existed in the experimental set-up and were also meant to test the operation of the system (e.g., the internal cooling for use at high heat fluxes). One run was made using a tube sample from a pressurized water reactor (PWR) steam generator.

3.2.1 Blank Runs

The results of the blank tests are listed in Table 3. The change in solution conductivity during a typical blank run is shown in Figure 4.

Table 3: Results of Blank Runs using Clean Tube - Conductance, Sodium and Chloride.

Run #	Heat Flux (kW/m ²)	Conductivity (μS/cm)			Sodium (mg/kg)			Chloride (mg/kg)		
		Initial	Final	% Diff.	Initial	Final	% Diff.	Initial	Final	% Diff.
12	144	88	88	0	3.6	3.6	0.0	--	--	--
18	119	150	150	0	3.4	3.4	0.0	--	--	--
19	119	104	104	0	3.4	3.4	0.0	--	--	--
20	119	111	56	-49*	3.3	2.3	-30	--	--	--
21	119	133	132	-0.8 [§]	3.8	3.9	+2.6	--	--	--
21B	119	133	71	-47**	3.9	1.9	-51	--	--	--
22	119	122	120	-1.6 [§]	3.4	3.4	0.0	--	--	--
22B	119	124	125	+0.8 [§]	3.4	3.4	0.0	--	--	--
23	119	107	105	-1.9 [§]	3.4	3.4	0.0	--	--	--
23B	119	101	103	+2.0 [§]	--	--	--	--	--	--
30	267	102	103	+1.0	3.8	3.8	0.0	5.7	5.9	+3.5
	383	103	104	+1.0	3.8	3.8	0.0	6.0	6.0	0.0
32	380	100	100	0.0	3.6	3.7	+2.8	5.9	6.0	+1.7

-- No analysis performed.

* Hideout due to leak at the plug at the end of the tube sample.

§ Difference arising from noisy conductance readings and drift in the probe output.

** Hideout in the thermocouple groove between the heater and the tube.

As can be seen from the table and the figure, no hideout took place at heat fluxes as high as 380 kW/m². The indication of hideout in one test was due to a small leak at the plug at the bottom of the tube sample. In two other cases, when a thermocouple was placed in a groove on the thermowell inside the tube sample to monitor the tube wall temperature, there was some leak of the solution into the groove and consequent hideout. In some runs noisy conductance readings indicated an apparent decrease in conductance, suggesting hideout, and in some runs there was an

apparent increase in the conductance. However, generally no hideout could be observed in the absence of a deposit coating on the tube. Chemical analysis of solution samples from the autoclave for sodium and chloride during the runs also confirmed the absence of significant hideout, where the conductance data did not show significant drops. This confirmed that the experimental set-up is suitable for studying hideout in tube deposits.

3.2.2 Hideout under Wire-Gauze Wrapping on Tubes

In three tests the tube was wrapped with one or two layers of 200-mesh stainless steel wire gauze to simulate a porous deposit. In the first test, the tube was wrapped with one layer of wire gauze. The conductance probe indicated no significant hideout during the test, but the conductance probe had been damaged during the test. The solution samples collected during the test showed a slight decrease in sodium concentration. The second tests used a tube wrapped with two layers of wire gauze. Appreciable hideout was seen (Figure 5). The third test used one layer of wire gauze wrapping. Even in this test significant hideout was seen. The results are summarized in Table 4.

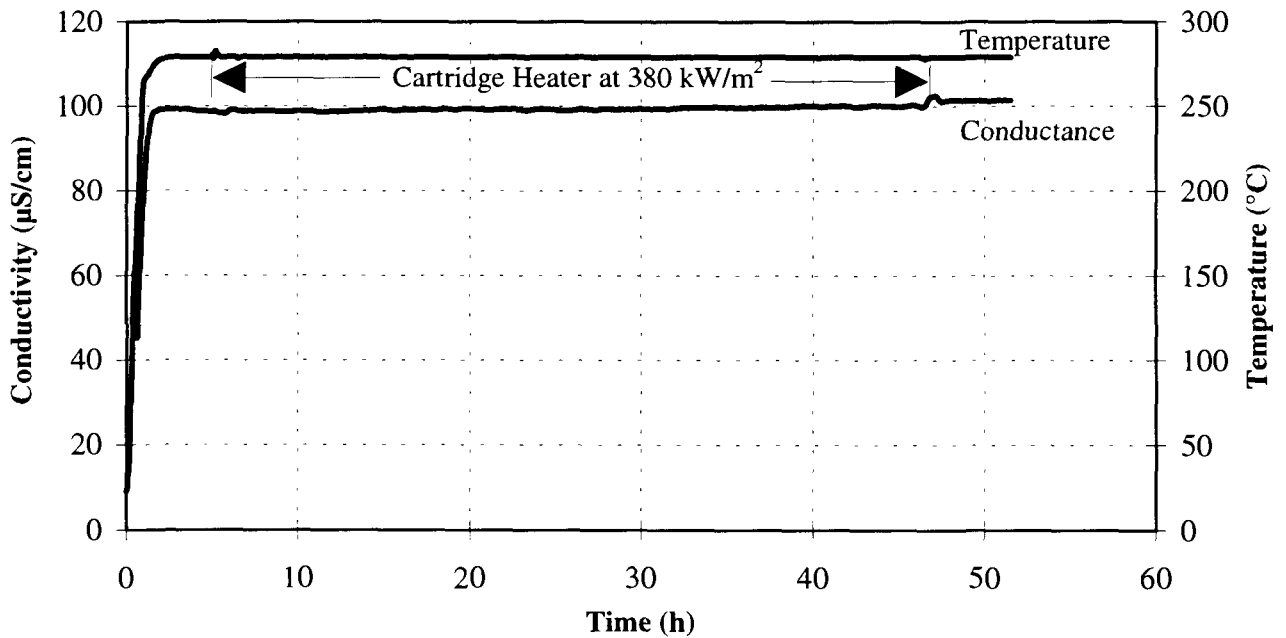


Figure 4. Hideout Test on Clean Tube - Run #32, 279°C, 5 mg Cl/kg as NaCl.

Table 4. Hideout on Tubes Wrapped with Wire Gauze.

Run #	No. of Layers	Heat Flux (kW/m ²)	Conductivity (μS/cm)			Sodium (mg/kg)		
			Initial	Final	% Diff.	Initial	Final	% Diff.
5	One	158	n.a.	n.a.		3.50	3.47	-0.8
6	Two	80	130.7	27.4	-79	3.30	0.64	-81
7	One	158	127.1	119.8	5.7	3.39	3.21	-5.3

The small difference in the hideout behaviour observed between the two tests on tube wrapped with a single layer of stainless steel wire gauze may have been due to the difference in the tightness of the wrapping. In the test with two layers of wrapping (Run #6), the hideout appeared to be proceeding very rapidly at 158 kW/m², so the heat flux was cut down to 80 kW/m² during the test. Even at the lower heat flux the hideout was extensive; about 80% of the solute was hidden out when a steady state was attained.

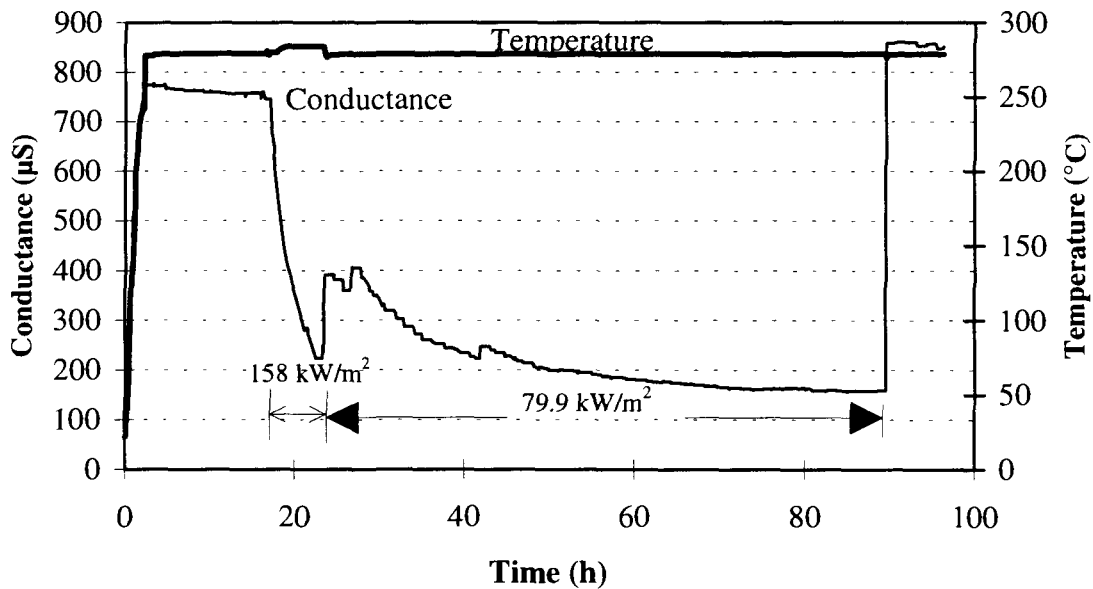


Figure 5. Hideout under Two Layers of Wire Gauze Wrapping - 279°C, 5 mg Cl/kg as NaCl.

3.2.3 Hideout on Magnetite-Coated Tubes

Table 5 summarizes the results obtained in the tests on tubes coated with magnetite. The magnetite coating was applied to obtain different thicknesses and porosities. The heat flux applied varied from 119 kW/m² to 387 kW/m². The change in conductance of the solution in the autoclave during the test, which indicates the progress of hideout, is shown in Figure 6 for a typical test, along with that for a blank test (Runs #31 and #30, respectively). Table 5 lists the conductance of the solution in the autoclave with and without heat flux, and the difference as a

percentage of the initial value (i.e., conductance before heat flux was applied.) The table also gives, where available, the results of analysis of the solution samples collected during the tests for sodium and chloride, and the change in concentration as a percentage of the value before heat flux was applied. A negative value for the difference indicates loss of solute from the solution in the autoclave by hideout. A difference of less than 1% should be considered as no hideout, since the accuracy of the measurements is not expected to be better than 1%. In general, the indication by the conductance probe should be considered to be more accurate, since the probe gives on-line, continuous measurements. However, drift in the probe had occasionally produced less reliable results than the off-line sample analyses (e.g., Runs #10, #14, #15, #24). Ten of the 21 runs listed in Table 5 showed significant hideout. Of the ten runs showing hideout, one run (Run #8) may have shown hideout because of Teflon tape applied at the bottom of the tube, to cover the area where the coating had flaked off a little on inserting the stainless steel plug. Also, at the end of the run, the tube was seen to have slipped down on the thermowell about 1 cm, which could also lead to some hideout if the top of the tube came near the liquid level in the autoclave. In most of the cases where no hideout was seen, the magnetite coating on the tube was unstable; in some cases the coating had completely peeled off in the heated zone and in others considerable flaking was seen. It is also possible that the conductance probe indication was not reliable in some runs due to a defect that developed in the probe (e.g., Run #4). A new conductivity probe was used from Run #8 onwards.

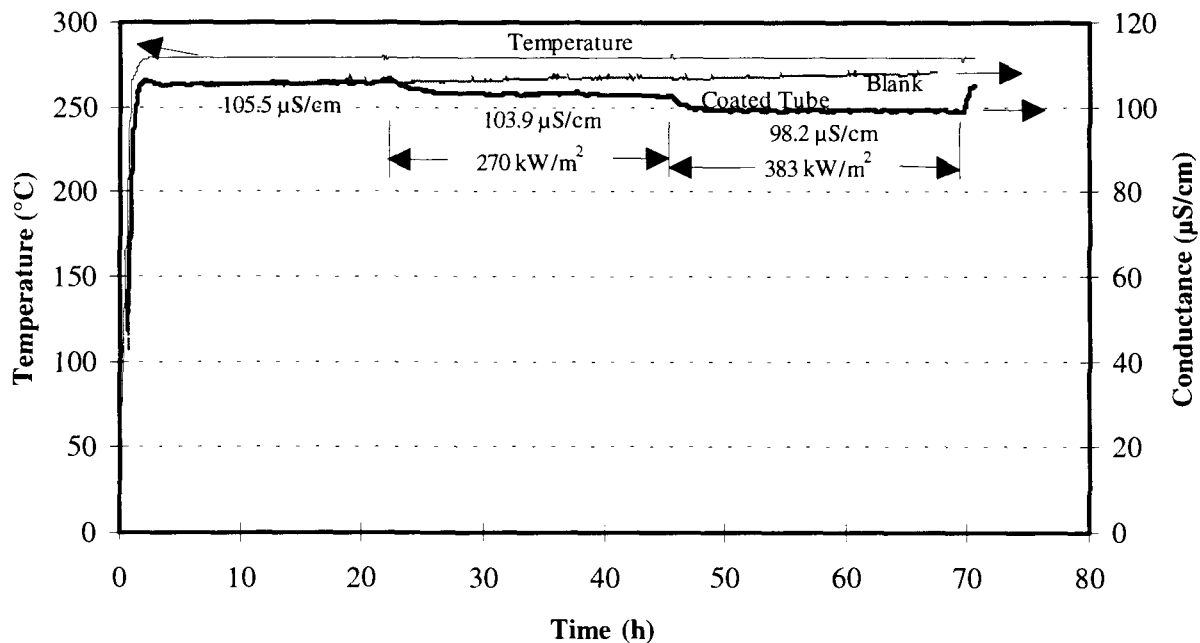


Figure 6. Hideout on Tubes with Deposit Coating - 279°C, 5 mg Cl/kg.

Table 5: Results of Hideout Tests on Tubes with Deposit Coating.

Run #	No. of Coats	Thick-ness (μm)	Porosity (%)	Heat Flux (kW/m ²)	Conductivity (μS/cm)			Sodium (mg/kg)			Chloride (mg/kg)		
					Initial	Final	% Diff.	Initial	Final	% Diff.	Initial	Final	% Diff.
1		--	--	158	131.0	131.0	0.0	--	--	--	--	--	--
2		--	--	158	126.2	126.2	0.0	--	--	--	--	--	--
3		--	--	158	111.7	111.7	0.0	--	--	--	--	--	--
4	>1	42.5	28	158	132.7	132.7	0.0	3.4	3.4	0.0	--	--	--
8	4	63	52	144	102.8	95.8	-6.8	3.5	3.2	-8.6	--	--	--
9	4	64	--	150	111.0	102.6	-7.6	3.3	3.3	0.0	--	--	--
10	4	64	--	150	92.0	98.0	+6.5	3.4	3.4	0.0	--	--	--
11	3	34	15	144	102.4	101.2	-1.2	3.4	3.4	0.0	--	--	--
14	4	--	--	119	111.6	110.9	-0.6	3.9	3.8	-2.5	--	--	--
15	1	23	58	119	104.5	103.6	-0.9	3.7	3.6	-2.7	--	--	--
16	3	51	56	119	104.7	103.7	+1.0	3.3	3.2	-0.3	--	--	--
17	3	51	--	119	103.8	103.9	+0.1	3.3	3.3	0.0	--	--	--
24	5	42	29	119	110.6	107.9	-2.4	3.5	3.5	0.0	--	--	--
25	2	40	62	119	108.2	108.1	-0.1	--	--	--	--	--	--
26	3	52	40	119	126.9	127.1	+0.2	--	--	--	--	--	--
27	4	19	--	387	143.5	137.6	-4.1	--	--	--	--	--	--
28	3	16	26	387	158.0	155.5	-1.6	4.6	4.1	-10.1	7.9	7.3	-7.6
29	5	72	38	267	126.4	117.4	-7.1	4.3	3.7	-13.9	7.2	2.4	-67
				387	125.6	100.1	-20.3	4.1	3.3	-19.5	6.6	5.4	-18.2
29B	5	72	38	380	108.9	104.1	-4.4	3.7	3.6	-2.7	6.1	5.3	-13.1
31	4	69	40	270	105.5	103.9	-1.5	3.7	3.5	-5.4	6.3	6.2	-1.6
				383		98.2	-6.9		3.4	-8.1		5.9	-6.3
33	3	31	39	200	94.7	93.0	-1.8	3.6	3.5	-2.8	5.8	5.5	-5.2

-- Measurement not made.

3.3 Post-Test Analyses of Tube Deposits

Some samples of deposit scraped off from the tubes used in tests that showed indications of hideout were analyzed using the SEM/EDX system. Specifically, the analyses aimed to determine the sodium and chloride content of the deposit. The autoclave was drained before turning the heat flux off in these tests. Several analytical techniques were applied to determine whether residual sodium and chloride could be observed in the deposit.

A typical EDX spectrum is shown in Figure 7. In this spectrum, which was acquired with the instrument set to maximize sensitivity to the lighter weight elements, it is clear that any signal arising from Na is overlapped by a much stronger Ni line. Furthermore, the background level in this region of the spectrum is poorly defined. While some EDX spectra seemed to suggest that trace amounts of sodium were present, attempts to deconvolute the spectrum and quantify the data were unsuccessful. No signals attributable to chlorine in the sample were ever observed. Taking various instrument and inherent sensitivity factors into account, the detection limits for EDX analysis were estimated to be on the order of 6500 mg/kg for sodium and 1000 mg/kg for chlorine, which are consistent with the relatively low sensitivity of EDX for the lighter elements. Table 5 shows that, according to conductivity data, 4.4% of the solute in the autoclave bulk solution hid out in the deposit during Run #29B. Assuming uniform hideout over the two-inch heated zone of the tube in the 72-μm-thick deposit, the deposit would contain 152 mg/kg Na

and 234 mg/kg Cl. These values would be even higher locally if hideout was limited to localized areas in the deposit and these localized areas were subjected to the analysis. Both of these values are much lower than the detection limits for the SEM/EDX technique employed.

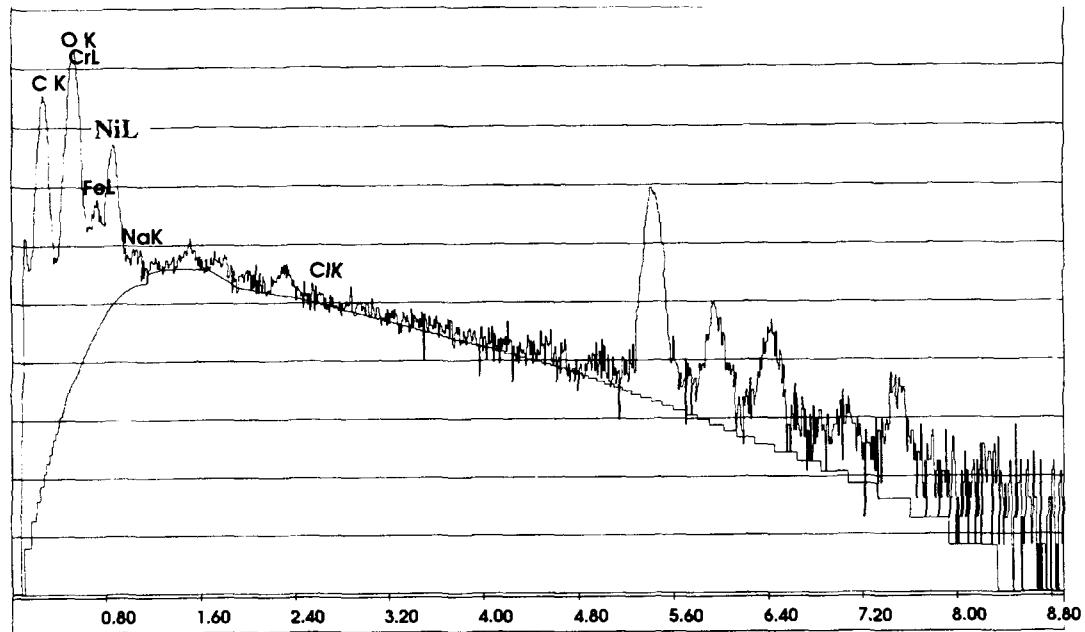


Figure 7. Representative EDX Spectrum of a Post-test Deposit Sample.

The possibility of analyzing deposit samples by secondary ion mass spectrometry was assessed but not pursued because of the amount of development work required to determine matrix and geometric effects on data acquisition.

Samples of the magnetite coating scraped from each of the tubes used Runs #8, #10 and #29 were dissolved in nitric acid and analyzed for sodium by ICP-AES. Unfortunately, the detection limits for sodium using this technique were only marginally better (3500 mg/kg) than those obtained using EDX; because of the small sample size and the amount of acid that had to be used to dissolve it, the concentration in the solution after the dissolution of the sample was very low.

Finally, scraped samples from the above tubes were submitted for neutron activation analysis of sodium and chlorine. Inconsistencies in replicate analyses of samples were attributed to a combination of environmental contamination, insufficient data for accurate blank corrections, uncertainties in the weights of sample containers and in the duration of irradiation, and to the low level of sodium and chlorine concentrations in the samples. Although the measured values were not statistically meaningful, these measurements did establish that the concentrations of sodium and chlorine in the deposits were less than 100 mg/kg, which is lower than indicated by conductivity data. These results suggest that any concentrated solutions resulting from hideout in the pores of the magnetic coating are not held in place when the tube is removed from the solution, whether the autoclave is drained while the heat flux on the tube is on or off. It was

expected that keeping the heat flux on the tube while draining would cause any solution in the deposit to boil dry due to the drop in pressure and to leave the solid solutes in the deposit.

Signs of chemical reactions were evident upon visual inspection of tube deposits from many of the tests. These signs ranged from small oval stains and rings to areas in which much of the magnetite coating had spalled off, leaving behind craters in the deposit layer. The distribution of stained areas in some experiments appeared to be identical to the distribution of the craters in other runs, suggesting that the craters developed from the stained areas. In the case of rings and stains, some were found to traverse the deposit oxide layer and be quite visible on the underlying metal. SEM/EDX studies of these stains showed a chromium content of up to 38 wt%, which is much higher than the 16 wt% in the underlying metal. This enrichment in chromium associated with stains was found in both the deposit layer and on the surface of the underlying metal.

An example of more severe deterioration of the deposit is shown in Figure 8, which depicts a spalled area on the tube recovered from Run #29B. The smooth outer areas in this figure correspond to the undisturbed deposit, and the spalled region is marked by roughly concentric steps through the deposit to the bottom of the crater. The elemental composition at the bottom of the crater was determined by EDX to be 28 wt% Fe, 37 wt% Ni and 35 wt% Cr. In other runs (#15 and #16) some craters were found to contain nodules of material in the central regions that contained up to 60 wt% chromium. This composition is richer in both Fe and Cr than the tube material, Alloy 600. The composition up the sides of the crater was essentially that of magnetite. A scratch was made in the coating in the vicinity of the crater to expose the underlying material. The deposit adjacent to the scratch, when examined by EDX, showed a composition (98 wt% Fe, 0.5 wt% Ni and 0.5 wt% Cr) similar to that of the deposit near the crater, whereas the exposed alloy surface at the base of the scratch showed the composition (72.5 wt% Ni, 8.5 wt% Fe, 16.7 wt% Cr) expected of Alloy 600. Thus the chromium enrichment is observed only at bottom of the craters and areas of staining; i.e., where the attack of the tube surface may have been initiated.

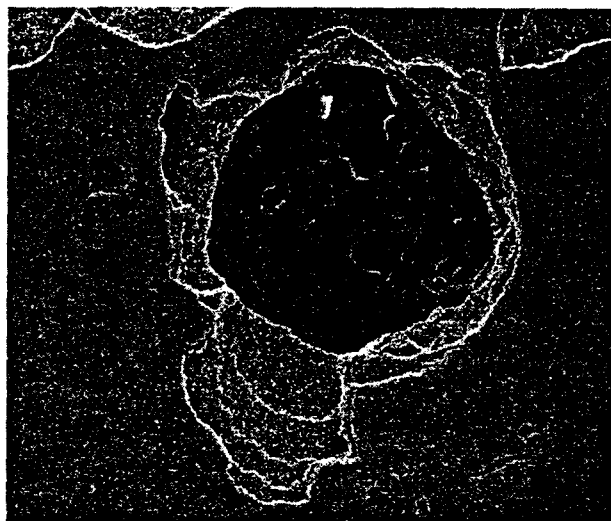


Figure 8. Scanning Electron Micrograph of Spalled Region on Tube Deposit After Hideout Run.

4. DISCUSSION

Decreased adherence of the coating to the tube was a serious problem during the tests. Often the deposit spalled off during the run, sometimes over the entire tube and sometimes just over the heated zone. For this reason, repeating a run using the same tube with the same coating gave very different results. For example, approximately 20% of the solute in the autoclave bulk solution hid out in Run #29 at 387 kW/m^2 , but continuation of the test with the same tube showed much less hideout (4.4%, based on conductivity data) in Run #29B (Table 5). The coating lost some of its integrity during Run #29, so that it peeled off appreciably during the next run, Run #29B. Preparing tubes with identical coatings was not achieved during this work. However, it was possible to identify some trends during the tests, which are analyzed in the following sections to determine the factors that affect the process of solute hideout in deposits on a heat-transfer surface.

As Table 5 shows, this study included variables such as deposit thickness, deposit porosity and heat flux, which were considered to be the important parameters influencing solute hideout. The extent of hideout expressed in Table 5 as a percentage of solute lost from the autoclave bulk solution is translated into a concentration factor under the tube deposit. The concentration factor is calculated assuming that the solute lost from the bulk solution exists in the deposit as a solution occupying the whole available volume within the deposit, that the available volume is defined by the geometrical volume of the deposit multiplied by the porosity, and that all concentration takes place over only the 2-inch (5-cm) heated zone of the tube. Where a porosity was not available, an average porosity of 50% was assumed. The concentration factor is the ratio of the concentration in the deposit so calculated to that in the bulk water after hideout.

4.1 Effect of Heat Flux on Hideout

The runs that consistently showed hideout were made at high heat fluxes, and no reliable indication of hideout was seen in runs at low heat fluxes ($<200 \text{ kW/m}^2$; Runs #1 to #26). Based on temperature measurements inside the tube and in the bulk water from one of the tests, the temperature drop across the deposit was estimated to be about 2°C at a heat flux of 200 kW/m^2 . Table 6 shows results of tests at different high heat fluxes for nominally similar properties of the deposit. Because of possible differences in the morphology of the coatings on the tube for nominally similar thickness and porosity, strict comparison of the extent of hideout can only be made using data obtained from the same tube and deposit coating, when disturbances affecting the integrity of the coating have been avoided. There are only two such sets of data: Runs #29 and #31. While comparison between the two sets does not appear to be informative, comparison of the data for the two values of heat flux in each of the runs shows that hideout occurred to a larger extent at the higher heat flux. The results from Run #29 show a dependence of the concentration factor of heat flux raised to the 2.8 power, while the dependence is 4.3 power in Run #31; i.e., hideout increases non-linearly with heat flux. The results for chloride in Run #29 appear to be anomalous.

The discrepancy between the two sets of results in Table 6 may not be due entirely to a possible difference in the morphology of the tube deposit. The hideout behaviour, as indicated by the

continually decreasing trend of the conductivity vs. time plot obtained for Run #29, suggested that hideout may have started to occur elsewhere in the test section, in addition to the hideout in the deposits, during the later stages at the higher heat flux. The hideout in the deposit at 387 kW/m² was probably significantly lower than shown in the table. Also, the results from

Table 6. Effect of Heat Flux on Hideout.

Run #	Thickness (μm)	Porosity (%)	Heat Flux (kW/m ²)	Concentration Factor		
				Conductivity	Sodium	Chloride
29	72	38	267	640	1250	6040
29	72	38	387	1830	1760	1640
31	69	40	270	134	483	143
31	69	40	383	617	724	563

chemical analysis for sodium and chloride appear to be abnormally high. The agreement between the values for the concentration factor during Run #31 derived from chloride analysis and the conductivity data is good, while the sodium analysis may have been in error.

4.2 Effect of Deposit Thickness and Porosity on Hideout

As stated out earlier, producing deposit coatings with the desired characteristics proved very difficult. Because of the poor adherence of the deposits, they tended to fall off the tube surface when solutions were boiled at high heat fluxes, and high heat fluxes were required for hideout to occur in the deposits. For this reason, the successful runs were very few in number. The correlation of hideout with deposit thickness or porosity is, therefore, difficult. The results of the few runs that could be used to deduce the effect of these parameters on the hideout process are listed in Table 7.

Table 7. Effect of Deposit Thickness and Porosity on Hideout.

Run #	Thickness (μm)	Porosity (%)	Heat Flux (kW/m ²)	Concentration Factor		
				Conductivity	Sodium	Chloride
28	16	26	387	950	5990	4510
27	19	n/a	387	1060	n/a	n/a
29B	72	38	380	397	243	1180
31	69	40	383	617	724	563

The limited amount of data that Table 7 provides show that the solute concentration factor resulting from hideout, as indicated by the conductivity data, decreases with increasing thickness. Since conductivity indications have been the most reliable, we would use only the data based on conductivity, using the chemical analysis data only for supporting the conductivity data. The apparent decrease in concentration factor with increasing deposit thickness suggests that the

concentration process was occurring in approximately the same volume, irrespective of the deposit thickness; the deposits in these tests all had about the same porosity. Therefore, only a part of the deposit contains the liquid, the remainder being occupied by steam.

Measurements of the thermal resistance of porous deposits under flow-boiling conditions have led to the same conclusion, i.e., that under boiling heat transfer, the pores of a deposit are filled with a mixture of steam and water [5]. The thermal conductivity of a magnetite deposit at 250°C is predicted to range from 0.14 to 1.4 W/mK, depending on whether the pores of the deposit are completely filled with steam or water. In comparison, the thermal conductivity measured for magnetite deposits on steam generator tubes removed from operating plants ranged from 0.23 to 0.96 W/mK under flow-boiling conditions, while measurements on synthetic magnetite deposits gave a thermal conductivity of 0.89 W/mK. For both cases, the measured thermal conductivity supports the hypothesis that the pores of a deposit are filled with a mixture of steam and water under nucleate boiling conditions.

There is not enough data to examine the effect of porosity on hideout, but indications are that the structure of the pores, rather than porosity itself, is a more important factor. The tests with the wire-gauze-wrapped tubes show a disproportionate increase in the hideout on increasing (doubling) the thickness of the wrapping (Table 4). There was a fourteen-fold increase in the hideout on the tube with two layers of wire gauze compared to the tube wrapped with a single layer, even though the heat flux in the former case was only half that in the latter. The double wrapping would produce tortuous paths in its pore structure. In contrast, in a single wrap the pores would be essentially straight and normal to the heat-transfer surface, and any solution concentrating under the wrap would be subject to facile release. The equivalent pore diameter of the wire-gauze wrapping would be approximately 50 μm , while that in the deposits is typically 1 μm . The bigger pore size in the wire-gauze wrapping would allow easy access of the solution to the heat-transfer surface, while the small pore size in the deposits would restrict the access of the solution and favour the formation of steam-covered areas. This would account for the big difference between the hideout behaviour of the magnetite-coated tubes and that of the wire-gauze-wrapped tube.

4.3 Under-Deposit Chemistry

In several tests, the autoclave was drained at the end of the hideout phase of the test, while the heat flux on the tube was still on, with the intention of retaining any solute hidden out in the deposits for analysis after the test. Although significant hideout was observed in some of these tests (e.g., Runs #29B and #33), no sodium or chloride was present at the detection level in the deposits scraped off the tube. This implies that any solute hidden out escaped to the bulk water when the autoclave was drained. This can happen if the solute is present in the deposits as a solution, and the vigorous boiling action that would result when draining the autoclave would expel the solution from within the deposits to the bulk water. The failure to detect any solute species in the deposit after the test does not, therefore, mean no hideout occurred, but it suggests that all the solutes were present as a solution and the concentration factors calculated from the data obtained during the hideout phase are very likely to be realistic.

However, the absence of detectable amounts of sodium chloride on the tubes after removal from the autoclave does not seem to tell the whole story. SEM/EDX analysis of stains and craters through the deposit show substantial concentrations of chromium, the only source of which is the underlying metal. Localized attack of the Alloy 600, apparently intergranular attack or pitting, seems to have occurred, which suggests an aggressive environment next to the tube surface. The nature and extent of the attack have not been studied in detail by surface examinations. The distribution of the stains and craters that have been observed is quite scattered and suggests that there are a limited number of pores that traverse the oxide deposit, in which the aggressive environment developed. The composition of the oxide deposit and of the corrosion film on the alloy could provide some clues as to the nature of the local environment. Analyses of corrosion films on Alloy 600 under a variety of chemistry and redox conditions have shown that under acidic to mildly alkaline conditions the corrosion film is enriched in chromium, while under alkaline conditions the film is depleted in chromium, owing to the preferential dissolution and mobilization [6]. The enrichment of chromium in the deposit near the craters may be interpreted in two different ways. The partially sensitized state of the Alloy 600 material of the tube makes the alloy sensitive to corrosion in an acidic environment. Moreover, since the inherent film on the tube surface was removed prior to the magnetite coating, active corrosion could be occurring under the deposit during the hideout tests, and it is conceivable that the chromium would be preferentially dissolved from the metal and taken up by the deposit. Alternatively, it could be argued that chromium should be mobile to reach the relatively thick deposit layers, which would suggest an alkaline environment. An alkaline environment can result from the preferential accumulation of sodium in the pores of the deposit during the hideout process. Further experimental evidence should be obtained to support the hypothesis that the environment resulting from hideout in the deposit was alkaline. Since the chromium enrichment occurred only in the craters, the hideout process must have been localized; segregated areas of solution accumulation and areas occupied by steam must have existed in the deposit over the heat-transfer surface, owing to the inhomogeneity of the coating. This supports the earlier deduction from the calculated concentration factors and from thermal conductivity data that the deposits contained a mixture of liquid solution and steam.

5. SUMMARY AND CONCLUSIONS

During the shutdown of steam generators, solutes hidden out in the various crevices and other flow-restricted areas are returned to the bulk water. Analysis of the chemistry of the bulk water during this hideout return is used to deduce the chemistry *in the crevices during operation*, and hence the expected corrosion behaviour. If significant hideout takes place in the porous deposits covering the steam generator tubes, their inclusion in the hideout return could introduce appreciable errors in the interpretation of crevice chemistry. This program, therefore, studied the hideout in deposits on the heat-transfer surface of steam generator tubing.

Tubes were coated with magnetite and heated electrically from the inside using an electric cartridge heater. The hideout from bulk water containing sodium chloride in an autoclave at 279°C was monitored on-line using a high-temperature conductivity probe in the autoclave and off-line by chemical analysis of solution samples from the autoclave. Significant hideout was observed only at a heat flux higher than 200 kW/m² (i.e., at an estimated temperature differential

greater than 2°C across the deposit) and the concentration factor in the deposit resulting from the hideout increased highly non-linearly with the heat flux (varying as high as the fourth power of the heat flux); a concentration factor as high as 2×10^3 was observed at a heat flux of 380 kW/m². The apparent concentration factor seemed to decrease with increasing deposit thickness, which would imply that the pores in the deposit were occupied by a mixture of steam and water. The conclusion from measurements of thermal conductivity of steam generator tube deposits in a separate study supports the existence of a two-phase (steam-water) mixture in the deposits.

Attempts to directly analyze the deposits after the hideout tests using surface analysis by the SEM/EDX system or chemical analysis by ICP-AES were unsuccessful in finding the expected amounts of the solutes. The expected concentration of the solutes in the deposit was too low for these techniques. Moreover, it is possible that the solutes escaped back to the bulk water during the termination of the tests, even though the heat flux was not turned off at that point; the vigorous boiling action may have blown out the solution from the deposit, since in many cases even the magnetite coating did not stay intact after the test. Localized corrosion, apparently intergranular attack or pitting, was observed under the deposit in some cases. The chromium enrichment in the areas of localized corrosion could be explained on the basis of a somewhat alkaline environment, to account for the required mobility of chromium. The alkaline environment probably resulted from preferential accumulation of sodium in the concentrating solution in the deposit. Alternatively, the increased susceptibility of Alloy 600 to corrosion in acid media, owing to the partially sensitized state of the tubes used for the tests, and the possibility of active corrosion resulting from the removal of the inherent film prior to placing the magnetite deposit on the tubes, may make the preferential dissolution of chromium and its incorporation into the deposit a possibility.

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APPENDIX 1

Table A1. Summary of Hideout Runs - Magnetite-Coated Tubes.
(Alloy 600 tubes, unless otherwise stated; 5 mg Cl/kg Solution (NaCl) at 279°C).

Run #	Date	Deposit	# of coatings & sint T (°C)	Heat flux (kW/m ²)	Drained (Hot/Cold)	Hide-out (yes/no)	Deposit Thickness (µm)	Deposit Loading (kg/m ²)	% Porosity	SEM/EDX (S/E)	Comments
1	95/11/29	Magnetite	-	158	cold	no					Tube sample from a PWR SG.
2	95/12/04	"	-	"	"	"					SS tube used. Deposit flaked off.
3	95/12/08	B951124*	1	"	"	"					Approximately 50% of deposit flaked off.
4	95/12/12		multiple	"	"	"	42.5	0.158	28	SE	
5	96/01/02	S.S. mesh	1 wrap	"	"	"					Conductivity probe deteriorated during run.
6	96/01/11	"	2 wraps	"	"	yes					Conductivity probe showed some drift at end of run.
7	96/01/16	"	1 wrap	"	"	yes(?)					Using I-600 tube from Run #6.
8	96/01/22	B951124*	4 coats each @ 800	143.7	hot	yes(?)	63	0.157	52	SE	Using new conductivity probe. Magnetite didn't flake, but did peel back at heat zone.
9	96/02/07	B951124*	4 coats each @ 800	150.3	cold	yes	64				Magnetite flaked when SS plug inserted. No flaking of deposit.
10	96/02/20	B951124*	4 coats each @ 800	"	hot	no	64			SE	Significant flaking. Tube used in Run #9.
11	96/02/26	B951124*	3 coats each @ 800	143.7	cold	no	34	0.151	15	SE	
12	96/02/29	none	none	143.7	"	no					Tube used in Run #12.
13	96/03/22	none	none	various	"	no					Test of Temp/cond. profile & sampling. Heater on from beginning of run. Bare I-600 tube.
14	96/04/01	B951219*	4 coats each @ 800	118.8	"	?					Deposit in contact with solution all flaked off. Deposit intact above solution level, but easily fell off when tube was handled.
15	96/04/10	B960304-05	1 coat	118.8	"	no	23	0.05	58	SE	Tube sintered at 700°C to produce thicker coat. Some pitting/stains of deposit in contact with solution, but deposit appears to be still intact.
16	96/04/22	1 & 2 B951219* 3 B9600404-05	3 coats 1 & 2 - 800 3 - 700	118.8	"	no	51	0.116	56	SE	

continued...

Table A1 (cont'd).

Run #	Date	Deposit	# of coatings & sint T (°C)	Heat flux (kW/m ²)	Drained (Hot/Cold)	Hideout (yes/no)	Deposit Thickness (µm)	Deposit Loading (kg/m ²)	% Porosity	SEM/EDX (S/E)	Comments
17	96/04/25	1 & 2 B951219*	3 coats 1&2 - 800	118.8	"	no	51				Continuation of Run #16 never stopped; pits/stains present at the end of run.
18	96/06/03	3 - B960404-05 none	none	118.8	"	no					Calibrated thermocouple placed in thermowell to measure inside wall temperature of the Alloy 600 tube.
19	96/06/10	none	none	118.8	"	no					As in Run #18.
20	96/06/13	none	none	118.8	"	no					Leak in plug.
21	96/06/17	none	none	118.8	"	no					Tube used in Runs #12 & #13. Groove for thermocouple. Wrong solution used (4.05 ppm Na instead of 3.24 ppm Na with 5ppm Cl).
21B	96/06/21	"	"	118.8	"	?					Same solution as in Run 21. Apparent hideout.
22	96/06/25	"	"	118.8	"	?					Tube used in Runs #12, #13 & #21.
22B	96/06/27	"	"	"	"						Continuation of Run #22.
23	96/07/15	none	"	118.8	"	no					Tube polished to a mirror finish and SS support had a groove for thermocouple.
23B	96/07/20	"	"	118.8	"	no					Tube from Run #23. SS support had no groove.
24	96/07/25	B960524	5 coats all 5 @ 800	118.8	"	no	42	0.154	29	S	Magnetite spalling off at the end of run.
25	96/08/12	B960528*	2 coats 1 & 2 - 700 final - 800	118.8	"	no	40	0.08	62	SE	Coating in heated zone pitted (circular). Edges of pits covered with yellow deposit.
26	96/08/19	B960528*	3 coats 1 - 800 2 & 3 - 700	118.8	"	no	52	0.164	40	S	Many small oval stains upon removal.
27	96/02/27	B960531*	4 coats 1-800 2,3 & 4 - 700	386.8			19	0.163	?		Tube 6. Rupture disc failure due to plugged condenser.
28	97/03/18	B960531*	3 coats 1-800 2&3-700	386.8	cold	yes	16	0.059	26		Tube V.

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