EFFECTS OF DEBRIS GENERATED BY CHEMICAL REACTIONS ON HEAD LOSS THROUGH EMERGENCY CORE COOLING SYSTEM STRAINERS

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The effect of debris generated during a loss of coolant accident (LOCA) on the emergency core cooling system (ECCS) strainers has been studied via numerous avenues over the last several years. The research described in this manuscript examines the generation and effect of secondary materials – not debris generated in the LOCA itself, but materials created by chemical reactions between exposed surfaces/debris and cooling system water. The secondary materials studied in the research were corrosion products from exposed metallic surfaces and paint chips that may precipitate out of solution, with a focus on the corrosion products of aluminium, iron, and zinc. The processes of corrosion and leaching of metals with subsequent precipitation is important because: (1) the surface area of exposed metal inside containment represents a large potential source term, even for slow chemical reactions; (2) the chemical composition of the cooling system water (boric acid, lithium, etc.) may affect corrosion or precipitation in ways that have not been studied thoroughly in the past; and (3) an eyewitness report of the presence of gelatinous material in the Three Mile Island containment pool after the 1979 accident suggests the formation of a secondary material that has not been examined under the generic safety issue (GSI)-191 research program.

This research was limited in scope and consisted only of small-scale tests. Several key questions were investigated: (1) do credible corrosion mechanisms exist for leaching metal ions from bulk solid surfaces or from zinc-based paint chips, and if so, what are the typical rate constants? (2) can corrosion products accumulate in the containment pool water to the extent that they might precipitate as new chemical species at pH and temperatures levels that are relevant to the LOCA accident sequence? and (3) how do chemical precipitants affect the head loss across an existing fibrous debris bed? A full report of the research is available.[1]

1. Johns, R.C., B.C. Letellier, K.J. Howe, and A.K. Ghosh, “Small-Scale Experiments: Effects of Chemical Reactions on Debris-Bed Head Loss”, Los Alamos National Laboratory report LA-UR-03-6415 (November 2003).

Experimental setup and methods

The research consisted of two components: (1) corrosion rate experiments conducted in batch reactors, and (2) head-loss experiments conducted in a closed-loop recirculating hydraulic test system.

Zinc corrosion tests

Corrosion tests were conducted in 1-liter containers. The candidate metal for the corrosion tests was zinc, and the tests were performed with zinc granules, zinc coupons, and crumbled inorganic zinc paint primer. The experimental variables were pH, temperature, immersion duration, and immersion solution. Corrosion rates were determined using the weight loss method; i.e. a quantity of material with a known surface area was immersed in a solution for a known duration, and the change in weight due to corrosion was measured. Samples ranging from about 1 g to 10 g were used, and the weight loss was determined by measuring the weight of the sample before and after immersion using procedures in Standard Method 2540-D.[2] Weight was measured with an analytical balance that had a resolution of 0.0001 g. In addition, the zinc concentration in solution after immersion was measured to provide a second, and independent, measurement of the mass of zinc lost.

The immersion solution for most experiments was a prepared solution containing 3.3×10^{-2} M boric acid (H_3BO_3) and 2.0×10^{-4} M lithium hydroxide (LiOH) in deionised water, to simulate the solution chemistry in the containment pool. The pH of the immersion solution was adjusted using HCl or NaOH, with the target pH typically being either pH = 7.0 or pH = 9.0. Some tests selected as a control group used only deionised water as the immersion solution. The first several sets of experiments used glass containers as the immersion vessel and the final set used polypropylene containers.

The target values for the immersion temperature were room temperature, 40°C, and 80°C. The room temperature experiments were conducted by leaving the immersion vessels on a laboratory countertop; the room temperature in the laboratory ranged from 22°C to 25°C during these experiments. The higher temperature experiments were conducted by placing the immersion vessels in a constant temperature laboratory oven capable of maintaining the desired temperature. The immersion duration ranged from 1 to 11.75 days. Many of the experiments were conducted in triplicate.

Head-loss tests

Head-loss flow tests were conducted in a small-scale (10-liter), vertical, closed-loop circulation, hydraulic test system built for measuring the head loss across a fiber-laden screen in a chemical environment typical of that found in the ECCS recirculation sump. Calibration tests were first performed to confirm that head losses induced by a debris bed in the small test system were consistent with previous experiments and with standard correlations documented in NUREG/CR-6224.[3] Subsequent tests examined the additional head loss incurred by the precipitation of dissolved

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2. *Standard Methods for the Examination of Water and Wastewater*, prepared and published jointly by the American Public Health Association, American Water Works Association, and Water Environment Federation, Washington, DC (1999).

3. Zigler, G., J. Bridaeu, D.V. Rao, C. Shaffer, F. Souto, and W. Thomas, “Parametric Study of the Potential for BWR ECCS Strainer Blockage due to LOCA Generated Debris”, United States Nuclear Regulatory Commission report NUREG/CR-6224, Science and Engineering Associates, Inc. Report No. 93-554-06-A:1 (January 1994).

metals within the closed circulation loop. These tests were performed with a solution chemistry of 3.3×10^{-2} M H_3BO_3 and 2.0×10^{-4} M Li^+ in deionised water, with pH adjusted to either pH = 7.0 or pH = 9.0 by the addition of HCl or NaOH.

Each test was started by filling the system with the recirculation fluid. The recirculation pump was started and adjusted to a low velocity. Shredded NUKON® fibrous insulation was slowly added and allowed to settle against the screen. Once the fibrous debris bed was in place and the head loss was determined to be similar to that predicted by NUREG/CR-6224, the recirculation velocity was adjusted to the predetermined value for that experiment. At that point, precipitation was artificially induced by adding a metal nitrate salt solution to the recirculation loop water. The metal ion concentration in the recirculation loop was significantly above the solubility limit, and precipitates formed almost immediately. The metal ion addition ranged from 5.0×10^{-5} M to 1.0×10^{-2} M. The first tests incorporated the simultaneous precipitation of aluminum, iron, and zinc metals. Head loss across a pre-established fiber mat was observed using pressure transducers located above and below the fiber mat. Temperature and pH were monitored continuously using in-line instruments. The tests were typically an hour in duration, although some longer tests were also conducted.

Results and discussion

Zinc corrosion tests

Six groups of tests were conducted, resulting in 62 separate experiments conducted under a variety of conditions. Each group of tests examined a specific set of independent variables, such as the effect of temperature and pH, the effect of material configuration, or the effect of immersion duration.

The experimental procedures were refined with each set of experiments and, as a result, the final set of experiments produced the most consistent corrosion rate measurements. Weight loss data from the final set of experiments is reproduced in Table 1. For this group of experiments, the material was zinc coupons and the solution chemistry was 3.3×10^{-2} M H_3BO_3, 2.0×10^{-4} M Li^+, and pH = 7, and each test condition was done in triplicate.

At room temperature, the coupons lost an average of 9.9 mg after 2 days and 17.1 mg after 4 days. These weight loss measurements correspond to a corrosion rate of 0.055 g/m^2·h averaged over 2 days and 0.046 g/m^2·h averaged over 4 days. The continued loss of weight between the second and fourth days suggests that the solution had not reached a saturated condition and that the corrosion rates measured by weight loss were representative of the true corrosion rates present under these experimental conditions. These corrosion rates were somewhat higher than those observed in earlier tests but are consistent with the observation that the time-averaged corrosion rate decreased as the zinc concentration increased in solution (in the first group of tests and at the same experimental conditions, the corrosion rate was 0.017 g/m^2·h averaged over 11.75 days).

Similar corrosion rates were initially observed for the coupons immersed at 40°C. These coupons lost an average of 10.4 mg after 2 days, corresponding to a corrosion rate of 0.057 g/m^2·h. After 4 days, however, the average weight loss had increased only marginally to 10.9 mg, causing the time-averaged corrosion rate to drop to 0.030 g/m^2·h. Since the coupons did not continue to lose weight between the second and fourth days, it appears that the solution had reached saturated conditions, which prevented further corrosion of zinc without the formation of corrosion products.

For this group of tests, the measured zinc concentrations in solution were consistent with the measured weight lost from the zinc coupons, providing two independent sets of measurements that result in similar corrosion rates.

Tests conducted at higher temperature and higher pH conditions were less successful at producing quantifiable corrosion rates, but were nonetheless successful at producing qualitative indications of corrosion. Many of the tests at these conditions resulted in a weight gain over the test duration, thus indicating the formation of a corrosion product with a higher molecular weight than the original material. In addition, many of these tests resulted in the formation of a black coating on the zinc granules and coupons, which could be scraped off. The black coating and the increase in weight indicate the formation of a corrosion product and are qualitative indicators of corrosion.

Table 1. Weight-loss measurements and corrosion rates for the sixth group of zinc corrosion experiments

Test no.	Temp (°C)	Time (h)	Beginning weight (g)	Final weight (g)	Weight change (g)	Corrosion rate ($g/m^2 \cdot h$)
6-1	40	48	3.4539	3.4403	-0.0136	0.0769
6-2	40	48	3.4752	3.4679	-0.0073	0.0410
6-3	40	48	3.8883	3.8780	-0.0103	0.0518
				Average:	-0.0104	0.0566
				Std. dev:	0.0032	0.0184
6-4	40	96	3.4306	3.4190	-0.0116	0.0330
6-5	40	96	3.4963	3.4880	-0.0083	0.0232
6-6	40	96	3.8794	3.8666	-0.0128	0.0322
				Average:	-0.0109	0.0295
				Std. dev:	0.0023	0.0055
6-7	22	48	3.4408	3.4298	-0.0110	0.0625
6-8	22	48	3.4763	3.4666	-0.0097	0.0545
6-9	22	48	3.7284	3.7194	-0.0090	0.0472
				Average:	-0.0099	0.0547
				Std. dev:	0.0010	0.0077
6-10	22	96	3.4526	3.4348	-0.0178	0.0504
6-11	22	96	3.7159	3.6979	-0.0180	0.0473
6-12	22	96	3.9052	3.8897	-0.0155	0.0388
				Average:	-0.0171	0.0455
				Std. dev:	0.0014	0.0060

The results from these experiments were compared to previous research conducted under similar conditions. Piippo *et al.*[4] measured zinc and aluminium corrosion rates using electrical resistance measurements with several test solutions. The solutions included: (1) distilled water that had been adjusted to pH values of 8.0 and 10.0 using LiOH and maintained in either aerated or de-aerated conditions and (2) a 0.1-M H_3BO_3 solution buffered at pH 9.2. For purposes of comparison, the test conditions in the current experiments are most closely comparable with the H_3BO_3 solution used by Piippo. In that solution, Piippo measured zinc corrosion rates of 0.05 g/m^2·h at 50°C, 0.03 g/m^2·h at 70°C, and 0.04 g/m^2·h at 90°C. Piippo et al. noted that their measured corrosion rates were consistent with previous results reported by van Rooyen[5] and Loyola and Womelsduff[6], which also experimentally measured corrosion rates of zinc in water containing H_3BO_3. The results of the current study, with corrosion rates of 0.055 g/m^2·h at 22°C and 0.057 g/m^2·h at 40°C, are consistent with rates measured in previous studies.

Piippo measured higher zinc corrosion rates under other experimental conditions. For most aqueous solutions, the corrosion rate increased by at least an order of magnitude when the temperature increased above the normal boiling point of water. In the H_3BO_3 solution, Piippo measured a zinc corrosion rate of 4.45 g/m^2·h at 110°C and 1.26 g/m^2·h at 130°C. However, the highest measured zinc corrosion rate in the Piippo report was a value of 11.27 g/m^2·h, which was measured in deaerated deionised water at 170°C, after the test materials had been exposed to hot steam at 300°C.

Several attempts were made to identify chemical and physical characteristics of the corrosion products. Visualisation with a light microscope demonstrated a change in appearance after immersion, with the zinc granules exhibiting a shiny, light-grey appearance before immersion and either a dull light-grey or dull black appearance after immersion. Scanning electron microscope (SEM) imaging identified the formation of a platelet structure, which was not characteristic of the original zinc material. Elemental composition by energy-dispersive spectrometry (EDS) and zinc content by mass balance both suggested that the corrosion product was about 60 percent zinc. Chemical composition by x-ray diffraction suggested the presence of zinc oxide but could not conclusively identify other zinc compounds. EDS identified the other elements present as oxygen (18 to 20 percent), silica (10 to 12 percent), carbon (6 to 10 percent), and aluminium (trace). No evidence of the presence of boron or lithium was observed. One of the species predicted to precipitate by water-chemistry modelling is $Zn_5(CO_3)_2(OH)_6$, which has an elemental composition of 60 percent zinc, 35 percent oxygen, 4 percent carbon, and 1 percent hydrogen. It is possible that the EDS analysis detected a combination of $Zn_5(CO_3)_2(OH)_6$, background metallic zinc, and other compounds that formed on the granules, including some silica-containing compounds.

4. Piippo, J., T. Laitinen, and P. Sirkiä, "Corrosion Behaviour of Zinc and Aluminium in Simulated Nuclear Accident Environments", Finnish Center for Radiation and Nuclear Safety report STUK-YTO-TR 123, (February 1997).

5. van Rooyen, D. "Hydrogen Release Rates from Corrosion of Zinc and Aluminum", BNL-NUREG-24532 informal report, pp. 1-37 (May 1973).

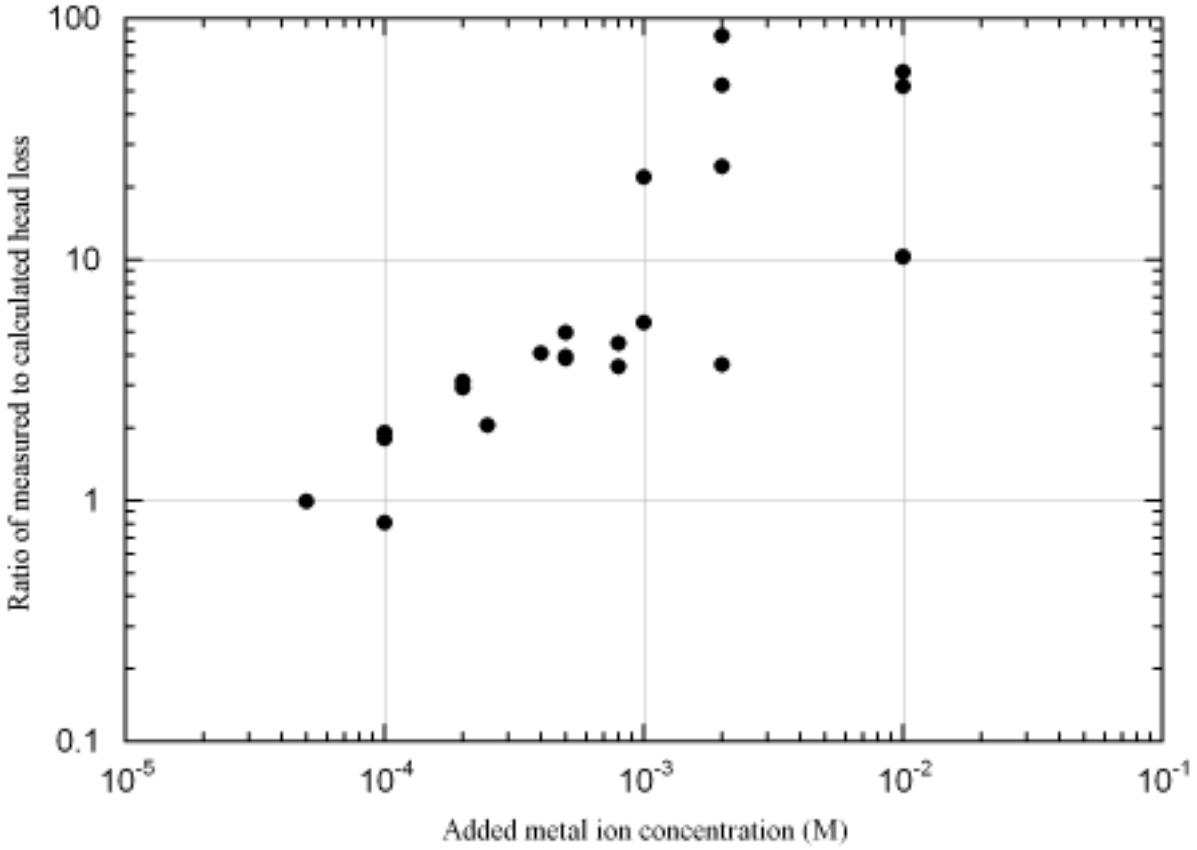
6. Loyola, V.M. and J.E. Womelsduff, "The Relative Importance of Temperature, pH, and Boric Acid Concentration on Rates of H_2 Production from Galvanized Steel Corrosion", Sandia National Laboratories report SAND82-1179, United States Nuclear Regulatory Commission report NUREG/CR-2812 (November 1983).

Head-loss tests

Previous experimentation has established a correlation for head loss through ECCS strainers caused by a bed of fibrous debris composed of NUKON® insulation. This correlation was documented in NUREG/CR-6224.[3] The primary focus of the current experiments was on the additional head loss due to the capture of insoluble corrosion products that may precipitate after the corrosion of metals in the containment structure, assuming a fibrous debris bed has already formed on the ECCS strainers. The first tests incorporated the simultaneous precipitation of calcium, aluminium, iron, and zinc compounds, which were each added at a concentration of 1.0×10^{-2} M. High head loss was observed almost immediately, and the test had to be terminated within 15 min because the head loss had exceeded 15 ft and the recirculation flow rate had dropped to almost zero. Subsequent tests were conducted with only one metal precipitate at a time.

In all, more than 20 experiments were conducted with various concentrations of metal precipitates. The head loss caused by a combined bed of fibrous debris and precipitated metals was compared to the head loss by a fiber-only debris bed. This comparison was made by calculating the fiber-only head loss with the NUREG/CR-6224 correlation using the NUKON® quantity and actual measured temperature and velocity at the end of each test. A ratio of head loss with and without precipitated metals was then calculated, and is presented in Figure 1 for each of the tests.

Figure 1. Ratio of head loss through a fibrous debris bed with and without chemical precipitates



The ability of a precipitate to cause additional head loss appeared at an added metal ion concentration of about 1.0×10^{-4} M (equal to a concentration of 6.5 mg/L of zinc or 2.7 mg/L of aluminium). These concentrations correspond to less than 100 lb of metal dissolved into 1 million gallons of water. The quantity of NUKON® used for preparation of the fibrous bed was 4.4 g (~1.5 cm^3/cm^2); therefore, the precipitate-to-fiber mass ratio at which additional head loss appeared was 0.015 for zinc. These results are significant because previous studies have reported that the sludge-to-fiber mass ratio at which additional head loss appears was 0.1 or higher.[3] Additional head loss from precipitates of corrosion products may be significant at mass ratios on an order of magnitude lower than reported for incompressible particulate debris.

The results were reasonably consistent and repetitions of tests under identical test conditions produced repeatable results despite the potential for wide variations resulting from test conditions that were difficult to control, such as the uniformity of the formation of the initial fibrous debris bed. Higher quantities of metal precipitate consistently led to higher head loss. The head loss through a mixed bed of precipitate and fibers was about an order of magnitude higher than that through a fiber-only bed when the added metal concentration reached about 2.0×10^{-3} M. Greater variability in the ratio of head loss with and without precipitates developed above an added metal concentration of about 1.0×10^{-3} M. Above this value, the measured head loss through the debris bed was substantial and taxed the ability of the recirculation pump in early experiments (the pumping system was modified in later experiments, leading to an improved ability to maintain a uniform velocity throughout the test). The greater variability in this ratio at low flow conditions suggests that the head loss caused by the precipitated material does not have the same dependence on velocity as other materials that have been studied. This different dependence on velocity may be due to the compressibility of the material, since other debris that has been studied has been incompressible.

Physical examination of the beds after the tests revealed the presence of a sticky, gelatinous coating on the entire surface of the bed. This continuous gelatinous layer appeared to cause more physical resistance to water flow than mixed beds containing fibers and discrete particles. Examination of the beds by SEM showed that material adhered to individual fibers, although the gelatinous materials were desiccated by the high vacuum in the SEM. This gelatinous coating may be compressible and may exhibit a significantly different head-loss relationship than that described in NUREG/CR-6224.

Conclusions

Leaching tests to evaluate the corrosion of metal materials were conducted at ambient and elevated temperatures and at two pH values (pH = 7 and 9) in the presence of an aqueous solution containing boric acid and lithium. The ability of metals to corrode under these conditions was observed. Evidence for corrosion included weight loss of the metal materials and accumulation of soluble metal ions in the water. The measured corrosion rates were similar to literature values from previous studies with similar chemical conditions.

High-temperature corrosion tests attempted in this study were clearly confounded by exceeding the solubility limits of zinc in solution. Because the immersion vessels were quiescent, it is possible that only the local concentration near the sample surface, and not the bulk concentration, exceeded saturation when crystallisation was initiated. This condition would not be expected in a system with flowing water, such as in the containment pool.

Chemical solubility relationships predict that chemical precipitation can occur at relatively low concentrations of dissolved metal; precipitation at low metal concentrations was confirmed in

laboratory testing. Chemical solubility relationships suggest that the precipitants are metal oxides and hydroxides, which can have flocculent characteristics, thus causing them to aggregate into amorphous masses that can plug a fibrous debris bed more efficiently than dust or dirt that may be present on the containment floor. Preliminary tests reveal that these metal precipitants can have a significant effect on the head loss through a fibrous debris bed on an ECCS strainer. Precipitation (noted as a rapid milky white change in water clarity) and measurable head loss have been observed for concentrations as low as 1.0×10^{-4} M. Higher metal concentrations caused head loss that was substantially greater than the head loss occurring through a fibrous debris bed without chemical precipitants. As a result, these tests suggest that secondary debris generation may be a significant issue that should be addressed with further research.

Referring to the three questions posed at the beginning of this manuscript, the following conclusions can be drawn from this research:

1. Credible evidence of a corrosion mechanism for zinc in conditions characteristic of the containment pool after a LOCA were observed. The measured corrosion rate was 0.055 $g/m^2 \cdot h$ at 22°C.
2. Theoretically, corrosion products could accumulate in the containment pool water to the extent that they might precipitate as new chemical species at pH and temperatures levels that are relevant to the LOCA accident sequence. The solubility of metal oxides and hydroxides is very low and the surface area of exposed metals within containment is substantial.
3. If precipitation occurs, the chemical precipitants could have a substantial negative effect on the head loss across an existing fibrous debris bed. Additional head loss was observed at added metal ion concentrations as low as 1.0×10^{-4} M, and the head loss increased by about an order of magnitude above that without metal precipitants when the added metal ion concentrations reached 2.0×10^{-3} M.

This investigation provided credible evidence for a sequence of events that could lead to excessive head loss across the ECCS sump strainers following a large LOCA. It should be noted, however, that the complete progression of events necessary to produce excessive head loss was not studied in an integrated manner. The scope of the experiments was limited to an analysis of the individual steps in the progression scenario; i.e. corrosion/leaching tests in a batch reactor and head-loss tests with artificially induced precipitation. Integrated testing of the complete progression of events is being explored as a follow-on activity.

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