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CORROSION TEST OF IRRADIATED URANIUM
IN MONOISOPROPYLBIPHENYL (RM-171)

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CORROSION TEST OF IRRADIATED URANIUM
IN MONOISOPROPYLBIPHENYL (RM-171)

INTRODUCTION:

The use of organic cooling media for nuclear reactors operating at high power levels predicates the use of a coolant which will not react violently with metallic uranium in the event of a fuel element failure. This report describes the testing, and subsequent examination, of two pieces of irradiated uranium which were immersed in mono-isopropylbiphenyl (MIPB) at high temperatures and pressures for periods of time up to twenty-five days. The uranium samples had different irradiation histories and cooling times.

Similar experiments had been performed with unirradiated uranium by the Corrosion and Coatings Operation, and it was wished to determine whether irradiated uranium would react with MIPB in a different manner.

SUMMARY:

A sample of uranium which had been irradiated to 5.50×10^{20} nvt and was six months cooled, was immersed in MIPB at 400 C and at a pressure not greater than 300 psi. Immersion under these conditions for 105 hours produced visible corrosion. Up to 342 hours of immersion was necessary to produce a similar amount of corrosion with a uranium sample which had received 1.47×10^{20} nvt, and was 3-1/2 years cooled.

The rate and severity of corrosion of irradiated uranium by MIPB was found to be no greater than for unirradiated uranium.

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SUMMARY: (Continued)

Radiation damage to the MIPB by the high exposure sample is believed to be the probable cause for the sample's more rapid corrosion. Longer immersions of both samples produced correspondingly greater corrosion.

DISCUSSION: Materials and Equipment

The MIPB used was obtained from the Monsanto Corporation. It was received as an isomeric mixture containing 62 percent in the meta, and 38 percent in the para configuration, and was used without any further purification.

One sample of uranium in the form of a cube weighing 25.43 g was obtained from a normal fuel element which, after being exposed to 1.47×10^{20} nvt, had been cooling for about three and a half years. This sample will hereafter be referred to as sample A.

Sample B was taken from an I & E production fuel element which had failed. This sample, weighing 23.96 g, was a quarter section of a 1/4 inch transverse wafer. It had received 5.50×10^{20} nvt and had cooled for six months. The material from which this sample was obtained was known to contain microcracks.

A high pressure bomb was obtained from the Corrosion and Coatings Operation for this test. The bomb and its associated equipment were prepared for remote handling.

The bomb cap was fitted with a thermocouple sheath and a gas venting line. These leads exited from the cell through the cap wrench handle. A 0-1000 psi pressure gauge was connected to the venting line. The gauge was graduated in units of 10 psi.

The venting system consisted of 1/16 inch I.D. tubing, which was controlled by a Hoke valve. The vented gases were returned to the cell atmosphere.

A cylindrical sieve basket was fabricated from 20 mesh, 0.017 wire stainless steel mesh. This basket would contain the uranium sample and its possible fragments, and would permit its entry into and removal from the bomb.

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DISCUSSION: (Continued)

A 1000 watt tube furnace was mounted to heat the bomb while it was being held in a vertical position by the cap wrench handle.

DISCUSSION: Experimental

The corrosion tests were performed in the following manner:

The uranium sample and 50 ml of MIPB were put into the bomb and, after being sealed, the bomb was brought up to temperature (400 C) as rapidly as possible. A Brown temperature recorder-controller held the bomb temperature to this value.

The temperature of 400 C was chosen for this experiment in the belief that it was probably a temperature higher than any for which an organic moderated reactor might be designed. Therefore, it would represent a conceivable "worst condition" of operation.

The gas pressure over the sample was read routinely. Whenever the system pressure was expected to exceed the prescribed maximum value of 300 psi before the next inspection period, the system was vented until the pressure dropped to 100 psi. At 400 C the vapor pressure of MIPB was found to be about 92 psi, so that this procedure prevented the free boiling of MIPB.

The reason for not allowing the system to attain pressures in excess of 300 psi is that hydrogen gas is one of the reaction products between MIPB and uranium. If much hydrogen was present in this experimental system, uranium hydride would be formed. It had been shown that uranium hydride is an intermediate compound necessary for the formation of uranium carbide in the MIPB environment.⁽¹⁾ In experiments with cold uranium it had been found that when the pressure was maintained below 300 psi the rate of formation of uranium carbide was slow. Therefore, since it was wished to duplicate those experiments which had been performed with cold uranium as closely as possible, this criterion was established.

(1) Private communication from H. C. Bowen, Corrosion and Coatings Operation, HLO.

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DISCUSSION: (Continued)

The less radioactive of the two uranium pieces was the first sample to undergo this corrosion testing.. Sample A had received 1.47×10^{20} nvt, and had been cooling for at least three and a half years. Radiation damage to the MIPB from this sample was expected to be small relative to the thermal decomposition which the organic liquid would experience at the temperature of operation of the reaction bomb. It was expected that radiation damage of the MIPB would produce reactive species which could enhance the corrosion rate of the uranium.

After sample A had been heated in the bomb for 90 hours, the system was allowed to cool. A sample of the organic phase was removed for analysis. It had turned a dark brown color and was quite viscous. The uranium sample was rinsed with acetone and examined. No evidence of corrosion could be seen. The bomb was rinsed out and reassembled with 50 ml of fresh MIPB.

This examination was repeated after another 252 hours. A coating of corrosion product was seen on the sample, but, as it did not appear to be very thick, the sample was re-immersed in fresh MIPB for another 252 hours.

After a total immersion of 594 hours considerable corrosion had occurred to sample A. Attack of the sample had been general, but was more pronounced at the corners. The sample was thickly coated with a type of corrosion product which is characteristic of uranium carbide. The corrosion product was fragile and broke away from the uranium easily (Figure 1).

The sample had been a cube with the dimensions 0.53 x 0.53 x 0.57 inches. With the loose corrosion product removed, its dimensions were now approximately 0.39 x 0.38 x 0.42 inches. The average dimensional change had been a loss of 0.15 inches in all directions.

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DISCUSSION: (Continued)

The sample was sectioned to expose the center for further examination. It presented an appearance which suggested that some intergranular corrosion had occurred (Figure 2). Before further examination could be pursued, the sample was lost.

The second sample, sample B, which had undergone exposure to 5.50×10^{20} nvt, and was six months cooled, was treated in a similar manner. After immersion in 50 ml of MIPB, the temperature was raised to 400 C and the system was vented whenever the pressure approached 300 psi.

After 105 hours the bomb was allowed to cool. Examination of the sample showed the presence of corrosion product to a slight extent. The run was continued for a total of 195 hours with the same charge of MIPB.

After 195 hours of immersion in MIPB under the experimental conditions, sample B was badly corroded. The piece of inner can wall was missing and no bare uranium metal could be seen. Figures 3a, b, and c show the appearance of the sample prior to, and after, 105 hours, and 195 hours of immersion in MIPB. The sample was sectioned, mounted, ground and polished. Figure 4 shows the cross sectional view of the sample. Note the thickness of the corrosion product and the convex shape of the unattacked metal. Micro examination revealed that there was no intergranular corrosion or pitting type attack. The corrosion was limited to general surface attack (Figure 5).

The high temperature reaction of MIPB with uranium under high pressure produces an end product of uranium carbide. This reaction product is fragile and is easily fragmented. When irradiated uranium reacts to produce such a compound, the liquid in which the reaction occurs may become contaminated. The reaction product may be fragmented into various sizes of particles, ranging down to extremely small ones. This type of contamination is of a particulate nature, and could be removed from the organic liquid by some such process as centrifugation or filtering.

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DISCUSSION: (Continued)

It is also possible that fission product species could be leached from the fragmented reaction product by the organic liquid. These fission products might then chemically combine directly with the MIPB or with its thermal and radiation-damage degradation products. Such contamination as this could be removed only by a more refined form of treatment, such as a solvent extraction, or adsorption column process.

Two samples of the MIPB were removed from the bomb after such contamination had presumably occurred. One sample was removed after 90 hours of reaction with sample A. The second sample was obtained after the 195 hour test on sample B. The samples were gamma counted before and after being centrifuged for two hours in a standard laboratory centrifuge. The results may be found in Table I.

TABLE IAnalysis for Centrifugeable Solids

<u>Sample A</u>		<u>Sample B</u>	
Before centrifuging (γ c/m)	2240		7.0×10^6
After centrifuging (γ c/m)	208		1.47×10^5
% of uncentrifugeable activity 8.5%			2.1%

The activity in the liquid left after centrifuging those solids, which were large enough to be removed, gives the relative amount of contamination which exists as something other than particles larger than a few microns. No attempt was made to further define the absolute nature of this contamination.

CONCLUSIONS:

An immersion time in MIPB of 105 hours produced a discernable layer of corrosion product on a piece of high exposure irradiated uranium. By comparison, an immersion time of 342 hours in MIPB was necessary to produce a similar amount of corrosion product on a relatively low exposure piece of irradiated uranium. In either case, the times under consideration are more than ample for the removal of an element failure from a reactor which

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CONCLUSIONS: (Continued)

is cooled with an organic media such as MIPB. These times necessary for significant corrosion to occur are also in agreement with experiments which have been performed with unirradiated uranium by the Corrosion and Coatings Operation.

The high temperature and pressure under which these experiments were performed will produce carbonaceous degradation products in MIPB. Radiation damage to an organic compound such as MIPB will produce free radicals. A combination of these effects could be expected to alter the normal chemical behavior of the pure compound.

The organic phase becomes contaminated by fission product reaction or adsorption by the organic degradation products, or by fragmentation of the corrosion product from the uranium. The finely divided corrosion product would then be distributed throughout the MIPB.

It was also shown that, of this radioactive contamination of the MIPB, between ninety and ninety-eight percent can be removed by centrifugation. Filtration studies were not pursued in this investigation.

Thus, this study has shown that a uranium fuel element failure in a MIPB liquid cooled reactor would not result in rapid and severe corrosion of the uranium. Most of the contamination of the coolant could be removed by physical means.

RL Brandt:mm

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MATERIALS DEVELOPMENT OPERATION

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HW-58143

Neg. #8315

Mag. 2X

FIGURE 1

Sample 171-A after 644 Hours Exposure to MIPB

Neg. #8322

Mag. 9X

FIGURE 2

Sample 171-A after Sectioning

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HW-58143

Neg. #7177

2X

a. Sample 171-B As Sectioned.

Neg. #8030

2X

b. Sample 171-B After 105 Hours Immersion.

Neg. #8091

2X

c. Sample 171-B After 195 Hours Immersion.

FIGURE 3

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Neg. #8285

2X

FIGURE 4

Sample 171-B Sectioned

Neg. #8515

250X

FIGURE 5

Sample 171-B. Corrosion Product - Uranium Boundary

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