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CONSIDERATIONS ON THE MEDIA
FOR DECAY STORAGE OF FUEL ELEMENTS

NOVEMBER, 1968

AEC RESEARCH &
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CONSIDERATIONS ON THE MEDIA
FOR DECAY STORAGE OF FUEL ELEMENTS

By

W. K. Winegardner
Chemical Technology Department
Chemistry and Metallurgy Division

for

FFTF Project

November 1968

BATTELLE MEMORIAL INSTITUTE
PACIFIC NORTHWEST LABORATORY
RICHLAND, WASHINGTON 99352

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CONSIDERATIONS ON THE MEDIA
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W. K. Winegardner

I. INTRODUCTION

This report was prepared in response to a Fast Flux Test Facility (FFTF) Division request for information and recommendations concerning the decay storage of irradiated fuel elements. The report first summarizes information concerning irradiated fuel element storage techniques. This summary is followed by a discussion of information related to fuel cladding corrosion problems that conceivably could result from decay storage after in-core exposure. Finally, all of the information, obtained primarily by literature review, is used as the basis for recommendations concerning storage media.

The work was prompted by concern over possible loss of fuel element cladding integrity during decay storage in air or water. The concern ranges from effects that could hinder fuel examination to those that could result in local regions of cladding penetration or even failure while handling fuel. This report is primarily related to the latter type of effect, since the facility is viewed simply as a cell or basin where fuel elements can safely be stored prior to chemical processing.

II. SUMMARY

Literature review revealed that it is common practice to store irradiated fuel in water-filled pools or basins. Such facilities inherently provide reliable heat removal and adequate shielding. Water is stable and can be cleaned of contaminants by ion-exchange. The ability to conveniently manipulate fuel elements under water has been established, and finally, a pool provides a certain degree of safety because it is a large heat sink. The acceptance by industry and the above combination of desirable features are the bases for the recommendation that the facility for storing spent FFTF driver fuel should also be a water-filled pool. Sealed cans, or storage in cans that are vented to exhaust ventilation filters, is recommended for failed fuel. The capability to suspend and manipulate thimbles or canisters in the water-filled pools should be provided. This would permit not only storage of potentially reactive, nonroutine, or future fuels without direct contact with water, but also could possibly provide backup should concern over storage of spent driver fuel in direct contact with water prove valid. Solids, including lead and aluminum-silicon alloy, have been considered or used as materials to facilitate heat removal from cans.

The recommendation of a water-filled pool, based on current acceptance and the combination of desirable features, assumes that it is necessary to select a storage medium now, so that conceptual design can proceed. Experience with high burnup fuels and higher coolant temperatures could reveal the inadequacy of the bases for this recommendation. In addition, literature review and phone calls failed to resolve instances of corrosion that can be related to the storage of irradiated fuel in water. It is felt that the concern arising from the preceding two statements is not presently sufficient

to override the combination of desirable features associated with water. However, it is recommended that the effects of water storage on stressed, sensitized cladding material with in-reactor exposure be investigated experimentally. Continued review of literature, especially for results from specific tests that are being conducted by Argonne National Laboratory personnel, is also recommended.

III. DISCUSSION

As indicated in the introduction, the report was prepared in response to a FFTF request related to the decay storage of irradiated fuel elements. Initial objectives included:

- A. Investigate corrosion and compatibility aspects of potential storage mediums and components to be stored.
- B. Investigate and recommend storage mediums and conceptual design features for storing:
 - 1. Irradiated driver fuel sections.
 - 2. Packaged pins, specimens, and hardware.
 - 3. Nonroutine and future fuels.

More specifically, investigations were to include at least the following storage mediums: water, inert gas or air, and oil. It was indicated that visual control of all phases of operation was desired. Irradiated driver fuel sections are assumed to be spent austenitic stainless steel clad fuel pins with oxide cores while nonroutine and future fuels are assumed to be sodium bonded, carbide, vented, and failed fuel elements.

Before proceeding, it should be noted that both of the above objectives include the term investigation. The investigation was primarily a literature survey: no experimental work was conducted. Any investigation of potential storage

media that is primarily a literature survey is inherently restricted by the fact that most of the information concerning corrosive aspects and major storage medium concepts is related to water. As will be revealed in the following section, this is simply because irradiated fuel elements have usually been stored in water-filled pools or basins.

A. STORAGE TECHNIQUES

An excellent survey article concerning storage techniques for irradiated fuel, edited by C. E. Guthrie, was presented in the fall, 1965 edition of Nuclear Safety.⁽¹⁾ This article stated that "The use of water-filled pools for storage of metal clad [not sodium bonded] inert fuels appears to be universally accepted as a safe practice... the words most often used [by reactor representatives] to describe a water-filled pool for storage of irradiated fuel were convenient, economical, and adequate." It was also stated that, "With the exception of the Enrico Fermi Fast Breeder Reactor [FERMI], the reactors that utilize fuel which is not metal-clad or which is bonded with sodium do not store their fuel in direct contact with water." Water can still be used to remove decay heat, but in general, cans, thimbles, or canisters are used to prevent contact.

Storage techniques for selected reactor plants are summarized below. Descriptive material was taken from References 1 and 2. Except for the Peach Bottom Reactor, reactors were selected because of the utilization in either the core or blanket of fuels that are bonded to the cladding with sodium or NaK. It should be noted that Reference 2 is an excellent summary of major fuel decay storage considerations: location, heat-removal, criticality control, shielding, and fission product containment and control.

FERMI.^(1,2) After cleaning, FERMI fuel subassemblies are transferred to a water-filled tunnel which connects with a cutup pool. In the cutup pool the assemblies are sealed in a can for leak check. The subassembly is then left in the can if after appropriate decay the fission product concentration of liquid from the can exceeds $5 \times 10^{-3} \mu\text{Ci}/\text{cm}^3$. If the subassembly is classified as a nonleaker (fission product concentration lower than $5 \times 10^{-3} \mu\text{Ci}/\text{cm}^3$) then it is removed from the can and transferred to a water-filled pool (decay pool) for storage. Leakers can also be stored in the decay pool in their sealed cans. Apparently, only blanket rods are fabricated with a sodium bond.

Dounreay Fast Reactor (DFR).⁽²⁾ The technique used at the DFR is to seal irradiated fuel in cans before they are removed from the reactor building. The cans contain lead to facilitate transfer of decay heat. Canned subassemblies are then stored in water decay pools located outdoors.

Hallam Nuclear Power Facility (HNPF).⁽¹⁾ It is stated in Reference 1 that "At Hallam, the sodium-bonded fuel is suspended in dry stainless steel thimbles that are cooled on the bottom and sides by immersion in concrete covered water-filled vaults in the reactor confinement building. The open-topped thimbles are sealed and shielded by concrete-filled plugs."

Experimental Breeder Reactor-II (EBR-II).^(1,2) EBR-II fuel is stored in a rack inside the containment vessel. The rack is submerged in sodium. It should be noted that EBR-II fuel handling is somewhat special in that fuel can be transported to an on-site processing plant with an inter-building coffin (fuel is cooled by forced argon flow during transfer).

Peach Bottom Reactor.⁽¹⁾ It is stated in Reference 1 that "The graphite clad uranium carbide fuel used at Peach Bottom is sealed in dry stainless steel cans and then submerged in a pool in a confinement building."

B. CORROSION AND COMPATIBILITY PROBLEMS

Investigation suggested five phenomena associated with in-core exposure that could initiate metallurgical changes in fuel cladding: radiation damage, sigma phase formation, temperature sensitization, mass transport, and sodium penetration. Permanent metallurgical changes arising from such phenomena could conceivably reduce cladding corrosion resistance in candidate storage media. Temperature sensitization refers to the enhanced possibility for intergranular corrosion arising from carbide precipitation (chromium carbides) upon exposure of 18-8 austenitic stainless steel in the 800 to 1500 °F range. Sigma phase formation refers to the formation of a hard, brittle, intermetallic, iron-chromium compound that can affect both mechanical and corrosion properties. The term "mass transport" is used to indicate the formation of ferritic surface layers by nickel (and chromium) depletion. The mechanism for sodium penetration remains undefined, but it is conceivable that sodium could penetrate by volume diffusion, grain boundary diffusion, or by microcracks. Sodium penetration could be a complex phenomenon. For example, it is stated in Reference 3 that "It is believed that the sodium does not penetrate into the grain boundaries as an aggregate, but as some complex alloy that does not necessarily markedly affect the physical properties of the metal but that can, under some stress and temperature conditions, affect the mechanism of failure or crack propagation." Before specific instances associated with sensitization, sodium penetration, etc. are discussed, it should be noted that potential detrimental effects could be initiated by phenomena other than those leading to corrosion. For example, severely reduced visibility or even inadequate cooling could conceivably result from the degradation, by radiation and temperature, of oil.

1. Review of ANL Literature

Several references suggesting potential corrosion effects were found in Argonne National Laboratory (ANL) reports and publications. The January 1967 ANL Reactor Development Program Progress Report⁽³⁾ presented results, arising from the analysis of 304 Stainless Steel (SS) which had been exposed to 1200 °F sodium, indicating potentially serious problems. Briefly, aluminum sulfate etchant penetrated deeper into samples exposed in sodium than in samples heat treated in vacuum. It was stated in this report that "The various tests on both tube and rods show chemically and metallographically that sodium penetrates 0.006 to 0.010 in. into the grain boundaries within only 7 days." It should be noted that based on an unsuccessful subsequent attempt to reproduce the above degree of penetration, it was "... tentatively concluded that the effect of sodium on stainless steel is dependent on impurities in the sodium." (p. 51)⁽⁴⁾

A second indication of a potential problem arose from the investigation of surface anomalies on EBR-II blanket tubing where "...it was suspected that water cleaning after sodium exposure could produce results of the nature observed [pattern of alternate bright and dark areas]."⁽⁵⁾ It was noted in Reference 5 that "Examination of the wall sections showed that the microstructures of the sodium-exposed and water-cleaned 304 SS samples were similar in nature to sodium exposed samples subjected to a modified Strauss solution. ... Apparently, it takes distilled water about 30 days to produce the same visual effects on 304 SS microstructure that can be accomplished in 15 min in modified Strauss solution" (p. 6).*

* *Tubing samples pretreated in 1200 °F flowing sodium for 5 days, washed in alcohol for 1 hour, exposed to laboratory air for 60 days, then immersed in 180 °F distilled water. Modified Strauss solution is apparently a boiling solution consisting of 30 g CuSO₄, 54.9 ml H₂SO₄, and 870 ml H₂O.*

continued investigation it was noted that "This [catastrophic effect with 15 min exposure to Strauss reagent] indicates that presensitized Type 304 stainless steel that is exposed to 1200 °F sodium when under stress has a microstructure very susceptible to intergranular corrosion" (p. 15).⁽⁴⁾

Observations such as the preceding apparently prompted additional series of tests. One of these is termed "Cyclic Testing of Stressed Capsules (Type 304L Stainless Steel) at EBR-II" in ANL literature.⁽⁶⁻¹⁵⁾ Briefly, "This work involves metallographic comparisons of as received Type 304L stainless steel tubing with samples from pressurized tubes which will be exposed to EBR-II primary sodium (700 °F), cleaned by standard EBR-II procedures and equipment, stored in air, and returned to the primary tank" (p. 52).⁽⁶⁾ The test is in progress.

In a second test series, "EBR-II Subassembly Recycle Test," the cladding from several fuel pins will be examined metallographically after five cycles (sodium exposure followed by washing and drying).^(7-10,13-15) This test is also in progress.

A third test series, "Metallographic Examination of Capsule-wall Material (Type 304 Stainless Steel) from Capsules Irradiated in Subassembly XG06," consisted of the examination of test capsules that were exposed in EBR-II core for about 18 months and then washed (some were stored in water for about 11 days).⁽⁶⁻¹¹⁾ It was reported in Reference 11 that, "The conclusion [arising from metallographic examination] was that the Type 304 stainless steel is normal considering the temperature history of the material" (p. 81).

A fourth series of tests, "Effect of Recycling Types 304 and 304L Stainless Steel Tubes" or "Cyclical Testing of Types 304L and 304 Stainless Steel Tubing Exposed to Sodium at 1100 °F, Washed in Water, and Stored in Air" is to evaluate the effect of storing tubing in air after exposure to 1100 °F

sodium.^(6-13,16) Some mechanical property data arising from this test is given in Reference 7, but, for the most part, conclusions are based on the measurement of electrical resistance after accelerated corrosion with modified Strauss reagent. This technique, sometimes used for investigating intergranular attack of stainless steel, is discussed in Reference 17. The test series, also apparently incomplete, is difficult to summarize because of its broad scope. Briefly, observations based on resistance measurements "... indicate the general desirability of not re-exposing Type 304L Stainless Steel to sodium after any storage in air. But the swift rise in resistivity of the 120-day dry air stored specimens [CaSO_4 -desiccated air]...leads to the conclusion that re-exposure of Type 304L Stainless Steel that has been exposed to dry air for an appreciable time should particularly be avoided" (p. 76).⁽¹¹⁾ Greater attack in dry air than in normal (humid) air is attributed to formation of oxides instead of hydroxides. It is further postulated that these oxides can then act as a seal at or in grain boundaries, trapping unreacted sodium. In addition, metallographic examination revealed sensitization upon re-exposure, in 1100 °F sodium, of 304L SS samples previously stored in air.⁽⁷⁾ Tube samples have been examined by electron microprobe but "The sensitivity...was inadequate, however, to arrive at conclusive results in detecting sodium in the grain boundaries" (p. 68).⁽¹³⁾ Although not specially related to this fourth test series, "...tests with the laser microbe showed no significant penetration of sodium into the grain boundaries" (p. 43).⁽¹⁰⁾ The type 304L SS samples used in this latter investigation had accumulated 1000 hr of exposure at 1200 °F.^(9,10) ANL also plans to furnish foils, exposed to various sodium environments, to Stanford Research Institute (SRI) for examination by electron microscopy.^(11,18) The SRI program is also discussed in the following section.

Finally, Idaho Chemical Processing Plant (ICPP) in-house corrosion tests are being conducted "...to determine if, after reactor exposure [EBR-II fuel] of the fuel elements to sodium, the corrosion rate of the stainless steel cladding is accelerated during storage in the water basin" (p. 56).⁽⁶⁾ To the best of my knowledge, no cladding penetration (as evidenced by the lack of significant release of fission products during leak test) has been observed since the start of the test in May or June, 1967.⁽⁶⁻¹⁴⁾

2. Other Sources of Information

Contact with the Defense Metals Information Center, Battelle-Columbus, revealed an Oak Ridge National Laboratory report that causes concern when water storage is considered.^(19,20) In this report it is stated that "The observations indicate that storage of sensitized, irradiated type 304 Stainless Steel in a high-humidity environment or under-water may lead to extensive intergranular corrosion."⁽²⁰⁾ The sensitization was the result of the in-reactor exposure (in a helium cooled loop) of cladding to temperatures of 1500 °F and other stainless steel components to temperatures ranging from 800 to 1200 °F. Literature review then revealed a second instance of attack where it was felt that the corrosive environment was water in which the components, Type 316 SS fuel element outer liners, were stored.⁽²¹⁾ No evidence of sensitization was found in this second instance, but, as stated in Reference 21, "failure mechanisms were influenced by the formation of a second phase, believed to be a sigma phase..." Prior to storage in water, these liners had been exposed to reactor coolant (nitrogen with 0.5% oxygen) for up to 2500 hr and 1200 °F.

Progress associated with the Stanford Research Institute (SRI) investigation, by electron microscopy, of interactions between 1200 °F sodium and Type 304 SS foils is reported in

Reference 18. It is concluded in Reference 18 that "The experimental technique of transmission electron microscopy in stainless steel foils after exposure to liquid sodium at 1200 °F has been successful in revealing several liquid-sodium, stainless steel reactions [grain boundary attack, formation of unidentified precipitates]."

Prior to this investigation, D. E. Bloomfield of FFTF had contacted several off-site persons concerning water storage of fuel elements and/or the possibility of attack of sensitized stainless steel. Letters received in response to these contacts are presented in Appendix A. Review of information arising from Mr. Bloomfield's contacts indicated that intergranular attack of stainless steels by water, with facilities to control pH and ion content, would be unexpected. No other potential compatibility problems, other than those described in the previous section on ANL literature, were suggested.

Information concerning storage concepts, penetration of stainless steel by sodium, and compatibility of stainless steel with potential storage media after sodium exposure was also requested from the Liquid Metals Information Center (LMIC). A portion of the excellent response by LMIC personnel is presented in Appendix B.

3. Remarks Concerning Reported Instances of Corrosion

It is well known that the exposure of stainless steel to certain media after carbide precipitation can result in intergranular corrosion. It must be remembered that tests using corrosive reagents, such as the ANL tests with Strauss reagent, are to indicate susceptibility. This does not mean that sensitization leads to intergranular attack in all active environments. Corrosives that are known to induce intergranular corrosion in austenitic stainless steel are listed on page 11 of

Reference 22. The list includes water + starch + sulfur dioxide and water + aluminum sulfate, but nothing appears that is representative of storage basin water. The applicability of ANL data arising from tests with Strauss reagent to FFTF spent fuel storage concepts is not totally clear. Much of the ANL work is still in progress. Continued review of literature, especially for results from tests such as the ICPP in-house corrosion test, or the "EBR-II Subassembly Recycle Test," is needed.

Telephone conversations with the authors of ORNL-3684 did nothing to resolve the concern arising from review of this document. The authors essentially reiterated what was published: "Admittedly, the evidence is fragmentary because it was obtained as a by-product of other experimentation, but the observations [severe intergranular attack] appear conclusive for the case of underwater storage...It is believed that these observations are of significant interest to reactor metallurgists and of sufficient importance to warrant a careful experimental verification." (20)

The second instance of attack reported in the previous section can be attributed to water contamination. Analyses of water revealed halide concentrations of 21 and 30 ppm [0.25 and 0.22 ppm chloride with no fluorine, bromine, or iodine breakdown], a content that "constitutes an environment which is potentially quite corrosive..." (21)

It is felt that the results of the SRI investigation using foils cannot yet be related to spent fuel storage concepts. It is stated in Reference 18 that, "While direct evidence of attack by the liquid sodium has been observed, additional studies will be necessary to fully determine the compatibility of 304 Stainless Steel with liquid sodium at 1200 °F with this new technique [electron microscopy]."

In a report of this type it is perhaps appropriate to note that some material was not included because of the unavailability of references to the reader. Reported instances of indications that some sodium can be retained by stainless steel after cleaning are known to the author (also see Appendix B). Review also revealed reported instances of corrosion of sensitized stainless steel upon exposure to the atmosphere. There is no intent to imply that all instances of attack of austenitic stainless steel (sensitized and unsensitized) by high purity water that are known to the author have been discussed.⁽²³⁾ These instances, arising from exposure to power reactor coolant environments and which suggest effects arising from radiation, were not mentioned simply because direct application to FFTF spent fuel storage plans is not totally clear. It is also perhaps appropriate to note that nothing was found to suggest that sensitization can reduce corrosion resistance to sodium.

Finally, review, admittedly speculative in nature, suggests other areas of concern. For example, reaction products arising from exposure of improperly cleaned or sodium impregnated components to moist air or water could possibly initiate attack. The assumption of stressed cladding resulting from fission gases and the formation of sodium hydroxide in surface microcracks immediately suggests the possibility of a caustic stress corrosion mechanism. Such attack could be both severe and insidious.

C. RECOMMENDATIONS

Instances of corrosion of sensitized stainless steel, attributed to underwater storage and storage in a high humidity environment have been discussed. Oil presents the potential problem of radiation and temperature degradation. In other words, literature review suggests the possibility of corrosion or compatibility problems with three of the

four candidate storage mediums. These possibilities have to then be weighed along with other considerations because conceptual design considerations obviously extend the bases for recommendations beyond corrosion problems. In other words, it becomes necessary to consider the fact that facilities must provide adequate heat removal, shielding and personnel protection, criticality safety, safety, reliability, and maintainability, and at the same time be compatible with auxiliary systems, e.g., the ventilation system.

When factors other than corrosion are discussed, review reveals that water has a combination of desirable properties. In general, water permits heat removal by natural convection and therefore has inherent reliability.* Guthrie reveals the adequacy of water-filled pools by the following: "The pool provides a high degree of shielding and convenience of fuel handling at relatively low cost."⁽¹⁾ Stader and Yevick state in Reference 2 that "The advantages of water are that it is transparent, is stable in high gamma-radiation fields, and can be cleaned of fission products by well-established ion-exchange techniques. Another advantage is that present fuel reprocessing plants are prepared to receive and unload fuel from water-filled shipping casks." Finally, as stated in Reference 1, "Because of the large heat capacity of the water pool, no high-pressure containment vessel [or perhaps confinement scheme] is required to reduce the effects of an accident in a fuel-storage pool to tolerable levels." The preceding combination of desirable properties and the general acceptance to date are the bases for the opinion that, in general, facilities for storing spent irradiated fuels should be water-filled.

* *It is assumed that there is prior, adequate decay storage in sodium to permit cooling by natural convection.*

1. Spent Driver Fuel Storage

One factor that will negate the advantages associated with the use of water for spent fuel storage is proof of the possibility of catastrophic cladding attack of fuel of the type proposed for initial core loadings (austenitic stainless steel clad fuel pins with oxide cores which are not sodium or NaK bonded). At this time it is felt that the unresolved instance of intergranular corrosion and other potential compatibility problems described previously are not this proof. One measure that should be taken in an attempt to protect against unexpected attack is control of water quality by ion exchange. Chloride concentration should be kept as low as practical since even low bulk concentrations (ppb range) can be concentrated by crevices.

2. Failed Fuel Storage

The methods used in the past in water pools to confine activity from ruptured fuel (sealed cans or storage in cans vented to the stack) appear appropriate.⁽¹⁾ Any storage facility will have to be designed to ensure appropriate containment; control of both the fission product content of the water and pool surface ventilation will be necessary. As previously indicated, radioactive material can be removed by ion exchange (additional methods are discussed in Chapter 7 of Reference 2). Water vapor should be compatible with the ventilation exhaust system.

3. Nonroutine and Future Fuels

For the types of nonroutine and future fuels considered, the major disadvantages associated with water is its well known reactivity with sodium and its incompatibility with carbides. As previously reported, it was stated in Reference 1 that "With the exception of the Enrico Fermi Fast

Breeder Reactor, the reactors that utilize fuel which is not metal clad or which is bonded with sodium do not store their fuel in direct contact with water." Except for vented fuels, which can be considered as leaky or failed fuel, methods that have been used to store "nonroutine and future" fuels were briefly described in Section III-A. In review, water is still frequently used to remove decay heat but, in general, cans or thimbles are used to prevent contact.

The ability to suspend thimbles in the water-filled pool(s) should be incorporated into conceptual design. The method of suspension would, of course, have to assure a fixed, critically safe array. The capability of installing and manipulating thimbles would not only permit flexibility in handling future fuels but also could possibly provide backup should concern over storage of spent driver fuel in direct contact with water prove valid. It is realized the use of cans, thimbles, or canisters could result in shielding and heat removal problems. Solids, including lead and aluminum-silicon alloy, have been considered or used as materials to facilitate heat transfer from cans. As stated in Reference 2, "At Dounreay spent fuel assemblies are sealed in lead-lined cans, and the lead is melted to provide a heat transfer medium." Facilities for some amount of decay storage in sodium inside the containment vessel could also assist in lessening heat transfer problems.

4. Specimen and Hardware Storage

Specimens and hardware that have been sensitized by high temperature sodium and then stored in water should not be reused in-reactor unless there is positive assurance that the storage resulted in no effects that could result in failure under in-core conditions. Obviously, the pool

is not the place to store components that could contaminate the water: e.g., nonmetallic gaskets that contain halides and improperly or partially cleaned hardware (sodium not totally cleaned from surfaces).

5. Experimental Program

The recommendation of using a water-filled pool assumes that it is necessary to select a storage medium now, so that conceptual design can proceed. The bases for this recommendation are the general acceptance of water and its combination of desirable properties. Experience with high burnup fuels and higher coolant temperatures, resulting in higher cladding stresses for nonvented fuel and metallurgical changes in the cladding, could yet reveal the inadequacy of these bases. The instance of extensive intergranular corrosion reported in ORNL-3864 remains unresolved.⁽²⁰⁾ It is therefore recommended that the effects of water storage on stressed sensitized cladding material with in-reactor exposure be investigated experimentally. Continued literature review is also needed.

It was also speculated that reaction products arising from exposure of improperly cleaned or sodium impregnated compounds to water could possibly initiate attack. As previously stated, the sodium penetration phenomenon is undefined, but instances of indications that sodium can be retained after cleaning are known to the author. Any experimental program should also be used to initiate the quantitative investigation of this phenomenon. It is anticipated that such an investigation may require exposure to high temperature sodium that has been contaminated with impurities such as carbon and oxygen followed by relatively long storage in air or water.

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APPENDIX A

LETTERS RESULTING FROM OFF-SITE CONTACTS

ARGONNE NATIONAL LABORATORY

February 19, 1968

Mr. Donald E. Bloomfield
Battelle Pacific Northwest Laboratory
P. O. Box 999
Richland, Washington 99352

Dear Don:

This will confirm our recent conversations concerning storage of FFTF fuel elements.

Provided the fuel elements are properly cleaned of sodium (e.g. by the method described in ANL-6687, p. 67 or ANL-6934, p. 13, or an equivalent) storage in demineralized water should have no deleterious effect on the integrity of the elements or their suitability as metallographic specimens in subsequent investigations. This conclusion applies equally to 304 and 316 stainless steel.

A program is underway at the EBR-II, which bears on some aspects of your questions. It is under the direction of Robert Neidner of the ANL Idaho Division. By copy of this letter, I am informing him of your interest in this matter. I am sure he will be willing to inform you of the results of the program.

I trust this information will be adequate for your needs. If there are other areas in which we may be helpful, feel free to call.

Yours truly,

Sherman Greenberg
Applied Corrosion Research
Metallurgy Division

SG:sw

CC: M. V. Nevitt
R. Neidner
P. G. Shewmon

ARGONNE NATIONAL LABORATORY
IDAHO FACILITIES

March 6, 1968

Don Bloomfield
Senior Development Engineer
Irradiated Fuel Handling Systems Group
FFTF Project - PNWL
P. O. Box 999
Richland, Washington 99352

Dear Mr. Bloomfield:

As you requested in our telephone conversation on March 5, 1968, I am summarizing my knowledge of the corrosion problems associated with sodium removal from 304 stainless steel using a water wash.

Because all of the EBR-II driver and experimental subassemblies are handled by the FCF and are normally subjected to a sodium removal procedure that involves a water wash, we have always had a vital concern as to the effects of the wash on the reactor components. A considerable amount of metallography has been performed on both irradiated and unirradiated sodium-exposed components. To date, no deleterious effects have been observed that are traceable to the washing procedures.

Our interest in this problem is a continuous one, and at the present time, two experiments are being conducted to further evaluate the problem. One of these experiments (being conducted by R. Neidner, ANL Idaho) involves recycling pressurized capsules from the primary reactor tank through the sodium removal step and back into the tank. To date, three such cycles have been completed and the experiment is ready for the final step, which involves putting the experimental subassembly into the reactor grid and irradiating the capsules, then performing a final sodium removal step before examining the capsules. Capsules from this experiment are examined metallographically after each cycle, and no deleterious effects have been observed. If you would like more information regarding this experiment, I suggest you contact Bob Neidner. The second experiment involves recycling a standard EBR-II driver subassembly that has reached terminal burnup (1.2 a/o) between the FCF and EBR-II. This experiment is about 30% complete.

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Sherm Greenberg of the Metallurgy Division at ANL-Illinois, has followed this compatibility problem very closely. In particular, he is familiar with Fred Smith's work on the program that Fred conducted at ANL-Illinois. Smith reported some rather drastic effects on 300 series stainless steel exposed to 1200° F sodium and then allowed to set in 180° F distilled water or under the atmospheric conditions at Illinois. Either Smith or Greenberg could best summarize this work for you, but I believe the effects that Smith observed were attributed to his particular set of experimental conditions and have not yet been duplicated anywhere else.

The General Electric Company, at ANL's request, has examined some of their tubing from capsules irradiated in EBR-II and subjected to the standard FCF sodium removal procedure. They reported no unusual conditions.

I hope that this will at least aid you in your quest for information. We would be most interested in any conclusions you reach and if we can be of any further help, please let us know.

Yours truly,

V. G. Eschen

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March 6, 1968

Mr. Donald E. Bloomfield
Battelle Northwest Laboratories
P. O. Box 99939
Richland, Washington 99352

Subject: Corrosion of Type 316 Stainless Steel

Dear Mr. Bloomfield:

This letter summarizes our two telephone calls relative to the corrosion of type 316 stainless steel in water. As I understand the question, you are interested in corrosion during underwater storage of two types of fuel elements - one type is a type 316 sheathed sodium cooled element which has been subjected to a temperature of approximately 1200°F in service; the 316 cladding on the other type of fuel elements has not been sensitized in operation. You indicated that the water system could be designed to control the temperature, pH, and chloride ion content. It was not clear what temperature the cladding will be due to decay heat from the fuel. Apparently, there is also sodium on the outer surface of some of the elements.

This type of question is somewhat difficult to answer without personal contact as I may not know all of the variables involved. The following comments are offered based on my understanding of the question:

1. I do not have experience with sodium in contact with stainless steel so I cannot advise you on this phase of the work.
2. We do have experience with austenitic stainless steel in

Mr. Donald E. Bloomfield

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water environment. There are three basic mechanisms of corrosion: Pitting, stress corrosion, and weld decay due to sensitization. Dealing with these in order, we operate stainless steel systems and components in de-ionized water without much concern for pit type corrosion provided the pieces are not stacked or in close contact to form a tight crevice.

We carefully consider the problem of stress corrosion in each stainless application as this type of attack can be severe under certain conditions. In this regard we base our evaluation on previous experience, reports from the Corporation, and the numerous technical articles and books on the subject. We have had an excellent plant experience in this regard primarily because our temperatures are usually below the threshold temperature and we carefully control the chloride ion concentration in the water. As you know, stress corrosion cracking of stainless steel is affected by chloride ion concentration, pH, temperature, tensile stresses, and possibly oxygen. High chloride concentrations, and high temperature with low pH's are the worst offenders. It would appear that for your situation, the variable may be the temperature of the stainless cladding. Fontana has published a curve (shown below) which we have found beneficial. Others have set the threshold at about 212°F, still others at 175°F. If the cladding temperature is below the threshold temperature, stress corrosion cracking will not occur.

The Eight Forms of Corrosion 101

Fig. 3-64. Effect of temperature on time for crack initiation in types 316 and 347 stainless steels in water containing 875 ppm NaCl. [W. W. Kirk, F. H. Beck, M. G. Fontana, Stress Corrosion Cracking of Austenitic Stainless Steels in High Temperature Chloride Waters, in T. Rhodin (ed.), "Physical Metallurgy of Stress Corrosion Fracture," Interscience Publishers, Inc., New York, 1959.]

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One example of this is the High Flux Isotope Reactor. We use deionized water, control the chloride ion to low limits, operate at 165°F maximum, and experience no stress corrosion of stainless components. We ran no R and D corrosion studies as accelerated corrosion tests for stress corrosion are of limited value. We did however, perform extensive corrosion tests on bimetal samples and have surveillance samples in the reactor to evaluate pitting of combinations of stainless to aluminum, aluminum to beryllium, etc. For your information I have attached a brief write-up of the water purification system for HFIR.

Turning to the third type of corrosion, weld decay, we use "L" grades or stabilized stainless steels to prevent this type of problem. The added cost of material is justified by the freedom available during heat treatment for dimensional stability and to permit the use of strong acids such as HNO₃ for decontamination should this become necessary. In your case, you have no choice of material, however, I would not expect you to have a problem in water with sensitized 316. I have attached a copy of an internal document on this subject for your consideration.

Yours very truly,

J. R. McGuffey

JRM:as

Enclosures

cc: E. C. Miller
File

APPENDIX B

LMIC RESPONSE TO REQUEST FOR INFORMATION

NOTE: A small portion of the following letter has been deleted because it contained information extracted from reports which had distribution limitations.

AEC offices and contractors can obtain the deleted portion by contacting the Liquid Metals Information Center, P. O. Box 1449, Canoga Park, California 91304.

Liquid Metal Engineering Center

*Operated for
the U.S. Atomic Energy Commission by Atomics International*

A Division of North American Aviation, Inc.

June 17, 1968

*In reply refer
68LMEC-359*

Mr. W. K. Winegardner
Corrosion Research Unit
Surface and Coolant Chemistry Section
Chemistry and Chemical Engineering Department
Pacific Northwest Laboratory
P.O. Box 999
Richland, Washington 99352

Dear Mr. Winegardner:

We are pleased to respond to your request for information on three subject categories as follows:

- (1) Storage concepts for irradiated fuel
- (2) Penetration of stainless steel by sodium
- (3) Compatibility of stainless steel cladding that has been exposed to high temperature sodium or NaK (either in a reactor or a loop) with water, air, inert gas or oil.

An excellent section on storage of irradiated fuel is contained in Chapter 7 of a book edited by J. G. Yevick, "Fast Reactor Technology Plant Design," published by the M.I.T. Press, Cambridge, Massachusetts (1966). A copy of this section is attached. We are also enclosing copies of pertinent sections on storage from five other reports listed below:

1. NAA-SR-9516 Rev., "Sodium Reactor Experiment Power Expansion Program Reactor Safety Analysis Report" (November 2, 1964)
2. NAA-SR-5700, "Final Summary Safeguards Report for the Hallam Nuclear Power Facility" (April 15, 1961)
3. ANL-7168 (Volume II), "Fast Reactor Test Facility (Faret) Volume II - Summary of Preliminary Safety Analysis (April 1966)
4. NP-10458, "Enrico Fermi Atomic Power Plant, Part B, Revised License Application AEC Docket No. F-16 Proposed Technical Specifications" (November 1962)

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Pacific Northwest Laboratory

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5. Proceedings of the Ninth Conference on Hot Laboratories and Equipment, Chicago, Illinois, November 7-9, 1961, pp 295-302, "Steam Cleaning System Liquid Metal Cooled Fuel Elements," by H. L. Brinkman and W. Wilson.

It should be realized that information on storage of irradiated fuel, per se, is outside the scope of our Center and would not be processed for storage in our data bank unless it appeared in a document containing information on sodium or NaK technology.

In regard to penetration of stainless steel by sodium, a report by A. Brasunas "Interim Report on Static Liquid-Metal Corrosion," ORNL-1647 (May 1954), provides some indication that sodium may penetrate Type 304 stainless steel but only to a shallow depth. The test specimens were exposed to sodium for 400 hours at 1000C (1832F). Voids in the steel, in the grains as well as in the grain boundaries, appeared close to the sodium exposed surface. Most of the voids were in the region extending about 0.001 inch from the surface. The author stated that these voids had previously contained either a brittle, a water soluble, or a low-melting phase which was removed in metallographic preparation; he did not, however, exclude the possibility of monodirectional diffusion.

V. S. Lyashenko et al., "Resistance to Corrosion of Austenitic and Ferrite-Pearlitic Steels in a Stream of Liquid Sodium at Temperatures of 600 and 700C," AEC-TR-5409 (June 1962), state that the corrosion of the austenitic stainless steels tested was characterized by transcrystalline corrosion and disintegration of grains in addition to intercrystallite effects. Attached are copies of tables in the report showing (1) the depth of corrosion in austenitic stainless steel specimens exposed to 600C sodium (oxygen, 0.02%) for 5500 hours, and (2) the composition of the austenitic stainless steel test alloys. The maximum corrosion depth observed was 150 microns or about 0.006 inch.

Mine Safety Appliance Research Corporation has conducted an extensive program to determine mechanical properties of stainless steel in sodium. Data on tensile strength, yield strength, creep strength and creep-rupture strength of Type 316 stainless steel in 1200F sodium and in 1200F helium (for comparison) are reported by R. C. Andrews and L. H. Kirschler in Topical Report No. 7, "Design Properties of One Heat of 2-1/4 Cr-1 Mo Steel and 316 Stainless Steel in High Temperature Air, Helium and Sodium Environments," MSAR-66-174 (September 1966). No appreciable difference was observed for each of the above-mentioned mechanical properties of 316 stainless steel exposed at 1200F to (1) clean sodium (10 ppm oxygen), (2) high oxygen sodium (200 ppm oxygen), and (3) helium. Exposure time for each test was 4000 hours. In AEC-TR-5409 (referenced above), Lyashenko et al. found that the tensile strength and relative elongation of the austenitic steels tested did not undergo any significant changes following 5500 hours exposure to 600C sodium. Thus, if any penetration of sodium

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into stainless steel does indeed occur, it does not appear to have any significant effect on the mechanical properties of the stainless steel.

You are probably familiar with ANL tests, reported in ANL-7302 (January 1967) and later progress reports, on penetration of 304 stainless steel tube by liquid sodium. In ANL-7302 the following statement is made:

"Sodium penetration tests were conducted by exposing 304 stainless steel tube and rod samples to 1200F for relatively brief times, cleaning the sample surface, etching it chemically, and observing the effects either metallographically or chemically. To isolate the effects of high temperature from those of sodium, control samples were exposed to vacuum at the same 1200F temperature. The various tests on both tube and rods show chemically and metallographically that sodium penetrates 0.006-0.010 in. into the grain boundaries within only 7 days. When the 0.018-in.-wall tube was etched, the sodium penetration led to its disintegration. There were inconclusive chemical indications that sodium penetrated as deep as 0.036 in. into a 5/8-in.-dia rod within 35.7 days. In either case, etchant reacts more strongly with samples that had been exposed to 1200F sodium than with samples pretreated in a 1200F vacuum. The longer the 304 stainless steel is exposed to 1200F sodium, the more reactive the surface is with aluminum-sulphate etchant."

In "Sodium Component Development Program Mass Transfer Investigations in Liquid Metal Systems," GEAP-5546, Quarterly Progress Report No. 2 for June-August 1967, R. S. Young and E. L. Zebroski stated that they had observed apparent exudation of sodium from samples of Type 316 stainless steel which had been exposed in a sodium loop at temperatures above 1000F for about 2100 hours. The samples were cleaned by washing with hot ethyl alcohol followed by washings in distilled water and acetone. After storage for 5 to 6 years, the samples were again examined and some were found to be covered with a white material. X-ray diffraction analysis indicated that the white substance was sodium sesquicarbonate. Attempts to speed up exudation from these samples, by heating to around 300F, were unsuccessful. The conclusion was drawn that the bulk of the sodium retained in Type 316 stainless steel after cleaning was concentrated in a region extending 1.5 mils deep from the surface.

Tests were also conducted at General Electric prompted by the results of Strauss tests performed at ANL on Type 304 stainless steels. The specimen exposed to carbon-contaminated sodium disintegrated within

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Pacific Northwest Laboratory

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10 minutes after the Strauss solution started boiling. The second test consisted of exposing a series of alloys, previously exposed to carbon contaminated sodium at 1050F in the Strauss solution for 5 minutes. Immediately after dipping the specimens in the hot solution, violent reaction occurred which copper coated them in a matter of seconds. A third test contained three Type 316 stainless steel tubes exposed to 1300F sodium (cold-trapped) for 2085 hours. After 24 hours in the Strauss solution, the specimens had 1.5 mils and 0.5 mil surface intergranular attack. The authors concluded that the degree of reaction of stainless steel in a Strauss solution was dependent upon the extent of carburization of the material surfaces. Their tests indicated no effect of sodium (cold-trapped) exposure on the susceptibility to intergranular attack of austenitic stainless steels in the Strauss solution. If the sodium was carbon contaminated, carburization of the stainless steel occurred, which resulted in an increase in carbide precipitation, and, subsequently, intergranular corrosion. If the bulk carbon buildup was essentially constant throughout the cross section of the material, as in Types 304 and 347 stainless steel, rapid attack throughout the material occurred. However, if the bulk carbon was the result of a "moving front," as in Type 316 stainless steel and Incoloy 800, the attack followed this front.

Stanford Research Institute personnel also have been investigating the liquid sodium penetration of grain boundaries in polycrystalline Type 304 stainless steel. Transmission electron microscopy was utilized in an attempt to distinguish between the various bulk, grain boundary, grain-boundary "tunnel," and surface diffusion processes. The results of this work are reported in SRI-Project PMU-6694-F, "Interaction of Liquid Sodium With 304 Stainless Steel," by J. W. Moberly et al., January 31, 1968. A copy of the conclusions from this report is enclosed.

In regard to your third question Yevick (referenced in paragraph 2) states that gases which have been considered for decay-heat removal of irradiated fuel range from inert gases for uncleaned fuel to air for cleaned fuel. The decay cooling of EBR-I fuel rods is an example of air cooling. Liquids considered for decay-heat removal include water, organics such as oils and Dowtherms, and liquid metals such as sodium and NaK.

Water has seen considerable use (Fermi, Hallam, etc.). The advantages of water are that it is transparent, is stable in high gamma-radiation fields, and can be cleaned by well-established ion-exchange techniques. Another advantage is that present fuel reprocessing plants are prepared to receive and unload fuel from water-filled shipping casks. The use of a different decay coolant would require a transition of

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coolants either at the reactor site or at the reprocessing plant. Organics present the potential problem of decomposition under high-temperature and gamma-radiation conditions.

In the austenitic solid-solution form, stainless steels are resistant to a large number of aqueous corroding media, although they are by no means uncorrodible, especially where the medium contains nitric acid together with chlorides or hydrochloric acid. When the 18-8 grade alloys are heated within or slowly cooled through the temperature range of 800-1500F, they undergo a structural change commonly referred to as carbide precipitation, which affects their subsequent resistance to corrosion. The precipitation of grain-boundary carbides causes the alloys to become sensitized and to lose a large part of their corrosion resistance by becoming extremely susceptible to grain-boundary attack. Although there have been no known instances where sensitized stainless steel has failed due to sodium attack, appreciable concern has been registered concerning stainless steel in the sensitized condition coming in contact with aqueous reagents. This concern arises from observations of failures of sensitized stainless steel components exposed to air and caused by the moisture content of the air. An excellent example of this type of failure is reported by J. A. Leppard in "Review of SCTI Bellows Failures," NAA-SR-MEMO-11970 (May 1966).

In a recently-issued report, "Long-Term Radiological Hazards Associated with Storage of Radioactive Material in HNPF Fuel and Storage Pit No. 3 Thimbles," AI-AEC-Memo-12664 (April 1968), W. B. Sayer states that the corrosion rate of Type 304 Stainless Steel is less than 0.001 in./yr in groundwaters similar in hardness and chloride content to that found at the HNPF. For a 0.012 inch thick fuel cladding, a corrosion rate of about 0.001 in./yr could be considered rapid.

We are happy to have provided you with the information you requested and trust that this information will aid you in accomplishing your immediate objectives. In return, we would like to enlist your support

Mr. W. K. Winegardner
Pacific Northwest Laboratory

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in helping the Liquid Metals Information Center attain the broadest possible coverage of topics falling within our purview. First, if you find any significant omissions in the list of pertinent references would you please notify us. Second, should your particular effort result in any publications related to liquid metal technology, and which are available to the Commission or its contractors, would you send a copy to the Liquid Metals Information Center. Kindly notify us before forwarding if there is a charge involved. Your cooperation in this matter will help us in providing the best service possible to our users and would be greatly appreciated. We are pleased to have assisted you and look forward to serving you again. To this end a brochure describing in some detail the services offered by our Center is also enclosed.

Sincerely yours,

R. A. Winborne

R. A. Winborne
Liquid Metals Information Center

J. J. Droher

J. J. Droher, Supervisor
Liquid Metals Information Center

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