

## CORROSION OF RESEARCH REACTOR AL-CLAD SPENT FUEL IN WATER

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### SUMMARY

A significant amount of aluminium-clad spent nuclear fuel from research and test reactors worldwide is currently being stored in water-filled basins while awaiting final disposition. As a result of corrosion issues, which developed from the long-term wet storage of aluminium-clad fuel, the International Atomic Energy Agency (IAEA) implemented a Co-ordinated Research Project (CRP) in 1996 on the "Corrosion of Research Reactor Aluminium-Clad Spent Fuel in Water." The investigations undertaken during the CRP involved ten institutes in nine different countries. The IAEA furnished corrosion surveillance racks with aluminium alloys generally used in the manufacture of the nuclear fuel cladding. The individual countries supplemented these racks with additional racks and coupons specific to materials in their storage basins. The racks were immersed in late 1996 in the storage basins with a wide range of water parameters, and the corrosion was monitored at periodic intervals. Results of these early observations were reported after 18 months at the second research co-ordination meeting (RCM) in Sao Paulo, Brazil. Pitting and crevice corrosion were the main forms of corrosion observed. Corrosion caused by deposition of iron and other particles on the coupon surfaces was also observed. Galvanic corrosion of stainless steel/aluminium coupled coupons and pitting corrosion caused by particle deposition was observed. Additional corrosion racks were provided to the CRP participants at the second RCM and were immersed in the individual basins by mid-1998. As in the first set of tests, water quality proved to be the key factor in controlling corrosion. The results from the second set of tests were presented at the third and final RCM held in Bangkok, Thailand in October 2000. An IAEA document giving details about this CRP and other guidelines for spent fuel storage is in pres. This paper presents some details about the CRP and the basis for its extension.

### IAEA CO-ORDINATED RESEARCH PROJECT

#### Background

In December 1994, a meeting of corrosion experts was held at IAEA headquarters in Vienna, as part of an ongoing CRP entitled: "Irradiation Enhanced Degradation of Materials in Spent Fuel Storage facilities". In this meeting, discussions were held with the IAEA and the European participants on spent fuel corrosion issues at SRS and other sites in the United States. As a result of this meeting and recommendations by an advisory group, the IAEA established a new Co-ordinated Research Project on "Corrosion of Research Reactor Aluminium-Clad Spent Fuel in

Water". The new CRP was designed to address several issues presented by vulnerability assessments conducted at some of the spent fuel storage sites. The objectives of the CRP were to:

- a) establish uniform practices for corrosion monitoring and surveillance;
- b) provide a technical basis for continued wet storage of research reactor spent fuel;
- c) collect data to help in the prediction of lifetimes of fuel handling tools and storage racks;
- d) establish a uniform basis for the characterization of water in fuel storage basins

Nine countries: Argentina, Brazil, China, Hungary, India, Pakistan, Russia (two different sites) United States, and Thailand were invited by the IAEA to participate in the CRP. Research agreements or contracts with institutes in these countries were put in place for work to be performed and the IAEA provided a detailed work package and standard corrosion test coupons to each participant.

The CRP was based on a corrosion surveillance programme developed and planned for several DOE spent fuel storage basins in the United States. With a limited budget, the IAEA version of the corrosion monitoring programme was smaller, and scaled down in the number of racks and coupons. The programme, however, was designed to develop basic information on corrosion of aluminium-clad alloys in spent fuel storage environments.

### **Details of the corrosion monitoring programme**

#### Racks, Coupons and Test Protocol

Two sets of racks with test coupons were prepared and distributed to the participants of this CRP at the: (a) 1<sup>st</sup> Research Coordination Meeting (RCM) held in Budapest, Hungary, in 1996 and (b) the 2<sup>nd</sup> RCM held in São Paulo, Brazil in 1998.

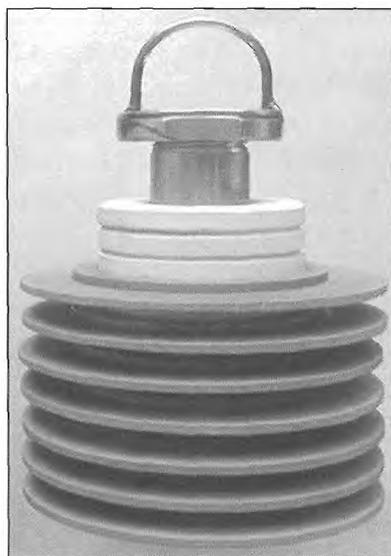


Fig.1. Photograph of Batch-I rack

The specimen stacking sequence was as follows, from top to bottom in racks of Batch-I (Fig 1.): AA1100; AA1100; AA6061; AA6061; SZAV-1; SZAV-1; AA6063-AA6063; AA6063-AA6063; SS316-AA6063; SS316-AA606.

The participants were instructed to expose these racks at representative spent fuel storage basins. They were also encouraged to either add coupons made of site-specific alloys to these CRP racks or to fabricate similar racks with site-specific alloy coupons. None of the participants added site-specific alloy coupons to the racks of Batch-I. China however prepared and exposed a separate rack with Chinese alloy coupons. This rack was exposed prior to the 2<sup>nd</sup> RCM. The Chinese rack, shown in Fig. 2, included coupons of alloys 305 Al, LT 24L and stainless steel 304-8K. Brazil manufactured a separate rack (IPEN rack and shown in Fig.3) This included a large number of coupons in different configurations, to evaluate the effect of fuel plate processing conditions on pitting, crevice and galvanic corrosion of Brazilian alloys (AA 1060, AA 6061, AA 6262), used in the manufacture of fuel elements for their IEA-R1 Research Reactor. The processing conditions included pre-oxidation and scratching of pre-oxidized coupons to simulate damaged fuel plate surfaces.



Fig. 2. Chinese rack of coupons

The number of racks distributed to each participant was limited, (just one or two). This precluded the removal of one rack after increasing periods of exposure. Consequently, the participants were asked to remove the rack from the basin water, inspect and photograph the coupons, reassemble the rack and return the rack to its original position in the storage basin, all this while preventing, if possible, the coupons from drying out.

The different participants adopted different procedures for interim inspection. In many cases invalidating their results. To avoid this in subsequent tests and to obtain more meaningful information, a test protocol was elaborated at the 2<sup>nd</sup> RCM.

Batch-II racks were also manufactured by KFKI from the original batch of alloys and these were handed out at the 2<sup>nd</sup> RCM. The procedure adopted for preparing the coupons of this batch was

slightly different. The surfaces of the coupons were mechanically polished as opposed to plain machine finishing (adopted for coupons of Batch-I). The racks of Batch-II (Fig. 4) did not contain the glass ampoules with the tiny tensile test type coupons and the numbers of coupons in each rack of Batch-II were fewer and arranged in the following order from top to bottom: SS 316 – AA 6063; SS 316 – AA 6061; AA 6063 – AA 6063; AA 6061 – AA 6061; AA 1100 – AA 1100.

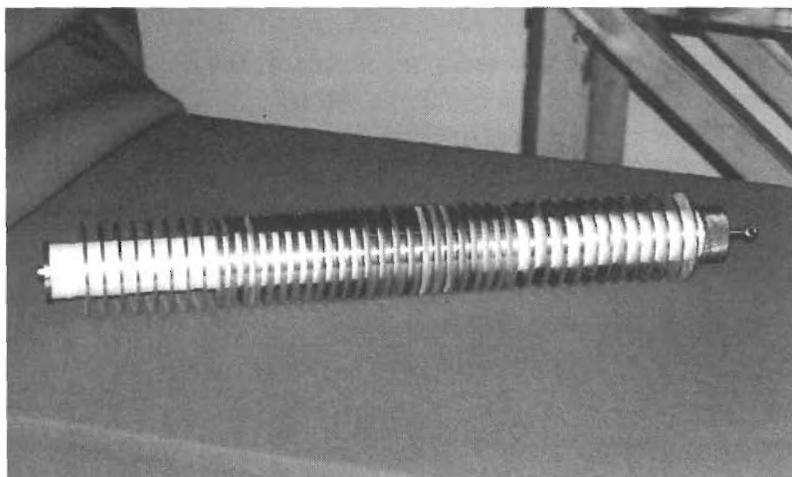


Fig. 3. The IPEN rack of test coupons

The AA 6063 coupons of this batch were passivated in water at 95°C for 24 hours and scratched; to simulate the effect of damaged surface oxide during handling. Two or more racks were handed out to the different participants. The racks were to be removed after one and two years of exposure in the storage basins.

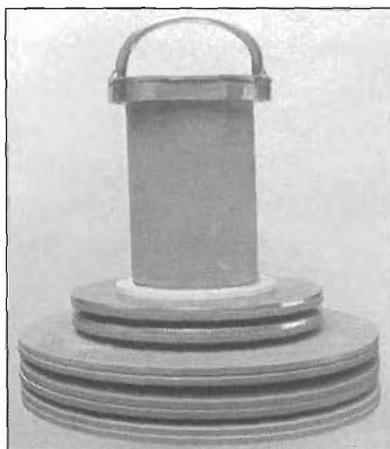


Fig. 4. Photograph of Batch-II rack.

The Argentine experience included the exposure of 6 racks at various storage sites. Four of the six racks (from Batch-II) included some Argentinean alloy coupons and two of the racks contained only Argentinean coupons.

In order to maximize the amount of information that could be obtained with the limited number of Batch-II racks handed out to each participant, interim inspections of the rack and the specimen was recommended, especially without disassembly of the racks and without allowing the racks to dry out. All these instructions constituted the Test Protocol, which is in Appendix 1. Guidelines for specimen evaluation also formed part of this Test Protocol.

## Results

At the second RCM in Sao Paulo, Brazil, results of tests carried out with IAEA Batch-I racks were presented. The third and final RCM of the CRP was held in Bangkok, Thailand in October 2000 and the results of all the tests carried out in the different participating countries were presented. A brief summary of the results presented at the two meetings is given below.

### CNEA-CAC, Buenos Aires, Argentina

Rack-1 was immersed in one of the open channels at the Ezeiza Fuel Cemetery (EFC), about one meter from a spent fuel assembly and the two are interconnected. The rack was inspected after 60 days of exposure. The surface was coated with a thin brown layer along with some dark particles. Corrosion products were visible on a number of coupons. Several white nodules were observed and when cleaned, revealed pits in the base metal. Pitting was associated with the particles. Crevice coupons were stuck together and were hard to separate because of corrosion. The SZAV-1 aluminium alloy showed a reduced tendency for corrosion and the 6061 aluminium/stainless steel galvanic couple was highly corroded. The channel water with a conductivity of 74  $\mu\text{S}/\text{cm}$  and chloride ion content of 14.8 ppm. was aggressive for storing aluminium-clad alloys

In mid-1998, six additional racks containing crevice and galvanic couple coupons were immersed at EFC, the RA3 decay pool, the RA6 reactor pool, and the RA6 decay pool. These pools provided a wide range of water chemistry conditions. Figs. 5 and 6 show variations in chemical parameters of the EFC and RA3 decay pool waters with time. The conductivity ranged from 227  $\mu\text{S}/\text{cm}$  in the EFC down to 1.8  $\mu\text{S}/\text{cm}$  in the RA6 reactor pool. The chloride ion content ranged from 16 ppm in the EFC to less than 0.5 ppm in the reactor pool. The racks immersed in the different basins contained coupons provided by the IAEA and aluminium alloy coupons used in the manufacture of Argentine fuel. Racks were immersed in the basins in mid-1998. Some were removed and examined in August 1999 and others in February, July, and September 2000. Severe pitting and crevice corrosion was noted on most of the aluminium coupons in the EFC, where the water was most aggressive. Much of the pitting on the external surfaces was caused by deposition of what was thought to be iron oxide particles from the corrosion of the carbon steel cover plates. Fig. 7 shows a coupon surface with crevice corrosion products and Figs. 8-12 the surfaces and cross-sections of different pitted Al coupons

Some pitting was noted even in the high purity water of the RA6 reactor pool. This pitting was always associated with deposited particles. Pits, 1-2 mm in diameter were produced in waters with no detectable amounts of chloride or sulphate and with conductivity less than 2  $\mu\text{S}/\text{cm}$ . The particles were thought to be iron oxide and were cathodic to the aluminium. Conditions inside some of the crevices resulted in oxide patches. Other areas seemed pickled, possibly by acidification of the water within the crevices. Small pits (0.1 mm in diameter) were found inside these crevices. Results of these investigations showed that the basin storage environment contained floating particles and it was not always possible to associate the degree of pitting with water composition.

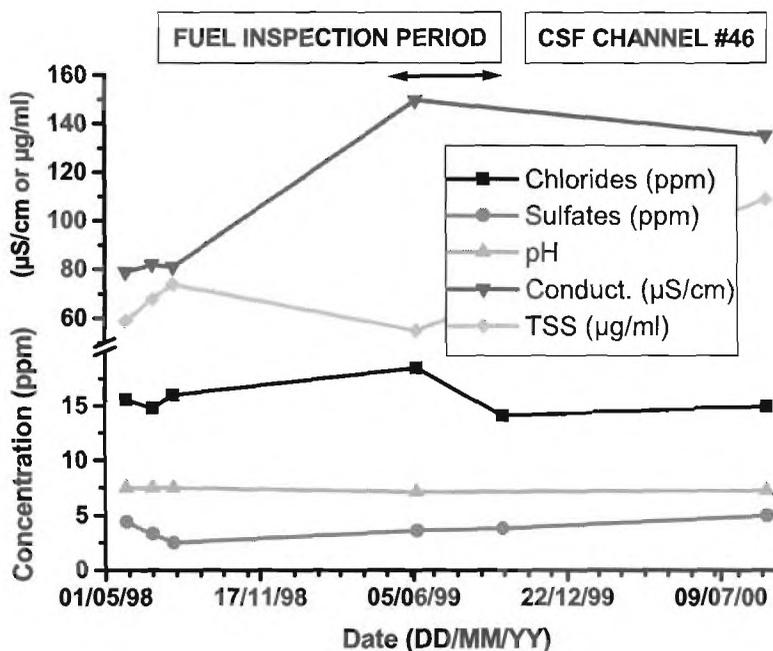


Fig. 5: Variation in water parameters in CSF #46

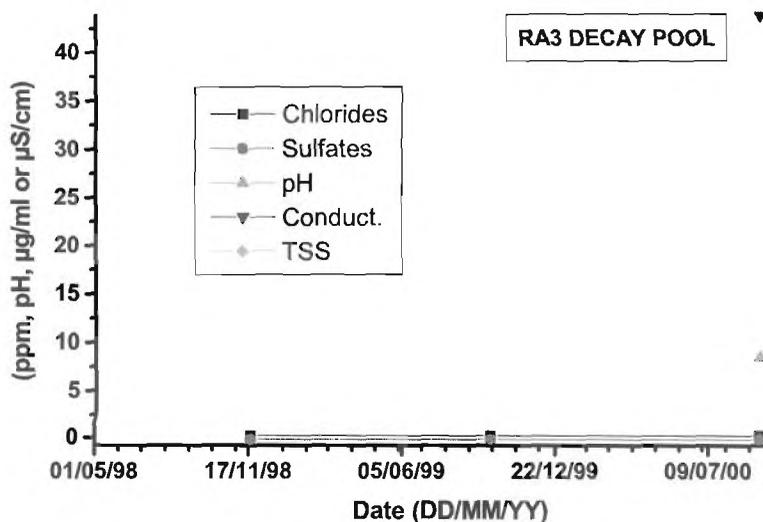


Fig. 6: Variation in water parameters in RA3 DP

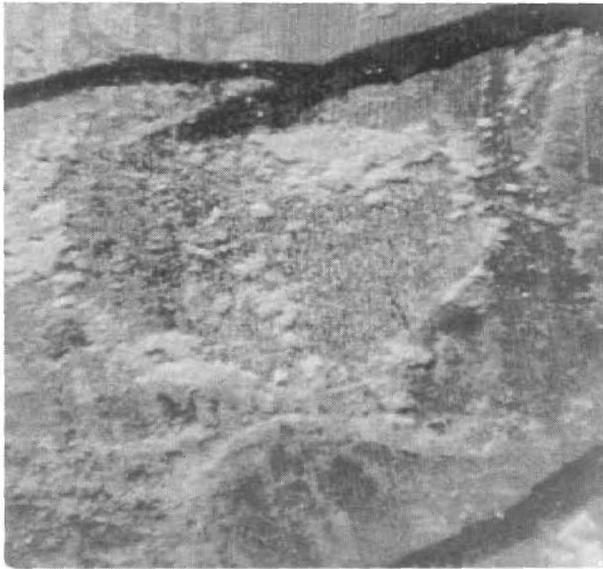


Fig. 7: Coupon 6064/64; crevice corrosion (18X)

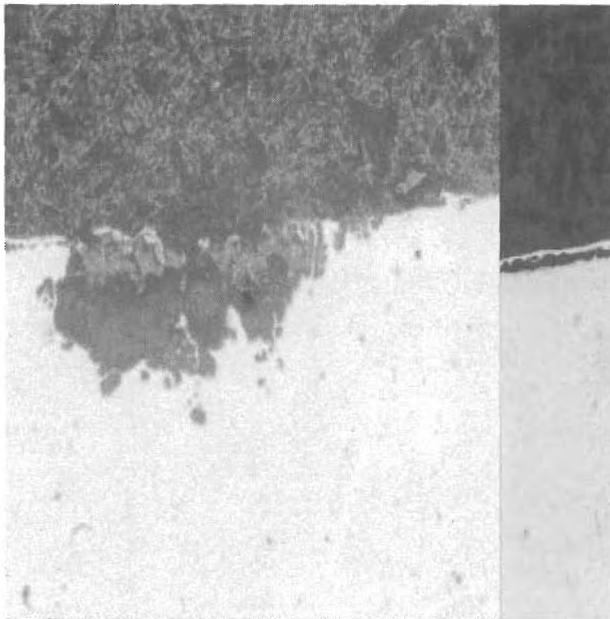


Fig. 8 Cross-section of an isolated pit 50X



Fig. 9: Uneven oxidation (x 1)

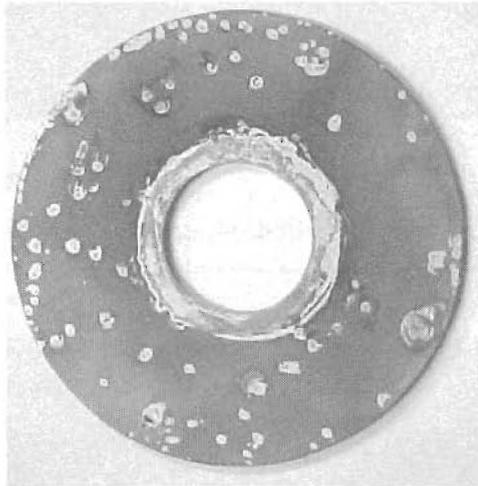


Fig. 10: Pitted surface (x 1)

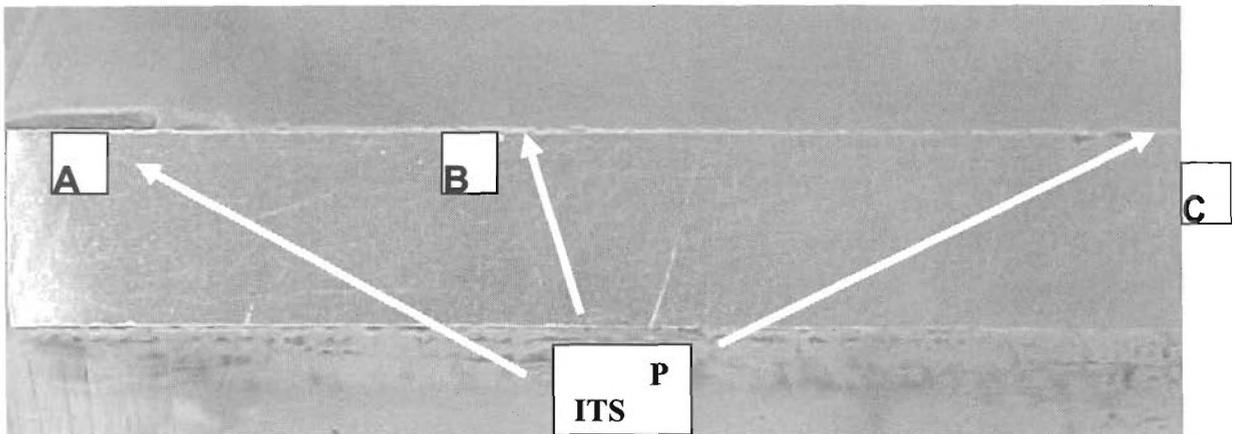


Fig. 11: Cross section of coupon revealing pits A, B and C. Coupon thickness: 3 mm

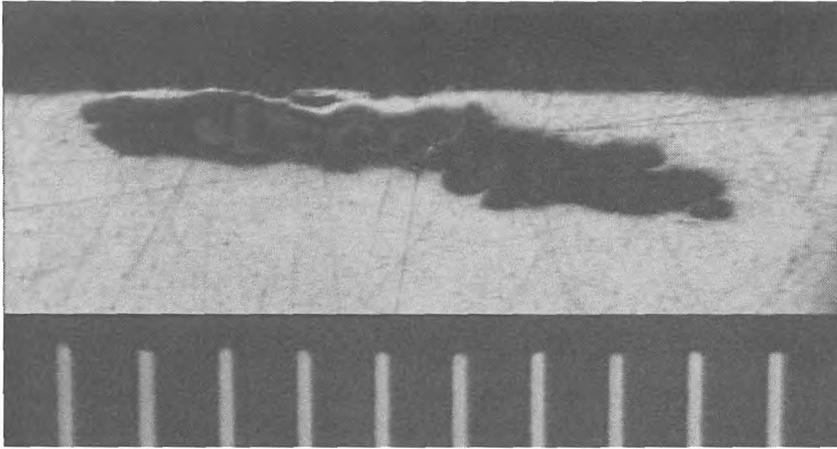


Fig. 12: Pit A from Fig.11 at higher magnification. One division equals 100  $\mu\text{m}$

Instituto de Pesquisas Energeticas e Nucleares (IPEN), Sao Paulo, Brazil

The IAEA coupon rack was immersed in September 1996. Water conductivity was maintained at  $<2.0 \mu\text{S /cm}$  and the pH in the range 5.5-6.5. The chloride ion concentration was  $<0.2 \text{ ppm}$  during the exposure period. No corrosion spots were observed on the coupons.

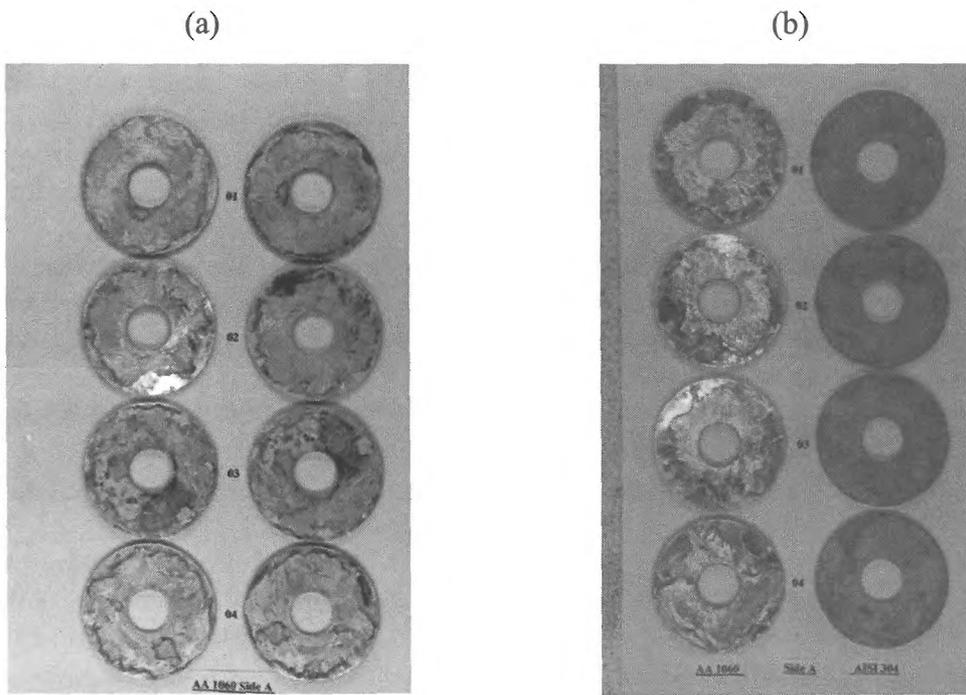


Fig. 13. Surface features on the facing surfaces of AA 1060/1060 crevice couple coupons and AA 1060/ AISI 304 galvanic couple coupons

The first inspection of the IAEA racks was conducted after about 6 months. All three galvanic and crevice coupons were stuck together, requiring forcible separation. The aluminium coupons were coated with a white/grey deposit typical of aluminium oxide. The pH was measured inside the crevice and it ranged between 4.0-4.5 (acidic). The glass ampoules had a brownish tint, indicative of radiation damage. After these observations, the coupons were re-assembled and returned to the basin for continued exposure. After an additional year of immersion, the rack was taken out of the water at a demonstration during the Sao Paulo RCM. It was disassembled and examined visually. The white oxide was observed in the crevice. In addition, small metallic particles were imbedded on the surface of some of the aluminium coupons. The particles were believed to be cathodic, possibly iron, as a halo existed around the particle/pit. This halo was bright and appeared to be free of corrosion. IAEA Batch-II racks 2 and 3 were immersed in the basin in August 1998 and were removed in October 1999. Examination of the coupons, revealed a few small pits within the crevices of the galvanic coupons and more pits at the contact points with the ceramic separators. Some loose deposits were seen on the top surfaces of the coupons. The top surfaces of the AA 1100/1100 couples had similar deposits and more than 50 pits < 1 mm in diameter. The bottom surfaces were free of pits.

An IPEN rack containing Al 1060, Al 6061, and Al 6262 alloys, used in Brazilian fuel element manufacture, was also immersed in the basin. After 16 months exposure, observations showed that some pitting occurred on the uncoupled coupons, mostly on the top surfaces. The aluminium couples were stained inside the crevices, but not pitted as shown in Fig.13a. The stainless steel/aluminium galvanic coupons were much more severely corroded ( Fig.13b). Additional laboratory tests were conducted to determine whether increased levels of silver (Ag) in the basin water could have increased the corrosion of the aluminium cladding in the IPEN basin. Results indicated no pitting, but an increase in darkness of the surface oxide colour with increase in silver concentration.

#### China Institute of Atomic Energy, Beijing, China.

Five racks were immersed in the spent fuel storage pool, and included the rack given by the IAEA in 1996. The basin water was not constantly circulated and it was purified once a year using an ion exchange system. The conductivity of the basin water ranged from 3-10  $\mu\text{S}/\text{cm}$  and the chloride ion content <0.1 ppm. A dark grey oxide layer, from general oxidation, developed on most of the outer surfaces of the coupons in contact with the water. The colour darkened and the layer thickened with time. Crevice corrosion products formed in the crevices between coupons. No pitting was observed on coupon surfaces, but some pitting occurred on the outer rim of the coupons, due to edge effect.(Fig.14)

#### KFKI Atomic Energy Research Institute, Budapest, Hungary

The results obtained from evaluation of Rack-1 coupons indicated that there were differences in corrosion resistance among the different aluminium alloys. Corrosion of these alloys in the water at the KFKI spent fuel storage pool was found to be more dependent on the materials than on the duration of exposure to the pool water. They concluded that a duration of 1 to 2 years in high quality water was probably insufficient to observe major differences in corrosion behaviour. The results obtained from Rack-2 and Rack-3 revealed no significant differences between the coupons immersed for one and two years in the pool water.

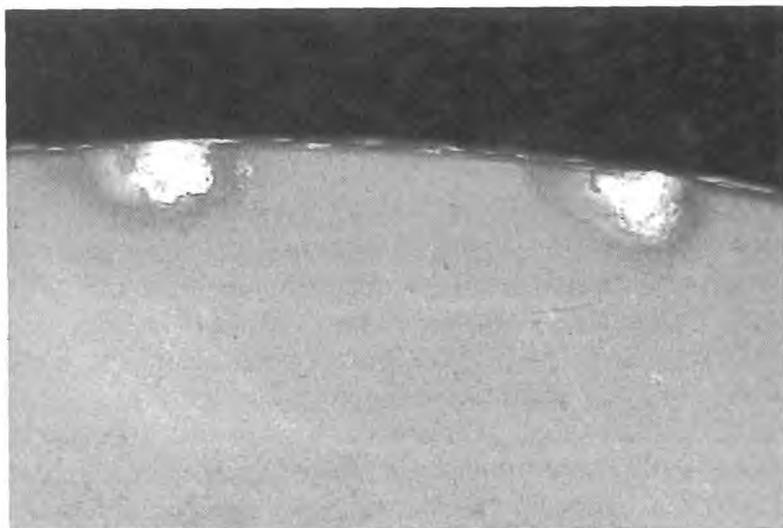


Fig. 14. Coupon of Rack-2 showing pits

Bhabha Atomic Research Centre, Trombay, India

Rack-1 was immersed in the Mumbai fuel storage basin in January 1997. It included a sample of Al-1S alloy fabricated at the research centre and used as a galvanic couple sample. After 8 months, several of the crevice coupons were taken apart and the pH inside the crevice found to be 3-4, while the bulk water pH was 5. Some staining was noted inside the crevice. The Al-1S sample coupled to stainless steel showed some white corrosion products on the rim of the sample. Other coupons showed no visible corrosion. The second inspection was made after 13 months exposure. The rack and coupons were generally free of corrosion products, except for some white product on the rim of the Al-1S specimen and some corrosion inside the crevice. The water conductivity ranged from 2-16  $\mu\text{S}/\text{cm}$  and the pH from 5.9-6.3 during the test. Chloride ions were always  $<2.0$  ppm. The two additional racks received at the second RCM were immersed in the pool for 742 days.

The coupons were disassembled and photographed during the interim inspection. Unlike the coupons received in Budapest, which had only a machined surface finish, these highly polished coupons showed no deep pits or crevice corrosion. Basin water conductivity was maintained at 3.5-16  $\mu\text{S}/\text{cm}$  with chloride ion contents less than 2 ppm. The excellent corrosion resistance of these coupons was attributed primarily to the surface finish and improved purity of the water.

Pakistan Institute of Nuclear Science and Technology, Nilhore, Islamabad

Rack-1 was immersed in November 1996 and withdrawn for a short time on a monthly basis for visual inspection. No pitting corrosion was seen on any coupons during the 12 inspections. Conductivity of the water ranged from 0.1-0.8  $\mu\text{S}/\text{cm}$  with pH between 4.8-6.1. Chloride ion content was  $<0.5$  ppm. Racks -2 and -3 containing two galvanic couples and three crevice couples were exposed in October 1998. Rack -2 was withdrawn after one-year. Conductivity of the basin water was always  $<1$   $\mu\text{S}/\text{cm}$ , chloride ion content  $<0.05$  ppm and pH 5.5-6.3.

Coupons were examined every month and discoloration was noted due to general oxidation. No pits were seen on any of the exposed surfaces. Once the coupons were disassembled, some pitting was seen under the ceramic washers of the galvanic coupons. Pitting was observed under

the washers of the crevice coupons also. There was no detectable visual change in the stainless steel coupons. The water conductivity was maintained between 0.1-0.7  $\mu\text{S}/\text{cm}$ .

#### Research Institute of Atomic Reactors, Dimitrovgrad, Russia

The steel lined storage basin at the research reactor uses ion-exchange technology to keep the water purified to 1.4-1.7  $\mu\text{S}/\text{cm}$  and chloride ion content <20 ppb. Inspections of Rack-1 were made after 6 months and 14 months. The rack was removed from the pool, dismantled, the coupons weighed, photographed, reassembled and the rack re-immersed in the basin. The coupons freely exposed to water had a uniform grey colour, typical of general corrosion on some aluminium alloys. Some 0.02 mm deep pits were seen on the exposed surface as well as some small red and brown particles protruding from the coupon surfaces. In addition, a few small 0.03 mm deep pits were seen at the contact line between the 100 mm disk and the 70mm disk. There was evidence of crevice corrosion between the coupled coupons. The weight gain of the alloys as a function of time showed a parabolic behaviour in accordance with an equation  $Y = ax^2 + bx + c$ .

Racks -2 and -3 were disassembled, coupons examined, weighed, and re-assembled for continued exposure. Some pitting was detected only at the grain boundaries of polished coupons. This was thought to be due to grain boundary etching and not caused by the storage pool environment. Some crevice corrosion was noted between the crevice coupons and some impurities were seen on the top sides of some of the coupons without pitting. The change in mass of all aluminium coupons was measured and found to be parabolic with time, as expected from the conductivity of the water and the general corrosion or oxidation of the alloys.

#### Russian Research Centre, Kurchatov Institute, Moscow, Russia.

During the first 18 months period, the water conductivity ranged from 1.9-7.6  $\mu\text{S}/\text{cm}$  and the chloride ion concentration, between <0.05-0.3 ppm. The corrosion rack was immersed in the RR-8 fuel storage basin and interim inspections were conducted after 6 and 12 months. The basin is lined with stainless steel and has aluminium storage racks. The purification system uses ion-exchange filters. Uniform surface oxide with no pitting corrosion was observed during these two inspections. Subsequently, Rack-1 was withdrawn at periodic intervals, disassembled, and photographed until a total exposure of 1254 days. The coupons were weighed, examined, re-assembled and immersed again. Two additional racks furnished by the IAEA were immersed in April 1998. The coupons in Rack -2 were examined after 367 and 551 days exposure and those in Rack -3 after 725 days exposure. These coupons were also weighed, photographed and examined for corrosion.

On most of the coupons, general corrosion resulted in a dull grey-white film, with additional corrosion products within the crevices. No pitting corrosion was seen on the outer surfaces of the coupons, except on the outer rim of some of the Al-6061 crevice coupons and the Al-6063/SS-316 galvanic coupons. Corrosion along the rim is quite common, as machined, high energy surfaces are more prone to corrosion. No corrosion was observed on the aluminium specimens inside the glass ampoules.

## Office of Atomic Energy for Peace, Bangkok, Thailand.

The rack was immersed in the fuel storage pool in November 1996. The coupons were examined visually every 30-40 days for the first 6 months. During the first inspection, carried out after 40 days, the crevice coupons were found stuck together. A few corrosion nodules were detected after about 4 months. After 6 months exposure, 3 coupons were examined and pits 10-40  $\mu\text{m}$  were found. On the crevice side, the pit density was higher (4-6 pits/ $\text{cm}^2$ ), compared to the non-crevice side (1 pit/ $\text{cm}^2$ ). However, the pits on the non-crevice side were deeper. The Al-1100 coupons had higher corrosion resistance compared to SZAV-1 alloy, and the Al-6061 alloy exhibited the highest corrosion, among the three aluminium alloys. The water conductivity ranged between 1.5-4.8  $\mu\text{S}/\text{cm}$ , the pH between 6-8 and the chloride ion concentration, between 0.1-0.8 ppm.

Two additional racks of corrosion coupons were immersed in the fuel storage pool in March 1998. The conductivity of the water ranged between 1-6  $\mu\text{S}/\text{cm}$  during the exposure period and the chloride ion content in the 0.1-0.5 ppm range. No pitting corrosion was observed on the exposed surfaces of the coupons, except along the outer rim.

### **General Comments**

- The pH of the water and the specimens inside the glass ampoules did not show any changes. These specimens were designed to evaluate radiation effects.
- *The colour of the exposed aluminium alloy surfaces varied from metallic bright to dark grey. The extent to which the surface darkened was dependent on the alloy composition.*
- Sediments were observed on the top surfaces of many coupons.
- A number of participants reported corrosion along the outer rim of the coupons. This would be expected from end grain attack on cut surfaces.
- Highly polished coupons were more resistant to corrosion than as-machined coupons.
- The crevice/galvanic couples were often stuck together with corrosion products requiring forcible separation.
- The pH in the crevice was generally 0.5 to 1.0 points less than in the bulk water.
- Pits <0.5mm in diameter were observed on the aluminium surfaces at regions in contact with the ceramic separator.
- Sediments on the top surfaces of aluminium alloy coupons caused pitting. No pits were observed on the bottom surfaces of these coupons.
- Surface features of coupons exposed for 13 months were similar to those exposed for 25 months. This time scale had no significant effect on Al coupon corrosion.

- The crevices of the aluminium alloy couples were stained, but not pitted, whereas, the aluminium/stainless couples were heavily pitted.

## Conclusions

A large database on corrosion of aluminium-clad materials has been generated from the CRP and the SRS Corrosion Surveillance Programmes. An evaluation of this data indicates that the most important factors contributing to the corrosion of the aluminium are:

- High water conductivity (100-200 $\mu$ S/cm);
- Aggressive impurity ion concentrations ( $\text{Cl}^-$ );
- Deposition of cathodic particles on aluminium (Fe, etc.);
- Sludge (containing Fe,  $\text{Cl}^-$ , and other ions in concentrations more than ten times the concentrations in the water);
- Galvanic couples between dissimilar metals (stainless steel/aluminium aluminium/uranium, etc);
- Scratches and imperfections (in protective oxide coating on cladding);
- Poor water circulation.

These factors while operating both independently and synergistically may cause corrosion of the aluminium. The single most important key to preventing corrosion is maintaining good water chemistry. Water conductivity near 1 $\mu$ S/cm generally ensures that the aggressive impurity ions like chlorides are in the parts-per-billion range. When chemistry is maintained in this regime, corrosion of aluminium alloys is minimized.

Good water chemistry alone does not always guarantee that corrosion will be prevented as seen by the extensive testing conducted in the Argentine storage pools, where iron oxide particles deposited from the water caused pitting even in high purity water. This has been seen in other fuel storage basins. Corrosion mechanisms involved in this pitting can be both galvanic and/or oxygen depletion cells.

The CRP has succeeded in making sure that all the participating countries became more aware that the successful wet storage of aluminium-clad spent fuel is not automatic, but requires diligence in maintaining high quality water conditions. Moreover, from papers written and published during the CRP by the participants and their presentations at International Conferences, the whole research reactor community is now more fully aware of the susceptibility of aluminium cladding to localized corrosion and the measures that can be taken to minimize it.

## Extension of the CRP

Extension of the CRP was considered necessary and important to broaden the database for aluminium alloy corrosion in spent fuel storage basins and especially from basins that have demonstrated significant corrosion problems. This will provide additional and much needed insight into the fundamentals of localized corrosion. A better understanding of the fundamental mechanisms will allow the prediction of corrosion rates under different combinations of

environmental parameters, enabling storage pool operators to better control those parameters necessary for safe and efficient interim storage of aluminium-clad spent fuel.

Extension of the CRP will also enable more comprehensive research in the following areas:

- Evaluate effect of dust sediments on corrosion of coupons and its implication on corrosion of fuel cladding.
- Identify aluminium alloys and other metals presently used in spent fuel basins and evaluate effect of specific bimetallic couples.
- Evaluate effect of hydrodynamic conditions on coupon and fuel cladding corrosion.
- Evaluate effect of water parameters on localized corrosion of aluminium fuel cladding in the wide range that exists between known poor water chemistry conditions and optimum conditions.

## **Appendix 1.**

### **Test Protocol**

Definitions –Corrosion rack is the assembled set of coupons, spacers, central support tube and hanger. Basin, pool, pond is the contained volume of water used for storage of spent fuel. In some cases the pool also contains the reactor core.

### **Pre-assembly**

- Unpack, handle with surgical gloves (with any talcum powder on the outside washed off).
- Weighing of individual coupons is optional.
- Rack numbering system. This is up to the individual participant because site-specific coupons of either large or small size may be added. The numbering system chosen should be logical, carefully recorded at the beginning of testing and adhered to throughout the programme.
- Photograph the front and back of each coupon and the overall assembled rack.
- No cleaning of coupons is required if the rack was received in a sealed plastic bag. If, however, the rack has been handled without gloves, cleaning will be necessary.

### **Assembly**

- Assemble coupons in specified order – large coupons on top with the deliberately scratched side facing up.
- Add extra coupons of site-specific materials, noting their position and recording all relevant information on them.
- Tighten the top hanger-nut by hand until tight and then give a further 10 degree turn.
- Attach wire/nylon rope to the hanger on the rack (remember aluminium and steel form a galvanic couple).

### **Immersion in storage basin**

- Lower/position the corrosion rack into the water in the vicinity of spent fuel if possible.
- Position below water surface and above basin floor. Do not allow the rack or any of the coupons to touch sidewalls, floor, sludge or metallic components.
- Record the location (depth, distance from walls etc.). Make a sketch to remind you and for the final report.
- Measure the radiation field intensity at periodic intervals underwater near rack (in R/h, or Sv/h).
- Sample the water at the immersion depth as near to the rack as possible.
- Indicate flow conditions near rack, (flowing or static), rate of or frequency of renewal of water in basin.
- Observe if loose particles are present on coupon surface, and if they appear to be causing a corrosion problem, please describe.

### **Exposure interval**

- Place Rack -2 and -3 into the water as soon as possible.
- Record the date of immersion.
- Leave Rack - 2 in the basin water for one year from the immersion date.
- Leave Rack - 3 in the water for continued exposure. You will be notified when to remove this rack after an evaluation of results from Rack - 2 examinations have been carried out.

### **Removal and examination of coupons**

- Withdraw the rack assembly from basin
- Measure pH of water on the external surface of coupons.
- Compare with the pH of the bulk water sample.
- Photograph the overall rack assembly prior to disassembly. Photograph all points of interest showing significant corrosion phenomena, including any corrosion of the edges.
- Remove the individual coupons from rack assembly.
- Photograph front and back of each individual coupon. A small card with a note of the material, immersion time and identity number, photographed together with the coupon surface would be very useful.
- Count the number of pits.
- Provide written observations of specific corrosion phenomena for individual coupons, including ease of removal (ease of separation of crevice/bimetallic coupons), amount and type of loose deposits, staining, discoloration, pitting, tenacious or loose oxides, raised embedded particles felt with a gloved finger, etc.
- Separate crevice coupons and measure pH on the inside faces of coupons, compare with bulk basin water pH and with that on the outside surface.

### **Post storage detailed examination**

- Decontaminate coupons with a *chloride-free* detergent and rinse with deionized water.

- Air dry/wipe.
- Use a 50% solution of phosphoric acid, if necessary, to clean/dissolve oxide from pits to conduct metallographic evaluation. Immerse for a short interval only. Remove from the solution as soon as bubbles start to increase. Further exposure will dissolve the base metal.
- Ensure that there are no oxides in the pits before determining the true pit depth.
- Conduct metallographic evaluation on the deepest pit(s). Measure pit depths with a stylus or using the calibrated focusing technique.

## **Evaluation**

All participants are requested to focus on localized corrosion mechanisms and remember that the primary purpose of the CRP experiments is to relate what is seen on the racks to performance of fuels in the storage basins

### Pitting corrosion

- Pits should be photographed.
- Pit depth should be determined, if a stylus device is available, or if a calibrated microscope stage is available.
- Final evaluation should focus on the deepest pits and should be carried out by sectioning and polishing, as in the preparation of all metallographic coupons.
- The section should be through the deepest part of the pit.
- Some attempt should be made to count the pits along with measurement of their diameters and depths. A stochastic approach can be used.

### Crevice corrosion

Opening a crevice couple destroys or drastically changes the crevice features Therefore, it should be opened and evaluated only once. If you reuse the couple, the surfaces should be machined and/or polished to reproduce the surface finish of the coupons.

- Visual and photographic inspection, together with the determination of the pH using pH paper is the first step.
- Metallographic examination should be carried out and some evaluation of the area of attack should be made.
- Evaluation of pitting in the crevice should follow the above instructions for pitting.

### Galvanic corrosion

Galvanic couples should be treated in precisely the same way as crevice couples.

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