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RADIOMETALLURGICAL EXAMINATION OF K5E
FUEL FAILURES WITH BEEHIVE GROWTH
IN SPIRES

OTHER OFFICIAL CLASSIFIED INFORMATION

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RADIOMETALLURGICAL EXAMINATION OF K5E
FUEL FAILURES WITH BEEHIVE GROWTH IN SPIRES

July 19, 1968

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RADIOMETALLURGICAL EXAMINATION OF K5E
FUEL FAILURES WITH BEEHIVE GROWTH IN SPIRES

INTRODUCTION

On September 18, 1967, KE Reactor experienced a failure in a K5E fuel element in tube 2863, exposure 634 Mwd/t. On September 24, 1967, two additional failures occurred in tubes 2865 and 3256, exposures 653 and 822 Mwd/t, respectively. All of these elements, clad with X8001 alloy were from the same canning lot, QG-829E; 2863 and 2865 were 51-piece columns and 3256 was a 35-piece column. Following is a summary of the operating data for the three columns.

<u>Tube No.</u>	<u>Tube Factor</u>	<u>Exposure (Mwd/t)</u>	<u>Tube Power (KW)</u>	<u>Outlet Temp (°C)</u>	<u>Inlet Temp (°C)</u>	<u>Flow (Gpm)</u>
2863	1.314	634	1841	111	19.5	76.4
2865	1.324	653	1871	112	19.5	75.6
3256	1.171	822	1652	103	19.5	74.9

All fuel elements from this canning lot were termed rupture-prone and were discharged. The elements from five columns including column 2863, but neither columns 2865 nor 3256, were sent to 105-C Basin Fuel Examination Facility for further examination. An incipient failure was found and sent to Radiometallurgy in an effort to determine the cause of failure. Because the elements sent to C-Basin were normal production material, they were not identified as to position or column; therefore, the column from which the incipient failure came is not known.

During the period when Lot QG829E was canned, problems of AlSi non-wetting in the spire well near the male end were being experienced in all K5 geometry fuel models. If there was a defect in the male (spire) weld such as a pinhole, water entering the defect would encounter no AlSi barrier in the non-wetted spire well area and a continuous water path to the core would be established causing eventual fuel rupture. Because of this problem with K5 geometry fuel, the initial phases of this investigation were directed toward determining the point of water entry in the failures. The first section of this report will deal with this phase of the investigation.

During the course of examination, the spire was found to contain beehive-shaped growths approximately 1/8-inch high and 1/8-inch in diameter at the base. The incipient failure contained four of these growths protruding from the spire wall. Closer examination showed that the spire contained numerous other "beehives" in less advanced stages of growth. The ruptured element from 2863 also contained a "beehive". The second section of this report covers

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examination of several "beehives" and presents information concerning the possible cause or causes of growth.

SUMMARY AND CONCLUSIONS

Explanation for Possible Water Entry Points

No water path could be found in the male or female weld of either the incipient failure or 2863 although excessive corrosion of the spire and the greater amount of uranium oxide present toward the male end suggest that water entry occurred at this end. The amount of uranium oxide present around the spire in failure 2865 indicates that water entry occurred to the spire initially, but the relative location of water entry was not established.

Conclusions

- 1) Water entry occurred initially in the spire in at least three of the four failed elements.
- 2) The relative amounts of uranium oxide present along the spire suggests that initial water entry occurred toward the male end in the incipient failure and in 2863. Water entry in 2865 occurred in the spire but the relative location was not established. No investigation was performed on element 3256.
- 3) No water path could be found in the male or female welds of either the incipient failure or 2863. The welds of these two elements were the only ones examined.

Examination of "Beehive" - Growths and Spire

Of the four elements of Lot QG829E which failed, only two, the incipient failure and 2863, were known to contain beehive-shaped growths in the spire. Although the incipient failure was from one of five columns of which 2863 was one, the column in which this failure occurred is not known. The incipient failure did not occur, however, in either of the other two columns 2865 or 3256 which experienced failures. Because this fuel was normal production material, position numbers were not stamped on them. The coloration of the film on the elements indicated that all of the elements were located toward the downstream end of their respective columns.

Failure 2863 and the incipient failure were examined extensively as they were known to contain beehive growths. Some areas of the spires were quite badly corroded but the extent of corrosion varied along the spires, with some areas revealing relatively little corrosion. The spires on 2865 and 3256 were given a visual examination, with 2865 disclosing a partially obstructed spire due to deformation of the aluminum spire material and 3256 disclosing a spire free from any blockage. The cause of the blockage observed in 2865 was not investigated.

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Although the beehive-shaped growths varied somewhat in size, they were approximately 1/8-inch high and 1/8-inch in diameter at the base. The spire on the incipient failure contained four "beehives" protruding from the spire surface; 2863 contained one beehive. Two of the beehives in the incipient failure were located toward the male end, the two others toward the female end. In 2863 the beehive was located toward the male end. Autoradiograph of one beehive from each the incipient failure and 2863, revealed no uranium present in them. The inner portion of the beehives were filled with a white granular material found to be an aluminum oxide and the outer shell was composed of a considerable amount of metallic aluminum intermixed with corrosion products.

The beehives probably were not the cause of failure, but evidence gathered in this investigation and in the review of documents suggests a higher-than-normal temperature in the spire as a contributing factor toward beehive growth. This evidence includes the presence of intergranular corrosion in the spire, a thicker-than-normal oxide film on the spire wall, a corrosion attacked "hot area", a report of a similar phenomenon occurring on the OD of a solid fuel in a KER Loop, ⁽¹⁾ and communications with BNW personnel, ⁽²⁾ who have produced this type of corrosion in the laboratory.

Conclusions

- 1) Beehive corrosion is a result of aluminum corrosion product buildup.
- 2) Indications from the two examined beehives are that water did not permeate to the core through the AlSi beneath the beehive. The AlSi was not attacked from the can wall side, and no uranium^{oxide} pocket was seen beneath the beehive.
- 3) Autoradiographs of two beehives revealed no uranium.
- 4) Higher-than-normal temperatures in the spire are suggested by intergranular corrosion on the spire-water interface, by a thicker-than-normal oxide film on the spire surface, which would tend to increase the spire temperature, and by a corrosion-attacked hot area in the spire.
- 5) Microphotographs of pre-irradiated spires from those used in canning lot QG829E, disclosed that aluminum intermetallic particle distribution and particle size were well within the limits of specifications.
- 6) The cause of beehive corrosion is not known, but it does appear to be associated with higher-than-normal operating temperatures in the spire.

TEST DETAILSExamination for Possible Water Entry Points

The incipient failure is shown in Figure 1. The black slash mark at the base end of the element (to the right in sub-figures A and B), indicates the direction of each 90° rotational movement for each subsequent subfigure view of Figure 1. Limited general uniform corrosion and minor pitting of the OD and the self supports occurred but the degree of corrosion is so minor as to be of no concern. Swelling of the OD between the end cap and self supports occurred at both ends of the element. This swelling near the ends has been seen in failures which have water entry on either the ID or OD.

Transverse sections of the element were made about 3/4 inch from each end cap as indicated in Figure 1. Figure 2 shows the cross-sections in their as-cut conditions. The amount of uranium oxide buildup is nearly equal on the OD at both locations, but much more oxide is present on the ID near the male weld, suggesting that water entry occurred nearer the male end. Cracks radiating from the uranium oxide around the spire were probably induced as a result of stresses produced in the uranium by the volume increase of uranium oxide. The ragged edges on the ID and OD clad resulted from aluminum smearing during the cutting operation. At this point it appeared that water entry had occurred through the male weld. In an effort to confirm or disprove that it had, the end cap was ground through in a plane perpendicular to the element's longitudinal axis. No water entry path was found even though the weld was inspected at various stages during the grinding operation. No void was found in the weld at any stage of the grinding. Prior to grinding, the weld had been carefully inspected and no suspicious areas were found.

As explained previously, a weld defect alone is not enough to allow water entry to the core. There must also be a continuous path through the AlSi to the uranium core. In the event that the pinhole was quite small and had been smeared over during the grinding process, the male end cap was sectioned longitudinally, the two sections of which can be seen in Figure 3. Because there was no suspicious area on the weld, a random cut was made, and the sections were ground into, in an effort to locate a possible non-wet area in the spire well near the male end. The AlSi, indicated by the arrows in Figure 3, is intact in those areas which would most likely show non-wetting in the event that water entered through the male weld. Necking down of the spire was caused by uranium oxide formation on the ID of the core. Cracking of the AlSi on the OD near the base end, Figure 3, was probably a result of uranium oxide formation also. Although water entry appears to have initiated near the male end, it does not appear to have entered through the male weld. The failure from 2863 was sectioned 1/2-inch from the male and female

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A

0°

B

Transverse Cuts Shown by Vertical Black Lines
90°

C

180°

D

270°

FIGURE 1 AS-RECEIVED PHOTOGRAPHS OF INCIPIENT FAILURE

AEC-RL RICHLAND, WASH.

A. Sectioned 3/4" from Male Weld - Extensive Uranium Oxidation Around Spire

B. Sectioned 3/4" from Female Weld - Uranium Oxidation Not Nearly As Extensive Around Spire as 3/4" From Male End

FIGURE 2. TRANSVERSE SECTIONS OF INCIPIENT FAILURE

FIGURE 3
MALE END CAP OF INCIPIENT FAILURE

ends, Figure 4. As was the case with the incipient failure, much more uranium oxide is seen around the spire near the male end than near the female end, suggesting initial water entry near the male end. As a result of the uranium oxide formation on the OD, the cladding necked down and split as shown by the arrow in Figure 4. Water entry occurred at that point only after uranium oxide had formed beneath the cladding, and therefore the split should not be thought of as an initial point of water entry.

The welds on both the male and female ends were ground into, but no pinholes were observed. The end caps were sectioned longitudinally in half, but no suspicious possible water entry point in the AlSi was seen. Although the point of water entry in both the incipient failure and 2863 appear to be at or near the male end, no water entry path could be found. Failure 2865, which was sectioned transversely $1\frac{1}{4}$ inches from the male end, revealed extensive uranium oxidation around the spire to much the same extent as the two failures discussed previously. Core corrosion was much more extensive around the spire than around the OD, suggesting that initial water entry occurred in the spire.

Examination of "Beehive" Growths

Two of the four beehive growths seen in the spire of the incipient failure are shown in Figure 5. A beehive growth was also found in failure 2863. These growths are approximately $1/8$ -inch high and $1/8$ -inch in diameter at their base. They are composed of a shell containing some elemental aluminum from the spire and some corrosion product. The inner portion of the beehives contain a white granular-looking material composed primarily of aluminum and oxygen. An electron microprobe analysis of the corrosion product revealed aluminum and oxygen with a possibility of calcium or iron. The latter two elements appeared only in trace quantities at one point in the sample (an electron microprobe can analyze a point 1.5-3 microns in diameter), but their presence could not be determined at other points in the sample. Carbon was found, but since the mountant used to prepare the sample contains carbon, the sample could have easily been contaminated by the mountant. No evidence of copper, chromium, silicon, nickel, magnesium, or manganese could be found. The elements selected for examination were those which could have entered the aluminum during its fabrication, either as alloying elements or as impurities or from those impurities known to be in the reactor cooling water. The above explanation is not complete without reservations concerning the method of sampling because of the method's non-representative nature. A single sample, sufficiently small, was taken from the beehive to assure minimum irradiation problems during analysis. Using only a single sample limits reliability. Although several locations on the sample were probed, obtaining a representative sample would require that a larger number of points be analyzed since such a small area is probed. Funding limitations prevented a more exhaustive examination.

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1/2" from
Female
End Cap

1/2" from
Male End
Cap

FIGURE 4 TRANSVERSE SECTIONS TOWARD MALE AND FEMALE END CAPS
AEC-RL RICHLAND, WASH.

A. Two Beehives Protruding from Spire Wall. Growths Are Approximately 1/8" High

B. Longitudinal View of Corrosion of Core. Beehives Are in Background

FIGURE 5 SPIRE WITH BEEHIVES - INCIPIENT FAILURE

The beehive in Figure 5, indicated by the X was chosen for examination. The element was sectioned transversely, and grinding proceeded on the beehive. Several successive steps in the grinding can be seen in Figure 6, with the final grinding step shown in subfigure C. Before proceeding with an explanation of these beehives, it will be necessary to explain the presence of certain artifacts in the photographs of Figure 6. First, the ragged edges on the water surface of the spire in subfigures A and B are the result of aluminum smearing during sectioning. These ragged edges have disappeared in subfigure C. Second, the bubbles rising from the tip of the beehive were entrapped in the mountant during sample preparation. During the mounting operation, some gas in the beehive was forced out through the tip by the mountant. There is no reason to believe that the gas was anything except air as the tip of the growth is visibly porous and air was readily accessible to the inside pores within the beehive.

As mentioned previously, the inner portion of the beehive contained a corrosion product of aluminum and oxygen, which was concentrated mainly toward the top of the beehive. The lower portion, particularly that portion adjacent to the AlSi, is quite porous. A large void which has an open lead to the outside of the beehive is shown adjacent to the AlSi layer. Whether or not the void was present in the spire prior to irradiation is a matter of conjecture, but evidence, to be shown later in this report, suggests that the void is a result of beehive corrosion and is not the cause.

The arrow in subfigure A of Figure 6 indicates an area believed to be the base of a beehive whose upper portion has been removed. Localized corrosion at this point originated at the water-spire interface and proceeded inward toward the AlSi. This particular corrosion area has not reached the advanced stages of corrosion, but it does contain a small amount of the white granular-looking material characteristic of the material seen in the beehive. Microscopic examination and comparison between the beehive and the other indicated corroded area revealed visual similarities between the two. Also, under polarized light the materials in both appeared identical.

The AlSi layer directly beneath the beehive was intact, Figure 7, although some damage was incurred to it along the core side. However, no porosity through the AlSi was seen beneath the beehive and probably no water entered to the core through the AlSi. If water had entered through the AlSi, one would expect to see a greater amount of uranium oxide directly beneath the beehive than actually is present. The amount of uranium oxide around the spire on that transverse section is quite uniform. An autoradiograph showed the beehive to contain no more radioactive material than the adjacent aluminum. This would support the premise that the AlSi did not allow water to penetrate into the core nor did it allow uranium oxide to diffuse out.

B. Ground into 1/6 of Growth B. Ground into 1/4 of growth

C. Ground to Middle of Growth

FIGURE 6 TRANSVERSE SECTION OF "BEEHIVE" - SHAPED GROWTH

FIGURE 7 ALSTI BARRIER BETWEEN GROWTH AND URANIUM CORE

AEC-RL RICHLAND, WASH.

250X

75X

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A second element designated 2863 because the failure occurred in tube 2863 was also examined and found to contain one beehive growth about two inches from the female end, Figure 8. Figure 9 shows a view looking down onto the beehive after grinding through to its center. The beehive is quite similar to the one examined in the incipient failure. The AlSi layer is intact as before, and an autoradiograph again revealed no uranium in the beehive. A closeup, Figure 10, 40X, of the beehive shows that the white corrosion product within the beehive has darkened considerably as at least a month elapsed between the time the photographs of Figures 9 and 10 were taken. One of the most interesting facets of this beehive is the nearly continuous shell of aluminum surrounding the corrosion product. Flow lines observed in the aluminum indicate that the corrosion product swelled the aluminum forcing the aluminum to plastically deform and form the bulk of the beehive shell. The outer shell of the beehive in the incipient failure also contained some aluminum but not nearly as much as observed in 2863. A film similar in appearance to the film on the spire surface is present over much of the surface of the beehive, but this film is broken badly, Figure 11. Because the film on the spire surface is continuous, the broken film on the beehive surface suggests that the film was present before the beehive grew, and that during growth the somewhat brittle film broke to alleviate the stresses created by elongation during growth.

Corrosion of a similar nature to the beehives has been observed previously both in the laboratory and in a special test in a KER loop.⁽²⁾ Battelle Northwest Labs have produced blistering on sheet aluminum X8001 alloy aluminum by subjecting it to temperatures near 350°C for several hours. The "blisters" were nearly identical to the beehives in size and shape with the "blisters" assuming a somewhat flatter inverted cone geometry. The "blisters" contained a "white powdery-looking material" thought to be hydrate of aluminum oxide.⁽¹⁾

Two solid X8001 aluminum jacketed elements discharged from KER Loop 3 on July 11, 1959, revealed numerous blisters. These elements, were authorized to operate at a maximum surface temperature of 315°C. Failure of the two elements was caused by an accelerated corrosion attack of the aluminum alloy can wall which started at the base of the supports. The severe corrosion attack started in the dead space under the supports where accumulated oxide film reduced the heat transfer and caused localized overheating of the can wall surface.

The corrosion was limited to the can wall; only limited deterioration of the AlSi layer and core occurred. The two elements had numerous large "wrinkled blistered" areas in the cladding around the support bases. The blisters were filled with a white corrosion product and were covered with a thin wrinkled skin of the outer can wall.⁽²⁾

FIGURE 8 "BEEHIVE" GROWTH AND "BULGES" IN 2863

Female
End ↑

Male
End ↓

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A. Top View - Looking Down
Into Spire 4.6X

B. Transverse Section Through
Beehive 4.6X

FIGURE 9 BEEHIVE GROWTH IN SPIRE OF FAILED ELEMENT 2863

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FIGURE 10
Beehive Growth Corrosion

Near Tip of Beehive

250X

Section on Left Side of Beehive

250X

FIGURE 11 PHASES PRESENT IN BEEHIVE GROWTH

AEC-RL RICHLAND, WASH.

Examination of Spire

Element 2863 contained several large bulged areas in the spire, Figure 12, which were found to have been caused by uranium oxide buildup beneath the clad in certain localized areas. The bulges were located toward the male end of the element where a greater percentage of uranium oxide had formed. Adjacent to the bulges was a third, but somewhat smaller "bulge", with what appears to be a hole in its top. This smaller "bulge" may have been a beehive growth, or it may have resulted from uranium oxide build-up beneath the clad. No further examination was performed on this bulge because at the time of its discovery, the beehive growth in this spire was considered to be of greater importance. Following examination of the beehives and subsequent examination of the spire (to be discussed later), sufficient funds were not available for further investigation.

The white-spotted area at the top of Figure 8 is indicative of a hot area. Excessive corrosion occurred in that area as can be seen by the reduction in clad thickness. Figure 5 also shows considerable clad reduction, and at one point, corrosion occurred to the AlSi. General appearance of the spires from the incipient failure and 2863 suggests that temperatures in the spire were higher than normal. Just how much higher is not known. Generally speaking, the spire of an element irradiated to 600 Mwd/t is relatively smooth.

Examination of the transverse section of the spire of failure 2863 shown in subfigure 13 of Figure 9 revealed pitting and intergranular corrosion. This corrosion was found at various points over the entire cross-section. Figure 13 reveals intergranular corrosion of the aluminum at one such point. The grains of aluminum are clearly outlined even though the aluminum in this photograph has not been etched. Intergranular corrosion has been noted on both the OD^{3.4.5} and spire⁵ in "hot areas", suggesting again that elevated temperatures occurred in the incipient failure and 2863. Spheroidization of the AlSi, which generally occurs at elevated temperatures did not occur in the metallographically examined portion of the spire. However, the white-spotted area believed to have been the hottest portion of the spire was not examined for AlSi spheroidization. Intergranular corrosion will occur at lower temperatures than AlSi spheroidization but both effects are indicative of higher-than-normal operating temperatures in the aluminum.

Figure 14 shows one of several typical pits which also were found on the same transverse section of the spire as the beehive growth. Corrosion product was observed on the edges of nearly all of the pits. Whether or not the entire pit at one time contained corrosion material is not known, but several other attacked areas, completely filled with corrosion product, were seen.

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↑
Female End

Male End
↓

Top View of Bulge

Transverse Section Thru Bulge

FIGURE 12 BULGE IN SPIRE OF FAILED ELEMENT 2863
AEC-RL RICHLAND, WASH.

75X

250X

"Blocked" Area from Subfigure Above

750X

FIGURE 13 INTERGRANULAR CORROSION ON SPIRE - UNETCHED

AEC-RL RICHLAND, WASH.

250X

Polarized Light 250X

750X

FIGURE 14

PIT NEAR SPIRE-WATER INTERFACE

Corrosion in X8001 aluminum alloy has been known to occur because of aluminum intermetallic particle segregation,⁽¹⁾ leaving some areas in the aluminum matrix depleted of nickel while at the same time other areas are becoming enriched in nickel. When this occurs, a potential is established between the two distinct areas with the anodic area experiencing corrosion. To determine if nickel segregation may have been present in either or both of the pre- and post-irradiated spires, the microphotographs of the aluminum from the spire were examined, Figure 15. Very limited nickel segregation was seen in areas in the post-irradiated spire where intergranular corrosion was prevalent. Aluminum intermetallic phases in both the pre- and post-irradiated spires are of the same general particle size and distribution. Although a major portion of the large black "spots" in the post irradiated material resulted from bits of oxide film tearing from the spire surface or from etchant which was not washed away, some of these "spots" are sources of localized corrosion. Those areas of localized corrosion, generally speaking, are near the spire-water interface where intergranular corrosion is prevalent. An examination of numerous photographs of the pre-irradiated aluminum disclosed that this material falls well within the specifications for aluminum intermetallic particle size and uniform particle distribution. The spires used to fabricate the failed element come from lot B-4-10-62.

Figure 16 shows aluminum intermetallic particle distribution in an area which has undergone extensive intergranular corrosion. Generally speaking, the particles are uniformly distributed throughout the grains, although in some areas segregation appears to have occurred in the form of higher-than-normal intermetallic concentration at the grain boundaries. Segregation of this type could essentially nullify the corrosion resistant properties provided by the uniform intermetallic dispersion. Because segregation was limited in the post irradiated spire and was absent in the pre-irradiated spires, the actual segregation probably occurred in-reactor.

Beehive Corrosion at KW Reactor

KW Reactor recently experienced a failure in 125 enriched fuel, lot XU005L, which contained beehive-shaped growths on the aluminum spire. The failure, from position 12 and the elements from positions 11, 13, and 14 all contained beehive-shaped growths. cursory examination of the failure in the technical viewing pit at KW Reactor revealed no cause for failure other than that suggested by the presence of the beehives. These elements containing beehives were in the region of the column in which the spires' surface temperatures were calculated to be the highest. Outlet coolant temperature in this tube was one of the three highest prior to shutdown.

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Typical Pre-Irradiation Spire Condition 250X

Post-Irradiation Spire Condition 250X

Most of Large Black Particles are Bits
of Film Torn Loose from Spire-Water
Interface during Sample Preparation

FIGURE 15 DISPERSION OF ALUMINUM INTERMETALLIC PHASES

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ETCHED IN HF 75X

Note: Uniform Aluminum Intermetallic Dispersion Within Grains

FIGURE 16 INTERGRANULAR CORROSION ON SPIRE - ETCHED

AEC-RL RICHLAND, WASH.

At the time of the failure of the KW elements a relationship between the KW and KE beehive failures relating to spire history was suspected. An investigation revealed that the spires used in the KW and KE failed elements were fabricated approximately a month apart. Spire history on these two lots could not be traced to either heat number or ingot, but the possibility that the two lots were fabricated from the same ingot or heat is remote.

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