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MICRORADIOGRAPHY OF POROSITY

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IN ROVER FUEL ELEMENTS

DATE AUG 28 1973

For The Atomic Energy Commission L. R. BUNNELL

Bram C. Feldman

JANUARY 1968

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Division of Classification

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MICRORADIOGRAPHY OF POROSITY
IN ROVER FUEL ELEMENTS

By

L. R. Bunnell

January 1968

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MICRORADIOGRAPHY OF POROSITY
IN ROVER FUEL ELEMENTS

L. R. Bunnell

January 1968

ABSTRACT

Radiographic detection of a vacuum-impregnated stain was used to reveal pore networks in as-manufactured and hot-gas tested ROVER elements from WANL and Y-12. Access of the stain was controlled to simulate hydrogen access to bore-connected pores in these elements. Pore networks revealed by this method in untested samples were most prevalent at positions corresponding to the greatest porosity in tested elements. The study also revealed details of fuel-bead migration, empty fuel-bead cores, and associated pore networks.

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MICROGRAPHY OF POROSITY
IN ROVER FUEL ELEMENTS

L. R. Bunnell

INTRODUCTION

This document reports on efforts to improve our understanding of the influence of the pre-existing pore networks on the development of corrosion patterns in graphite-matrix fuel by hot hydrogen. We previously developed a method for revealing connected pores by radiographic detection of radiological stain.⁽¹⁾ We used this method to look at a recent untested and tested element from WANL and Y-12. Various methods have been used to check elements for porosity; gas-flow tests, whole-body and incremental, are used for production checks. Incremental weight-loss measurements after hot-gas-testing define zones of most extensive attack.

Corrosion of the graphite-matrix fuel elements by hot hydrogen is one of the factors limiting the operating lifetime of reactors of the ROVER type. During reactor operation, differential pressures develop between neighboring bore holes as a result of the very high hydrogen flow. While differential pressures are not very high, the distance between bores is small with the result that pressure gradients are quite large. The NbC bore coating used to protect the graphite invariably contains cracks which provide access to the graphite below. Access to the graphite per se is not extremely detrimental if reaction products have to be removed to expose the graphite surface to fresh hydrogen. A system of connected pores, however, can provide a flow path which accelerates corrosion substantially.

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SUMMARY AND CONCLUSIONS

Stain radiography produces a graphic representation of any pore networks which are connected to coating defects in ROVER elements in the as-manufactured condition and as affected by hot-gas testing. The stain access was controlled to simulate the route of the access during hot-gas tests. The stain was successfully detected, and the results of the experiment agreed with weight-loss data furnished with the samples.

The use of the method outlined in this report revealed the following features:

- Porosity and the connection between pores to form networks
- Development of corrosion patterns during hot-gas testing
- Empty fuel-bead cores and associated pore networks deep in the graphite
- Details of fuel-bead breakup during hot-gas testing.

In general, the midband region of heavy corrosion during hot-gas testing corresponds to the location of the most extensive pore networks observed in as-manufactured elements. Although the WANL and Y-12 hot-tested elements were similar in general appearance, the WANL elements evidenced a corrosion peak between station 12 and station 20. In the hot-tested Y-12 element, defects were most extensive in the midband region.

DISCUSSION

The elements examined are described in Table I. Appendix A, the cover letter we received with these samples, contains more detailed information.

This series of samples was composed of 28, three-inch sections of fueled elements. These included nine samples each from a Y-12 and WANL corrosion-tested element (Stations 9-12, 14-17, 19-22, 22-27, 29-32, 34-37, 39-42, 44-47, 48-51), and five samples each from a Y-12 and a WANL as-coated element (Stations 9-12, 19-22, 29-32, 39-42, 48-51).

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TABLE I. *Brief Characterization of Elements Examined*

<u>Element No.</u>	<u>Coating Batch No.</u>	<u>Condition</u>
99-28802 (Westinghouse)	B72	Untested
99-30997 (Westinghouse)	C319	Tested 60 min at 2350 °C
99-75066 (Y-12)	8947	(a)
99-75522 (Y-12)	8965	Tested 60 min at 2350 °C

(a) *This element is described in Appendix A as an untested element, but had been tested and shipped inadvertently. Since it is similar to the other test Y-12 element, it is not included in this report.*

Since definition of pore networks was our prime reason for examining these elements, normal metallography was performed on only a few samples. The following section describes the work on the sample set.

EXPERIMENTAL METHOD

A method was developed to identify the access routes for hydrogen during testing, as far as this can be done in a room-temperature environment. This required a radiological stain which we could impregnate into a bulk sample and whose access we could easily control, so that impregnation would occur only through cracks in the bore coating. A stain which meets this requirement is a 50-50 (vol%) mixture of tetrabromoethane (for hazards associated with using tetrabromoethane, see Ref. 2) and Maraglas resin.

The sample-preparation process was as follows.

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Longitudinal Samples

The bore-to-bore interconnection is best revealed in a longitudinal sample, that is, a sample radiographed perpendicular to the extrusion axis. The longitudinal samples were prepared in the manner described below and depicted in Figure 1.

- (a) A 1 inch sample was cut from the 3-inch lengths supplied.
- (b) All external surfaces, i.e., flats and ends, were painted with resin to prevent access by the impregnant other than through the bore coating.
- (c) The samples were vacuum-impregnated with a 50-50 (vol%) mixture of tetrabromoethane and Maraglas resin.
- (d) The staining mixture was allowed to drain from the bore holes.
- (e) The samples were mounted in Maraglas resin and held at 160 °F for 16 hr to cure the resin and immobilize the tetrabromoethane.
- (f) The longitudinal samples, 3/16 in. thick, were cut from the 1 in. pieces as shown in the drawing.
- (g) An epoxy (Devcon Type F) was applied to the ends for support during thinning.
- (h) The sections were thinned to 0.008 in. and ultrasonically cleaned to remove debris.
- (i) Radiography was done with a tungsten tube operated at 8 kV and 15 mA, with a helium beam path between X-ray tube and specimen. Average exposure time was 20 min. The film used was Eastman High Resolution Plate. After processing (5 min development in D-19 developer, with agitation, fixed and dried in film dryer), the microradiographs were ready for examination and photography. The macrographs in this report were made from the original plates by direct enlargement.

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FIGURE 1. Steps in Sample Preparation

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Transverse Samples

While the network of porosity is usually symmetrical about the center hole, transverse sections, radiographed parallel to the extrusion axis, are easier to interpret. The samples are easier to prepare, and are prepared by the same steps as above, except that no epoxy reinforcement (Step g) is necessary.

OBSERVATIONS

Figure 2 is a composite of macroradiographs ($\sim 12X$) for the stations indicated along the length of Element 99-28802. For compactness, the bore-hole portions were cropped from the original photographs. Small variations in the web width are caused by small errors in grinding to the exact center line of the bore holes.

Some of the samples broke during thinning; for that reason, portions of some macrographs are missing. Sample breakage occurred while we were still perfecting the sample preparation method. The current methods, as described, will produce samples without the breakup.

The black solid lines in Figure 2 are NbC bore liners; the black circles are UC_2 fuel-bead cores. The black lacy networks are the radiological stain in pores. Due to the difference in the photographic processing, all micrographs discussed in the report are mirror images of their counterparts on macrographs; and have undergone reversal so that a black image on the macrograph is white on the micrographs.

Element 99-28802 (Westinghouse, untested, refer to Figure 2 with supplemental Figures 3-6) At station 9, 10 there is a small amount of porosity in the outer regions of the element. The same holds true for Station 19,20 (see transverse view of Station 19, Figure 3). At Station 29,30 the pore networks are more extensive and larger. (See micrograph of area A, Figure 4.) The same is true at Station 39,40, where we also see an empty-bead defect. A broken fuel bead swelled early in processing,

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cracking surrounding graphite, and was later leached free of uranium. (See microradiograph of area B, Figure 5.) At Station 48,49 the networks are not as extensive as at Station 29, 30 and 39,40, but some pores are better aligned (see area C, Figure 6).

Element 99-30997 (Westinghouse, hot-gas tested, refer to Figure 7, with supplemental Figures 8-16) At Station 9,10, there are bands of extensive porosity, especially in the area close to the NbC coating. The area surrounding the center bore hole is usually not very porous. The connection between inner six and outer twelve bore holes is seen in Figure 8, a micro-radiograph of area A in Figure 7, Station 9,10. The most extensive porosity in this element is revealed at Station 16, Figure 9. This sample was so corroded that it was impossible to prepare a longitudinal sample to compare with other stations. A transverse section is self-supporting and serves to illustrate the appearance at this station.

Figure 10 shows a transverse section at Station 19. The center bore hole region is still tight even though in this case the bore coating is no longer in place. The other holes are surrounded by extensive porosity. Similar pore distribution is shown in a longitudinal section at Station 20,21 in Figure 7. Station 26,27 appears less porous than 20,21, with its porosity in the central region of webs instead of close to the bore coating interface. At Station 30,31 there is extensive porosity in the middle of the web between outer twelve and inner six bore holes. At Station 34,35 the attack is quite extensive, especially near defects, as in the enlargement (B), Figure 11. At Station 39,40 the attack is mostly in the bore coating interface as in the enlargement of (C), Figure 12. Massive fuel-bead migration first appears in a transverse section from Station 44, Figure 13. To make a photographic print which

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satisfactorily reveals both fuel by the bore channel and porosity in the outer portion, we lightened the inner portion. This printing procedure washes out some porosity near the center. Notice that the migration pattern is slightly off-center.

The porosity in an area at Station 44,45, where fuel-bead breakup is not too extensive (D) is seen in Figure 14. Fuel-bead breakup obscures most of the porosity at higher numbered stations. The enlargement of (E), Figure 15, shows the various stages of fuel-bead breakup. Figure 16, a transverse macrograph, shows a fully developed migration pattern at Station 48.

Element 99-75522 (Y-12, hot-gas Tested, refer to Figure 17, with supplemental Figures 18-20) At Station 10,11 (Figure 17) the porosity is about the same as that in an untested element. By Station 15,16 the effects of hot testing appear (see enlargement A, Figure 18). A transverse view at Station 19 (Figure 19) shows corroded annular voids around some bore channels in inner six and outer twelve positions. Station 20,21 also shows annular ring defects, as does Station 29,30 to a greater extent (see enlargement B, Figure 20). Station 34,35 has annular rings, reduced in extent. At Station 30,40 porosity is concentrated adjacent to the bore coatings. Station 44,45 also exhibits this behavior. After Station 44,45 fuel-bead breakup masks images due to porosity.

COMPARISON OF RESULTS

Figure 21 is a plot of weight-loss profiles for the two elements. These results are consistent with our radiographic observation of greater porosity between stations 25 and 40 on the Y-12 element. Fuel migration after Station 43 makes interpretation difficult, hence we do not see high-end corrosion very clearly. The real difference between these elements, however, is at Station 14-17, where Y-12 weight loss is only

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about one gram, while WANL weight loss is over eight grams. Referring back to Figure 9, we see this is an area of exceptionally heavy corrosion. The center seven bores are interconnected, and the outer bores have their heaviest corrosion, either toward other bores or toward external surfaces, suggesting an attack mode which is assisted by bore-to-bore gas communication. Unfortunately, the untested samples did not include this (14,17) section. However, such extensive corrosion is more likely the result of a random than a systematic defect unless other elements of the same vintage show a similar corrosion habit.

ACKNOWLEDGEMENTS

Throughout the performance of this work, the critical eye of E. M. Woodruff in helping to analyze the results and the hand of D. H. Parks in preparing the samples were invaluable.

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FIGURE 2. A Composite of Sections from 12X Macroradiographs Along the Length of Element 99-28802 (Westinghouse, untested). Station Locations are Indicated Below Each Radiograph.

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11

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19

10X

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FIGURE 3. Transverse Macroradiograph from Station 19 of Untested WNL Element. Note Empty-Bead Defect (Arrow). The Objects in the Bore Holes are Reinforcement Which We Found Were Not Needed.

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Tetrabromoethane
Stained
Resin
Coating

75X

FIGURE 4. *Microradiograph from Area A, Station 29, 30, in Figure 2*

Empty-
Bead
Defect

75X

FIGURE 5. *Microradiograph from Station 48, 49, Area B in Figure 2, Showing Empty-Bead Defect*

Aligned
Porosity

75X

FIGURE 6. *Microradiograph from Station 39,40,
Area C in Figure 2, Showing Aligned
Porosity*

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FIGURE 7. A Composite of Sections from Macroradiographs, Along the Length of Element 99-30997 (Westinghouse, tested)

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75X

FIGURE 8. *Composite Microradiograph from Station 9,10
Area A in Figure 7*

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10X

FIGURE 9. *Transverse Macroradiograph from Station 16.
Note Extensive Corrosion, Which Connects Holes
Near the Center of the Element.*

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19

10X

FIGURE 10. Transverse Macroradiograph from Station 19

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75X

FIGURE 11. *Microradiograph from Station 34,35, Area B in Figure 7, Showing Corrosion Near Defect*

75X

FIGURE 12. *Microradiograph from Station 39,40, Area C in Figure 7, Showing Corrosion Near Coating*

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44

10X

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FIGURE 13. Macrograph from Station 44, Showing Initial Fuel-Bead Breakup. Reinforcement Material Is in Some Bore Holes

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75X

FIGURE 14. *Composite Micrograph from Station 44,45, Area D
in Figure 7*

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Test
Gas
Flow
Direction

75X

FIGURE 15. *Microradiograph from Station 48,49, Area E in Figure 7, Showing Some Stages of Fuel-Bead Breakup--*

Bead a: Uranium on its way to the bore-coating interface diffused around the bead.

Bead b: Uranium decorated the PyC on the downstream (hot) end of the bead.

Bead c: Uranium penetrated the fuel-bead shell and moved away through the graphite, toward the downstream end.

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48

10X

FIGURE 16. *Transverse Macroradiograph at Station 48, Showing Fully Developed Migration Pattern*

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Figure 17. A Composite of Sections from 12X Macroradiographs, Along the Length of Element 99-75522 (Y-12, tested)

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75X

FIGURE 18. *Composite Microradiograph from Station 15,16,
Area A in Figure 7*

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10X

FIGURE 19. *Transverse Macroradiograph from Station 19.*

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75X

FIGURE 20. *Microradiograph at Station 29,30, Area B in Figure 17, Showing Corrosion at an Extensive Ring Defect. Note Cluster of Loose Beads at Lower Left*

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FIGURE 21. Weight-Loss Data for the Two Tested Elements,
as a Function of Station Number

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APPENDIX A
INFORMATION PROVIDED BY WANL
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TABLE A-I. Sample Data

<u>Element No.</u>	<u>Coating Batch</u>	<u>Time, min</u>	<u>Wt. Loss, g</u>	<u>Sample Location</u>	<u>Calculated Wt. Loss per Sample, g</u>
99-75522	8965	60	14.7	9-12	0.000
				14-17	1.125
				19-22	1.163
				24-27	0.440
				29-32	0.362
				34-37	0.437
				39-42	0.596
				44-47	1.817
99-30997	C319	60	21.8	48-51	2.178
				9-12	0.593
				14-17	8.372
				19-22	0.826
				24-27	0.155
				29-32	0.020
				34-37	0.128
				39-42	0.630
99-75066	8947	No Test		44-47	1.485
				48-51	1.643
				9-12	
				19-22	
				29-32	
99-28802	B72	No Test		39-42	
				48-51	
				9-12	
				19-22	
				29-32	

TABLE A-II. Coating Profiles

<u>Batch</u>	<u>Station Number</u>									
	<u>5</u>	<u>10</u>	<u>15</u>	<u>20</u>	<u>25</u>	<u>30</u>	<u>35</u>	<u>40</u>	<u>45</u>	<u>50</u>
<u>Coating Thickness, mils</u>										
C319	1.50	1.60	1.76	1.90	2.00	2.24	2.34	2.60	2.54	2.22
B72	1.25	1.27	1.30	1.30	1.35	1.40	1.50	1.67	1.70	1.77
8947	1.15	2.02	2.10	2.10	2.02	2.05	2.10	2.14	2.15	2.02
8965	1.30	1.67	1.80	1.80	1.80	1.80	1.80	1.80	1.80	1.80

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