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REPORT

QUARTERLY PROGRESS REPORT
ROVER GRAPHITE STUDIES
JANUARY, FEBRUARY, MARCH, 1966

THE STAFF OF
CERAMICS AND GRAPHITE RESEARCH SECTION

CLASSIFICATION CANCELLED

DATE AUG 8 1973

For The Atomic Energy Commission

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Chief, Reactor, Space and Technology Branch
Division of Classification

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(per 7/22/82 Duff/Caudle memorandum)
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APRIL, 1966

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C-91, Nuclear Reactors for Rocket Propulsion (M-3679)

QUARTERLY PROGRESS REPORT
ROVER GRAPHITE STUDIES
JANUARY, FEBRUARY, MARCH, 1966

By

The Staff

of

Ceramics and Graphite Research Section

April, 1966

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OPTICAL MICROSCOPY - E. M. Woodruff

Previous microscopy studies were conducted on portions of ROVER elements representing the condition during several stages of processing or after a subsequent hot test. To provide a more valid basis for comparing coating adherence on fueled and unfueled elements and to examine the development of porosity in the graphite matrix during hot tests, a series of full-length fueled and unfueled elements were collected at LASL and shipped to Battelle-Northwest in January. Identifying numbers and test histories of the elements are given in Table I.

TABLE I. Test Histories of Examined Elements

	Element Serial No.	Coating Batch No.	Hot-Gas Test Condition	Weight Loss, %
Unfueled				
	1878-AA-264	D6-23	Not tested	---
	1878-AA-220	D6-23	2400 °C for 6 min	0.7
	1878-AA-244	D6-23	2400 °C for 30 min	1.2
	1878-AA-265	D6-23	2400 °C for 30 + 30 min	2.6
Fueled				
	287-14181	2046	Not tested	---
	287-14161	2046	2250 °C for 7 min	0.49
	287-14164	2046	2250 °C for 20 min	0.81
	287-14170	2046	2250 °C for 20+16 min	2.64

Although different test temperatures were used for unfueled and fueled elements and the duration of the longest hot-test was 60 min for unfueled and 36 min for fueled elements, the two sets of samples showed quite similar weight losses.

Specimens were taken from each element at 10 in. intervals for optical and electron microscopy. All micrographs of a given element were taken along the same channel at the position indicated in Figure 1. Figures 2 through 5 are optical micrographs of transverse sections of each station sampled.

Adherence of the coating on the untested, unfueled element (Figures 2a through f) is good with the exception of a small separation between the coating and graphite at Station 40. On the fueled, untested element (Figures 2g through l) only the coatings at the ends show no separation. The graphite surface along separations at Stations 10, 20, 30, and 40 has much more relief than the relatively smooth contact line of adherent coatings. Pitting in the graphite is common along the separation and is particularly noticeable in the pyrolytic carbon fuel coating in Figure 5k. The pits suggest a catalyzed chemical reaction may have occurred during coating. Such a reaction could lower graphite strength at the surface where it is subjected to thermal stresses imposed by the coating.

Coating thickness on unfueled elements increases from about 0.01 mm at the reactor inlet end (Station 1) to about 0.14 mm at the exit end. Spalling of NbC grains at the bore surface during the 30 and 60 min hot tests (Figures 4 and 5) reduced the exit thickness in some regions. Coatings on the fueled elements are more uniform in thickness, increasing from 0.03 mm at the inlet to 0.05 mm at Station 10, remaining constant through Station 40, and decreasing to 0.04 mm at the exit end.

Grain size of the coating increases from the inlet to Station 20 on the unfueled elements. At Station 20, grains extend across the entire width of the coating; and, therefore, intergranular cracks form more direct openings between the coolant channel and graphite substrate than at other positions. Severe graphite corrosion is evident at Station 20 in the tested unfueled elements.

Porosity that develops during hot testing is generally less extensive on the fueled elements than on the unfueled. On fueled elements, the more severe localized attack, evidenced by penetration to the outer element surfaces, may explain the similar weight losses. Preferential removal of binder is extensive in the unfueled elements; whereas, in the fueled graphite, microcracks at binder-filler interfaces open up and the adjacent binder regions remain intact.

Examination of this series of elements is continuing in the electron microscope.

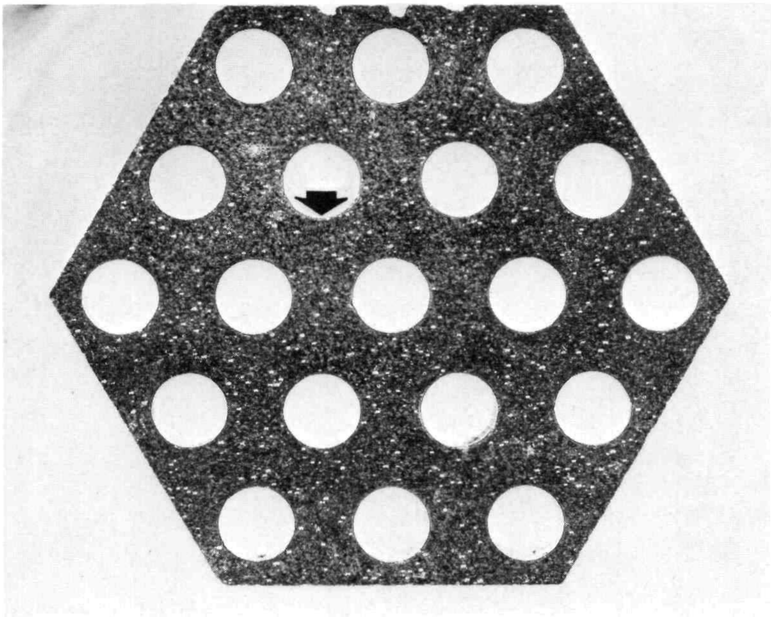

FIGURE 1. Rover Element Showing Sampling Position

(Arrow indicates location of areas viewed in Figures 2 through 5. Notches at top indicate element face on which the original element number was stamped and are used to orient sample for channel location. Station 10 on untested element 287-14181.)

Unfueled Untested | Fueled Untested

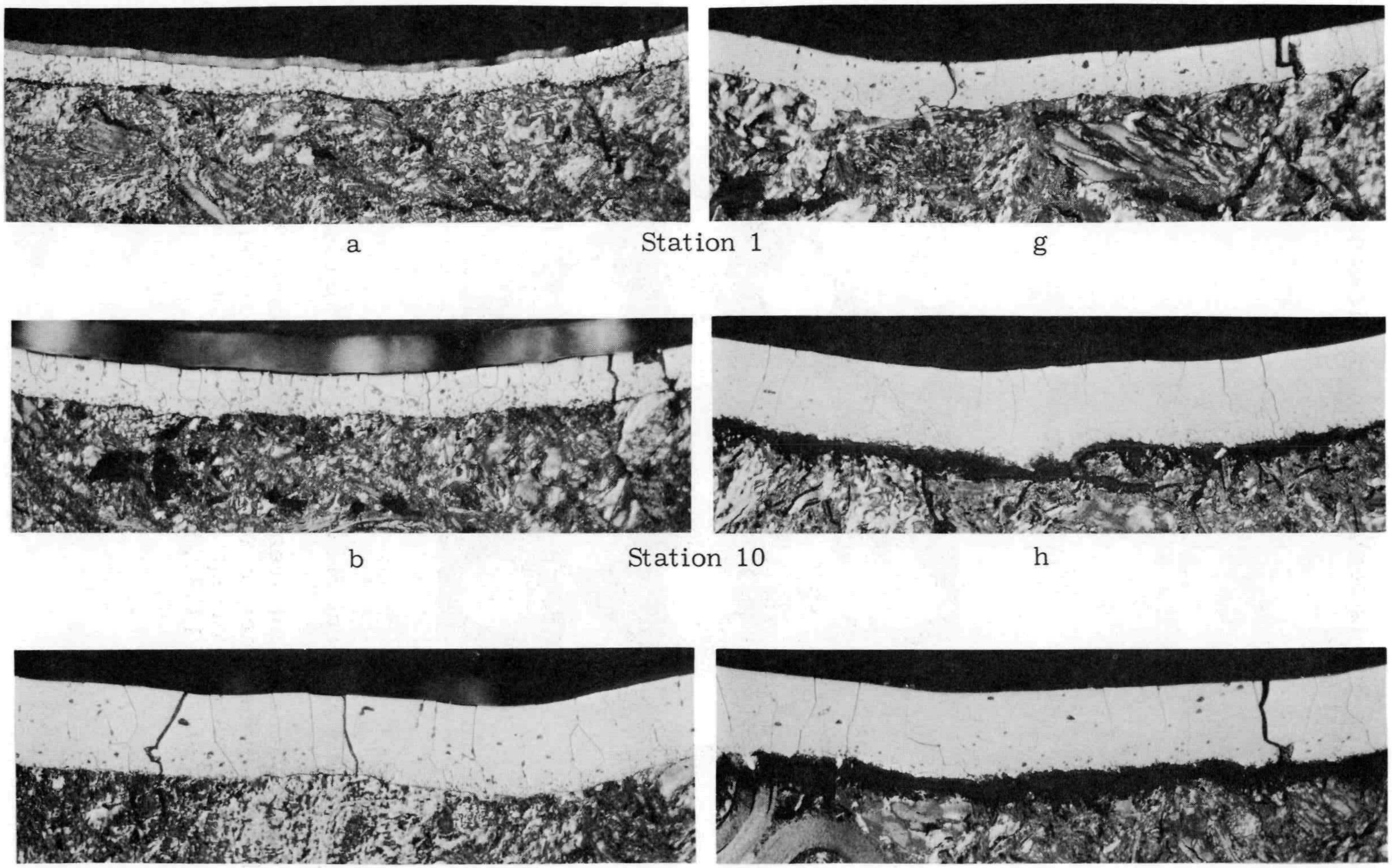

FIGURE 2.

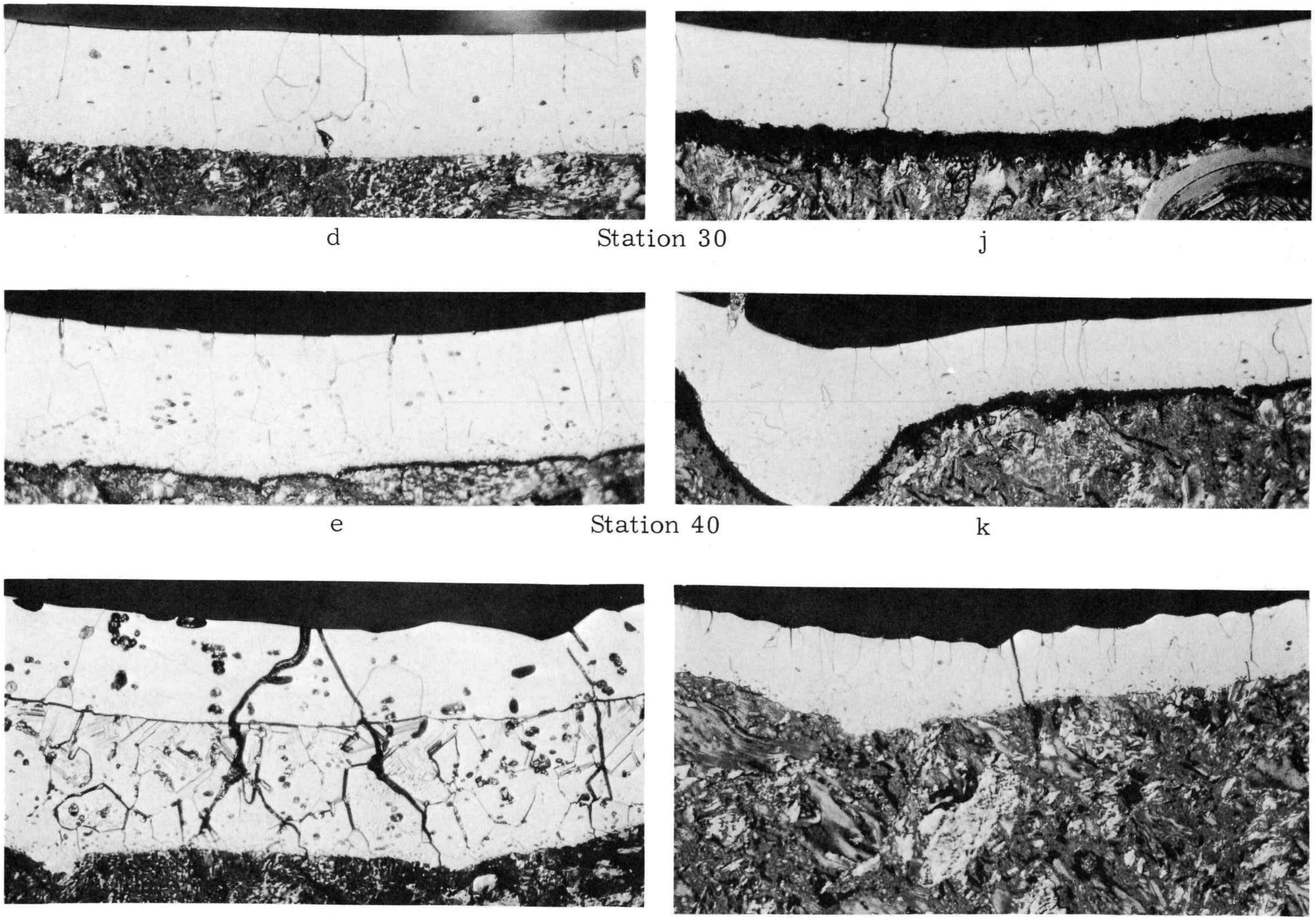

FIGURE 2. (contd.) Figures 2a Through 2f are of Element 1878-AA-264, 2g Through 2l are of Element 287-14181
250X

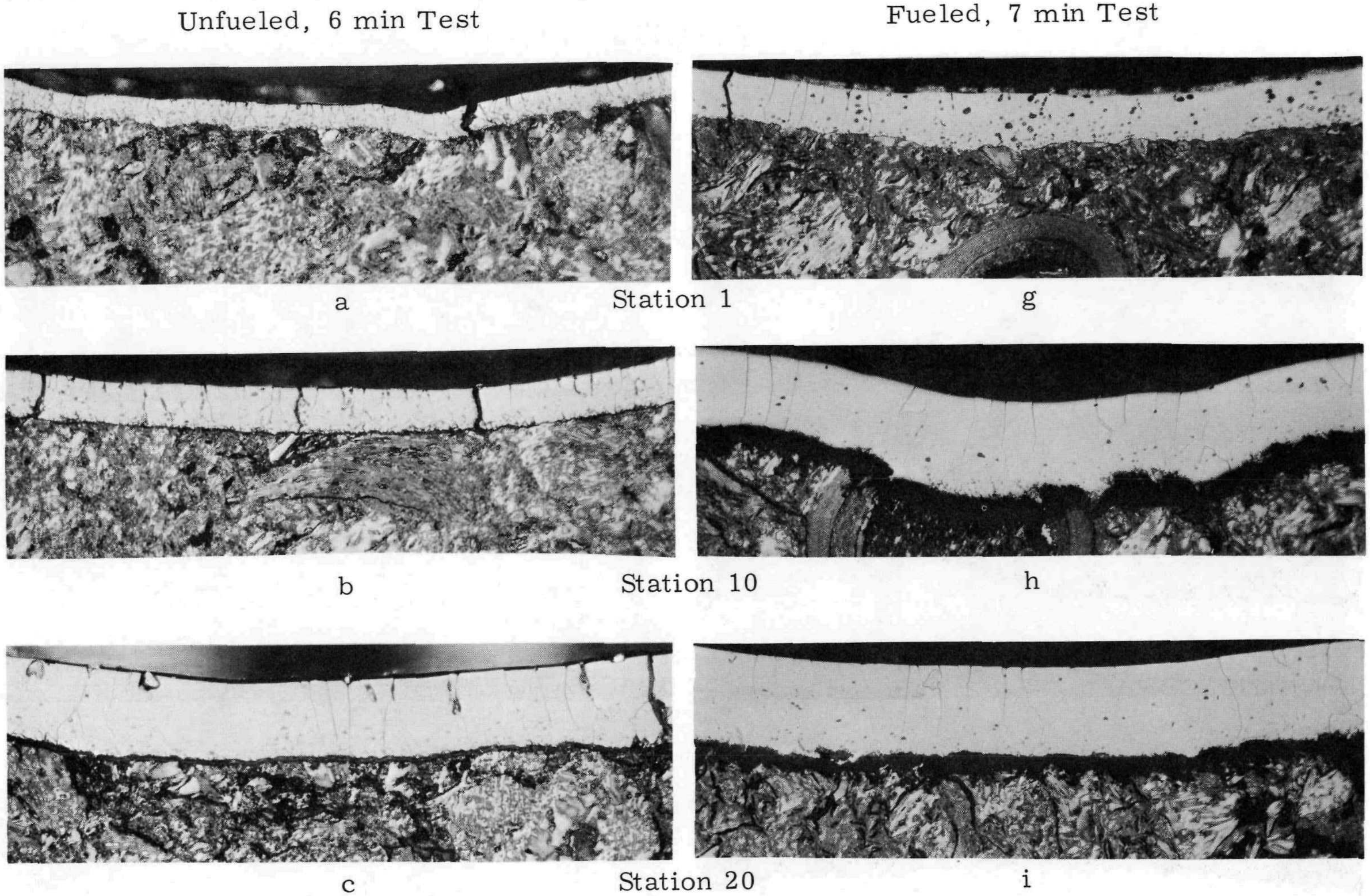
Unfueled, 6 min Test
Fueled, 7 min Test
a
Station 1
g
b
Station 10
h
c
Station 20
i

FIGURE 3.

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Unfueled, 6 min Test

Fueled, 7 min Test

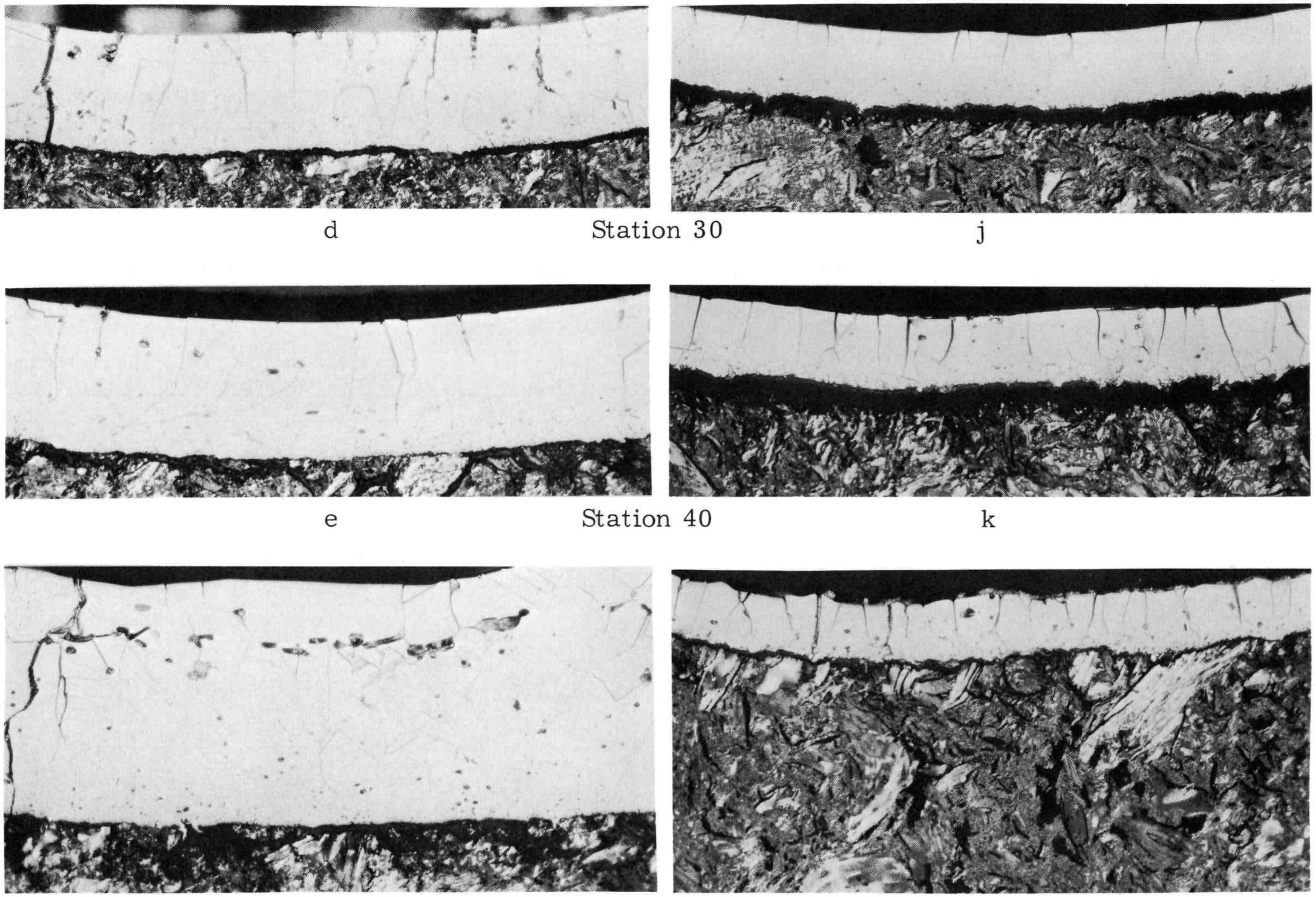

FIGURE 3. (contd.) Figures 3a Through 3f are Element 1878-AA-220, 3g Through 3l are of Element 287-14161 250X

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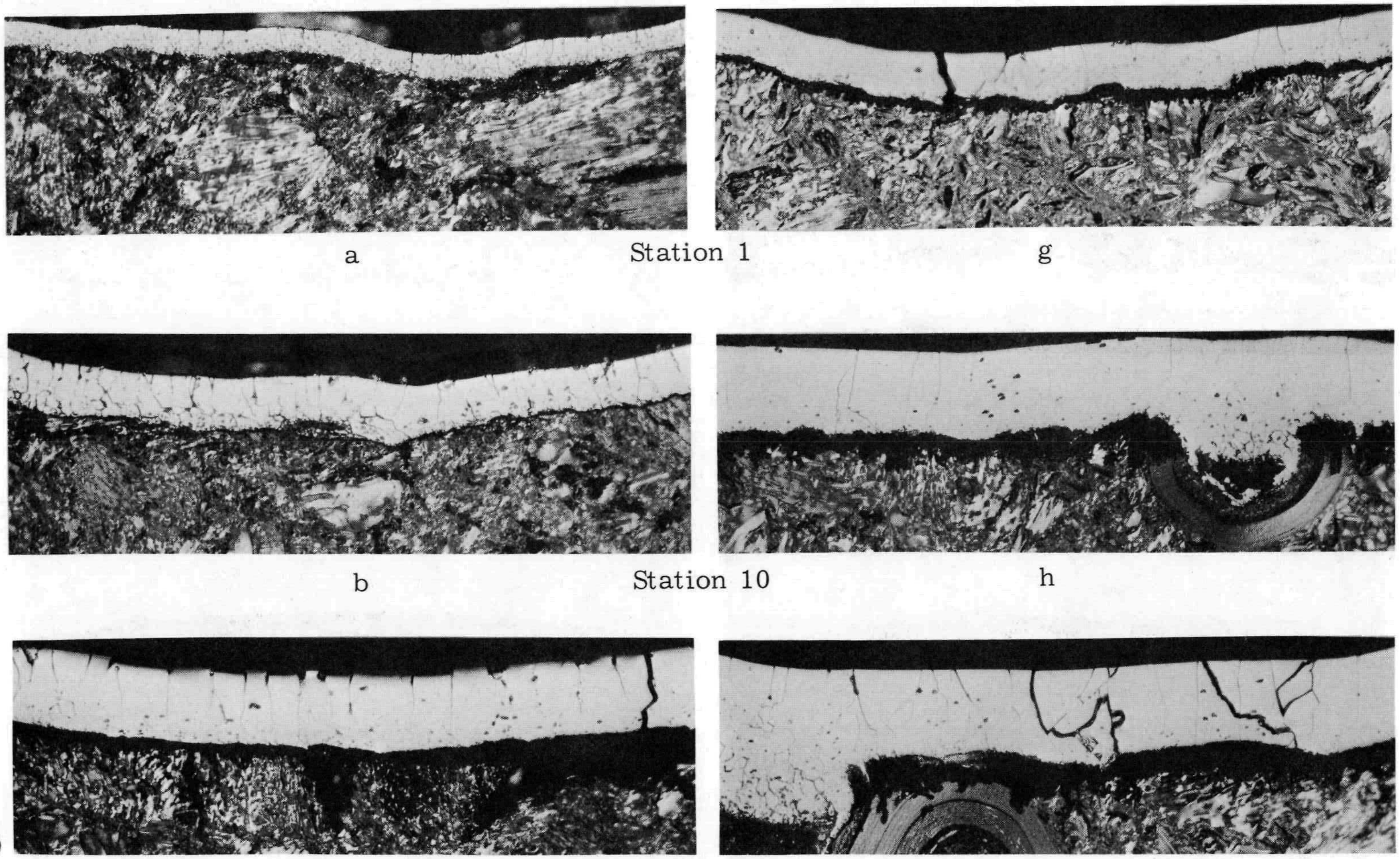
Unfueled, 30 min Test

Fueled, 20 min Test



a Station 1 g

b Station 10 h

c Station 20 i

FIGURE 4.

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Unfueled, 30 min Test   Fueled, 20 min Test

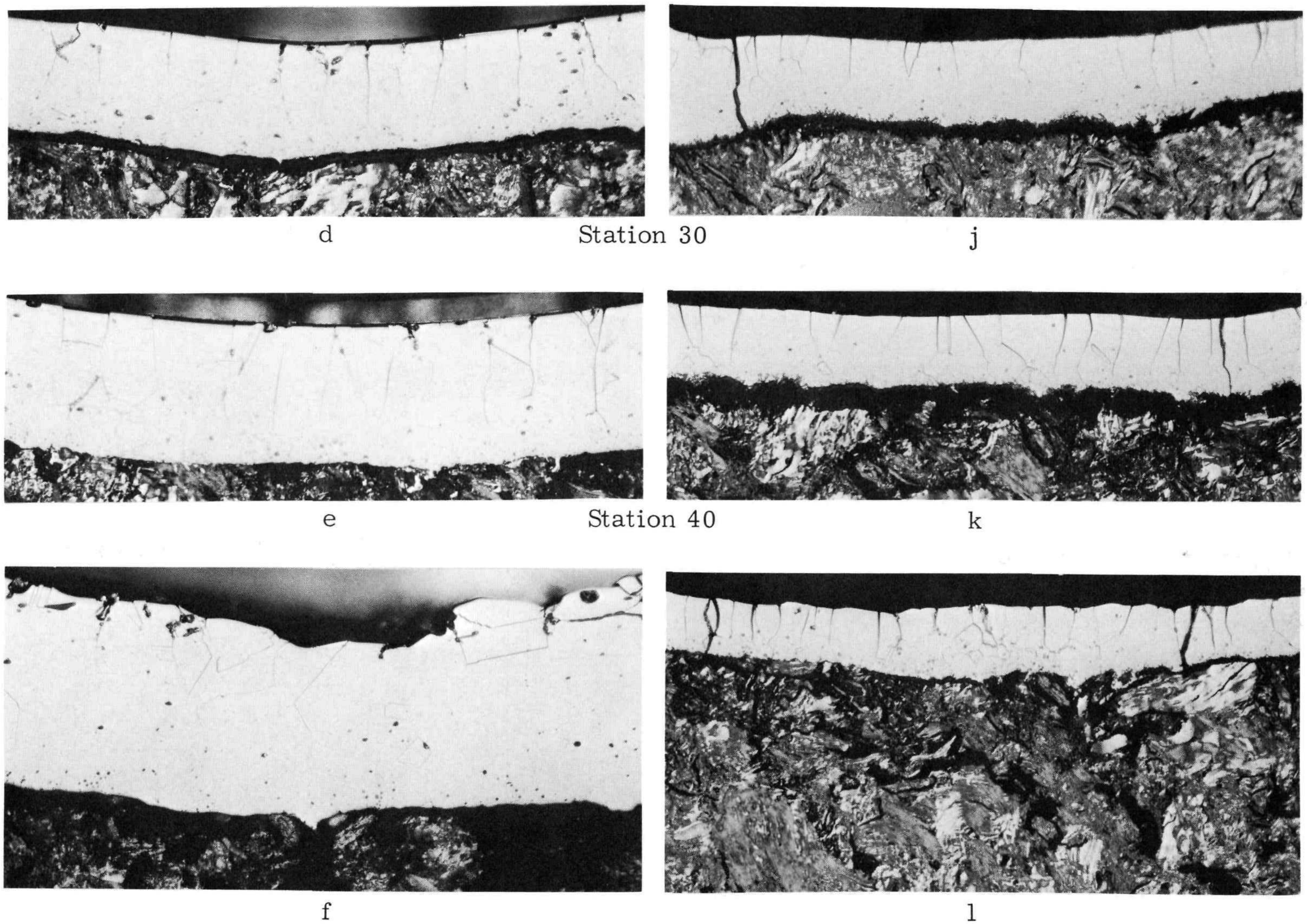

FIGURE 4. (contd.) Figures 4a Through 4f are of Element 1878-AA-244, 4g Through 4l are of Element 287-14164
250X

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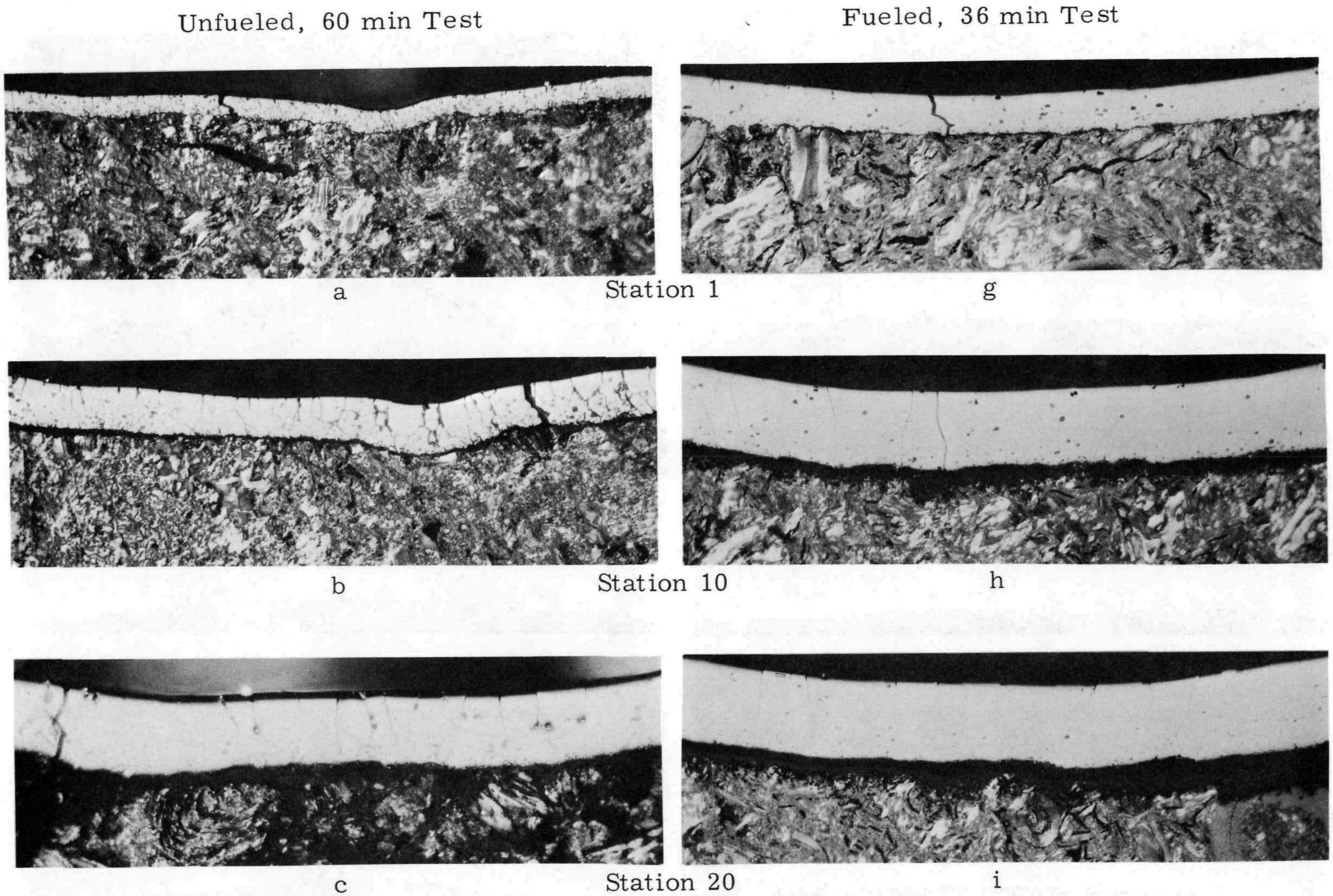
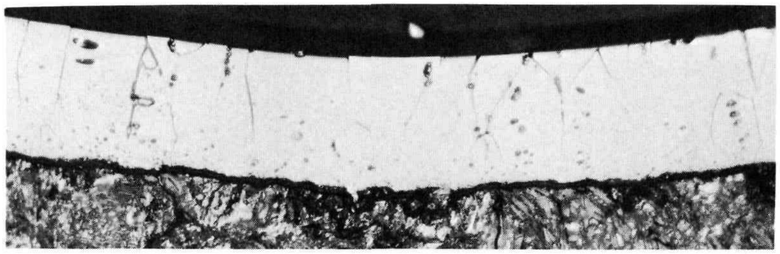
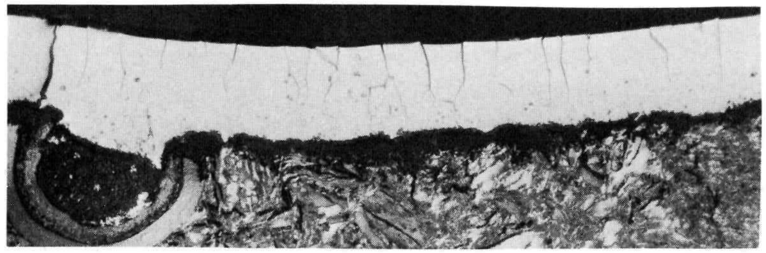
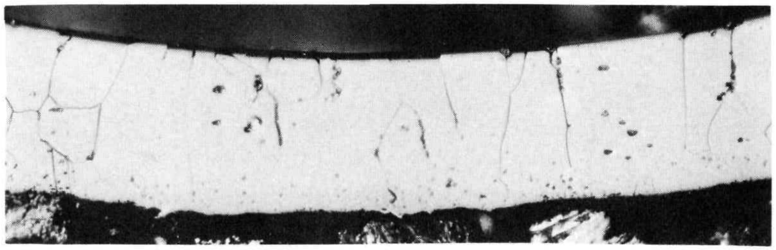
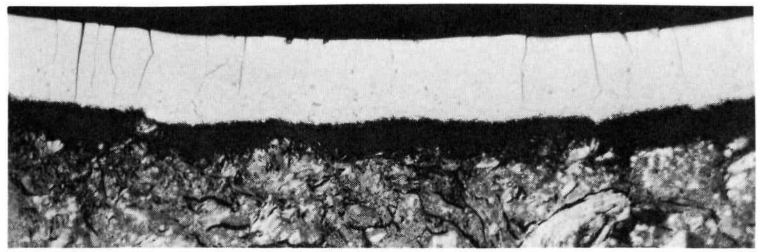

Unfueled, 60 min Test
Fueled, 36 min Test
a
Station 1
g
b
Station 10
h
c
Station 20
i

FIGURE 5.

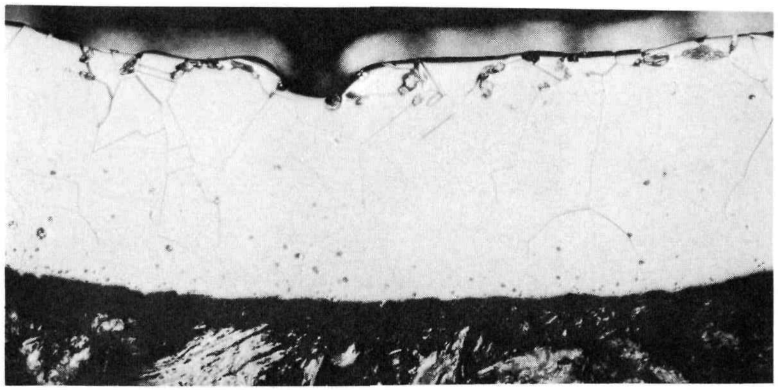
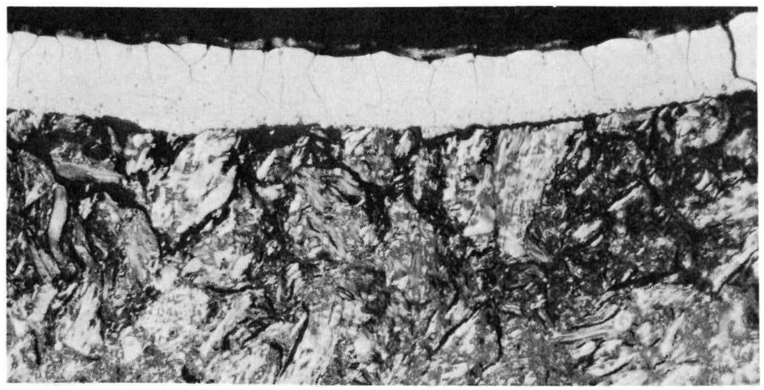
Unfueled, 60 min Test | Fueled, 36 min Test

d Station 30 j

e Station 40 k

f Station 52 l

FIGURE 5. (contd.) Figures 5a Through 5f are of Element 1878-AA-265, 5g Through 5l are of Element 287-14170
250X

MICRORADIOGRAPHY OF ROVER ELEMENTS - L. R. Bunnell

The microradiographic techniques described in the previous report[1] have been refined. The modifications developed over the past few months are:

- Use of a photographic emulsion with higher contrast and very small grain size. The film now in use is capable of magnification to 500X with useful retention of detail.
- Modification of thinning techniques to allow the preparation of samples approximately 0.006 in. thick with surfaces quite highly polished and free of surface uranium-carbide contamination.

For this purpose of evaluating microradiography as a means of examining the ROVER elements, the specimens discussed in the previous report[1] were thinned from 0.020 to 0.006 in. and radiographed. Since previous work had given an indication of the quantity of fuel leaving the beads in various stages of processing, the most recent studies were intended to evaluate the applicability of microradiography in detecting small-order fuel migrations. Figure 6 is one of the microradiographs which was used to evaluate the applicability of the technique. This and other radiographs demonstrate that fuel migrations of small order can be detected; and radiography appears to be a promising technique.

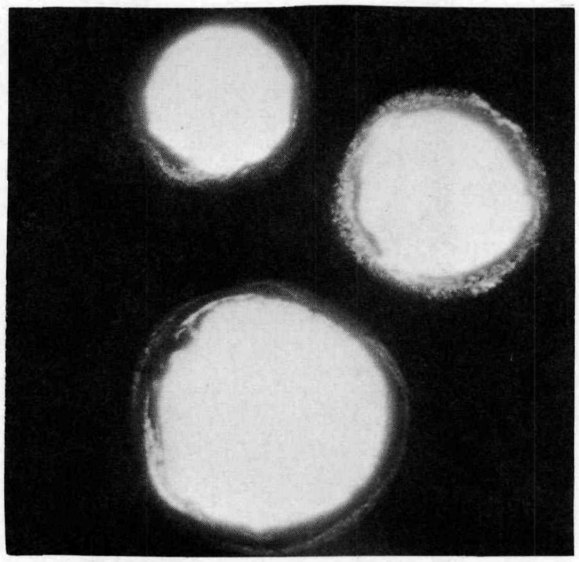


FIGURE 6. Microradiograph of Section of Element 27-408-13114 (Fueled, Coated, Untested) Showing Small-Order Fuel Migration
Section Thickness = 0.008 in.
250X

(1) Quarterly Progress Report on Rover Graphite Studies, October, November, December, 1965, BNWL-212. (Confidential)

Examination of radiographs of the unfueled element of the samples discussed in the previous section showed the presence of an NbC rooting system at the NbC-graphite interface. These roots are seen at 250X magnification in Figures 7 and 8, which are microradiographs of opposite ends of an unfueled, untested fuel element, No. 1878-AA-264. Note the fine structure of the root tips. Very few of these roots are observed by conventional optical microscopy, and those which are observed have not been as extensive as found by radiography. This may be due to the tips being broken off in polishing or their low reflectivity may make the tips impossible to view. This rooting system may be of importance in coating adherence since preliminary observations have noted a lack of this rooting system in the fueled elements.

The current work includes microradiography of the fueled elements with emphasis on the bore coating-graphite interface.

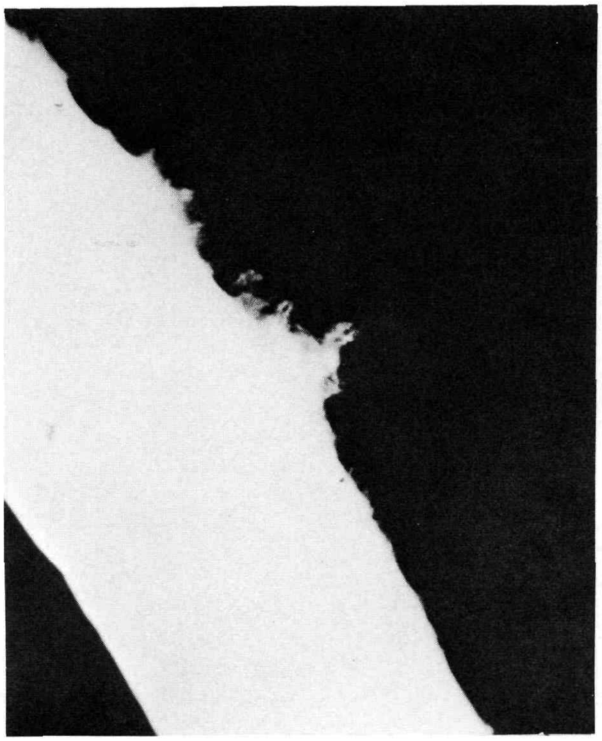


FIGURE 7. Microradiograph of NbC Bore Coating Near the Coater Inlet Showing Extensive Rooting Section Thickness: 0.008 in.

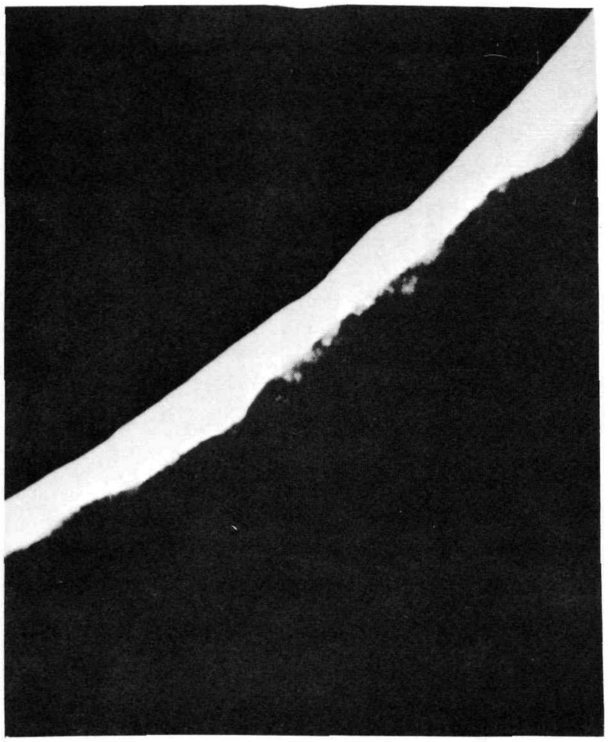


FIGURE 8. Microradiograph of NbC Bore Coating Near the Coater Outlet Showing Less Rooting Section Thickness: 0.008 in.

BASIC FUELED-GRAPHITE STUDIES - C. E. McNeilly

A brief investigation of the nitriding of NbC_{1-x} compositions was undertaken to determine the effect of nitriding upon various properties of interest to ROVER coatings. Samples with nitrogen composition as high as $NbC_{0.75}N_{0.19}$ were easily produced. The effect on thermal expansion and melting point was negligible. All other high-temperature tests indicate that the nitrogen is driven off within one-half hour at 2200 °C at 1 atm pressure, so work along these lines has been discontinued.

The exhaust fume hood for the coating apparatus has been delivered and is being installed. The only other item necessary for the coating work is the fixture to hold the 50-cm-long fuel element sections in the furnace. This item is under construction.

The range of possible values of the various parameters included in the coater can be summarized as follows:

$NbCl_5$ delivery rate	- 1 g/hr to 1.0 Kg/hr (anticipated normal use - 100 g/hr)
$H/NbCl_5$ ratio	- 1.6×10^{-3} to 6×10^{3} (anticipated normal value - ~0.5)
%$NbCl_5$ in atmosphere	- 5×10^{-4} to 55% (anticipated normal value - 1 to 3%)
HCl%	- 0 to 90 % (anticipated normal value - 10 to 20%)

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