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HW-84556

MASTER

QUARTERLY PROGRESS REPORT
ROVER GRAPHITE STUDIES
JULY, AUGUST, SEPTEMBER, 1964

THE STAFF OF REACTOR AND FUELS LABORATORY

OCTOBER 15, 1964

HANFORD LABORATORIES

HANFORD ATOMIC PRODUCTS OPERATION

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C-91, Nuclear Reactors
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By

The Staff of Reactor and Fuels Laboratory

October 15, 1964

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Migration Studies of Uranium Through Pyrolytic Carbon - C. E. McNeilly

The investigation of uranium migration through pyrolytic carbon coatings on uranium dicarbide particles has been continued using hot stage reflection electron miccroscopy. In order to overcome the problem of loss of image quality in the microscope due to evaporation of the graphite matrix, several specimens were prepared by pneumatically impacting a dispersion of coated particles in various refractory metal powders. The particles in the tungsten matrix cracked on impaction; however, those in the tantalum, niobium, and rhenium matrices appeared to be intact.

Because of the severe work hardening of rhenium during polishing, it has been impossible to obtain a satisfactory sample for examination. Time lapse motion pictures have, however, been obtained for tantalum and niobium matrix samples.

No reaction between UC_2 and the PyC coating was observed in either case, although it was quite apparent that the PyC coating on the particles in the niobium matrix evaporated at a much faster rate than those in a tantalum matrix.

The most recent work has been directed towards a better understanding of the formation and adherence of the NbC coating used on the Rover graphite materials. Since coatings can be successfully applied to unfueled, but not fueled graphite, hot stage microscopy is being used to detect any difference in the two materials. Time lapse motion pictures of fueled and unfueled specimens containing a transparent niobium coating, have been taken and do show some differences. Similar pictures are being obtained on uncoated samples and comparisons between the four films will be made.

Niobium Carbide Coated Graphite Studies

X-ray Investigation - L. D. Johnson

This X-ray investigation consisted of a crystallite orientation study of the fueled and unfueled graphites (Samples 45-2181 and 01-1685-105, respectively) and an examination of the diffraction patterns of the NbC coatings on the graphite.

The crystallite orientations were found by X-ray transmission using the Norelco type 52495 pole figure device. The average crystallite orientation, θ, was calculated from the dependences of the (002) diffraction line intensity on sample position and orientation. The two transverse directions were assumed to have equal concentrations of c axes. The relative crystallite orientation is 0.22 and 0.28, for the unfueled and fueled graphites, respectively. The unfueled sample was therefore significantly more anisotropic than the fueled graphite.

An attempt was made to determine the particle orientations in the NbC coatings by X-ray reflection, but the rough surface texture and high X-ray absorption cross-section of the coatings made this analysis unfeasible.

Reactor Rates - R. L. Gibby

A study of the reaction between niobium and graphite has been initiated to determine why high quality niobium carbide coatings can be routinely applied to reactor grade graphite and not to graphite containing PyC coated UC_2 fuel particles.

Experimental techniques are:

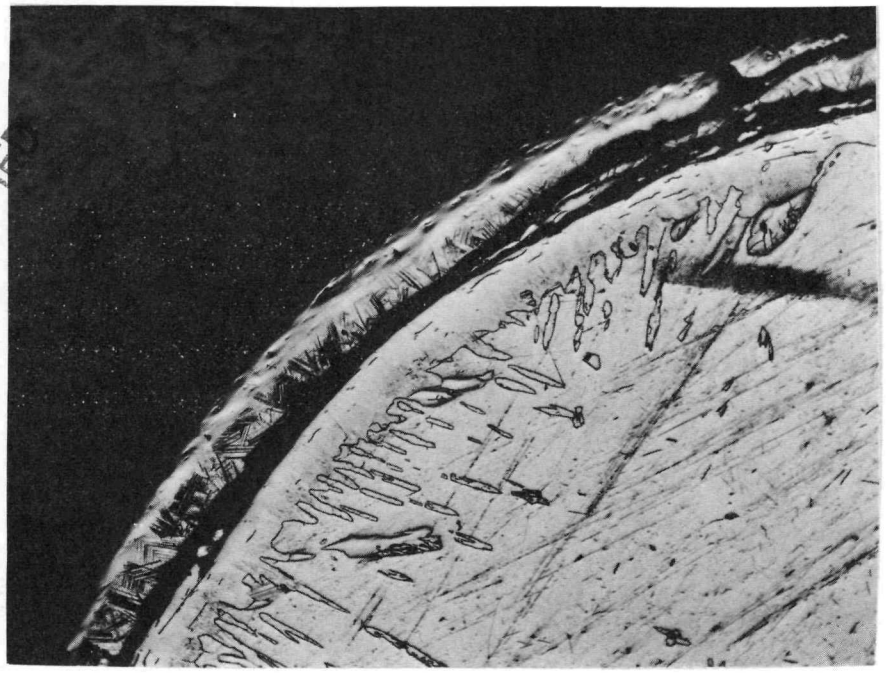
1. press fitting a small niobium pin into a graphite sleeve (fueled or unfueled) forming a diffusion couple
2. soaking the couple at an elevated temperature for a measured time
3. examining the reaction zone with X-ray diffraction, metallography and microhardness measurements

At present, four samples have been reacted at two different temperatures. Two samples were heated for 4 hr at 1600 C, while two other samples were heated 4 hr at 1900 C. In each case, one sample contained fueled graphite so that a comparison could be made between the reaction rates of niobium and fueled and unfueled graphite.

Reactions occurred in all four specimens but were more limited at the lower temperature. At the circumference of the niobium pins two distinct reaction products were observed (Figure 1). Microhardness measurements, with a Knoop indentor and a 50 g load, indicated that both phases had essentially the same hardness, with values between 1350 and 1780. The niobium metal, on the other hand, tested under identical conditions, had a Knoop Hardness of 96. In some cases, reaction products were located at niobium grain boundaries (Figure 2). Knoop hardness measurements gave values of 1350 to 1780 for these reaction products.

At both temperatures there appeared to be no apparent difference in the reaction rates of niobium with fueled and unfueled graphite. Although the reaction products have not yet been identified, it is highly probable that they are Nb_2C and NbC, since these are the only reported carbides of niobium.

In continuing envestigations a polished flat diffusion couple is to be used in conjunction with inert boundary markers.



100X

FIGURE 1

Reaction Zone at Circumference
of Niobium Pin Heated 4 hr at 1900 C

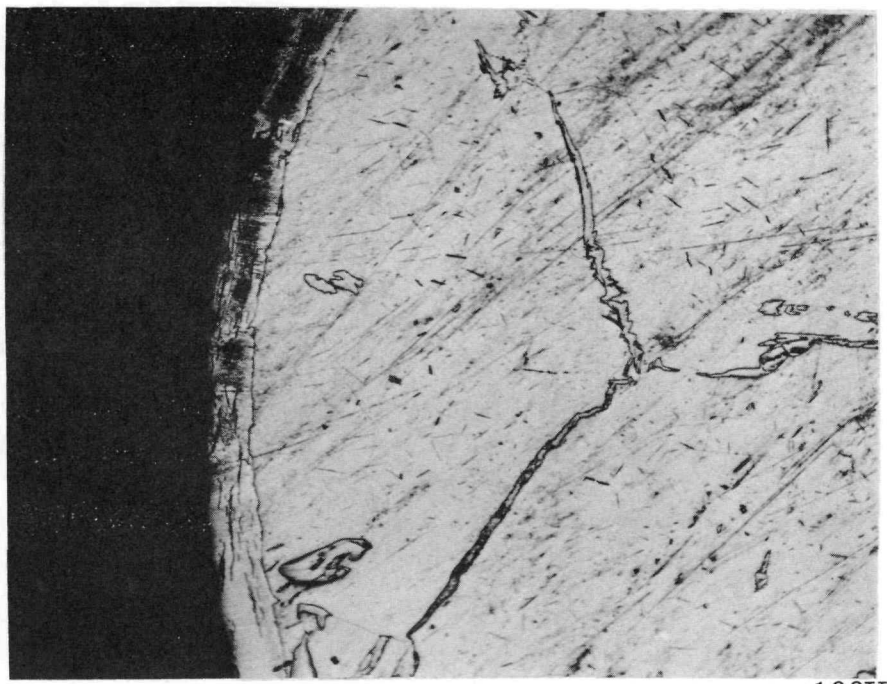


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FIGURE 2

Reaction Products in Niobium
Grain Boundaries After 4 hr at 1900 C

Neg. 441, 442

Magnetic Force Welding of Graphite[1] - C. H. Shaw

Feasibility studies on magnetic force welding of fueled to unfueled NASA Rover graphite elements have shown that at least partial bonding can be accomplished. Figure 3 is illustrative of the maximum bonding achieved to date.

Evaluation of most joints produced to date has been by metallographic techniques using the sensitive-tint photographic process on those joints of particular interest. Physical testing (tensile strength) of joints has been limited to only a few samples. The samples tested ranged in strength from 100 to 200 psi (based on total joint area), Metallographic examination of tensile samples after fracture, however, showed that the actual diffusion bond areas were quite small (less than 30%) or nonexistent.

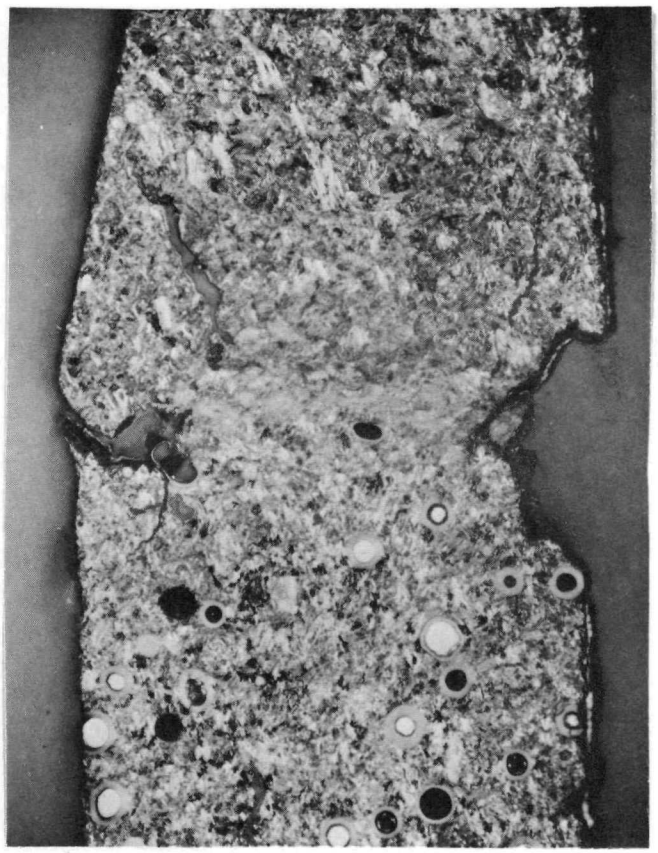

FIGURE 3
NASA Graphite Weld Test Sample

1. Progress Report: Fueled Graphite Studies, April, May, June, 1964, HW-83980. (~~Confidential~~)

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