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PROGRESS REPORT ON FUELED GRAPHITE STUDIES
JANUARY 1965

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February 10, 1965

Los Alamos Scientific Laboratory
P. O. Box 1663
Los Alamos, New Mexico

Attention: Dr. D. P. MacMillan

Gentlemen:

PROGRESS REPORT ON FUELED GRAPHITE STUDIES
JANUARY 1965

Electron Microscopy

A specimen comprising fueled graphite particles dispersed in a niobium matrix was heated and photographed in the reflection stage of the electron microscope. A time lapse motion picture was taken while the sample was being heated at approximately 1900 C for four hours, the same time-temperature conditions previously used in diffusion couple studies.

Resolution in the time lapse pictures is rather poor, but a series of still photos taken after heating reveal considerable detail. Various areas of the sample can be identified as Nb₂C, and HfC, as well as unreacted Nb. The graphite in most cases lost its identity. A second specimen now ready for in-microscope heating will be photographed on high resolution plates during heating to allow selection of the area which appears to offer the most information on the reactions between the Nb and the graphite.

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Replicas of hot-tested fueled and unfueled samples were examined in the electron microscope. The samples were from a series of fueled and unfueled graphites representing as-machined, leached, coated, and hot-tested steps in processing. Observations made during examination of this series by optical microscopy were discussed in the December progress report.

Electron micrographs traversing the NbC bore-coating were assembled for the hot-tested, unfueled sample. Grain boundaries are evident in the NbC near the bore; however, they become less distinct near the center of the 20-30 μ thick coating. Pits and microcracks become more predominant features near the NbC-graphite interface.

Pits frequently take the form of circular patterns having the size and shape of carbon blacks present in graphite binder adjacent to the NbC-graphite interface. NbC in a region adjacent to a graphitized coke filler particle was notably free of circular pit patterns. It is proposed that these features are NbC pseudomorphs after the carbon-black structure, since their outline takes the form of blacks, but internally they appear to be composed of NbC.

Microcracks in the NbC are $<0.05 \mu$ wide. They are concentrated near and roughly parallel to the graphite-NbC interface, but follow a very irregular and discontinuous pattern. A continuous microcrack 0.1 to 0.2 μ wide separates the NbC and the graphite at the interface.

The NbC bore-coating replicated on the hot-tested, fueled sample is relatively free of structural detail; however, the area examined did not include any of the large cracks noted optically and mentioned in the December report. Examination of the graphite matrix revealed evidence of the transformation of binder carbon and carbon blacks to a graphitic structure. Evidence of a similar transformation associated with fuel migration through pyrolytic carbon coatings on fuel particles is well documented. However, in this case, fuel migration continued into the matrix altering binder regions to a point where the characteristically spherical carbon blacks are not distinguishable. The transformed regions are characterized by a mosaic of randomly oriented graphite plates surrounding UC_2 grains which are 2 to 8 μ in diameter. Much of the original graphitized coke structure can still be identified within these regions which suggests that the uranium migration and resulting graphitization progressed preferentially through the less graphitic binder component of the matrix.

Examination of replicas of other samples in the series is in progress.

Magnetic Force Joining

The strength of JOZ graphite and JOZ graphite welds was further evaluated by the direct pull tensile test. Preliminary results indicate that the nominal weld strength is approximately one-fifth that of the parent material.

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Tensile test specimens are shown after fracture in Figure 1. The graphite weld samples were prepared by magnetic force resistance butt welding two solid cylinders, the ends of which were machined to form opposing cone frustums. The reduction in cross section allowed for sufficient pressure and current density during joining, while maintaining a suitable area for reliable strength measurements. The stress concentration effect originating from this "notch" design was evaluated as follows: (1) solid cylinders of JO2 graphite were tested to determine its nominal tensile strength; (2) similar cylinders were machine notched to simulate the welded specimens, and the notch tensile strength determined; and (3) the stress concentration factor, K, was calculated according to the equation:

$$K = \frac{\sigma(\text{nominal})}{\sigma(\text{notch})},$$

where $\sigma(\text{nominal})$ is the mean tensile strength of the graphite cylinders and $\sigma(\text{notch})$ is the mean notch tensile strength of the machined specimens. In this manner, the tensile strength of the graphite weld could be corrected for the notch effect, and directly compared to that of the parent material.

Results are summarized in Table 1. The mean tensile strength of the JO2 graphite cylinders was 2650 psi, as compared to 1960 psi for the machine-notched specimens. The notch induced stress concentration factor, 1.35, was relatively low, as expected from the brittle behavior of graphite. Moreover, this value can be considered maximum (within the limits of the test) when employed as a correction factor for the welded specimens, since the notch depth was significantly greater for the machined samples (0.187 inch vs. 0.136-inch). Applying this factor to the mean notch tensile strength of the welded specimens (380 psi), an average weld strength of 510 psi was obtained. This value is approximately one-fifth the nominal strength of the parent material; the notch tensile strengths of the welded and machined specimens are also related by the same ratio (380 to 1960 psi).

The nominal tensile strength of JO2 graphite found in this study is approximately twice the handbook figure (1200-1400 psi) used in previous tensile strength comparisons, but compares well with recent commercial data (3000 psi).⁽¹⁾ In addition, the reported JO2 graphite weld strength agrees well with that published previously⁽²⁾, even though widely different specimen configurations were employed.

- (1) Great Lakes Carbon Corp., Commercial Data on JO2 Graphite, as published in Materials in Design Engineering, Vol. 61, No. 1, January 1965, p. 24.
- (2) Progress Report on Fueled Graphite Studies, November 1964, edited by F. W. Albaugh, HW-84551 (Confidential).

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The relatively lower strength of the JOZ graphite welds is presumed to be partially due to insufficient bonding during joining and to stress risers inherent in the material or initiated during welding. Any evaluation of the over-all weld strength will necessarily reflect the number, magnitude, and nature of these flaws. Because of the lack of complete weld reproducibility, it could not be established that a metallographically sound weld had the same properties as one that was destructively tested. A more meaningful strength test would be to determine the specific strength of specimens machined from a metallographically sound section of the weld to eliminate or minimize the stress concentration effects. Development of a testing technique, preferably transverse bending, for reliably testing these small and brittle specimens would be required. However, application of a test of this nature, in conjunction with tests involving the entire weld cross section, would provide information on both the maximum achievable weld strength, as well as the extent of achieving this strength.

Now that a higher degree of reproducibility has been achieved in graphite joining, it is planned to extend the testing to Rover materials.

Very truly yours,

W. J. Albaugh
W. J. Albaugh

Manager
Reactor & Materials Technology

FW Albaugh:kb

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Table 1

TENSILE STRENGTH DATA FOR JOZ GRAPHITE AND JOZ GRAPHITE WELDS

Specimen Type ^(b)	No. of Specimens	Notch Tensile Strength (psi) ^(a)			Tensile Strength (psi) ^(a)		
		Range	\bar{y}	α	Range	\bar{y}	α
Cylinder	5				1700-3500	2650	700
Machine- Notched	8	1500-2400	1960	275			
Welded	9	150-500	380	125	200-675 ^(c)	510 ^(c)	170 ^(c)

(a) \bar{y} = arithmetic mean; α = standard deviation

(b) See Figure 1 for specimen configurations.

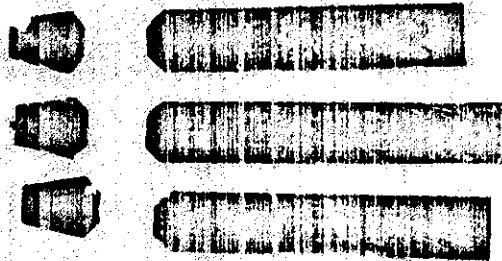
(c) Calculated from $S = KSi$, where S = tensile strength, Si = notch tensile strength, and K = stress concentration factor. K determined from ratio of cylinder tensile strength (2650 psi) to notch tensile strength of machined specimen ($K \approx 1.35$).

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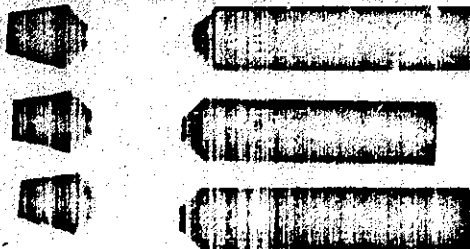


0650253-3

(a)

Magnetic Force Welded Specimens

($D_1 = 0.312$ in., $D_2 = 0.176$ in., 45° Frustum Taper)

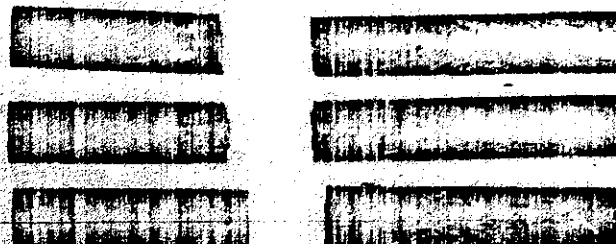
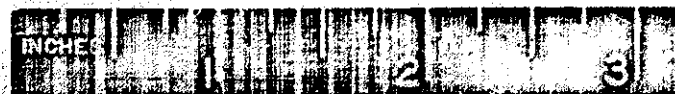


0650253-2

(b)

Machine-Notched Cylinders

($D_1 = 0.312$ in., $D_2 = 0.125$ in., 45° Frustum Taper)



0650253-1

(c)

Graphite Cylinders

($D_1 = 0.312$ in.)

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FIGURE 1

JOZ GRAPHITE TENSILE TEST SPECIMENS (AFTER FRACTURE)

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0650253-3 (a)
Magnetic Force Welded Specimens
($D_1 = 0.312$ in., $D_2 = 0.176$ in., 45° Frustum Taper)

0650253-2 (b)
Machine-Notched Cylinders
($D_1 = 0.312$ in., $D_2 = 0.125$ in., 45° Frustum Taper)

0650253-1 (c)
Graphite Cylinders
($D_1 = 0.312$ in.)

FIGURE 1

JOZ GRAPHITE TENSILE TEST SPECIMENS (AFTER FRACTURE)

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