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Westinghouse Astronuclear Laboratory

CORROSION OF GRAPHITE SUPPORT BLOCKS
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PREPARED BY:

J. T. Lamartine

J. T. Lamartine

GROUP 1

~~EXCLUDED from automatic
downgrading and declassification~~

APPROVED BY:

L. L. France

L. L. France
Supervisory Engineer

Classification cancelled (or changed to _____)

by authority of _____

by *H. F. C.* TIC, date _____

E. L. Layland

E. L. Layland, Manager
Materials Engineering and Specifications

SPECIAL REVIEW
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ABSTRACT

Since graphite reacts extremely rapidly with hydrogen at the temperatures and pressures of reactor operation, the support blocks for the NRX-A2 reactor were protected from hydrogen corrosion by a coating of niobium carbide applied to all exposed surfaces. As a part of an extensive quality control program, a section of one support block from each coating batch was corrosion tested in hydrogen at conditions approximating NRX-A2 operating conditions. This report describes the corrosion testing program and correlates results of these tests with non-destructive and destructive tests such as adherency tests, coating stoichiometry, coating discoloration, coating defects, and others. The results and data presented in this report are confined to the information available at the termination of NRX-A2 support blocks evaluation. Subsequent work yet to be reported has furnished answers to certain of the questions raised as a result of this work.

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INTRODUCTION

One of the crucial areas of the NERVA engine on which the success of operation is directly dependent is the core axial support system. In the NRX-A design, this system is comprised of a tie rod and support block arrangement in which the core is divided into clusters of fuel elements, each of which is supported at the exhaust end by an interlocking support block. Each of these support blocks, in turn, is tied to the core support plate at the inlet end of the core by one or more tie rods that pass through accommodating unfueled elements in the fuel clusters.

Design demands dictate that each block must independently support the elements clustered on it while operating in the high-pressure, high-temperature hydrogen environment of the reactor exhaust that passes through its flow channels. For the NRX-A series of reactors, ATJ graphite was chosen as the support block material. Since graphite reacts extremely rapidly with hydrogen at the temperatures and pressures of reactor operation, the support blocks are protected from hydrogen corrosion by a coating of niobium carbide applied to all exposed surfaces. The importance of this coating cannot be over emphasized. To fulfill its protective function, it must be of adequate thickness, continuous, defect free, and adherent to the graphite substrate. Above all, it must be able to withstand the high-temperature, high-pressure hydrogen reactor environment without allowing attack of the underlying structural graphite.

Support blocks for the A-2 reactor were coated with NbC at the Westinghouse Astrofuel Facility located at Cheswick, Pennsylvania. Regular support blocks were coated, eight at one time in two tiers of four, in the furnace designated as "B". The irregular periphery blocks, types J3 through J9 were coated five at one time in furnace "A"; a J4, J5, and J8 in the top tier and a J3 or J9 and a J6 or J7 in the bottom tier.

Experience with NbC coated fuel elements has indicated that the protective nature of the coating when exposed to reactor environmental conditions cannot always be predicted by pre-test

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coating property measurements.^{1, 2} This observed variability in coating behavior coupled with a marked lack of knowledge about the coating deposited on unfueled graphite prompted the imposition of an extensive quality control program on NbC-coated support blocks for the NRX-A2 reactor.

Briefly, these requirements, which applied to both regular and irregular blocks, were as follows: (3)

1. One block from each coating furnace lot was destructively evaluated for coating thickness profile.
2. At least one hexagonal lobe from the destructive sample from each lot was corrosion tested in high-temperature, high-pressure hydrogen for five minutes in conjunction with the IA fuel element corrosion test.⁴
3. All coated blocks were visually examined for coating defects.
4. All coated blocks were given a tape test to determine coating adherency.
5. All coated blocks were radiographed through the end and sides to assure coating continuity and to detect internal flaws.
6. Prior to reactor hardware production, both furnaces were qualified by subjecting one entire furnace lot of blocks to the tests outlined in Items 1 through 5.

Over the entire A-2 block production run, approximately 320 coated regular support blocks and about 60 coated irregular support blocks were evaluated. This memorandum describes the corrosion testing program and correlates results of these tests with non-destructive and destructive test data.

DESCRIPTION OF CORROSION TEST

To ascertain the behavior of the NbC protective coating when exposed to the high-pressure, high-temperature hydrogen environment expected to exist in an operating reactor, sections of coated support blocks were tested in the hydrogen exhaust stream of the high-

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temperature fuel element corrosion test apparatus. In this test, a fuel element is placed in a pressure vessel and is self-resistance heated to a very high temperature. High pressure hydrogen gas is blown through the element coolant holes to test the corrosion resistance of the element bore coating. As a result of passing through the heated element, the exhaust hydrogen is heated to temperatures approaching the element operating temperature.

The corrosion resistance of coated support blocks was evaluated in this hot gas stream. Because of space limitations in the exhaust plenum of the furnace and a test hydrogen flow rate equivalent to that through only one 19-hole segment of a support block, whole support blocks could not be conveniently and meaningfully tested. Therefore, 19-hole, one sixth hex segments of a support block were used for the test sample. The lobe sample is illustrated in Figure 1. From the figure it can be seen that the test segment was the full height of the support block and therefore possessed the top and bottom end coating. Three of the six hex sides were coated and three were bare graphite as a result of sectioning.

The graphite holder that adopted the block lobe sample to the exhaust fixture is shown in Figure 2. Two support schemes for supporting the block have been used. In the first, Figure 2A, the block sample was supported at the interlock step by a ledge in the holder. In the other scheme, the block was held in place by a coated end stop of the design illustrated in Figure 2B with the ledge acting as a redundant support member.

This sample holder was inserted into the element exhaust chuck, as shown in Figure 3, in a manner such that the top face (inlet) of the block was in contact with the exhaust end face of the element. The chuck assembly was secured in the down stream electrode by the diffuser tube.

The prime use of the hydrogen test facility during this study was to define the corrosion behavior of production fuel element lots. As defined by specification,^{4,5} these tests were rigidly controlled and fell into two classes, the IA and IC tests. Since almost exclusive use was made of

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the furnace for these tests, support block testing was restricted, for the most part, to these conditions. Test conditions, as outlined in the specification, are presented in Table I.

Although the element operated at rather precise and measured temperatures, little has been ascertained about the temperature at which the support block section operated, since the exhaust plenum was not equipped with instrumentation or sight ports. Gas flow rates and pressures were measurable and were as indicated in Table I. On the basis of one experiment in which thermal capsule wires were incorporated into both the support block specimen and specimen holder, it was determined that in the IA element test the upstream end of the block achieved a temperature between 2100 and 2195°C. The exhaust end of the block reached temperatures between 2000°C and 2100°C. The degree of confidence in these values, however, is low since in this particular test the element failed after 1 minute and 45 seconds of operation, thus introducing the possibility of a temperature excursion just prior to failure.

DISCUSSION OF RESULTS

During the course of this study, in excess of 75 coated support block samples were corrosion tested. The majority of these tests were run for 5 minutes at the IA condition. A limited number were run at the IC condition and several at temperatures in excess of the IA temperature. The effects of hot hydrogen on the support block coating, as defined by these tests, are presented in the following sections.

General Behavior

In general, after testing, the NbC coating was bright, clean, and free from defects other than those resulting from chips present prior to the corrosion test or from hydrogen attack on the three uncoated hex sides. In instances where the fuel element under test with the block sample failed, the block sample usually exhibited unpredictable behavior. For the most part, block sections tested with elements that exhibited gross failure also failed. Failure

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was typically by severe attack at the upstream end interface and was accompanied by the build-up of foreign black deposits on the block coating. In other cases where element failure occurred, droplets of materials that have been identified as containing UC and Nb were observed to have collected on the downstream end face of the block.

The causes of block failures accompanying element failures are difficult to evaluate. On one hand they might have been caused by the release of uranium from the fuel element which in turn reacted with the NbC coating on the block to form an alloy that melted at a temperature below the test temperature. Certainly, the existence of the UC-Nb droplets on corrosion tested blocks indicates that melting of the fuel had occurred. On the other hand, both block failure and fuel melting might have been caused by severe arcing that occurred when element corrosion progresses to the point where the electrical heating circuit became interrupted. Whatever the cause, the possibility exists that an element failure in an operating reactor might in time precipitate a support block failure. Resolutions to the problem are difficult due to the nature of the corrosion apparatus and the unpredictability of element failure. More work in this area is clearly required. The degree of dependence of block behavior on element behavior, test apparatus and conditions must certainly be considered. The results may not be typical of reactor conditions.

Excluding block failures caused by element failures, all except one of the samples tested survived the five-minute corrosion test, (either IA (2280°C) or IC (2120°C) with no apparent signs of the coating having been attacked by the hot hydrogen. The exception was one section from a badly eroded irregular block that definitely failed the IA test, as a result of coating inadequacy. Erosion during the coating process is thought to be associated with a reaction between HCL or Cl in the coating gas stream and deposited NbC with the result that coating is slowly etched away (this generally occurs on the inlet surface). A photograph of this section after test is presented in Figure 4. Failure of the coating occurred on the upstream end face adjacent to the coated exterior surfaces. Prior to test, the coating appeared to be extremely thin in this area as a result of erosion during the coating process. Coating thickness measurements made on a section immediately adjacent to the corrosion tested section showed that the coating on the end face was on the order of 0.8 mils

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thick near the side walls tapering to about 2 mils thickness in the center of the face. It is postulated that failure resulted from an inadequate thickness of the NbC protective coating. This postulate was substantiated by corrosion testing a section from the same support block on which the minimum coating thickness was on the order of 1.2 mils. This section survived the five-minute IA test with definitely no signs of attack. The postulate is further substantiated by the fact that on the failed section, attack was confined to the outside edge of the end plane where the coating was thinnest. No damage was observed on any surfaces where the coating thickness was greater than 1.2 mils. These limited observations indicate that under conditions approximating the IA corrosion test conditions, the protective value of the NbC coating becomes questionable between 0.8 and 1.2 mils thickness for at least eroded coatings and possibly for all coating on support blocks. The minimum measured coating thickness on all of the other blocks tested was in excess of 1.2 mils and a specification minimum of 2.0 mils was established for reactor components.

Effect of Coating Defects on Corrosion Behavior

Intuitively, it was felt that discontinuities (chips, etc.) in the protective NbC coating will compromise the ability of the support blocks to successfully withstand the operating environment. Experiments in this area have confirmed this premise and have suggested that the degree of attack is dependent upon the size of the defect, location of the defect, and operating time and temperature.

During the course of quality control corrosion testing, block segments embodying defects of several sizes and locations were evaluated. All of the defects selected for evaluation were on the periphery of the upstream end face either on the flow channel wall or between flow holes (see Figures 5 and 6). Defect sizes ranged from less than $1/32$ " max. dimension to about $1/8$ " max. dimension. All of these tests were run at the IA test condition, i.e., five minutes in gas stream that passed through an element operating at 2280°C surface temperature. Post-test examination revealed that gross corrosion attack was extremely limited when the defect size was less than $1/32$ " max. dimension regardless of location. Between $1/32$ " and $1/16$ " original defect

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size, the affected area was somewhat larger and appeared to be more severe when located between holes than it was when located on the channel wall.

Post-test photographs of a typical sample containing an approximately 1/16" defect (Figure 6) is presented in Figure 7. Examination of Figure 7 shows that although the defect was enlarged slightly by the corrosive gas, attack was by no means extensive. A void or cavity under the defect approximately 1/16" deep was formed by reaction of the graphite with the hydrogen.

Results of an experiment designed to define the extent of attack resulting from defects on the top and bottom end planes of support blocks have shown similar undercutting. In this experiment, four intentionally defected samples were corrosion tested for five minutes in the IA test. Two of the samples had an intentional 0.050" x 0.025" defect adjacent to the center hole on the upstream end plane and two had similar defects adjacent to the center hole on the downstream end plane. See Figures 8 and 9. After testing, none of the original defects in the coating were found to have increased in size; see Figure 10. Undercutting of the graphite, however, occurred in all cases to approximately 1/8" dia. x 1/8" deep, substantiating previous observations. See Figure 11.

The occurrence of this undercutting, coupled with visual and metallographic examination of tested defected samples, suggests a mechanism for progressive attack at coating defect sites. Reaction of hot hydrogen with the exposed graphite creates a void. As the reaction continues, the inert coating is undermined, leaving it free standing. When this undermining progresses to the point at which forces on the cracked coating exceed its strength, the coating collapses, thus exposing an even greater area of unprotected graphite to attack and subsequent undermining. Through alternate undercutting and coating collapse, damage to the support block should progress as a function of the circumference of defect area. Assuming this mechanism of attack prevails, it follows that the rate and magnitude of attack will be strongly dependent on the operating temperature and gas pressure, since both of these parameters influence graphite corrosion rates and thus, the rate of undercutting. In addition, the strength, adherence to the graphite, and structural integrity of the coating should all influence the rate of attack at the defect sites. Reduction in any of these properties should cause

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the undercut coating to fall away prematurely resulting in more rapid enlargement of the area of exposed graphite to the gas stream.

These particular properties of deposited niobium carbide are difficult to define. By tape test, coatings may be determined to be either adherent or non-adherent on a gross basis without benefit of ability to interpolate varying degrees of adherency. Definition of inherent coating integrity and strength presents a formidable, if not impossible, task. Coupling these areas of ignorance with uncertainties as to the actual temperature, flow rate, and composition of the hydrogen gas stream in the corrosion test, it becomes apparent that quantitative predictions of the effect of coating defects in reactor hardware are not possible at present.

Non-Adherent Coatings

The adherence of the coating on the top face, the bottom face, and sides of every NRX-A2 support block was evaluated via a tape test. In this test, masking tape (of controlled origin, date, etc.) was applied to the coated surface, rubbed to promote maximum adherency, and then rapidly stripped away. Any observed coating removed was deemed cause for rejection.

During the course of the A-2 support block coating campaign, over 380 blocks were tape tested. Of these, two blocks were rejected for non-adherency. Both of these blocks were of regular type configuration, and were coated in the same furnace run, one in the top tier and one in the bottom tier. The other six blocks in the furnace run passed the adherence test with no coating removal.

Metallographic examination of a section through one of these blocks showed that a definite gap existed at the interface between the graphite and niobium carbide. Furthermore, fracture of NbC fingers or roots that penetrate into porosity in the graphite at the clad-graphite interface was observed. These observations are illustrated in Figure 12. For comparison, a photomicrograph of a section through a block with an adherent coating is presented in Figure 13.

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Another property observed to be unique to the non-adherent coatings was a marked difference in the surface crack network inherent to NbC deposits made on grades of graphites that have low coefficients of thermal expansion. As illustrated in Figure 14, the crack network on a typical adherent coating is extremely fine and closely spaced. As shown in Figure 15, the crack network on the non-adherent coatings was observed to be much more coarse and more widely spaced. These cracks are formed after coating, during cooling from the coating temperature, due to tensile forces that are generated in the coating because its coefficient of thermal expansion or contraction is greater than that of ATJ graphite. The occurrence of the coarse, widely spaced cracks in the non-adherent coatings suggests that the coating was non-adherent or only partially adherent as deposited; therefore, shrinkage during cooling was not as strongly restrained by an interface bond. The exact nature of the interface bond between the base graphite and NbC coating remains to be adequately defined. Sections from the two blocks defined by the tape test as having non-adherent coating behaved extremely poorly when tested in the IA corrosion test. As shown in Figure 16, loss of coating from a non-adherently coated block was extensive after the five-minute test and attack of the graphite was extremely severe.

Coating Adherence after Corrosion Test

As a followup to the quality control tape test, each of the corrosion tested samples was similarly tested to define the adherence of the protective coating after exposure to the five-minute IA corrosion test. Prior to the corrosion test, all samples were found to have adherent coating. After corrosion testing, however, the coating on about 80% of the blocks tested was found to be non-adherent. Criteria for the post-corrosion tape test was the same as for the pre-corrosion tape test, i.e., any removal of coating was considered non-adherent. The percentage of blocks that showed non-adherence on the upstream face, where contact with the element face restricted hydrogen flow and where temperature was greatest, was less than for the exhaust end where exposure to the hot hydrogen test gas was presumably greater and temperatures were less. The amount of coating removal from the exhaust end face varied from between 10% to 100% of the total coated surface area. Tape test of the coated sides of the corrosion sample, which were subjected to little flowing hydrogen

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exposure during the corrosion test revealed no instances of non-adherency.

As shown in Figure 17, a photomicrograph of a section through a corrosion-tested sample, a definite gap at the NbC graphite interface exists after corrosion testing and is indicative of graphite attack at the coating interface by the hydrogen environment at the test conditions. The fact that the NbC deposits are known to be extensively cracked suggests a method of hydrogen infiltration to the graphite substrate. As previously mentioned, this cracking is the result of differences in thermal expansion between the graphite and the carbide and occurs after coating during the process of cooling from the coating temperature (2100°C) to room temperature. It has been assumed that when a coated graphite component is heated to temperatures equal to or in excess of the coating temperature, the differences in thermal expansion again prevail and these cracks close and seal the surface against hydrogen penetration. Since the temperature achieved by the support block in the corrosion test has been estimated to be in the range of 2000 to 2195°C, at best, it is highly possible that the cracks on the carbide are not completely sealed during the test. To hydrogen atoms these cracks should appear as virtual boulevards to the graphite substrate. Further credence is lent to this method of hydrogen attack of the substrate by the observation that coating non-adherence is more prevalent on the bottom face of corrosion-tested blocks where temperatures are lowest during testing. Hydrogen attack of the graphite coating interface then appears to be responsible for coating non-adherency on corrosion-tested support blocks. The implications of these observations become important when future long-life and restartable reactor engines are considered. For operation at the A-2 conditions, as approximated by the IA corrosion test, this attack and resultant coating non-adherence does not appear to compromise the ability of coated blocks to survive for five minutes. When a small number of once-tested corrosion test lobes were rerun a second and third time in the IA test, however, results were extremely variable and the incidence of block failure was high. Although the number of retests was small, the variable results of the tests, the high incidence of non-adherency in IA corrosion tested samples, and results observed with the two blocks found to be non-adherent after coating demand that a detailed study be made to better define the restart and lifetime capabilities of coated support blocks with a view toward advanced reactor requirements.

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A second mechanism of coating adherence degeneration exists, namely, the creation of a void or gap at the interface due to the diffusion of either carbon or hydrocarbons through the coating and its subsequent removal by the gas stream. To date, this mechanism has received little attention with regard to support block behavior; however, calculations indicate the serious nature of such a mechanism with regard to void creation and subsequent loss of adherence of coatings which are not operated in compression, i.e., coating in areas other than channels.

Coating Stoichiometry Changes

A comparison of the carbon to niobium ratio of the coating on A-2 production support blocks before and after IA corrosion testing shows a general reduction in the carbon content of the NbC. Typical before and after C/Nb values are presented in Table II. This reduction in the carbon content of the coating can only be attributed to the reducing action of the high temperature hydrogen and suggests a possible limit to the useful lifetime of the NbC as a protective coating. If the NbC is depleted of carbon at a rate greater than the diffusion rate of carbon through NbC, in some finite time a composition of Nb and C will be reached that melts at the temperature of operation. At this point, either the molten metal will be blown away by the flowing gas exposing the underlying graphite to catastrophic attack or the metal will rapidly react with the graphite as a result of enhanced carbon-niobium diffusion rates in the molten state, and solidify into a new protective layer. These processes are, of course, conjectural. For five-minute operation at conditions approximating the IA test conditions, NbC coatings appear to be more than adequate from the standpoint of carbon loss. As reactor operating times become longer in future designs, however, the possibility of carbon loss lifetime limitations should be investigated.

Corrosion Behavior of Discolored Coatings

As a result of a contaminant in the furnace environment during the coating process, deposits are occasionally produced that are discolored. This discoloration has ranged from a pale straw color through blues, reds, greens up to a rather dull black. Investigation of this discoloration has revealed that it is an extremely thin film typical of thin oxide or nitride interference films that occur on such

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metals as Zircaloy-2. It cannot be seen by metallographic examination of sections through the coating thickness. Carbon to niobium ratio determinations made on badly discolored support blocks have fallen in the range of values determined for non-discolored coatings.

Results of IA corrosion tests performed on several discolored support block sections indicate that the discoloration in no way detracts from the corrosion resistance of the coating. Discolored coatings performed in every way as well as non-discolored coatings under the IA test conditions. During the test, the discoloring contaminant is either reduced by the test hydrogen or dissolved in the coating since after the test formerly discolored blocks are bright and clean.

Interchannel Communication

Radiographic inspection of coated support blocks revealed that in several blocks, small holes existed in one or more coolant channel walls allowing communication between adjacent channels. Close examination of radiographs in each case indicated that the breakthroughs between channels were completely coated with NbC. As shown in Figure 18, metallographic sectioning substantiates this observation and clearly shows that the communicating holes are coated. Class IA corrosion tests on several coated support block sections containing coated channel communicating holes have demonstrated that the defects do not compromise the ability of the component to survive in the hot hydrogen environment.

Coating Transfer

It was observed in about 90% of the support block corrosion tests that the NbC coating from the downstream end of the test fuel element was transferred to the upstream end face of the support block. A typical example of this phenomenon is illustrated in Figure 19. This transferred coating was tightly adherent to the block, defying removal in most instances by tape stripping and scraping with a knife blade. A photomicrograph of a polished section through the transferred coating is illustrated in Figure 20.

It is postulated that adherence of the element end to the NbC on the support block is effected by pressure bonding. As previously discussed, corrosion testing was conducted with the support block sample in intimate contact with the downstream end face of the fuel element. Review of the fixturing scheme previously illustrated in Figure 2 reveals that, in the test, the block sample was held at the downstream end by the ledge support in the block holder and at the upstream end by the element end which, during the test, becomes immovable in this element chuck as a result of thermal expansion. Under these conditions, no dimensional allowance is made for linear thermal expansion of the block samples; therefore, forces between block and element are generated when the block sample expands. Under these forces, bonding occurs at the coating interface. Since coating adherence is significantly greater on support blocks than it is on fuel element ends (non-wafer tipped elements), the coating strips from the element at the points of bonding when the components are separated at the termination of the test.

Another observed effect of these expansive forces has been the occurrence of cracks at the base of the interlock step by which the sample is supported in the downstream direction. As shown in Figure 21, these cracks originate at the junction of the step and the side wall of the block and run at a 45° angle toward the upstream end. In many instances, it is felt these cracks have been the direct cause of extensive corrosion in the interlock areas. By supporting the test sample at the downstream end during the test rather than at the step, this cracking and the attendant corrosion problem were solved. The expansive forces, however, were not eliminated; therefore, neither was coating transfer.

An indication of the magnitude of the force generated by expansion of the block segment was obtained by measuring the load required to cause the cracking observed in the interlock step. This was accomplished by loading a block segment supported at the interlock in a steel holder (similar to the graphite test holder) in an Instron testing machine. Results with three coated samples showed that cracking occurred at a load of about 500 pounds. Accordingly, it may be assumed that the force generated in the test is at least 500 pounds. Since the room temperature strength of graphite is less than its strength at corrosion test temperatures, the actual force is probably greater than this value.

It must be stated that these limited observations do not warrant the formulation of more definite conclusions. Estimations of the stress on the element end or on the element step cannot be made since actual contact areas are not known. No efforts have been made to establish the temperature and pressure required to initiate bonding between the element and block coatings since this was beyond the scope of the subject investigations.

CONCLUSIONS AND RECOMMENDATIONS

From the results of this investigation, the following conclusions and recommendations are made:

1. Niobium carbide coatings deposited by the Cheswick Astrofuel Facility on ATJ graphite support blocks appear to be adequate to preclude attack by hydrogen at temperatures at least to about 2195°C for times to at least five minutes at gas pressures and flow rates consistent with the IA corrosion test conditions. Remaining to be defined, however, is the behavior of coatings at higher temperatures and longer exposures.
2. Considerable doubt exists regarding the effect of a fuel element failure in reactor operation on the ability of the NbC coating to protect the support block graphite from attack by hydrogen. Additional work should be conducted in this area.
3. It appears that under operating conditions approximating the five minute corrosion test conditions, the protective value of NbC coating on ATJ support blocks becomes questionable at a thickness less than 0.0008 to 0.0012 inches.
4. Due to the progressive nature of hydrogen attack of graphite at coating defect sites and the general inability to predict the interrelationship between operating conditions and defect size with current experimental equipment, it is recommended that support blocks with visible coating defects not be used for reactor hardware.

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5. Tape testing (determining adherence by quickly stripping normally adherent masking tape from the coated surfaces) appears to be a reliable technique for detecting gross non-adherence of the NbC coating to ATJ graphite support blocks.

6. The protective value of non-adherent NbC coatings at conditions approximating the IA corrosion test is substantially less than that of adherent coatings.

7. In general, adherent NbC coatings on support blocks become less adherent when corrosion tested in the five minute IA test environment. Additional time at the same conditions precipitates extensive attack of the graphite and failure of the test sample. It is not clear, at present, whether this effect is unique to the IA corrosion test since test temperatures border on coating temperatures; therefore, cracks inherent to the coating may not be completely closed, or whether the effect is an inherent property of the coating. The implications of this observation are important when higher power, longer operating, and restartable reactor engines are considered. Additional work in this area is clearly required.

8. As a result of exposure to the environment of the IA corrosion test, NbC coatings become depleted of carbon. For five-minute operation at conditions approximating the IA corrosion test conditions, carbon loss should not compromise the ability of the NbC coating to perform its protective function.

9. Discoloration in the form of interference films that are formed on NbC deposits during the coating process does not affect the performance of the coating in the IA corrosion test.

10. In a hydrogen environment at temperatures extant in the IA corrosion test, adherence between the NbC on the element end and the NbC on the support block may occur. Separation of the block from the element will cause coating to be removed from the element. This effect might be important in future reactors where restart capability is desired. If the coating on the element ends is transferred to the support blocks during cool-down after power operation as a

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result of bonding and subsequent element shrinkage, then the restart operation might be endangered due to removal of the protective coating from the element ends. Additional study of this effect is recommended to establish temperatures and pressures at which bonding is initiated, to establish what effect coating transfer has on element life, and to develop methods to preclude coating transfer.

11. Serious limitations have been imposed upon these studies due to the nature of the corrosion test apparatus. The behavior of support block corrosion tests, at times, was greatly affected by the performance of the elements used in the tests. Test pressures, temperatures and hydrogen flow rates available for study of the corrosion behavior of support blocks during corrosion testing were unavailable. It is recommended that a more definitive study be initiated to establish the lifetime of coated support blocks in hydrogen as a function of temperature. In addition, the restart capability as a function of time and temperature must be determined.

12. Flow channel defects that allow interchannel communication do not affect the ability of coated support blocks to survive in the IA corrosion test environments provided that the defects are completely coated.

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3. WANL Specification PDS 30065-B, "Protective Coating"
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TABLE I
HOT-HYDROGEN CORROSION TEST PARAMETERS

<u>PARAMETER</u>	<u>CONDITION</u>
Average rate of rise of exit gas temperature at startup	60-90° R/sec., based on element surface temperature
H ₂ flow rate at startup	Full flow
Startup time - ambient to element operating temperature	45 - 60 sec.
Time at temperature	5 minutes
Operating power (element only - block not electrically heated)	Consistent with flow, pressure, and temperature
Element temperature (3 inches from block)	2120° C (IC) 2280° C (IA)
Exit gas pressure	560 psi
Rate of gas flow through 19 holes	0.0463 lb./sec.
Average rate of decrease of exit temperature at shutdown	80 - 90° R/sec.
H ₂ flow rate at shutdown	Full flow to ambient temperature
Time in furnace after shutdown	Consistent with safe handling

If arcing, element failure, or other malfunction is identified as influencing the results of the test, the test will be voided and a replacement test performed.

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TABLE II

CARBON-NIOBIUM RATIOS OF REPRESENTATIVE SUPPORT BLOCKS
BEFORE AND AFTER 5-MIN. CORROSION TEST IN HOT HYDROGEN

<u>BLOCK NUMBER</u>	<u>BEFORE TEST</u>	<u>AFTER TEST</u>
HT-53 (J7)	.892	.905
HT-985	.912	.909
HT-36	.920	.882
HT-890	.972	.886
HT-40	.952	.878
HT-48 (J8)	.932	.911
HT-42 (J6)	.945	.820
HT-29 (J5)	.927	.913
HT-1003	.900	.909
HT-407	.910	.905

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Figure 1 - Regular support block and 19-hole,
one-sixth hex corrosion test segment.

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FIGURE 2 - CROSS SECTION OF SUPPORT SCHEMES FOR SUPPORT BLOCK LOBES

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FIGURE 3 - ASSEMBLY USED FOR CORROSION TESTING OF SUPPORT BLOCK LOBES FOR
NRX - A2

WANL - TME - 1173

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Figure 4 - Upstream end face of support block section
that failed the IA corrosion test.

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Figure 5 - Upstream end face of corrosion test support block dose showing typical coating defect between holes on block periphery.

Figure 6 - Upstream end face of corrosion test support block lobe showing typical coating defect on peripheral hole wall at center left edge.

Figure 7 - Upstream end face of section shown in Figure 6 after corrosion testing at IA conditions for five minutes showing attack at defect area. Other dark areas are NbC coating that was transferred to the support block from the fuel element during the test.

Figure 8 - Upstream end plane of corrosion test sample showing typical defect in NbC coating prior to testing. Defect size is 0.050" x 0.025" (inlet end).

Figure 9 - Downstream end plane of corrosion test sample showing typical defect in NbC coating prior to testing. Defect size is 0.050" x 0.025" (outlet end).

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Figure 10 - Post-test photograph of sample shown in Figure 9 illustrating typical defect after testing for five minutes in 2280°C H₂. No increase in coating defect has occurred. (Outlet end)

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Figure 11 - Post-test photograph of sample shown in Figure 10 after removal of undercut NbC. Depth of graphite removal is approximately 1/8". (Inlet end)

250X

As Polished

Figure 12 - Photomicrograph of section through non-adherent coating on regular support block as defined by tape testing.

200X

As Polished

Figure 13 - Photomicrograph of interface of adherent coating
on support block as defined by tape test.

8X

Figure 14 - Crack network in adherent coating on a support block.
Note that not all of the fine cracks are revealed.

5X

Figure 15 - Crack network on non-adherent coating on a support block.

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Figure 16 - Support block with non-adherent coating after five-minute IA corrosion test.

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200X

As Polished

Figure 17 - Photomicrograph of section through coating of support block that failed tape test after IA corrosion test showing gap between NbC and graphite substrate.

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Figure 18 - Coated interchannel communication defects.

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Approx. 2X

Figure 19 - Upstream end face of sample corrosion tested at IA conditions illustrating typical transfer of NbC coating from element end to block sample end.

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200X

As Polished

Figure 20 - Photomicrograph of section through support block after IA corrosion test showing adherence of NbC coating transferred from fuel element. Block coating is thick coat surrounding graphite, element coating is thinner section angling toward upper right corner of photograph.

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Figure 21 - Support block segment that cracked at the interlock step during corrosion testing.

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