CONFIDENTIAL
RESTRICTED DATA
Atomic Energy Act - 1954

Records
SNPO-C

Subcontract NP-1

WANL-TME-1939

July 1, 1969

Westinghouse Astronuclear Laboratory

THE MICROSTRUCTURE OF NbC COATING

(Title Unclassified)

MASTER

CONFIDENTIAL
RESTRICTED DATA
Atomic Energy Act - 1954

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

~~CONFIDENTIAL~~
~~RESTRICTED DATA~~
~~UNCLASSIFIED~~

47

Subcontract NP-1

WANL-TME-1939

July 1, 1969

[Handwritten signature]

NOTICE
This report was prepared as an account of work sponsored by the United States Government. Neither the United States nor the United States Energy Research and Development Administration, nor any of their employees, nor any of their contractors, subcontractors, or their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness or usefulness of any information, apparatus, product or process disclosed, or represents that its use would not infringe privately owned rights.

Westinghouse Astronuclear Laboratory

MASTER

THE MICROSTRUCTURE OF NbC COATING

(Title Unclassified)

Classification cancelled (or changed to) _____
by authority of _____
by *H.F.C.* TIC, date **SEP 12 1973**

PREPARED BY:

E. L. Kochka

E. L. Kochka, Engineer
WNCO Coating Engineering

APPROVED BY:

C. M. Hollabaugh

C. M. Hollabaugh, Supervisor
WNCO Coating Engineering

SPECIAL REVIEW
DETERMINATION
Class: *U*
Reviewers: *DBW*
Class: *U*
Date: *4-26-82*

~~GROUP - 1
EXCLUDED FROM AUTOMATIC DOWNGRADING
AND DECLASSIFICATION~~

~~INFORMATION CATEGORY
C.R.D.
H.R. Ref 7/22/69
AUTHORIZED CLASSIFIER DATE~~

~~CONFIDENTIAL~~
~~RESTRICTED DATA~~
~~UNCLASSIFIED~~

DISTRIBUTION OF THIS DOCUMENT UNLIMITED

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency Thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

PAGE BLANK

TABLE OF CONTENTS

<u>Section</u>		<u>Page</u>
1.0	INTRODUCTION	1-1
2.0	NbC DIFFUSION COATING STRUCTURES	2-1
3.0	NbC METHANE COATING STRUCTURES	3-1
4.0	DISCUSSION	4-1
APPENDIX A:	PHOTOMICROGRAPHS OF NbC COATINGS	A-1
REFERENCES		

LIST OF ILLUSTRATIONS

<u>Figure</u>		<u>Page</u>
2-1 (CRD)	Microstructure of a Diffusion NbC Coating from Run F173 (200X, Etched). (U)	A-3
3-1 (CRD)	Microstructure of GEM NbC Coating from Run B211/B213 (200X, Etched). Type - Phase IVA. (U)	A-5
3-2 (CRD)	Microstructure of a GEM NbC/GED NbC Coating from Run G104 (200X, Etched). (U)	A-7
3-3 (CRD)	Effect of Heat Treatment on GEM NbC Coating from Run B207 (400X, Etched). (U)	A-9
3-4 (CRD)	Microstructure of GEM NbC Coating from Run B216 (200X, Etched). Type - IDa, WEFF. (U)	A-11
3-5 (CRD)	Microstructure of GEM NbC Coating from Run B233 (200X, Etched). Type - IDa, WEFF. (U)	A-13
3-6 (CRD)	Microstructure of a GEM NbC Coating Run Showing a Typical LASL Mid-band Structure (Y-12 Coating Run 0908 Using LASL Chemistry and LASL Temperatures, 400X, Etched). (U)	A-15
3-7 (CRD)	Microstructure of GEM NbC Coating from Run G103 (200X, Etched). Type - Phase IVB or Pewee I, WEFF. (U)	A-17
3-8 (CRD)	Microstructure of GEM NbC Coating from Run B231 (200X, Etched). Type - Phase IVB or Pewee I, WEFF. (U)	A-19
3-9 (CRD)	Microstructure of a GEM NbC Coating from Run F400 (200X, Etched). Type - Modified LASL, WNCO. (U)	A-21
3-10 (CRD)	Microstructure of GEM NbC Coating from Run C408 (200X, Etched). Type - Intermediate Hydrogen, WNCO. (U)	A-23
3-11 (CRD)	Microstructure of GEM NbC Coating from Run B239 (200X, Etched). Type - LASL Chemistry, WEFF. (U)	A-25

LIST OF ILLUSTRATIONS (CONTINUED)

<u>Figure</u>		<u>Page</u>
3-12 (CRD)	Microstructure of GEM NbC Coating from Run C417 (400X, Etched). Type - LASL Chemistry, WNCO. (U)	A-27
3-13 (CRD)	Microstructure of GEM NbC Coating from Run L327 (400X, Etched). Type - LASL + Hi HCl, WNCO. (U)	A-29
3-14 (CRD)	Microstructure of GEM NbC Coating from Run L349 (400X, Etched). Type - Pewee 2 Candidate, WNCO. (U)	A-31

LIST OF TABLES

<u>Table</u>		
2-1 (CRD)	NbC Diffusion Coating Parameters (U)	2-2
3-1 (CRD)	NbC GEM Coating Parameters (U)	3-4

~~CONFIDENTIAL~~

1.0 INTRODUCTION (U)

(U) A presentation was made to the SNPO Fuel Committee on March 6, 1969, relating the as-deposited coating structure to coating parameters. The Committee requested that a report be prepared based on that presentation.

(CRD) Prior to the Pewee I reactor, all fuel elements were NbC coated by a diffusion-controlled process. The rate of coating deposition is controlled in this process by the rate of carbon diffusion from the matrix to the outer surface of the deposited coating. The rate of carbon diffusion through the coating is a function of temperature and coating thickness. Temperatures in excess of 1600°C are required to deposit two (2) or more mils of NbC on graphite at a reasonable rate by a diffusion-controlled process.

(U) Diffusion coatings have two characteristics that adversely affect the corrosion test performance of an element. These are (1) the loss of adherence that results from supplying carbon from the matrix and (2) the large cracks present in element areas operating below deposition temperatures that result from differences in the coefficients of expansion of the coating and matrix.

(CRD) The methane coating process supplies carbon from the gas stream to form the coating that is deposited from 1000 to 1300°C . This coating is well-rooted in the matrix and has a much finer crack structure than a diffusion coating of equivalent thickness. However, this process is more sensitive to changes in gas composition and coating temperature than the diffusion process, and these changes are apparent in the different coating structures they produce. The first part of this report will trace the development of NbC coating through differences in coating structures based on parameter changes.

~~CONFIDENTIAL~~

2.0 NbC DIFFUSION COATING STRUCTURES (U)

(CRD) The diffusion (HED or GED)* coating process gives a large-grained columnar coating. A typical NbC diffusion coating is shown in Figure 2-1. Since the matrix supplies the carbon for the formation of this coating, a gap exists between the coating and the graphite substrate. This gap develops when the coating thickness exceeds about 0.7 mil and becomes greater with increasing thickness.

(CRD) Because of the high coating temperatures and differences in the thermal expansion coefficients of the coating and the matrix, large cracks develop as the element cools from coating temperatures. Since the entire element is heated above 1600°C, these cracks form over the entire length of the coating. In the corrosion test the midband of these elements is run at a temperature below the coating temperature, and open cracks are present, exposing graphite to hydrogen. Therefore, a characteristic of a diffusion-coated element is a high midband weight loss unless the cracks have been filled with a molybdenum overcoating. Molybdenum overcoating is an additional low temperature coating operation that does not alter the basic NbC structure.

(CRD) The parameters that have been used in the diffusion coating of elements with niobium carbide are given in Table 2-1. These parameters all produce the same type of structure shown in Figure 2-1. There may be differences in grain size that result from differences

(CRD) * Coating processes for the NERVA fuel element are described by the following: HED, GED, HEM, or GEM. The H and G refer to the end of the element as described on the fuel element drawing or, in more common usage, the H end is the hot or coated end of the element and the G end is the cold or serial numbered end of the element. In coating, the H or G indicates that the coating gases enter that end of the element first and flow toward the other end. D indicates a diffusion coating process and M a methane coating process.

TABLE 2-1 (CRD)

NbC DIFFUSION COATING PARAMETERS (U)

Type	Gas Flow (LPME)				Temperature ($^{\circ}\text{C}$)			Time (hrs.)	Wt. Gain (gms.)
	A	HCl	H ₂	Salt	0	25	50		
A-2 HED	0.80	0.80	0.063	0.065	1900	1900	1800	6.5	35
A-6 HED	0.86	0.80	0.071	0.071	1600	1650	1730	14	50
A-6 GED	0.86	0.80	0.071	0.071	1450	1575	1750	14	50
PW-1 GED	0.94	0.50	0.136	0.060	1370	1530	1780	14	70

~~CONFIDENTIAL~~

~~RESTRICTED DATA~~

in coating temperatures or rates (higher coating temperatures give wider grains) when coatings are compared over a wide range of temperature, but the basic structure remains the same. Benefits gained in changing from the HED to a GED coating or changing process parameters are related to profile control, matrix-coating interaction, and the relationship between coating temperatures and operating temperatures rather than to changes in the coating structure.

~~CONFIDENTIAL~~

~~RESTRICTED DATA~~

3.0 NbC METHANE COATING STRUCTURES (U)

(U) The final reports on Test Specification IDa⁽¹⁾, IVA⁽²⁾, and IVB⁽³⁾ trace the early development of NbC GEM coating. This report compares the coatings developed in the test specifications and shows differences in structure based on parameter changes in work performed at and since that time. The nominal parameters of the NbC GEM coating processes discussed in this report are shown in Table 3-1.

(CRD) The purpose of Phase IVA was to assess the progress made in GEM NbC coating at WEFF, Y-12, and LASL using a standard set of high void fraction fuel elements. WEFF and Y-12 used similar gas compositions except that Y-12 used NbBr_5 and HBr , instead of NbCl_5 and HCl , and coated at slightly higher temperatures. LASL GEM coating conditions differed and were used to deposit the NbC GEM coating as a basecoat for NbC GED overcoating.

(CRD) Complete control of the GEM coating process was not obtained during work on Phase IVA. Coating thickness peaks occurred near station 40 using a gas chemistry with 0.020 LPME of methane, and it appeared that the methane in the gas stream had been depleted to the point where further temperature increases could not increase the hot end thickness. Y-12 compensated for this by increasing run time and WEFF elements were recoated to increase the hot end thickness.

(CRD) A coating typical of those produced during Phase IVA is shown in Figure 3-1. These coatings had very rough surfaces and occasionally contained nodular growths that extended into the coolant channels and impeded flow. The nodules were of two types: (1) those arising from accelerated deposition around carbon flakes and (2) those occurring as a single crystal of NbC. The base coating was fine grained near the inlet and became coarser and somewhat porous beyond station 40. Because of the lower temperatures used in recoating to increase hot end thickness, the overcoat has the fine-grained structure typical of the mid-band of the base coating.

~~CONFIDENTIAL~~
~~RESTRICTED DATA~~

(CRD) Corrosion test data showed that the GEM NbC-coated elements performed better when coating thicknesses were greater than one mil at station 20 and over 2.5 mils at station 50. However, many tested elements showed breakage and/or cracking near the hot end. Cracks in the graphite matrix were also observed in the metallographic examination of the as-coated elements and result from the abnormally high stresses created by thick adherent coatings with a thermal expansion coefficient significantly higher than the matrix. These cracks, which are not present in the LASL elements coated by the GEM/GED process, have a deleterious effect on element performance. The additional deposition of GED NbC over the GEM coating relieves the stress on the matrix by using some of the underlying graphite to form the coating and reduce the adherence of the coating to the matrix. This type of coating is shown in Figure 3-2.

(CRD) Examination of GEM bore coatings after corrosion testing revealed grain growth of niobium carbide. No correlations have been made between time, temperature, and growth rate, but it appears that significant grain growth occurs above 2100°C . Figure 3-3 shows the change that takes place in an NbC coating after one hour at 2300°C . This figure shows both a two-phase coating and an as-coated nodule. After heat treatment, there is dissolution of carbon, homogenization of the coating, and the presence of void areas at the intersections of grain boundaries in addition to fewer but larger grains.

(CRD) The Phase IVA work was followed by IDa. In the work on Test Specification IDa, the coating temperature was limited to 1140°C to prevent the profile maxima given by process gas stream depletion. Variations in gas composition, furnace temperature, and run time were explored to produce selected NbC coating profiles suitable for GEM NbC coating of high void fraction Pewee I fuel elements. Below 1140°C the deposition rate can be increased by increasing methane flow to give uniform thickness profiles.

~~CONFIDENTIAL~~
~~RESTRICTED DATA~~

(CRD) The coatings produced in runs B216 and B233 are shown in Figures 3-4 and 3-5. These coatings are fine grained, dense, and leaf-like with good penetration into the matrix. The differences between these two runs is that B233 had a higher concentration of active species in the gas stream and a faster deposition rate than B216. The richer gas stream entering the element caused most of the deposition to occur near the inlet end of the element, giving more rapid depletion and a thinner hot end.

(CRD) Phase IVA served as a preliminary to the larger Phase IVB program and a comparative evaluation of the NbC fuel element coating processes of WEFF, Y-12, and LASL on a common matrix manufactured through leaching at WNCO. These elements were coated by an NbC GEM process at WEFF, an NbC HED process and an NbC GEM + heat treatment process at Y-12, and an NbC GEM/GED process at LASL. The elements coated at WEFF had thinner coatings at the cold end and lower weight gains than the Y-12 GEM elements. The elements coated by the GEM/GED process at LASL and by the HED process at Y-12 had thinner hot end coatings than either the WEFF or the Y-12 GEM coatings. All of the HED coated, half of the GEM/GED, and some of the GEM coated elements were overcoated with molybdenum before they were corrosion tested. Nominal Phase IVB coating conditions used at the three facilities are compared in Table 3-1.

(CRD) An evaluation of the corrosion performance showed a mid-band behavior in order of increasing weight loss as (a) LASL, (b) WEFF, and (c) Y-12. Corrosion loss was due to binder attack in both the WEFF and Y-12 elements and was virtually eliminated by molybdenum overcoating. The mid-band performance of these elements was related to crack density (number of cracks per inch) rather than mid-band coating thickness. Crack densities could be related to coating structure, grain size, and stoichiometry differences between the coatings produced at the three facilities. The WEFF and Y-12 coatings had a more equiaxed grain structure and a higher stoichiometry than the LASL coating. The LASL coating at station 20 had a very fine-grained dense layer of NbC at the coating-graphite interface and a columnar structure extending from the initial layer. It was the dense layer that halted the propagation of some of the cracks that was responsible for the better mid-band performance of the LASL elements.

TABLE 3-1 (CRD)

NbC GEM COATING PARAMETERS (U)

Type	Run No.	Gas Flow (LPME)					Temperature (°C)			Time (hrs.)	Wt. Gain (gms.)	Beta Backscatter Thickness, mils		
		A	HCl	H ₂	CH ₄	Salt	8.5	27	52			C/Nb		
												4.5	22.5	49.5
WEFF IV A	B211	4.5	0.75	0.08	0.020	0.087	1013	1055	1103	22	52	0.3	0.7	1.6
Y-12 IV A	5444	4.5	0.75	0.08	0.020	0.088	1060	1135	1175	30	112	1.1	1.9	2.6
LASL IV A	B5-178	2.2	0.42	0.04	0.042	0.052	1035	1100	1150	6	47	1.2	1.7	0.1
WEFF IV B	B229	4.5	0.75	0.08	0.060	0.087	980	1050	1100	14	85	0.7	1.0	2.7
Y-12 IV B	8976	4.5	0.75	0.06	0.020	0.089	1035	1075	1140	6	90	0.8	2.3	2.7
LASL IV B	B5-219	2.2	0.56	0.19	0.042	0.078	1100	1180	1260	7	68	1.2	2.2	0.9
WEFF PW-1		4.50	0.75	0.18	0.06	0.09	1030	1065	1085	14	69	1.2	1.3	1.3
LASL Chem. (WNCO)		1.78	0.56	0.19	0.04	0.08	1077	1178	1306	7	78	1.3	2.4	1.3
LASL Chem.- LASL Temp. (Y-12)		1.71	0.54	0.15	0.03	0.06	1050	1200	1260	8	- -	1.2	2.7	1.4
LASL Chem.- Y-12 Temp. (Y-12)		1.71	0.53	0.15	0.03	0.06	1040	1095	1115	15	- -	1.2	1.8	2.2
WNCO Hi HCl		1.78	0.64	0.19	0.04	0.08	1080	1175	1335	8	100	1.4	2.2	2.5
WNCO PW-2 Dev.	L349	1.79	0.64	0.19	0.041	0.091	1072	1151	1301	10	113	1.6	2.4	3.3

~~CONFIDENTIAL~~
~~RESTRICTED DATA~~
~~XXXXXXXXXX~~

(CRD) An example of the LASL IVB mid-band coating structure is shown in Figure 3-6. WEFF IVB coating structures are shown in Figures 3-7 and 3-8. The dense layer adjacent to the matrix and the columnar-like grains are evident in the LASL coating. The WEFF structure is a random, equiaxed, fine-grained structure with some tendency toward a laminated coating. (Note in making comparisons that the photomicrograph of the LASL coating is at 400X and those of the WEFF coatings are at 200X.) The IVB chemistries were used in coating elements for the Pewee I reactor.

(CRD) WANL continued to develop NbC GEM coatings and to explore variations in coating parameters. The first step was to keep the argon, hydrogen chloride, and methane constant and increase the hydrogen to salt ratio to 3 to 1 or a little beyond the 2.5 to 1 used by LASL. The effect of this change as seen in Figure 3-9 was to develop the dense layer adjacent to the matrix with good penetration into the matrix. However, the remainder of the coating was very fine grained with laminations and modular growths. By reducing the hydrogen to salt ratio to 2 to 1, the nodular growth disappeared, but the structure showed greater lamination in the mid-band region. The width of the dense layer adjacent to the graphite also decreased. Photomicrographs typical of the intermediate hydrogen runs are shown in Figure 3-10.

(CRD) The next step was to convert all flows to correspond to those used by LASL and to adjust the temperatures accordingly to avoid the dropoff in thickness that LASL desired in their NbC GEM coating for overcoating by the NbC GED process. This change produced the coating shown in Figures 3-11 and 3-12. There is a reasonably thick dense layer adjacent to the matrix with random fine grains above this layer at the cold end of the element. The coating became more columnar at the hot end (station 50). This type of coating (which also dropped off in thickness toward the hot end) was used as the base coat

~~CONFIDENTIAL~~
~~RESTRICTED DATA~~
~~XXXXXXXXXX~~

CONFIDENTIAL
RESTRICTED DATA

in the early stages of work on Test Specifications 3012 and 3017*. Efforts to increase the hot end thickness by temperature adjustments alone were not successful. Therefore, the hydrogen chloride concentration was increased in an attempt to inhibit deposition at the cold end and have a stream richer in niobium species for coating the hot end of the element. The results of this change are shown in Figure 3-13. The dense inner layer of coating was retained and a narrow columnar structure was developed nearer the inlet of the element. Grain width increased slightly toward the hot end of the element.

(CRD) Although this chemistry gives a very desirable structure (as evidenced by the very low mid-band weight losses of recent corrosion tested elements), it was difficult to control the hot end thickness profile with the high deposition rates that were used. The thicker coatings that were requested for the cold end of the element depleted the methane and it was not available for coating at the hot end. Recent modifications drop temperatures slightly and run for a longer period of time. These changes give both the structure and profile required for the base coat on Pewee 2 candidate elements. An example of this type of coating is shown in Figure 3-14.

(CRD) * These test specifications are concerned with the development of hot end over-coating.

Test Specification 3012 deals with development of a ZrC HED coating on the hot end of an NbC GEM coated element. Test Specification 3017 deals with the development of a ZrC GEM coating over an NbC GEM coated element. These coating combinations are candidates for the Pewee 2 reactor.

CONFIDENTIAL
RESTRICTED DATA

4.0 DISCUSSION (U)

(CRD) In looking at photomicrographs of GEM NbC coatings produced over a period of time with different coating parameters, different coating structures are observed. Most of the coating runs that produced these structures were designed to give a particular coating profile independent of structure. However, it was noted during the evaluation of Phase IVB elements that the LASL structure gave better mid-band performance than the coatings produced at Y-12 or WEFF. Therefore, an effort was made to duplicate this structure at WEFF and later at WNCO.

(CRD) A comparison of structures and run parameters indicate that the GEM NbC coating process is very sensitive to changes in gas composition and temperature and a single change made to improve the structure may adversely affect the profile. Therefore, it is necessary to adjust one or more additional parameters until the condition is found that will give both the structure and profile that are desired.

(CRD) A number of generalizations can be made on the effects of changing individual parameters, assuming all other parameters are constant at nominal values and the temperature is increasing in the direction of gas flow.

(CRD) A high methane concentration will give a very fine-grained deposit at the inlet end of the element. As the temperature increases, the grains become larger. If inlet temperatures are high enough, most of the deposition will occur near the inlet, removing carbon from the gas stream and enriching it in hydrogen. There is always the possibility of increasing the hydrogen to salt ratio to a value where metal deposition can occur. In general, an increase in hydrogen, salt, or methane flow will increase the NbC deposition rate at a constant inlet temperature.

(CRD) Of the critical gases, hydrogen, salt, or methane, the methane concentration exerts the greatest control on the process. Proper control of the methane concentration and the furnace temperature profile will lead to the desired distribution of coating along the length of an element. Too high a methane concentration and too high a temperature may give the nodular deposits seen in B207 unless the hydrogen concentration is high enough to inhibit

~~CONFIDENTIAL~~
~~RESTRICTED DATA~~
~~XXXXXXXXXX~~

breakdown of the methane. On the other hand, too low a methane concentration will result in its early removal from the gas stream and cause the deposition rate to be irresponsive to increasing temperatures. This usually results in decreasing hot end profiles.

(CRD) In addition to hydrogen, HCl also appears to inhibit breakdown of methane.

An increase of HCl flow by less than 10% increased the population of fine columnar grains in the coating and dropped the carbon to niobium ratio from above 0.90 to around 0.87 along the length of an element.

(U) Because of the number of variables that affect the process (8 or more), the effects of process changes cannot be effectively predicted. The above observations relate process parameters and structure to a small degree, but they need to be followed by a detailed chemistry of the coatings as a function of position and a more thorough analysis of the process that would give the gas composition at any position in an element. These data could then be assembled in a useful form that would give a better understanding of the process and of the effects of changes in individual parameters.

~~CONFIDENTIAL~~
~~RESTRICTED DATA~~
~~XXXXXXXXXX~~

APPENDIX A (CRD)

PHOTOMICROGRAPHS OF NbC COATINGS (U)

CONFIDENTIAL
RESTRICTED DATA

Atomic Energy Act - 1954

Astronuclear
Laboratory

Station 50

Station 40

Station 30

Figure 2-1. (CRD) Microstructure of a Diffusion NbC Coating from Run F173 (200X, Etched) (U)

CONFIDENTIAL
RESTRICTED DATA

Atomic Energy Act - 1954

~~CONFIDENTIAL~~
~~RESTRICTED DATA~~
~~GROUP 1~~

Type	NbC GEM
Flows - Ar	4.50 ⁺ LPME
HCl	0.75 LPME
H ₂	0.077 LPME
CH ₄	0.020 LPME
Salt	0.080 LPME
Total Flow	5.427 LPME

Run No.	B-211/B-213
Run Time	14.0 hrs.
*Temp. S.P. 1	(-8.5)955 °C
S.P. 2	(9.0) 980 °C
S.P. 3	(27.0)1030 °C
S.P. 4	(52.0)1060 °C
Wt. Gain	83.0 gms.

⁺ Standard liters per minute per element.

* Numbers in parentheses refer to element stations.

~~CONFIDENTIAL~~
~~RESTRICTED DATA~~
~~GROUP 1~~

CONFIDENTIAL
RESTRICTED DATA

Atomic Energy Act - 1954

Astronuclear
Laboratory

Station 50

Station 30

Station 10

Figure 3-1. (CRD) Microstructure of GEM NbC Coating from Run B211/B213
(200X, Etched). Type - Phase IVA (U)

CONFIDENTIAL
RESTRICTED DATA

Atomic Energy Act - 1954

Type	GEM NbC	GED NbC
Flows - Ar	4.50 ⁺ LPME	0.94 ⁺ LPME
HCl	0.75 LPME	0.50 LPME
H ₂	0.077 LPME	0.140 LPME
CH ₄	0.060 LPME	- - - LPME
Salt	0.088 LPME	0.60 LPME
Total Flow	5.475 LPME	2.18 LPME

Run No.	G104 GEM	G104 GED
Run Time	14 hrs.	8 hrs.
*Temp. S.P. 1	(-11) 904 °C	1151 °C
S.P. 2	(6) 994 °C	1204 °C
S.P. 3	(25) 1039 °C	1376 °C
S.P. 4	(50) 1037 °C	1670 °C
Wt. Gain	83.0 gms.

Figure 3-2. (CRD) Microstructure of a GEM NbC/GED NbC Coating from
Run G104 (200X, Etched) (U)

Type	GEM NbC
Flows - Ar	4.50 ⁺ LPME
HCl	0.75 LPME
H ₂	0.077 LPME
CH ₄	0.020 LPME
Salt	0.088 LPME
Total Flow	5.435 LPME

Run No.	B-207
Run Time	18.4 hrs.
*Temp. S.P. 1	(-11) 931 °C
S.P. 2	(1) 1025 °C
S.P. 3	(25) 1104 °C
S.P. 4	(50) 1162 °C
Wt. Gain	116.9 gms.

⁺ Standard liters per minute per element.

* Numbers in parentheses refer to element stations.

As-Coated Nodule

Nodule - 1 hour at 2300°C

Figure 3-3. (CRD) Effect of Heat Treatment on GEM NbC Coating
from Run B207 (400X, Etched) (U)

**CONFIDENTIAL
RESTRICTED DATA**

Atomic Energy Act - 1954

Astronuclear
Laboratory

Type	GEM NbC	
Flows - Ar	4.11	⁺ LPME
HCl	1.00	LPME
H ₂	0.12	LPME
CH ₄	0.12	LPME
Salt	0.12	LPME
Total Flow	5.47	LPME

Run No.	B216
Run Time	8.0 hrs.
*Temp. S.P.1	(-11) 907 °C
S.P.2	(6) 968 °C
S.P.3	(25) 1033 °C
S.P.4	(50) 1073 °C
Wt. Gain	84.1 gms.

⁺ Standard liters per minute per element.

* Numbers in parentheses refer to element stations.

**CONFIDENTIAL
RESTRICTED DATA**

Atomic Energy Act - 1954

Station 50

Station 30

Station 10

Figure 3-4. (CRD) Microstructure of GEM NbC Coating from Run B216
(200X, Etched). Type - IDa, WEFF (U)

CONFIDENTIAL
RESTRICTED DATA

Atomic Energy Act - 1954

Astronuclear
Laboratory

Type	GEM NbC
Flows - Ar	2.58 ⁺ LPME
HCl	2.00 LPME
H ₂	0.30 LPME
CH ₄	0.300 LPME
Salt	0.297 LPME
Total Flow	5.477 LPME

Run No.	B233
Run Time	5.0 hrs.
*Temp. S.P. 1	(-11) 918 °C
S.P. 2	(6) 1014 °C
S.P. 3	(25) 1047 °C
S.P. 4	(50) 1042 °C
Wt. Gain	84.9 gms.

⁺ Standard liters per minute per element.

* Numbers in parentheses refer to element stations.

CONFIDENTIAL
RESTRICTED DATA

Atomic Energy Act - 1954

Station 50

Station 30

Station 10

Figure 3-5. (CRD) Microstructure of GEM NbC Coating from Run B233
(200X, Etched). Type - IDa, WEFF (U)

CONFIDENTIAL
RESTRICTED DATA

Atomic Energy Act - 1954

Astronuclear
Laboratory

Type	GEM NbC
Flows - Ar	1.71 ⁺ LPME
HCl	0.54 LPME
H ₂	0.15 LPME
CH ₄	0.032 LPME
Salt	0.059 LPME
Total Flow	2.491 LPME

Run No.	0908
Run Time	8.0 hrs.
*Temp. S.P. 1	(-) 950 °C
S.P. 2	(3) 1055 °C
S.P. 3	(29) 1200 °C
S.P. 4	(43) 1260 °C
Wt. Gain	74.3 gms.

⁺ Standard liters per minute per element.

* Numbers in parentheses refer to element stations.

CONFIDENTIAL
RESTRICTED DATA

Atomic Energy Act - 1954

Station 49.5

Station 22.5

Station 4.5

Figure 3-6. (CRD) Microstructure of a GEM NbC Coating Run Showing a Typical LASL Mid-band Structure (Y-12 Coating Run 0908, Using LASL Chemistry and LASL Temperatures, 400X, Etched) (U)

Type	GEM NbC
Flows - Ar	4.50 ⁺ LPME
HCl	0.75 LPME
H ₂	0.077 LPME
CH ₄	0.060 LPME
Salt	0.088 LPME
Total Flow	5.475 LPME

Run No.	G103
Run Time	14.0 hrs.
*Temp. S.P. 1	(-11) 900°C
S.P. 2	(6) 970°C
S.P. 3	(25) 1045°C
S.P. 4	(50) 1103°C
Wt. Gain	104.4 gms.

⁺ Standard liters per minute per element.

* Numbers in parentheses refer to element stations.

Station 50

Station 30

Station 10

Figure 3-7. (CRD) Microstructure of GEM NbC Coating from Run G103
(200X, Etched). Type - Phase IVB or Pewee I, WEFF (U)

**CONFIDENTIAL
RESTRICTED DATA**

Atomic Energy Act - 1954

Astronuclear
Laboratory

Type	GEM NbC	
Flows - Ar	4.50	⁺ LPME
HCl	0.75	LPME
H ₂	0.077	LPME
CH ₄	0.060	LPME
Salt	0.090	LPME
Total Flow	5.477	LPME

Run No.	B231
Run Time	14.0 hrs.
*Temp. S.P. 1	(-11) 907°C
S.P. 2	(1) 982°C
S.P. 3	(25) 1045°C
S.P. 4	(50) 1097°C
Wt. Gain	88.8 gms.

⁺ Standard liters per minute per element.

* Numbers in parentheses refer to element stations.

**CONFIDENTIAL
RESTRICTED DATA**

Atomic Energy Act - 1954

CONFIDENTIAL
RESTRICTED DATA

Atomic Energy Act - 1954

Station 50

Station 30

Station 10

Figure 3-8. (CRD) Microstructure of GEM NbC Coating from Run B231
(200X, Etched). Type - Phase IVB or Pewee I, WEFF (U)

CONFIDENTIAL
RESTRICTED DATA

Atomic Energy Act - 1954

**CONFIDENTIAL
RESTRICTED DATA**

Atomic Energy Act - 1954

Astronuclear
Laboratory

Type	GEM NbC
Flows - Ar	4.50 ⁺ LPME
HCl	0.75 LPME
H ₂	0.28 LPME
CH ₄	0.060 LPME
Salt	0.090 LPME
Total Flow	5.680 LPME

Run No.	F400
Run Time	14.0 hrs.
*Temp. S.P. 1	(-12) 985°C
S.P. 2	(1) 1052°C
S.P. 3	(26) 1072°C
S.P. 4	(52) 1100°C
Wt. Gain	73.5 gms.

⁺ Standard liters per minute per element.

* Numbers in parentheses refer to element stations.

**CONFIDENTIAL
RESTRICTED DATA**

Atomic Energy Act - 1954

Station 50

Station 30

Station 10

Figure 3-9. (CRD) Microstructure of a GEM NbC Coating from Run F400
(200X, Etched). Type - Modified LASL, WNCO (U)

Type	GEM NbC
Flows - Ar	4.50 ⁺ LPME
HCl	0.75 LPME
H ₂	0.18 LPME
CH ₄	0.060 LPME
Salt	0.090 LPME
Total Flow	5.580 LPME

Run No.	C408
Run Time	14.0 hrs.
*Temp. S.P. 1	(-12) 978 °C
S.P. 2	(1) 1030 °C
S.P. 3	(26) 1075 °C
S.P. 4	(52) 1095 °C
Wt. Gain	71.2 gms.

⁺ Standard liters per minute per element.

* Numbers in parentheses refer to element stations.

Station 50

Station 30

Station 10

Figure 3-10. (CRD) Microstructure of GEM NbC Coating from Run C408
(200X, Etched). Type - Intermediate Hydrogen, WNCO (U)

**CONFIDENTIAL
RESTRICTED DATA**

Atomic Energy Act - 1954

Astronuclear
Laboratory

Type	GEM NbC	
Flows - Ar	2.20	⁺ LPME
HCl	0.56	LPME
H ₂	0.20	LPME
CH ₄	0.042	LPME
Salt	0.078	LPME
Total Flow	3.070	LPME

Run No.	B239
Run Time	7.0 hrs.
*Temp. S.P. 1	(-12) 1000 °C
S.P. 2	(6) 1088 °C
S.P. 3	(25) 1177 °C
S.P. 4	(50) 1302 °C
Wt. Gain	66.6 gms.

⁺ Standard liters per minute per element.

* Numbers in parentheses refer to element stations.

**CONFIDENTIAL
RESTRICTED DATA**

Atomic Energy Act - 1954

Station 50

Station 30

Station 10

Figure 3-11. (CRD) Microstructure of GEM NbC Coating from Run B239
(200X, Etched). Type - LASL Chemistry, WEFF (U)

**CONFIDENTIAL
RESTRICTED DATA**

Atomic Energy Act - 1954

Astronuclear
Laboratory

Type	GEM NbC
Flows - Ar	1.73 ⁺ LPME
HCl	0.56 LPME
H ₂	0.19 LPME
CH ₄	0.040 LPME
Salt	0.080 LPME
Total Flow	2.650 LPME

Run No.	C417
Run Time	7.0 hrs.
*Temp. S.P. 1	(-12) 1020°C
S.P. 2	(1) 1077°C
S.P. 3	(26) 1178°C
S.P. 4	(52) 1306°C
Wt. Gain	78.2 gms.

⁺ Standard liters per minute per element.

* Numbers in parentheses refer to element stations.

**CONFIDENTIAL
RESTRICTED DATA**

Atomic Energy Act - 1954

Station 49.5

Station 22.5

Station 4.5

Figure 3-12. (CRD) Microstructure of GEM NbC Coating from Run C417
(400X, Etched). Type - LASL Chemistry, WNCO (U)

CONFIDENTIAL
RESTRICTED DATA

Atomic Energy Act - 1954

Astronuclear
Laboratory

Type	GEM NbC
Flows - Ar	1.78 ⁺ LPME
HCl	0.64 LPME
H ₂	0.91 LPME
CH ₄	0.041 LPME
Salt	0.083 LPME
Total Flow	2.734 LPME

Run No.	L327
Run Time	8.0 hrs.
*Temp. S.P. 1	(-12) 990°C
S.P. 2	(1) 1080°C
S.P. 3	(26) 1175°C
S.P. 4	(52) 1335°C
Wt. Gain	99.9 gms.

⁺ Standard liters per minute per element.

* Numbers in parentheses refer to element stations.

CONFIDENTIAL
RESTRICTED DATA

Atomic Energy Act - 1954

Station 49.5

Station 22.5

Station 4.5

Figure 3-13. (CRD) Microstructure of GEM NbC Coating from Run L327
(400X, Etched). Type - LASL + Hi HCl, WNCO (U)

**CONFIDENTIAL
RESTRICTED DATA**

Atomic Energy Act - 1954

Astronuclear
Laboratory

Type	GEM NbC
Flows - Ar	1.79 ⁺ LPME
HCl	0.64 LPME
H ₂	0.19 LPME
CH ₄	0.041 LPME
Salt	0.091 LPME
Total Flow	2.752 LPME

Run No.	L349
Run Time	10 hrs.
*Temp. S.P. 1	(-12) 976°C
S.P. 2	(1) 1072°C
S.P. 3	(26) 1151°C
S.P. 4	(52) 1301°C
Wt. Gain	113.2 gms.

⁺ Standard liters per minute per element.

* Numbers in parentheses refer to element stations.

**CONFIDENTIAL
RESTRICTED DATA**

Atomic Energy Act - 1954

Station 49.5

Station 22.5

Station 4.5

Figure 3-14. (CRD) Microstructure of GEM NbC Coating from Run L349
(400X, Etched). Type - Pewee 2 Candidate, WNCO (U)

REFERENCES (U)

1. L. M. Slater, Final Report, Test Specification IDa, "GEM Coating Development," WANL-TME-1845, July 29, 1968.
2. J. P. Chasko, Final Report, Fuel Development Test Specification Phase IVA, "Qualification Coating Process," WANL-TME-1847, January 1969.
3. M. B. Blinn, Final Report, Phase IVB, "Fuel Coating Development," WANL-TME-1887, January 1969.