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WANL-TME-969

EVALUATION OF FOIL-TYPE
PYROLYTIC GRAPHITE INSULATING SLEEVES
(Title Unclassified)

R. C. Goodspeed
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Mr. R. W. Schroeder
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Attention: J. Lombardo

Subject: WANL-TME-969, Evaluation of Foil-Type Pyrolytic Graphite Insulating Sleeves.

Gentlemen:

The subject report describes the results of the evaluation of experimental NERVA reactor insulating sleeves made from a highly flexible, anisotropic, pyrolytic graphite foil, known as Grafoil-100. These Grafoil sleeves appear to have a great potential as replacements for the conventional vapor deposited and machined pyrolytic graphite sleeves. Besides the inherent advantages of uniformity and the absence of deposition defects, they appear to have satisfactory axial load deformation and resilience characteristics, and resistance to abrasion, thermal gradients, thermal shock, and hydrogen corrosion at 2000°F (1100°C). The areas of concern that remain are the higher and somewhat unstable thermal conductivity and the excessive hydrogen corrosion at 3000°F (1650°C).

Very truly yours,

H. F. Faught

H. F. Faught
Program Manager
NERVA Nuclear Subsystem

bb
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WANL-TME-969

EVALUATION OF FOIL-TYPE PYROLYTIC GRAPHITE INSULATING SLEEVES

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(Title Unclassified)

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ABSTRACT

Conventionally vapor deposited and machined pyrolytic graphite thermally insulating sleeves for the NERVA reactor have been very difficult to manufacture to specified requirements, resulting in high rejection rates with attendant high costs and poor delivery. Therefore, two experimental lots of NERVA reactor insulating sleeves made from a highly flexible, anisotropic, pyrolytic graphite foil, known as Grafoil-100, were evaluated as potential replacements. The Grafoil sleeves were submitted by High Temperature Materials, Inc. Both lots were evaluated for general appearance, dimensional control, density, bonding, resistance to abrasion, chemical impurity content, thermal conductivity as a function of temperature and of the number of thermal cycles, resistance to thermal gradients, resistance to thermal shock, resistance to hydrogen corrosion, and axial load deformation and resilience behavior. Also, Grafoil-100 in the foil form was investigated for density, chemical impurity content, thermal conductivity and tensile strength.

The results indicate that Grafoil sleeves have a great potential as replacements for the conventional vapor deposited pyrolytic graphite sleeves. Besides the inherent advantages of uniformity, the absence of deposition defects, the possibility of being made in longer lengths, and a considerable reduction in procurement lead time and cost, they appear to have satisfactory axial load deformation and resilience characteristics, and resistance to abrasion, thermal gradients, thermal shock, and hydrogen corrosion at 2000°F (1100°C). The areas of concern that remain are the higher and somewhat unstable thermal conductivity and the excessive hydrogen corrosion at 3000°F (1650°C). The vendor is being encouraged to solve these problems.

INTRODUCTION

Thermally insulating sleeves capable of withstanding extreme conditions of temperature and neutron flux in a hydrogen environment are required in the NERVA reactors to prevent excessive thermal gradients in the unfueled elements and to limit heat flow into the tie rod coolant channels. These sleeves (Figure 1) which are sandwiched between the unfueled graphite central elements and the stainless steel tie rod liner tubes along the full length of the elements will be exposed to temperatures as high as 4000°F (2200°C) on their outside surfaces in contact with the unfueled central element of a cluster in one part of the reactor and to temperatures as low as minus 250°F (minus 160°C) on their inside surfaces in contact with the tie rod liner tubes in another part of the reactor. Radial temperature gradients across a wall thickness of about 0.065 inch may be in excess of 3200°F (1780°C).

To date, these insulating sleeves have been made from pyrolytic graphite because of its extremely low across-grain thermal conductivity and its ability to withstand the imposed environmental conditions of temperature and neutron flux. However, the required quality and reliability have been difficult to obtain because of the unique combination of pyrolytic graphite's anisotropy, stringent reactor design requirements, and present state-of-the-art in the pyrolytic graphite manufacturing industry.

Pyrolytic graphite, made by the thermal decomposition of a carbonaceous gas at low pressure and the subsequent deposition of carbon on a heated mandrel, has a hexagonal structure (Figure 2) and a very high degree of anisotropy typical of graphite single crystals. The crystallographic "a-b" planes, composed of carbon atoms bonded

to carbon atoms, are deposited parallel to the substrate surface, regardless of its shape. The "c" axis is perpendicular to the "a-b" planes, which are held together only by weak van der Waal's forces. The carbon-carbon bonding and free electron transfer within the "a-b" planes (with-grain direction) account for pyrolytic graphite's extremely high with-grain thermal conductivity and tensile strength and its low with-grain electrical resistivity and thermal expansion. The weak bonding and lack of free electron transfer between planes (across-grain direction) account for its extremely low across-grain thermal conductivity and tensile strength and its high across-grain electrical resistivity and thermal expansion. Thus, as-deposited pyrolytic graphite sleeves are made by depositing pyrolytic graphite on appropriate cylindrical male mandrels (up to 1000 per furnace lot). Each as-deposited sleeve consists of cylindrical planes (i. e., in the with-grain or circumferential and longitudinal directions) and low strength and conductivity between the planes (i. e., in the across-grain or radial direction).

This anisotropy, which is responsible for the low thermal conductivity in the across-grain direction, is also the major cause of manufacturing problems and component failures. As a part with a radius of curvature, such as the sleeves, is cooled from its relatively stress-free state at deposition temperature, thermal stresses develop due to the anisotropy of across-grain to with-grain thermal contraction, the ratio of which may exceed 10/1 for the temperature range of 3550°F (1950°C) to room temperature. The magnitude of these stresses increases with increasing deposition temperature and increasing ratio of deposited thickness (t) to radius of curvature (r) of internal surface (i. e., t/r ratio). If the residual stress in the with-grain direction of the pyrolytic graphite exceeds the with-grain tensile strength, tensile failure in the form

of cracks occur. If the residual stress in the across-grain direction exceeds the across-grain tensile strength, the material delaminates. Because of pyrolytic graphite's high with-grain tensile strength and low across-grain tensile strength, the material usually fails by delaminating parallel to the basal planes. Such is the case for the sleeves which have a t/r ratio (0.440) greatly in excess of the 0.05 suggested for delamination-free components, and which generally contain multiple delaminations.

Large differences in deposition thickness frequently occur within individual sleeves (Figure 3) deposited per the present state-of-the-art processes. Such preferential deposition means that the basal planes are progressively re-oriented; they are parallel to the substrate surface at the substrate surface and parallel to the last deposited surface at the last deposited surface. Thus the machining of a sleeve from the as-deposited product invariably intersects many basal planes and one or more of the delaminations. The resulting sleeves have a reduced insulating capability and are prone to spalling during reactor assembly and operation.

Large microstructural differences of two types also frequently occur during deposition. The first type is a difference between the vertical extremes of the furnace (Figure 4) and are due to inherent gradients of temperature, pressure, composition, etc., within a furnace. The second type is a difference from the substrate surface to the last deposited surface (Figure 5) and is due to inadequate process control. Both may occur within a single furnace run, resulting in a variety of microstructures within a component and from component to component. Even larger microstructural differences have occasionally occurred from furnace load to furnace load.

Another problem area which has resulted in the rejection of many otherwise acceptable sleeves, is the presence of such unacceptable deposition defects as

excessively large nodules (i. e., isolated and abnormally large growth cones - Figure 6a) and sooting (i. e., alternate layers of amorphous and pyrolytic graphite - Figure 6b).

While substantial improvements in quality have been realized in the more recently manufactured sleeves by improving the uniformity of deposition thickness and the type and uniformity of microstructure, these two areas still remain problems which largely limit manufacturing yields and sleeve quality and reliability.¹

One of the approaches taken to alleviate the problem of procuring satisfactory insulating sleeves was the evaluation of Grafoil-100 and of insulating sleeves made from Grafoil-100 (Grafoil sleeves). This material, manufactured by High Temperature Materials, Inc., is a flexible, highly anisotropic graphite foil, which is made from pyrolytic graphite. The specific manufacturing details are proprietary. It possesses chemical inertness, thermal stability, and low thermal conductivity across its thickness, and can be rolled, wrapped, pressed, embossed, or otherwise formed into useful shapes and components. The manufacturer's listed properties² are given in Table I. This material in nominal 0.005 and 0.010 inch thicknesses was evaluated for general appearance, thickness, density, change in density under compression, microstructure, lattice parameters, chemical impurity content, tensile strength, and thermal conductivity.

An experimental lot (lot 1) of five Grafoil sleeves was manufactured by High Temperature Materials, Inc., by wrapping 0.010 inch thick Grafoil-100, coated with an epoxy resin, around male mandrels, and heat treating them to between 1470° and 1830°F (800° and 1000°C) while still on the mandrels, to carbonize the epoxy binder. These initial Grafoil sleeves were evaluated for general appearance, dimensions,

density, chemical impurity content, microstructure, thermal conductivity, and thermal stability.

The results of the preliminary evaluation were so encouraging that 20 more Grafoil sleeves (lot 2) were procured for a more extensive evaluation. These were made by essentially the same manufacturing processes except that 0.005 inch thick Grafoil-100 was used and the outside surface of each sleeve was machined to size. Lot 2 sleeves were evaluated for general appearance, dimensions, density, abrasion resistance, thermal conductivity, thermal gradient and thermal shock resistance, hydrogen corrosion resistance, and axial load deformation behavior. The inadequacy of the 1470° to 1830°F (800° to 1000°C) heat treatment became apparent when a sleeve exhibited severe signs of degradation during thermal shock testing. Those sleeves from lot 2 which had not already been used were then heat treated to 3630°F (2000°C). Because of the limited number of sleeves and time, some of the data was not regenerated using the heat treated sleeves. Therefore, the condition of the lot 2 sleeves, "as-received" or "heat-treated," shall be defined throughout this report.

Recently, a production lot of more than 800 Grafoil sleeves was procured from High Temperature Materials, Inc. Some of these sleeves are presently being evaluated, and the results will be reported in an addendum to this report.

CONCLUSIONS

The extensive evaluation of two experimental lots of NERVA reactor insulating sleeves made from Grafoil-100 by High Temperature Materials, Inc., has resulted in the following conclusions:

1. Grafoil sleeves have a great potential for replacing the conventional vapor deposited and machined pyrolytic graphite sleeves in NERVA reactors.
2. Besides the inherent advantages of uniformity, the absence of deposition defects, the possibility of being made in longer lengths, and a considerable reduction in lead time and cost, Grafoil sleeves appear to have satisfactory axial load deformation and resilience characteristics, and resistance to abrasion, thermal gradients, thermal shock, and hydrogen corrosion at 2000°F (1100°C).
3. The only areas of concern are the Grafoil sleeves' marginally high and somewhat unstable thermal conductivity and the excessive hydrogen corrosion at 3000°F (1650°C) which may have been influenced by the high impurity content.
4. Further determinations of thermal conductivity and hydrogen corrosion, by improved experimental techniques, are required on Grafoil sleeves representative of mass production techniques and manufactured to a specification controlling density, chemical impurity content, minimum temperature of heat treatment, etc., before the severity of these two potential problem areas can be realistically ascertained.

RECOMMENDATIONS

The following recommendations are made:

1. Further determinations of thermal conductivity and hydrogen corrosion should be made on mass-produced Grafoil sleeves, manufactured to specification requirements of density, chemical impurity content, minimum temperature of heat treatment, etc., using improved experimental techniques.
2. The effects of foil thickness, density, chemical impurity content, and heat treatment on the thermal conductivity of Grafoil sleeves should be determined.
3. The effects of irradiation on thermal conductivity, dimensional stability, and bond integrity of Grafoil sleeves should be determined over as large a temperature range as possible, by a pre-post irradiation study.
4. The same material and technique used for making Grafoil sleeves should be applied to the manufacture of such other NERVA reactor insulating components as tiles and washers.

RESULTS AND DISCUSSION

Part I: Grafoil-100

The Grafoil-100 material was a uniform, black, graphitic appearing foil, which was highly flexible and which could be easily formed into various shapes (Figure 7). Its nominal thicknesses were 0.005 and 0.010 inch. Attempts to accurately measure the thicknesses indicated that the material was quite compressible and that the measured thicknesses depended on the method of measuring. Thickness measurements obtained by using micrometers varied from 0.005 to 0.006 inch and from 0.010 to 0.011 inch.

As with the foil's thickness, its density varied depending on the method of measurement. Immersion density values ranged from 1.20 to 1.40 gms/cc (53 to 62 per cent of the theoretical density of graphite). However, the validity of these values is uncertain, as bubbles kept forming on the immersed samples as the immersion medium (water) continued to penetrate into them. Thus, the immersion density increased with time and appears to be relatively higher than the manufacturer's listed values of 0.96 to 1.28 gms/cc (42 to 57 per cent of theoretical). The foil's density, as determined using a mercury porosimeter (zero psi) was 1.112 gms/cc (49 per cent of theoretical). The standard deviation of the density was 0.025 gms/cc. These mercury porosimeter results appear to be in good agreement with the manufacturer's listed range.

The foil density also varied with applied pressure from 1.112 gms/cc at a porosimeter pressure of zero psi to 2.071 gms/cc (greater than 91 per cent of theoretical density) at a porosimeter pressure of 5000 psi. This data are presented in Figure 8.

Samples of foil, mounted in Hysol containing particles of aluminum, were examined at magnifications up to 500X. In contrast to pyrolytic graphite material, no growth cone structure was observed (Figures 9a and b). Many "micro-delaminations," less than 0.02 inch long and roughly parallel to the surfaces were present. However, the surfaces seemed to be continuous. The apparent non-uniformity of thickness may be related to the vacuum impregnation mounting technique. Figure 9b illustrates the material's mode of failure (i.e., it delaminates under tension and buckles under compression).

The lattice parameters, " c_0 " (unit cell dimension in the c axis direction) and L_a (average crystallite diameter in the a-b plane), were determined on both the 0.005 and 0.010 inch thick foils by standard x-ray diffraction techniques. The values of c_0 (obtained by extrapolating from the c_0 values obtained for the 002, 004, and 006 crystal plane reflections) ranged from 6.7142 to 6.7139 Å. These values indicate a very high degree of perfection within the crystallites (see Table II). The values of L_a ranged from 260 to 352 Å. In spite of the perfection within the crystallites, they were somewhat more randomly oriented than in annealed pyrolytic graphite, as might be expected from the manufacturing processes. Thus, Grafoil-100 appears to be composed of highly ordered crystallites of graphite bonded together with a somewhat more random orientation than observed in vapor deposited pyrolytic graphite.

Chemical analysis of two foil samples, using standard ashing and emission spectrographic analysis techniques, indicated an extremely high impurity content compared to the manufacturer's indicated impurity level of less than 0.1 per cent.² The ash determinations on the two samples were 1.72 and 1.55 per cent and the major impurities consisted of aluminum (2190 and 2490 ppm), iron (865 and 1165 ppm), and silicon (3010 and 3030 ppm). The complete analyses are presented in Table III. The

impurities were presumably all contained in the binder used to bond crystallites, as such concentrations of impurities within the crystallites would preclude their high degree of perfection as indicated by the c_o lattice parameter measurements and is not typical of massive vapor deposited pyrolytic graphite (used as the raw material for the production of Grafoil-100).

Tensile data were also generated on both the 0.005 and 0.010 inch thick Grafoil-100 materials (Table IV). The test specimens of the design shown in Figure 10 were carefully cut to prevent the occurrence of places where edge tearing might preferentially start. Each specimen was then carefully aligned in the grips to minimize uneven loading and the associated initiation of a tear at one side and its propagation across the specimen. The average measured tensile strengths of 660 psi for the 0.005 inch thick foil and 585 psi for the 0.010 inch thick foil are below the manufacturer's listed values of 1000 to 1200 psi², but are sufficient for the intended application. The standard deviations of 87 and 31 psi respectively indicate that the specimen design, preparation, and testing technique were satisfactory.

Finally, the room temperature thermal conductivity of Grafoil-100 (across its thickness) was determined by calculation from the measured ratio of its thermal diffusivity to that of ATJ graphite in the with-grain direction. Assuming the heat capacity of both Grafoil-100 and ATJ graphite to be the same at room temperature, the following equation is derived from the standard relationship between thermal conductivity and thermal diffusivity:

$$K_{c_G} = K_{ab_{ATJ}} \frac{P_{ATJ} \cdot \chi_{c_G}}{P_G \cdot \chi_{ab_{ATJ}}} \quad (1)$$

where K_{cG} = thermal conductivity of Grafoil-100 in "c" direction
 (in BTU-Ft/Hr-Ft²-°F)

K_{abATJ} = thermal conductivity of ATJ graphite at room temperature
 in with-grain direction = 98.5 BTU-Ft/Hr-Ft²-°F

P_G = density of Grafoil = 1.11 gm/cc

P_{ATJ} = density of ATJ graphite = 1.75 gms/cc

X_{cG}/X_{abATJ} = ratio of thermal diffusivity of Grafoil-100 in "c" direction
 to thermal diffusivity of ATJ graphite in with-grain
 direction = 0.0059 and 0.0053 for 0.005 and 0.010 inch
 thick foil respectively.

Substitution of the above values into equation (1) results in room temperature thermal conductivity values of 0.92 and 0.82 BTU-Ft/Hr-Ft²-°F. These conductivity values are slightly lower than the conductivity of pyrolytic graphite at room temperature and equivalent to the conductivity of pyrolytic graphite at 1000°F (535°C).

Part II: Grafoil Sleeves

General Appearance

The Grafoil sleeves from both lots 1 and 2 appeared to be uniform within a sleeve and from sleeve to sleeve within each lot (Figure 11a and b). However, the outer surface of lot 1 sleeves, which had not been machined, was considered superior to and less subject to spalling than the machined outer surface of lot 2 sleeves. The lot 2 sleeves did not seem to be especially subject to spalling, even after a gentle rubbing, as any particles protruding from the surface were not separated by repeated flexing (Figure 12). While no unbonded or unwrapped areas were observed on the outer laps of the lot 1 sleeves (and occasionally these outer laps could not even be identified), some tearing of the foil occurred on the inner surface of each sleeve. Bonding in the lot 2 sleeves (in both the as-received and heat treated condition) appeared to be adequate, with no unbonded or unwrapped areas or tearing of foil observed on either the outer or inner laps. The lot 2 sleeve outer laps were even more difficult to identify than those of the lot 1 sleeves. The inner laps of the lot 2 sleeves were plainly visible, but were essentially dimensionless since the sleeves had been heat treated on mandrels.

Dimensions and Density

The dimensions and densities of both lot 1 and lot 2 Grafoil sleeves are listed in Table V. All 20 sleeves from lot 2 were measured in the as-received condition although sleeves 8 through 20 were not measured until after the EML-44 component vibration test.⁷ The differences in the inside diameters between the two lot 2 groups are due to differences in the measuring technique rather than to dimensional changes from the vibration testing. The average density of the 20 lot 2 sleeves was

1.163 grams/cc, or approximately 55 to 60 per cent of the density of the conventional vapor deposited pyrolytic graphite sleeves, as compared to the average density of 1.04 grams/cc for the 5 lot 1 sleeves. This difference in the average density is attributed to a tighter, more uniform wrapping of the foil in the lot 2 sleeves. The standard deviation of the density of the 5 lot 1 sleeves and the 20 lot 2 sleeves was 0.055 and 0.047 grams/cc respectively.

Microstructure

Grafoil sleeves from both lots 1 and 2 were mounted in Hysol by vacuum impregnation, ground back about 1/4 inch, polished by the technique given in Appendix I, and metallographically examined to determine their microstructure and the degree of bonding between the layers of foil. An examination of several lot 1 sleeves indicated that they were manufactured with simple lap joints at both the inner and outer surfaces. Considerable variation in the degree of bonding existed from one lot 1 sleeve to another, as illustrated in Figures 13 and 14. In one sleeve the joints between layers of foil were quite pronounced and occasionally even unbonded areas existed (Figures 13a and 14a). In another sleeve the majority of the layers of foil appeared to be satisfactorily bonded to each other (Figures 13b and 14b). The densities of the 2 sleeves were 0.97 and 1.11 grams/cc respectively. However, the inner and outer laps of all the lot 1 sleeves showed indications of poor bonding and a tendency to unwrap. Otherwise the inner and outer surfaces appeared to be continuous and undisturbed. Measurements of the foil's thickness obtained from Figure 14 ranged from 0.0069 to 0.0087 inch.

The examination of lot 2 sleeves indicated that they were manufactured in the same manner as the lot 1 sleeves, with the exception of having a much improved

inner lap design illustrated in Figure 15. The general structure and degree of bonding were uniform, both within a sleeve and from sleeve to sleeve (Figure 16a and b) and the majority of the layers of foil (including the inner and outer laps) were satisfactorily bonded to each other. Thus, both the inner and outer surfaces appeared to be continuous and undisturbed. Measurements of the foil thickness obtained from Figure 16 ranged from 0.0050 to 0.0056 inch.

Resistance to Abrasion

Lot 1 Grafoil sleeves were not evaluated for resistance to abrasion. However, they did demonstrate a reasonable integrity and resistance to wear, unwrapping, spalling, etc., during handling, inspection, and evaluation.

Lot 2 sleeves were evaluated for resistance to abrasion, such as they would receive during inspection and assembly, by thoroughly examining thirteen of the as-received sleeves after they had been tested in both lateral and vertical vibration in the EML-44 component vibration test⁷ and after they had been completely inspected by Quality Control. All of the thirteen sleeves appeared to have withstood the associated handling with only minor scoring and tearing of the inner layer of foil. No signs of dimensional changes, wear, unwrapping, severe spalling, and severe scoring were observed.

Both as-received and heat treated sleeves from lot 2 were also brushed per standard practice for vapor deposited sleeves. The outside surfaces of both types of sleeves were subject to scoring, minor spalling, and even occasionally a localized unwrapping of the outside lap, when brushed with a stiff bristled brush (Figure 17). The inside surfaces were subject to considerably more spalling when brushed with a stiff wire brush. However, no unwrapping of the inside laps was observed. Such

brushing is an extreme test for Grafoil sleeves, but it demonstrated the sleeves' integrity and degree of bonding of both the inner and outer laps.

Chemical Composition

The impurity content of 1 sleeve from lot 1 was determined using standard ashing and emission spectrographic analysis techniques. The data are presented in Table VI. The ash determination of 1.58 per cent is in excellent agreement with the ash determinations of 1.72 and 1.55 per cent on the Grafoil-100 samples. Likewise, the content of each specific impurity is very similar to that found in the Grafoil-100 samples, with the minor exceptions of magnesium, tantalum, and zirconium. It is evident that no major contamination resulted during the manufacturing process from the bonding agent.

As lot 2 sleeves were manufactured from the same lot of Grafoil-100 for which the chemical analysis was available, an analysis was not obtained for the lot 2 sleeves. Based on manufacturer's assurances, it is expected that all Grafoil sleeves procured in the future will have an ash content of 0.3 per cent or less.

Thermal Conductivity

The thermal conductivity of both lot 1 and 2 Grafoil sleeves has been determined in the radial direction by Southern Research Institute using their standard equipment and technique.^{8,9} The thermal conductivity for 2 lot 1 sleeves, as determined at mean radial temperatures of about 2000°F and 3000°F, (1100° and 1650°C), was moderately higher than that for the conventional vapor deposited and machined pyrolytic graphite sleeves¹ (Figure 18). The limited data, while inconclusive, suggested that the conductivity of the Grafoil sleeves was inversely proportional to their density.

Thermal conductivity data were obtained on three lot 2 sleeves in the as-received condition as a function of mean radial temperature from about 500°F (260°C) to about 3000°F (1650°C) and of the number of retest cycles (Table VII). The thermal conductivity again increased with temperature but was moderately higher than that of the lot 1 sleeves and considerably higher than that of the conventional vapor deposited and machined pyrolytic graphite sleeves. The following linear equation was obtained from the first cycle data (20 points from the three sleeves), using a least squares fit to the standard multiple regression program:

$$K_c = 0.198 + 0.08157 \times 10^{-3} T \quad (2)$$

where

$$K_c = \text{radial thermal conductivity in BTU-Ft/Hr-Ft}^2\text{-}^\circ\text{F}$$

$$T = \text{average radial temperature of sleeve in } ^\circ\text{F}$$

See Figure 19. The standard deviation of the thermal conductivity at the average temperature (1656°F, 900°C) was 0.053 BTU-Ft/Hr-Ft²-°F. A less apparent relationship existed between thermal conductivity and sleeve density for these lot 2 sleeves than existed for the lot 1 sleeves. However, a comparison of the data from both lots of sleeves indicated that the conductivity increased with increasing density.

The second cycle thermal conductivity data were significantly higher than for the first cycle, but could not be fitted to a curve because of the scatter. However, further cycling did not cause further appreciable changes. The following linear equation was obtained from all of the conductivity data, using the multiple regression technique:

$$K_c = 0.29374 + 0.06669 \times 10^{-3} T \quad (3)$$

The standard deviation of the thermal conductivity at the average temperature (1612°F , 875°C) was $0.113 \text{ BTU-Ft/Hr-Ft}^2\text{-}^{\circ}\text{F}$. As can be seen from Figure 20, the thermal conductivity still increases with increasing temperature, but not as rapidly. The increase in conductivity from the first to the second cycle was originally credited to changes in the sleeves as they underwent the imposed heat treatment. This viewpoint appeared to be supported by the fact that further changes did not occur upon further cycling. An examination of the cycled sleeves indicated that some of the inner and outer layers of foil had been torn locally (Figure 21), which may also account for part of the increase in the conductivity. Any further conductivity testing of Grafoil sleeves will be done by an improved technique to prevent such damage.

Resistance to Thermal Gradients

The ability of the Grafoil sleeves to withstand severe and repeated thermal gradients was evaluated by examining the three sleeves from lot 2 which had been exposed to gradients of about 4000°F (2200°C) at the outside surface to about 2000°F (1100°C) at the inside surface during thermal conductivity testing. The sleeves, numbers 3, 6, and 7, were cycled three, five, and two times respectively. No adverse effects due to the thermal gradients, such as unbonding, unwrapping, etc., were observed visually. However, some unbonding between the layers of foil was observed metallographically. The structure of sleeves 3, 6, and 7 (both before and after thermal conductivity testing) are shown in Figures 22, 23, and 24 respectively. Such minor unbonding within the sleeves is not expected to appreciably affect their performance within NERVA reactors. The disturbed condition of the outer and inner laps was caused by somewhat unsatisfactory thermal conductivity test techniques and not by the thermal gradients.

Resistance to Thermal Shock

Grafoil sleeves from lot 2 were evaluated for resistance to thermal shock between a) room temperature and 4530°F (2500°C) and b) room temperature and liquid nitrogen temperature (minus 320°F, minus 195°C). Several sections of as-received sleeves were inductively heated to 4530°F (2500°C) in less than a minute, and severe damage (i. e., blistering and swelling of the outer layers of foil, collapsing of the inner layers of foil, and some unwrapping of the laps) occurred due to sudden internal gas formation from thermal decomposition of the bonding agents, binder, etc., (Figure 25a). This process was then repeated, using several sections of a sleeve heat treated to 3630°F (2000°C). This time only a small blister on the inside surface and some unwrapping of the inner lap were observed (Figure 25b), indicating that the heat treatment was beneficial. Another section of heat treated sleeve was similarly tested to 3630°F (2000°C), with even less damage occurring (Figure 25c). One small blister was observed on the inside surface.

Several other sections of both as-received and heat treated sleeves were immersed in a liquid nitrogen bath until the boiling subsided. Immediately after removal from the bath, there were no detrimental effects observed on any of the sleeves, regardless of their previous heat treatment. However, as each sleeve warmed up to room temperature, the foil layers near each end separated until the outer layers split and the inner layers collapsed (Figures 26a and b). Again these results were independent of heat treatment. A second such cycle compounded the damage greatly (Figures 26c and d). While the sleeves withstood the cooling cycle, they apparently failed during warmup due to the rapid expansion of liquid nitrogen which had penetrated between the layers of foil during the immersion.

The resistance to thermal shock of Grafoil sleeves, which have been heat treated to 4000°F (2200°C) or above, to complete the thermal decomposition of the bonding agent, binder, etc., should be entirely satisfactory under the environmental conditions within the NERVA reactors (i. e., they will not be exposed to liquid hydrogen). Thus, all Grafoil sleeves procured in the future will have a 4000°F (2200°C), minimum, heat treatment specified as a requirement.

Hydrogen Corrosion Resistance

The comparative hydrogen corrosion resistance of lot 2 Grafoil sleeves, in the as-received condition, and conventional vapor deposited and machined pyrolytic graphite sleeves was determined at about 2000°F and 3000°F (1100° and 1650°C). The experimental test setup is illustrated in Figure 27. Each sleeve was individually heated to temperature by the 1/2 inch ID graphite tube heater, which was niobium carbide coated to protect it from hydrogen corrosion. Hydrogen gas was flowed through the annulus between the heater and the sleeve. The temperature of the sleeve was determined optically at a point 2-3/8 inches from the gas inlet end by means of a hole in the heater tube. As the hydrogen gas was not preheated, the recorded temperature was only approximate (i. e., the inlet end of the sleeve was colder and the outlet end was hotter). The precise variation of the temperature along the length of the sleeves has not been adequately determined to date. Other corrosion test parameters of hydrogen pressure and flow were 400 psi and 250 cfh, respectively.

No visually apparent corrosion occurred on either the conventional pyrolytic graphite sleeve or on the Grafoil sleeve during a nine-minute test at 2000°F (1100°C), except for possibly a slightly increased tendency towards spalling of the machined surface of the Grafoil sleeve. The respective weight losses of 0.0057 grams for the conventional vapor deposited sleeve and 0.0065 grams for the Grafoil sleeve were

approximately equal. However, because of the difference in weights, the respective per cent weight losses were 0.08 and 0.16.

No visually apparent corrosion of the vapor deposited sleeve was observed after a ten-minute test at 3000°F (1650°C). However, the Grafoil sleeve exhibited severe corrosion (i. e., loss of material, resulting in a highly irregular surface quite subject to spalling) at the gas outlet end (Figure 28), where the temperature was undoubtedly in excess of 3000°F (1650°C). This excessive corrosion may possibly be related to the high impurity content of the Grafoil material (i. e., preferential corrosion may have occurred at the highly impure boundaries between crystallites, which were then eroded away). The respective weight losses were 0.0171 grams for the vapor deposited sleeve and 0.5216 grams for the Grafoil sleeve and the respective per cent weight losses were 0.24 and 13.3. While the combined test conditions of temperature, pressure, and flow were extreme, the data indicate that hydrogen corrosion resistance might be a problem at or above 3000°F (1650°C).

Axial Load Deformation and Resilience Behavior

Axial load deformation and resilience data were generated on three of the heat treated Grafoil sleeves from lot 2. Each sleeve was tested in axial compression in a fixture which contained it on both the outside and inside (Figure 29), as an initial attempt to obtain this data on unrestrained sleeves resulted in failure of the sleeves (as illustrated in Figure 30) at loads ranging from 17.6 to 35.0 pounds. A load of approximately ten pounds (~ 47 psi) was applied to each sleeve and then released. This same process was repeated for loads of approximately 20, 30, and 40 pounds, which are all well in excess of the loads expected in actual operation. Total and plastic deformations for the four inch long sleeves (Table VIII and Figure 31) were obtained directly from the recorder chart (Figure 32). At 40 pounds (~ 188 psi) the total and plastic deformation were about 0.0235 and 0.0105 inch respectively. None of the sleeves jammed in the fixture, or otherwise showed indications of damage.

APPENDIX I: Metallographic Preparation of Pyrolytic Graphite Foils

The pyrolytic graphite foil samples are mounted in a prepared liquid Hysol thermal setting plastic, containing one part by weight Hysol hardener, number 3410, and ten parts Hysol resin, number 2038. The mounted samples are then loaded into a small vacuum - oven to remove all entrapped air and cured under vacuum for two hours at a constant temperature of 140°F (60°C).

The samples are prepared for metallographic examination by grinding on 240, 400, and 600 grit silicon carbide papers successively. Water is used on the first two grades of paper. The third is used dry. The samples are then hand ground on three zero emery paper.

The preliminary polishing is done with diamond abrasive (6 μ , 3 μ , and 1 μ) on Buehler Met cloth. The intermediate polish is performed on Micro cloth with a mixture of Linde B and 2 per cent chromic acid in water using heavy pressure. The final polish is done on Buehler Rayvel cloth with a distilled water slurry of magnesium oxide using light pressure.

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TABLE I: Manufacturer's Listed Properties of
Grafoil-100²

Property

Physical Data

Density	0.96 to 1.28 gms/cc
Purity	99.9 + %
Ash Content	< 0.06 %

Thermal Data

Thermal Conductivity ("c" direction) at 1000°F	0.3 to 0.6 BTU-Ft/Hr-Ft ² -°F
Thermal Shock Resistance	Excellent

Mechanical Data

Ultimate Tensile Strength	1000 to 1200 psi
Elastic Modulus (in tension)	0.184×10^{-6} psi

TABLE II: Lattice Parameters of Grafoil-100

<u>Material</u>	<u>Co*</u> <u>(Å)</u>	<u>La*</u> <u>(Å)</u>
Perfect Graphite ³ (Theoretical Values)	6.7079 + 0.0007Å	infinite
Natural Flake Graphite ³	6.719Å	3100Å
Grafoil Sample Number 1	6.7142Å	317 - 352Å
Grafoil Sample Number 2	6.7139Å	260 - 318Å
As-Deposited ^{4,5,6} Pyrolytic Graphite	6.82 - 6.84Å	250 - 325 Å

*Co is the unit cell dimension in the "c" axis direction

La is the average crystallite diameter in the "a-b" plane

TABLE III: Spectrographic Analysis of Grafoil-100

<u>Element</u>	<u>Foil No. 1 (ppm)</u>	<u>Foil No. 2 (ppm)</u>
Ag	5.2	1.7
Al	2190	2490
Au	<5	<5
B	0.5	0.2
Ba	17	16
Be	<0.2	<0.2
Bi	<2	<2
Ca	38	17
Cd	<5	<5
Co	7.2	6.7
Cr	7.5	7.8
Cu	33	23
Fe	865	1165
In	<5	<5
Mg	72	50
Mn	55	55
Mo	<2	<2
Nb	<0.2	<0.2
Ni	24	23
Pb	19	4.7
Sb	<2	<2
Si	3010	3030
Sn	<2	<2
Sr	2	2
Th	<20	<20
Ti	37	21
Ta	59	53
V	24	39
W	36	37
Zn	29	37
Zr	4.1	1.6
Ash	1.72%	1.55%

± 30% relative

TABLE IV: Tensile Strength of Grafoil-100

<u>Sample Number</u>	<u>Area (inches²)</u>	<u>Load (pounds)</u>	<u>Tensile Strength (psi)</u>
0.005 inch thick Grafoil			
1	1.58×10^{-3}	1.02	647
2	1.53	1.01	660
3	1.52	0.65	427*
4	1.73	1.17	677
5	1.77	--	--**
6	1.42	0.73	515
7	1.24	0.73	589
8	1.59	1.29	812
9	1.41	1.00	709
10	1.76	1.19	678
Average = 661 psi			
Range = 515 to 812 psi			
Standard Deviation = 87 psi			
0.010 inch thick Grafoil			
1	2.94×10^{-3}	1.36	463***
2	3.02	1.66	550
3	3.07	1.45	472***
4	2.98	1.87	627
5	2.94	1.71	582
6	2.66	1.60	602
7	2.69	1.64	387***
8	2.87	1.48	516***
9	2.85	1.60	561
10	2.99	1.40	468***
Average = 584 psi			
Range = 550 to 627 psi			
Standard Deviation = 31 psi			

*Sample was poorly aligned in grips

**Sample was destroyed during set-up.

***Samples showed signs of tearing prior to testing and broke in the fillet areas.

TABLE V: Dimensions and Density of Grafoil Sleeves*

<u>Number</u>	<u>Length (inches)</u>	<u>OD (inches)</u>	<u>ID (inches)</u>	<u>Weight (grams)</u>	<u>Density (gms/cc)</u>
Lot 1 Sleeves					
1	4.047	0.358	0.249	3.5691	1.04
2	4.047	0.358	0.249	3.7096	1.08
3	4.047	0.358	0.249	3.7960	1.11
4	4.047	0.356	0.249	3.2922	0.97
5	4.047	0.356	0.249	3.3333	0.98

Average = 1.04 gms/cc
 Range = 0.97 to 1.11 gms/cc
 Standard Deviation = 0.061 gms/cc

Lot 2 Sleeves

1	4.005	0.360	0.250	3.986	1.152
2	4.005	0.359	0.249	3.950	1.144
3	4.006	0.359	0.249	4.485	1.299
4	3.991	0.358	0.248	3.887	1.136
5	3.997	0.360	0.248	4.018	1.149
6	3.997	0.358	0.248	3.885	1.134
7	4.005	0.357	0.249	3.904	1.157
8	4.005	0.361	0.246	3.817	1.066
9	4.007	0.358	0.245	4.308	1.221
10	4.003	0.360	0.247	4.115	1.166
11	3.997	0.358	0.246	4.237	1.211
12	3.999	0.358	0.245	3.928	1.119
13	3.995	0.360	0.248	4.054	1.177
14	4.002	0.360	0.246	4.228	1.188
15	3.991	0.359	0.246	3.990	1.129
16	4.012	0.358	0.248	4.053	1.173
17	4.009	0.361	0.247	4.164	1.165
18	4.005	0.357	0.246	4.077	1.188
19	4.003	0.360	0.247	4.085	1.162
20	4.005	0.359	0.247	3.925	1.123

Average = 1.163 gms/cc
 Range = 1.066 to 1.299 gms/cc
 Standard Deviation = 0.047 gms/cc

*All values are for sleeves in the as-received condition.

TABLE VI: Spectrographic Analysis of Grafoil Sleeve

<u>Element</u>	<u>Sleeve (ppm)</u>
Ag	0.8
Al	2300
Au	--
B	1.1
Ba	--
Be	<0.2
Bi	<2
Ca	35
Cd	<20
Co	3.3
Cr	4.3
Cu	9.2
Fe	950
In	<20
Mg	175
Mn	49
Mo	<2
Nb	<0.5
Ni	24
Pb	12
Sb	<5
Si	2900
Sn	<2
Sr	0.2
Th	--
Ti	20
Ta	<5
V	35
W	30
Zn	35
Zr	12
Ash	1.58%

+ 30% relative
-

TABLE VII: Radial Thermal Conductivity of Lot 2 Grafoil Sleeves⁹

Sleeve Number	CYCLE No. 1		CYCLE No. 2		CYCLE No. 3		CYCLE No. 4		CYCLE No. 5	
	Mean Temp. (°F)	KC (BTU-Ft/ Hr-Ft ² -°F)	Mean Temp. (°F)	KC (BTU-Ft/ Hr-Ft ² -°F)	Mean Temp. (°F)	KC (BTU-Ft/ Hr-Ft ² -°F)	Mean Temp. (°F)	KC (BTU-Ft/ Hr-Ft ² -°F)	Mean Temp. (°F)	KC (BTU-Ft/ Hr-Ft ² -°F)
3	484	0.268								
	1028	0.297								
	1629	0.361								
	470	0.244								
	1015	0.295								
	1617	0.322								
	2038	0.490*	427	0.531	507	0.336				
	2483	0.408	1438	0.572	1561	0.283				
	2957	0.437	2471	0.628	2466	0.425				
6	470	0.242								
	992	0.342								
	1595	0.377								
	2154	0.445	535	0.319	519	0.170	491	0.774*	504	0.310
	2582	0.468	1448	0.505	1537	0.232	1588	0.531	1486	0.457
	2973	0.508	2672	0.591	2493	0.264	2433	0.612	2573	0.542
7	520	0.181								
	1043	0.213								
	1626	0.252								
	2002	0.307	455	0.625						
	2465	0.326	1516	0.527						
	3009	0.368	2446	0.491						

*Values are questionable

**ID thermocouple appeared to be reading low, probably due to poor contact

TABLE VIII: Axial Load Deformation Behavior of Lot 2 Grafoil Sleeves*

Sleeve Number	Load (pounds)	Deformation in Four Inch Sleeve	
		Total (inches)	Plastic (inches)
8	4	0.0030	0.0010
	20	0.0092	0.0024
	30	0.0146	0.0048
	40	0.0240	0.0104
9	10	0.0066	0.0020
	20	0.0114	0.0042
	30	0.0162	0.0064
	40	0.0222	0.0094
10	10	0.0056	0.0016
	20	0.0094	0.0032
	30	0.0136	0.0050
	40	0.0240	0.0110

Crosshead speed = 0.1 in/min

*Values read directly from recorder

FIGURE 1 NERVA REACTOR INSULATING SLEEVE

604134A

FIGURE 2 HEXAGONAL CRYSTAL STRUCTURE OF GRAPHITE

604135A

FIGURE 3: Non-uniform Deposition Thickness of As-Deposited
Pyrolytic Graphite Sleeves 1X

a) Top of Furnace Load

b) Bottom of Furnace Load

FIGURE 4: Microstructural Differences between Vertical Extremes
of a Furnace Load of Pyrolytic Graphite Sleeves 25X

a)

b)

FIGURE 5: Microstructural Differences throughout the Deposited Thickness of Pyrolytic Graphite Sleeves · 25X

a) Nodule

b) Sooting

FIGURE 6: Typical Deposition Defects in
Pyrolytic Graphite Sleeves 25X

FIGURE 7: General Appearance and Formability of Grafoil-100 1X

FIGURE 8 DENSITY OF GRAFOIL - 100 AS A FUNCTION OF APPLIED PRESSURE

WANL-TME-969

a) 0.005" Thick
Grafoil-100

b) 0.010" Thick
Grafoil-100

FIGURE 9: Microstructure of Grafoil-100 50X

604136A

FIGURE 10 DESIGN OF GRAFOIL-100 TENSILE SPECIMENS

a) Lot 1 Sleeve

b) Lot 2 Sleeves

FIGURE 11: General Appearance of Grafoil Sleeves 1X

FIGURE 12: Flaking and Spalling Characteristics of Grafoil Sleeves ~1X

a) Poor Bonding

b) Satisfactory Bonding

FIGURE 13: Typical Structures of Lot 1 Grafoil Sleeves 8X

a) Poor Bonding

b) Satisfactory Bonding

FIGURE 14: Typical Microstructures of Lot 1 Grafoil Sleeves 50X

604137A

FIGURE 15 ARRANGEMENT OF INNER LAP OF LOT 2 GRAFOIL SLEEVES

a) 8X

b) 50X

FIGURE 16: Typical Structure and Microstructure of Lot 2 Grafoil Sleeves

Brushed End

FIGURE 17: Appearance of Grafoil Sleeve Brushed with a
Stiff Bristled Brush 1X

FIGURE 18 RADIAL THERMAL CONDUCTIVITY OF LOT 1 GRAFOIL SLEEVES

FIGURE 19 RADIAL THERMAL CONDUCTIVITY OF LOT 2 GRAFOIL SLEEVES

FIGURE 20 RADIAL THERMAL CONDUCTIVITY OF LOT 2 GRAFOIL SLEEVES

FIGURE 21: Appearance of Grafoil Sleeve Samples after Having Been Tested for Thermal Conductivity IX

a) Before

b) After

FIGURE 22: Structure of Lot 2 Grafoil Sleeve Number 3 Before and After Being Subjected to Severe and Repeated Thermal Gradients 8X

a) Before

b) After

FIGURE 23: Structure of Lot 2 Grafoil Sleeve Number 6 Before and After Being Subjected to Severe and Repeated Thermal Gradients 8X

a) Before

b) After

FIGURE 24: Structure of Lot 2 Grafoil Sleeve Number 7 Before and After Being Subjected to Severe and Repeated Thermal Gradients 8X

a)

b)

c)

FIGURE 25: Thermal Shock Damage to Lot 2 Grafoil Sleeves 1X

a) 1X

b) 8X

FIGURE 26: Liquid Nitrogen Damage to Lot 2 Grafoil Sleeves

c) 1X

d) 8X

FIGURE 26: Liquid Nitrogen Damage to Lot 2 Grafoil Sleeves
(continued)

FIGURE 27 APPARATUS FOR DETERMINING HYDROGEN CORROSION OF GRAFOIL SLEEVES

Grafoil Sleeve

Pyrolytic Graphite
Sleeve

FIGURE 28: Hydrogen Corrosion of Grafoil and Conventional Pyrolytic Graphite
Sleeves after 10 Minutes at 3000°F 1X

FIGURE 29: Fixture for Containing Grafoil Sleeves during Axial
Compression Testing $\sim 1/2X$

a)

b)

FIGURE 30: Type of Failure Exhibited by Unrestrained Grafoil Sleeves under Axial Compression 4X

FIGURE 31 AXIAL LOAD DEFORMATION OF LOT 2 GRAFOIL SLEEVES

604142A

FIGURE 32 RECORDER CHART OF AXIAL LOAD DEFORMATION TESTS ON LOT 2 GRAFOIL SLEEVES

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