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November 1969

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Westinghouse Astronuclear Laboratory

EFFECTS OF COMPOSITION AND PROCESSING
ON THE PROPERTIES OF NIOBIUM CARBIDE -
GRAPHITE COMPOSITE MATERIAL

(Title Unclassified)

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ABSTRACT

(CRD) A process was developed for the hot press fabrication, from powder, of crack-free, strong billets of NbC-graphite using acicular calcined petroleum coke as the graphite source. Thermal expansion measurements showed a reduction in expansion coefficient in the with-grain (WG) direction compared with material of similar composition made with isotropic graphite flour. The reduction was greater (15%) at the lower NbC content (65 w/o) than at the higher (75 w/o) content (5%). The reduction in expansion coefficient was accompanied by a substantial (35 to 40%) reduction in the across-grain (AG) room temperature flexural strength. AG creep deformation rates at 2500° C and WG room temperature flexural strength were not affected significantly. Post pressing the billets at 2700° C did not affect properties. Post pressing at 2500° C lowered the AG creep deformation rate at 2500° C. Lowering the composition from 75 w/o to 65 w/o decreased the WG expansion coefficient, decreased the AG compressive creep rate, and decreased the room temperature flexural strength, particularly in the AG direction. The composition effect was more pronounced with calcined petroleum coke composites than with flour composites. The averaged data is shown in Table 18. An analysis of quality control data including creep deformation rate, expansion coefficient and flexural strength performed on 49 vendor fabricated 75/25 post pressed calcined petroleum coke billets supported the conclusions reached in the experimental program.

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I. FOREWORD

(CRD) Several years of effort have been directed toward developing carbide-graphite composite materials at LASL¹⁾ and at WANL. This has culminated in its utilization in certain components in the hot end support section of experimental reactors. These components were machined from hot pressed compacts made from a mixture of nuclear grade graphite flour and niobium carbide powder to the composition 75 w/o NbC. The raw materials and processing have been optimized²⁾ and typical properties for the optimized process are given in Table 1. Averages of similar data on similar material made by optimum processing by 4 alternate vendors³⁾ are included in Table 1.

(CRD) The effects of raw material purity and morphology on properties have been studied at both LASL and WANL⁴⁾. Certain metallic impurities, such as iron, nickel, and cobalt are detrimental to composite creep properties if present in quantities greater than about 0.03%. Further, the creep properties were found to be degraded slightly when a more acicular graphite particle shape is used.

(CRD) The effects of processing on the properties of 75 w/o NbC composite made with flour have been evaluated²⁾ and several conclusions have been reached. The most critical parameter, from a process control point of view, is the compacting temperature. The pressings are normally made at 3050° C and 3000 psi and are held for 20 minutes. Lowering the temperature by 200° C lowered the density by 2%, doubled the creep rate and lowered the flexural strength by approximately 25%. Lowering the pressure to 1000 psi lowered the density by 4%, doubled the creep rate, and lowered the flexural strength by approximately 25%. Heat-up rate and temperature at which pressure is applied were found to be unimportant insofar as material properties were concerned. The use of fine, -325 mesh, flour decreased the density by 2%, tripled the creep rate and lowered the flexural strength by approximately 35%.

(CRD) A very limited number of billets containing 40 w/o NbC, 40 w/o TaC, 20 w/o graphite flour, in which the volume percent flour is identical with that of 75 w/o NbC material, had been fabricated and evaluated. The properties were very close to those of 75-25 material as reported in Table 1 except that the creep rate was somewhat lower. Good billet homogeneity was not achieved.

(CRD) Design considerations had indicated that the 75 w/o NbC 25 w/o graphite flour composite material was not completely satisfactory in the fuel tip in the hot end support area in that the mismatch between the coefficients of thermal expansion between it and the graphite could result in excessively high stresses. This required that the expansion coefficient in the with-grain (WG) direction be lowered. Further, improvements in creep rate are always desirable. Improvements in these properties should preferably not be made to the detriment of room temperature flexural strength since transient thermal tensile stresses are known to occur in the end use application, particularly in the across-grain direction.

(CRD) It was known from studies made at IITRI⁵⁾ that the WG coefficient of thermal expansion could be lowered by using, as the carbon type, either calcined petroleum coke or Ceylon graphite instead of graphite flour. Studies at both IITRI and WANL revealed that the WG expansion coefficient could be lowered by lowering the NbC content. At WANL,²⁾ a billet containing a lower NbC content, 50 w/o, had been fabricated, using graphite flour, and evaluated to demonstrate the effects of increasing free carbon content on composite properties. It was found to have a density of 92.6% of theoretical, a 2500° C - 4000 psi - 1 hour AG compressive creep deformation of 2.5%, an average WG expansion of $5.4 \times 10^{-6}/^{\circ}\text{C}$ from 25° to 2400° C, and a WG and AG flexural strength of 11,400 and 6100 psi, respectively.

(CRD) It was known that the creep rate could be lowered by simply compressing or upset forging, a billet at the same temperature at which the creep test is run. This is obvious from examination of the equation: $\epsilon = At^n$.

where ϵ is the creep strain, t is the time and A and n are constants. It has been shown that it is equal to approximately $2/3$ for 75 w/o NbC composite.⁴⁾ The equation had been shown to apply between 0.01 and 1.0 hours so that a straight line could be obtained on a log strain versus log time plot over this time span. For example, a billet which crept 4% at 2500° C under a 4000 pound compressive load ring during the first hour would be expected to creep only 2.4% during the second hour in accordance with the relationship.

(U) The goal of the IITRI study⁵⁾ was to lower the creep deformation of carbide-graphite composites and to make the material more isotropic. During the program, they inadvertently produced material that was quite anisotropic. A summary of their data that is pertinent to this investigation is listed in Table 2. It is apparent that the use of either calcined petroleum coke or Ceylon graphite has lowered the with-grain (WG) expansion coefficient but to the detriment of the hot strength in both directions and of the cold strength in the across-grain (AG) direction. It is considered possible that the use of coarser powder would not have resulted in this hot strength lowering. Further, Ceylon graphite was known to have a high impurity content and this factor could also have lowered the hot strength.

(CRD) The exact mechanism of the development of thermal expansion anisotropy was not known. However, it was obviously associated with an orientation of the hexagonal crystallites in the graphite phase since the carbide phase has an isotropic cubic crystalline structure. The a-b axes of the graphite crystals are aligned preferentially along the longest axes of the particles which are in turn aligned in the WG direction in the billet. This necessarily introduces opposing stresses in the 2 phases with the carbide phase being in tension in the WG direction, as the lower thermal contraction of the graphite needles restrains contraction of the NbC on cooling.

(CRD) WANL sponsored studies made at Carborundum early in 1968⁷⁾ indicated that the WG expansion coefficient of 75-25 composite is lowered by approximately 5% by using

calcined petroleum coke instead of graphite flour. The data showed that the WG expansion coefficient of the coke composite could be further lowered, by 10%, to 6.6×10^{-6} per °C, by subjecting the billet to a post pressing operation which consisted of holding the billet at 2700° C under an axial compressive load of 2000 psi for 1 hour. The billet was completely unrestrained during this operation; that is, the graphite die was removed prior to the post pressing operation. This lowering the expansion coefficient was achieved without detriment to the creep properties or the WG flexural strength. The AG flexural strength was not measured.

(CRD) The study at Carborundum⁷⁾ included a minimal effort with coke composites with a lower NbC content. For example, a 65 w/o NbC coke composite was found to have a WG expansion coefficient of 6.1×10^{-6} per °C.

(CRD) The present investigation was undertaken by and at WANL primarily to determine the property changes that result when the graphite flour raw material in 75 and in 65 w/o NbC composite is replaced with calcined petroleum coke and to determine the property changes that further result when the coke billet is subjected to an unrestrained post pressing operation. A total of 24 coke billets of 75 w/o NbC composition were fabricated, 12 of which were subjected to a secondary post pressing operation similar to that employed in the WANL-sponsored study at Carborundum. One flour billet of 75-25 material was fabricated and post pressed in a similar way. Four coke billets and 2 flour billets containing 65 w/o NbC were fabricated and half of them were given a post pressing treatment consisting of holding for 1 hour at 2500° C and 3000 psi. It was predicted that the lower post pressing temperature would improve the creep rate as well as the expansion characteristic. (One coke billet and one flour billet of the composition 35 w/o NbC 35 w/o TaC balance carbon were fabricated but not post pressed.) This composition contains the same volume percent of free carbon as does the 65-35 material.

(U) Each billet fabricated at WANL was evaluated with respect to its density, WG expansion coefficient, AG creep rate, and flexural strength and electrical conductivity in both grain directions.

(CRD) In addition to the major laboratory program, a creep test exchange program with LASL was completed. Billets of composition 40 w/o TaC 40 w/o NbC balance graphite fabricated at LASL and one billet of composition 75 w/o NbC balance graphite fabricated at WANL were creep tested in both laboratories under identical conditions. The data was then analyzed as a means of comparing the relative accuracy and precision in the 2 facilities.

(CRD) During the same period of time that the present program was underway, 5 batches of calcined petroleum coke billets, a total of 49, containing 75 w/o NbC were made as part of the requirement for fuel tip and support pedestal hardware for Pewee 2. This material was given the 2700° C post pressing treatment. The quality control data, consisting of creep and flexural strength on each billet and expansion coefficient on some of the billets is presented here to demonstrate process control during a production run. Electrical conductivity measurements were made on the flexural specimens that were machined from the first 29 billets. In addition, two of the billets were cut into creep, flexural strength, and expansion specimens in order to demonstrate material homogeneity by measuring the property spread as a function of specimen location within the billet.

II. MAJOR OBJECTIVES (U)

- (CRD) 1. The general aim of the program is to lower the with-grain coefficient of expansion and/or the across-grain creep deformation rate of a carbide-graphite composite with a minimum of detriment to the other properties.
- (U) 2. A specific aim of the program is to determine the effect on properties of replacing the graphite flour raw material in NbC-carbon composite material with calcined petroleum coke.
- (U) 3. Another specific aim of the program is to determine the effects of an unrestrained hot post pressing operation on the properties of NbC-graphite billets using calcined petroleum coke.
- (CRD) 4. Another specific objective of the program is to determine the effects of composition variation over the range 65 to 75 w/o (35 to 47 v/o) NbC on the properties of both NbC-flour and NbC-coke composites.

III. MAJOR CONCLUSIONS (U)

(CRD) The following property variations refer to 75 w/o NbC-graphite flour composite as baseline for items A through E.

A. WG THERMAL EXPANSION COEFFICIENT (U)

- (CRD) 1. May be decreased 5% by lowering the composition to 65 w/o NbC. ✓
(See Table 18.)
- (CRD) 2. May be decreased 5% by substitution of needle coke for graphite flour. ✓
(see Table 18.)
- (CRD) 3. May be decreased 20% by simultaneous substitution of needle coke for graphite flour and decrease of composition to 65 w/o NbC. (See Table 18.) ✓
- (U) 4. No effect was observed as a result of any post pressing operation at 2700° C or at 2500° C. (See Table 16.)

B. AG CREEP DEFORMATION (U)

- (CRD) 1. May be decreased 5% by lowering the composition to 65 w/o NbC. ✓
(See Table 18.)
- (CRD) 2. May be decreased by 20% by substitution of needle coke for graphite flour. ✓
(See Table 18.)
- (CRD) 3. May be decreased significantly by substitution of 50 w/o of the NbC with TaC. (See Table 14.)
- (U) 4. Is not appreciably effected by a deformation at 2700° C of a billet made with needle coke. (See Table 16.) ✓
- (CRD) 5. May be decreased by a post pressing deformation at creep test temperature. ✓
(See Tables 4 and 13.)

C. WG FLEXURAL STRENGTH (U)

- (CRD) 1. Is decreased by 10% by lowering the composition to 65 w/o NbC. (See Table 18.)
- (CRD) 2. Is not appreciably affected by use of needle coke. (See Table 18.)
- (CRD) 3. Is lowered by 15% by simultaneous lowering of the composition to 65 w/o and use of needle coke. (See Table 18.)
- (U) 4. Is not appreciably affected by post pressing. (See Table 16.)

D. AG FLEXURAL STRENGTH (U)

- (CRD) 1. Is decreased by 20% by lowering the composition to 65 w/o NbC. (See Table 18.)
- (CRD) 2. Is decreased by 35% by use of needle coke. (See Table 18.)
- (CRD) 3. Is decreased by 50% by simultaneous lowering of the composition to 65 w/o and use of needle coke. (See Table 18.)
- (U) 4. Is not appreciably affected by post pressing. (See Table 16.)

E. BILLET THEORETICAL DENSITY (CRD)

- (U) 1. Is increased by 2% by substitution of needle coke. (See Table 15.)

F. COKE CHARACTERISTICS (U)

- (U) 1. Billets fabricated from 2 different coke sources had essentially identical properties. (See Table 16.)
- (CRD) 2. Use of fine, -325 mesh, carbon lowers the creep strength. For example, use of -325 mesh needle coke increased the creep rate by 50% compared with -100 +325 mesh needle coke. The use of fine carbon also increases the flexural strength. (See Table 17.)

G. PROCESSING VARIATIONS (U)

- (CRD) 1. Cracked billets, inferior room temperature strength properties, and lowered WG expansion coefficients are obtained using needle coke when full die compaction pressure is applied at or below 2600° C. (See Table 16.)
- (CRD) 2. Provided the die compaction pressure is applied at 2900° C or higher, 75/25 needle coke billet properties are not greatly affected by die heating rate, die size, or rate of cooling in the die. However, slow heating yields a material with a somewhat better AG flexural strength at the same WG expansion coefficient level. (See Figure 33.)

H. BILLET HOMOGENEITY (U)

- (U) 1. Radiography indicates good homogeneity in NbC-coke billets using dry blending techniques. However, when half of the weight of the NbC was replaced by TaC, good blending could not be achieved using dry blending techniques.
- (CRD) 2. The longitudinal centers of some of the 75/25 needle coke billets had a higher WG expansion coefficient, a lower creep rate, and a lower WG flexural strength than the billet ends. See Figures 26 and 28 and also Figures 4B and 5B in Appendix B.

RECOMMENDATIONS (U)

A. BASED DIRECTLY ON RESULTS OF THIS PROGRAM (U)

- (CRD) 1. Consideration should be given by Design to the use of 65/35 needle coke composite in areas where the advantage of a 20% decrease in WG coefficient of thermal expansion is not outweighed by a 50% decrease in AG room temperature flexural strength.

IV. RECOMMENDATIONS

- (U) 2. Post pressing cannot be recommended as a means of reducing the WG coefficient of thermal expansion.
- (CRD) 3. Post pressing at near the end use application temperature is recommended provided improved creep deformation resistance is required by Design.

B. FURTHER RELATED STUDIES (U)

- (U) 1. Study the effects of hot die compaction temperature, pressure, and time on the properties of needle coke composite.
- (U) 2. Further study the effects of purity and grain size of the NbC phase on the properties of flour composite.
- (CRD) 3. Study the effects of coke particle size variation on the properties of needle coke composite using the new processing; i.e., the die compaction pressure is applied at or above 2900° C.
- (U) 4. Conduct a study to determine the feasibility of using electrical conductivity as a non-destructive quality control test.
- (U) 5. Study composites fabricated with flake-like graphite.
- (CR)D 6. Study the effects of NbS addition to the properties of NbC-graphite composites.

C. PROPERTY MEASUREMENTS (U)

(CRD) Measure the following properties of both needle coke and flour billets of both 75/25 and 65/35 composition:

- (U) 1. Thermal shock resistance
- (U) 2. Corrosion resistance
- (U) 3. Tensile stress-strain curves - up to 2500° C
- (U) 4. Thermal conductivity - up to 2500° C

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D. FURTHER RELATED STUDIES (U)

- (U) 1. Study effects of molybdenum and other metallic additions on anisotropy.
- (CRD) 2. Study effect of thermal treatment on degree of solid solution and hot strength of TaC-NbC-graphite composite material.
- (U) 3. Conduct literature search aimed at the selection of candidate materials for a finely divided inert dispersoid to be added for the purpose of increasing the hot strength of the NbC phase.
- (U) 4. Initiate a theoretical study of the microstress fields in composite material.

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V. BILLET FABRICATION (U)

(U) Raw material and fabrication data pertinent to each billet fabricated under this program are listed in Tables 3, 4, and 5.

RAW MATERIALS

(CRD) Minus 325 mesh NbC and TaC of sufficient purity so as to not affect composite creep properties adversely were used. The analyses for purity and particle size of the carbide powders used in this program are listed in Table 6. Photomicrographs of one lot of NbC powder and the lot of TaC powder are shown in Figure 1.

(U) Four different lots of calcined petroleum coke were procured from 2 different sources. The metallic impurities and sulfur contents are shown in Table 7. The metallic impurity contents are considerably higher than normally encountered in graphite flour. Lot A1 is more impure than the other lots. It was considered possible that the use of this lot might increase the creep rate of the composite whereas the other coke lot purities were thought likely to be adequate.

(CRD) The particle size analyses of the as-received coke lots are compared with typical nuclear fuel grade graphite powder in Table 8. The coke size fractions used to fabricate the individual billets are as specified in Tables 3, 4, and 5. The particle size distributions within the size fraction used for each billet are what would be expected from Table 8 except that the -100 +325 mesh fractions consisted of a 50:50 mixture of -100 +150 and -150 + 325 mesh fractions.

(CRD) Three of the billets listed in Table 3 contained lot A1 coke that had been graphitized prior to blending. Graphitization was done by holding for 2 hours at 3000° C. X-ray diffraction lattice spacing analyses indicated that more than 80% of the coke was graphitized after this treatment.

(CRD) Examination of the composite material photomicrographs in this report reveals that the calcined petroleum coke particles are definitely more acicular than the flour particles with no major difference in shape between the 4 coke lots.

PROCESSING (U)

(U) The powders were dry mixed in a V-blender without an intensifier bar. The durations of blending for the individual runs are given in Tables 3, 4, and 5. It was found that the 30 minute blend that had been found to be satisfactory when mixing NbC and graphite flour was inadequate with petroleum coke. Progressive improvement was effected by increasing the blending time to 2 hours, later to 4 hours and finally to 16 hours. Rubber stoppers were added to the mixture when blending for 4 or more hours.

(CRD) A separate blend was made for each billet and the entire charge was added at once, in a single operation, to the graphite hot pressing dies. The dies and plungers were machined from ATJ graphite. The die was 7.5 inches long by 6 inches outside diameter when producing the small 2 1/4 inch diameter billets. A larger die, 9 inches long by 6 3/4 inches diameter was used when fabricating billets with a diameter greater than 3 inches. The sides and the ends of the cylindrical die were covered with a 1/2" thick double insulating layer of carbon felt sewn in place with graphite yarn, as shown in Figure 2.

(U) The die was heated by direct 3000 cps induction coupling from a 10 inch inside diameter by 11 inches long water cooled copper coil. The coil could be lowered and raised with motorized jacks for convenience in work loading and unloading, as shown in the photograph in Figure 3. A power level of approximately 70 KW was required to maintain the 9 inch diameter die at pressing temperature.

(CRD) The apparatus was enclosed in a double-jacketed water-cooled stainless steel container shown in Figure 4. After charging, the chamber was purged with argon for 30 minutes at a flow rate of 10 scfh. The runs were completed under argon at atmospheric pressure. All billets were held in die-confined axial compression at 3000 ± 100 psi at $3050 \pm$

50° C for 20 minutes. A center bulge of approximately 1/4 inches on the diameter was obtained. This prevented removal of the billet from the die without breaking the die.

(U) Axial compression was applied to the bottom ram by a hydraulic jack. The die remained free to float so that double action was effected.

(CRD) A hole was drilled in the die on an angle with the vertical axis so that the inner edge of the bottom of the hole was 1/8 inch from the inside surface of the die and at the longitudinal center of the die. The temperature was monitored by sighting onto the bottom of this hole with a calibrated Pyro Micro Optical Model 95 single color pyrometer. Temperature control was maintained by manual manipulation of the saturable reactor controlled power. The system was calibrated at the melting points of the ZrC-C system, 2890° C, and the NbC-C system, 3220° C. On these bases, an overall temperature correction of 250° C was used with the large die. The observed maximum hold temperature was 2800° C.

(CRD) Difficulties were experienced during the early part of the program with billet cracking. Only small 2 1/4 inch diameter billets were fabricated at that time. The cracking occurred when lot A1 coke was used but not with lot A2. The reason for this is unknown. All small cracked billets were rejected and were not evaluated. This cracking problem was overcome by using a slower heating and cooling cycle.

(U) The billet size and the cycle type, fast or slow, are indicated in Tables 3, 4, and 5. The fast cycle was the same as that used with billets made using graphite flour: The total heat-up time was 70 minutes. After the 20 minute hold, both the power and the pressure were terminated. With the slow cycle, the power was cut back at 1300° C so that the heating rate was 10° C per minute from 1300 to 2300° C and 8° C per minute from 2300 to 3060° C. The billet was then cooled at the controlled rate of 8° C per minute down to 2300° C whereupon the power was turned off. The pressure was released at 3050° C immediately after the 20 minute hold.

(CRD) Upon scale up to the larger 3 1/4 inch diameter billets, the cracking problem reappeared even when using the slow cycle. After considerable experimentation, it was discovered that the cracking disappeared when the temperature at which full pressure was applied was increased to 2900° C, such as with billet 222 in Table 3. Application of the pressure at 2500° C produced cracked billets, such as billet 225. Crack-free billets with good properties were produced using either slow or rapid cycling provided the pressure was applied at 2900° C. Rapid cycle billet 224 was crack-free. Billet 225 was evaluated even though it was cracked.

(U) Several of the billets were examined microstructurally for microcracking in the carbide matrix. Billets 180, 182, 193, 212, 225, and 231 all definitely contained cracked carbide matrices; however, no macrocracking had been observed. Although some carbide matrix cracking could be found somewhere in nearly all billets examined, even those made with graphite flour, these particular billets showed definite microcracking in all areas examined. Typical microcracks are shown at 1000X magnification in Figure 5. Examination of billets 187, 197, 222, 224 and 227 showed them to each have a relatively low incidence of microcracks. No microcracks were found in billet 192 which was made using fine, -325 mesh, coke.

(U) The post pressing operations were carried out in the hot press. The billet was held in unrestrained axial compression between two ATJ graphite cylindrical rams 4 inches in diameter. The billet was heated by radiation from an inductively heated 4 inch I.D. by 9 inch long graphite sleeve. Clearance between the sleeve and the rams was 0.010 inches or less. The temperature was monitored by sighting the single color optical pyrometer onto the side of the billet at an angle through a window on the top of the furnace shell. No temperature correction was made except that for the sight glass since black body conditions prevailed. The 75-25 compositions were post pressed for 1 hour at 2700° C and 2,000 psi whereas the 65-35 and 35-35-30 compositions were post pressed for 1 hour at 2500° C and 3,000 psi except where otherwise indicated in Tables 3, 4, and 5.

VI. BILLET CHARACTERIZATION (U)

(U) The homogeneity of each billet was evaluated by means of radiography. The result for each billet is described in Tables 3, 4, and 5. Positive print copies of typically light through heavy banding are shown in Figure 6. Composite material is subject to a banding type of heterogeneity which is readily visible in the billet radiographs and less so in the polished macrosections. The phenomenon is caused by a difference in carbide content along the length of the billet giving a pronounced layering effect. It usually does not affect properties unless it is heavy. Banding was improved throughout the program as the blending time increased and is thought to be caused by inadequate blending. It has been shown that a coke composite requires a longer blending time to eliminate banding than does a graphite flour composite. In general, banding was not considered to be a problem in the large NbC-coke billets. Likewise, carbide agglomeration was not a problem in this program. No coke agglomeration, excepting banding, was found. The few billets that were so afflicted are so indicated in the descriptive Tables 3, 4, and 5.

(CRD) It was noted that the higher carbon content compositions 65-35, were more difficult to blend although reasonably homogeneous material was realized. The addition of TaC to the NbC-carbon mixture greatly decreased the degree of homogeneity attainable using a specific blending procedure. Improved blending procedures must be used to obtain homogeneous NbC-TaC-carbon mixtures.

(CRD) Photomicrographs of typical materials fabricated in this program are shown in Figure 7 through 20. Comparisons of the particle shapes of coke and flour can be made by examination of Figures 8, 17, 19, and 20. It is evident that the coke is more acicular than the graphite flour. Microcracking in the carbide matrix was much more prevalent in billets made by the older processing, with coke, in which full pressing was applied at 2500° C or below during hot pressing, than with billets made either with flour or with coke by the new process.

Cracking was also observed in the coke particles, particularly in the longer particles, in a direction transverse to the long axis. These latter cracks are visible in billet 153 in Figure 8. More readily visible higher magnification microstructures showing carbide matrix cracking are shown in Figure 5.

(CRD) Carbide phase microcracking was greatly minimized when pressure was applied at 2900° C during hot pressing. No cracking is evident in the billet 222 microstructure shown in Figure 14. Similarly, cracking was minimized when pregraphitized coke was used. Microstructures of composite made with graphitized coke are compared with material made with raw coke in Figures 12 and 13. Billet 182, in which full pressure was applied at 2350° C, shows some microcracking where as billets 187 and 222, which were made using graphitized coke and raw coke with proper processing, respectively, show a good microstructure with little or no cracking.

(U) Calcined petroleum coke from the 2 different sources may be compared with respect to particle shape by examination of the composite microstructures in Figures 7, 11, 15, and 16. It may be concluded there is no significant difference. Carbide matrix cracks may be noted in billet 157 in Figure 11 and in billets 180 and 231 in Figure 16.

(U) The microstructures obtained when using 3 different coke mesh fractions are shown in Figure 9. The larger particles appear to be less acicular. The microstructure of billet 192, which was made using minus 325 mesh coke, is shown in Figures 9 and 14. No carbide matrix microcracks were found in this billet.

(U) The effect of post pressing on microstructure may be ascertained by examination of Figure 10 as well as some of the other figures. No effects were evident.

(U) Figures 19 and 20 typify the poor homogeneity obtained in billets containing mixed TaC-NbC carbon composites. Heterogeneity was rarely evident in the macrographs of NbC carbon billets.

VIII. PROPERTY EVALUATION

(U) Chemical and spectrographic analyses of coke composite billets has shown that the sulfur content has been reduced to below 10 ppm and the metallic impurities have been reduced to significantly below the level introduced by impurities in the raw materials, presumably due to vaporization.

(CRD) A short study on the relative preferred orientation of 75-25 needle coke and graphite flour composite material was completed. A back reflection technique employing a "Siemens" texture goniometer was used. The a-b planes of the crystallites were found to be highly oriented in the plane perpendicular to the billet hot pressing direction, whereas the flour particles tended to exhibit an essentially random texture. In both cases, the NbC phase was characterized by a random orientation.

(CRD) Initial X-ray diffraction studies on composite material have indicated that the carbon phase in needle coke billets is more highly graphitized than in flour billets.

PROPERTY EVALUATION (U)

COEFFICIENT OF THERMAL EXPANSION (U)

(U) Thermal expansion measurements were completed in the W.G. direction for each billet. A typical plot of the raw data points is shown in Figure 21. Secant coefficients of expansion between 25° C and 2000, 2300, and 2500° C were calculated from the raw data and the resulting data for each cycle on each specimen from each billet is listed in Table 9. Usually, 3 cycles were completed. Although some determinations were taken to 2500° C, most were taken only to 2300° C. Comparison of the data at these 2 temperatures indicates that the decrease in secant expansion coefficient from 2300 to 2500° C averages less than 2%.

(U) The strain measurements during expansion were measured using an optical technique. Temperature was monitored with a 2 color optical pyrometer that had been calibrated at the eutectic melting point of a rhenium-carbon mixture, 2486° C.

(U) Small specimens, 1/4 inch square by 1.4 inches long were machined from the small, 2 1/4 inch diameter billets. These specimens were run 2 at a time in a graphite resistance heated furnace in argon. Longer specimens, 1/4 inch square by 3 inches long, were machined from the larger billets. Those specimens were run 10 at a time in a uniform temperature zone heated by graphite resistance elements under argon. The amount of permanent deformation was deduced from optical measurement of the specimen length after the furnace had cooled.

(U) During the early stages of the program, 3 cycles were completed and the third cycle was assumed to be characteristic for that particular specimen. In order to increase the rate of data production during the later stages of the program, the specimens were given a 1 hour 2500° C heat treatment followed by a single cycle, really the second cycle, expansion measurement. The permanent deformation caused by the heat treatment was measured with a hand micrometer.

(U) Specimen location with respect to the ends of the billet is recorded in Table 9.

(U) The data after billet 197 appeared more consistent. It is interesting to observe that the lower CTE obtained on the first cycle can be accounted for by the permanent shrinkage encountered in the first cycle.

CREEP DEFORMATION (U)

(U) Compressive creep deformation measurements were made in the AG direction on specimens from each billet. The raw data is listed in Table 10. The specimens which were 0.5 inches in diameter by 1 inch long were held between graphite rams under an axial compressive load of 4,000 psi at 2500° C for 60 or 90 minutes. The load was maintained on the specimen during cooling. Percent deformation was determined by measurement of the specimen length with a hand micrometer both before and after the run. Creep deformation

was also monitored continually as a function of time by an automatic plot of cross-head movement on a strip chart recorder. A log-log plot of strain versus time generally produced a straight line similar to that obtained ³⁾ with flour billets. The slope, n , of this line is listed for each run in Table 10. The total hot deformation recorded during the run deviated from the cold measurement by, on the average, only 2% of the cold value, with the hot value being the greatest. This indicates good accuracy using the hot method. The coefficient of variation of the difference between the hot and cold measurement was 9%. The 1 hour creep deformation by cold measurement was interpolated from the hot and cold deformation data and is included in Table 10. The coefficient of variation for different locations within the billet averaged 13%.

(U) The density of each creep specimen was determined by weight and linear measurements. The creep specimen density was, on the average, 0.3% less than its parent billet density. The coefficient of variation, from zero, of the difference between the creep specimen density and the parent billet density was 0.8%.

(U) Specimen location within the billet was recorded, in most cases. Locations are indicated in Table 10. Specimens were taken at about mid radius from the smaller 2 1/4 inch diameter billets. The specimens from the larger billets came from near the periphery except those for the study of property variance within the billet described later in this report.

(CRD) A creep test specimen exchange program between LASL and WANL was completed as a check on the validity of creep data from both laboratories. The resulting data, which is presented in Appendix A of this report, showed poor agreement during the first series, with WANL obtaining approximately twice the creep rate as LASL on 40-40-20 composite supplied by LASL. However, excellent agreement, within 10% of the creep value, was obtained later using 75-25 material supplied by WANL.

FLEXURAL STRENGTH (U)

(U) The room temperature flexural strength was determined at least in triplicate in both grain directions for each billet. The raw data is given in Table II. For those large billets which were fabricated by the new processing method, the coefficients of variation within the billet averaged 12% in both grain directions. The specimens, which were 0.20 by 0.25 inches in cross section and at least 1.7 inches long were broken in 4 point loading with an outer span of 1.5 inches and an inner span of 0.5 inches. Longer specimens were broken in 2 or 3 locations depending on length. Specimen orientation of the WG specimens was such that the grain was in the 0.25 inch direction and the load was applied to the 0.25 inch side or parallel to the pressing direction. Previous studies with flour billets indicated that WG specimen orientation did not affect the flexural strength by more than 10%.

(U) Specimen location within the billet was recorded. Table II indicates those WG specimens which were taken from near the end of the billet and those AG specimens which were taken from near the cylindrical periphery of the billet. The remainder of the specimens were taken from the interior of the billet.

ELECTRICAL CONDUCTIVITY (U)

(CRD) Prior to breaking the flexural strength specimens, their electrical conductivities were determined at room temperature using a simple voltage drop technique. The raw data is shown in Table II. Niobium carbide has an electrical conductivity approximately one order of magnitude greater than that of graphite. It is also roughly an order of magnitude stronger at room temperature than graphite. Since the carbide matrix is continuous, this suggests that a relationship exists between room temperature flexural strength and electrical conductivity and this has been found to be the case for 75-25 flour billets⁴⁾. This in turn suggests the use of electrical conductivity as a convenient non-destructive quality control measurement. Cracks in the carbide matrix should lower both the strength and the conductivity. Changes with composition should show a similar correlation.

(U) The electrical conductivity measurements were taken over the same 0.5 inch span along the specimen which later constituted the inner span during the flexure test. It should be noted, however, that the electrical conductivity measurement characterizes an average of the full cross-section whereas the flexural strength is primarily controlled by that portion of the cross section near the surface under tension. Hence, the flexural strength is greatly influenced by material heterogeneity and by surface flaws. The over-all coefficient of variations within large billets fabricated by the new technique averaged 8% and 7% in the WG and AG directions, respectively, indicating that, as expected, the conductivity measurement is more consistent than the flexural strength measurement.

(U) Electrical conductivities are plotted against flexural strengths in Figures 22 and 23. The correlation in the WG direction is evident, while it is much less pronounced in the AG direction. No effect of post pressing is evident.

PROPERTY VARIATION WITHIN THE BILLET (U)

(U) It is certain that temperature and pressure gradients do exist within the billets during die compaction and, if the billets are post pressed, again during that operation. A program was initiated to determine the effects of these gradients on local properties within the billet. Specifically, the effect of longitudinal location within the billet on WG coefficient of expansion and the effects of diametral and longitudinal location on AG creep and on room temperature flexural strength and electrical conductivity in both grain directions were determined. The specimen location plan is shown in Figure 24.

(CRD) Two 75-25 coke composite billets without post pressing, billet numbers 249 and 252, two similar billets, numbers 250 and 251, with post pressing, and two 35-35-30 billets without post pressing, numbers 238 and 242, were selected for this study. The latter two billets appeared, by radiography, to be the most heterogeneous billets made in the over-all program. All billets were die compacted using the new processing except billet 251 which was die compacted by the old processing. The raw data is shown in Tables 9, 10, and 11. The data is also shown in Figures 25 through 30 with respect to specimen location within the billet.

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(U) Each flexural bar was broken in 4 point loading in 3 locations using outer and inner spans of 1.5 and 0.5 inches, respectively. The strength data so obtained is indicated in the figures at the location of breakage. Prior to breakage, an electrical conductivity profile was taken along the entire length of the specimen in 1/4 inch increments using a 1/2 inch span for each measurement. This profile data is tabulated in Appendix C. The electrical conductivity data given in Table II is the value at the point of failure in the flexural test. The relationship between flexural strength and electrical conductivity for billets 238, 242, 249, 250, 251, and 252 is illustrated in the plots in Figures 1C and 2C in Appendix C.

(U) The expansion coefficient specimens were of the standard 3 inch long type. The creep specimens, which were 0.5 inches in diameter by 1 inch long, were also standard.

(U) The densities of each flexural specimen were determined by immersion. The resulting data revealed the longitudinal and the diametral density profiles and is so plotted in Figures 31 and 32.

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VIII. ANALYSES OF DATA (U)

(CRD) Certain firm conclusions and other tentative conclusions with respect to the effects of raw materials, process variations, and composition on properties can be reached by a careful analysis of the data which is summarized in Tables 12, 13, and 14. The fact that many of the 75-25 coke billets were fabricated by the old processing technique, in which full compacting pressure was applied below 2600° C, is a complicating factor. All 75-25 coke billets with identification numbers greater than 212 were made by the new compacting method in which full pressure was applied at 2900° C, with the exception of billet 225. All 65-35 coke billets were made by the new method, as was the single 35-35-30 coke billet, with the exception of 65-35 billet 180 which was made by the old method.

EFFECTS OF RAW MATERIALS (U)

(U) Several lots of NbC powder were used as indicated in Tables 3, 4, and 5. As could be expected in view of the relatively high purity of all of these lots, no property variation could be detected between billets made from different lots of NbC and this variable NbC purity is now thought to be under good control.

(CRD) The properties of 75-25 billets made with needle coke are compared with those of 75-25 billets made with graphite in Table 15. The graphite billets were fabricated at WANL under another program²⁾ as well as by four outside vendors³⁾. The needle coke billets in Table 15 were made by the new processing procedure. The data shows the coke billets to be approximately 2% more dense than the graphite billets. No significant difference is apparent in either the creep rate or the WG flexural strength. However, the AG flexural strength is lowered by 35% by using needle coke. The WG thermal expansion coefficient is lowered slightly, but not by more than 5%. The electrical conductivity shows an increase in the WG direction and a decrease in the AG direction. However, the use of coke causes an increase in the ratio of conductivity to strength in both grain directions as is evident by examination of Figures 22 and 23.

(CRD) The properties of 65-35 billets made with needle coke and graphite, respectively, may be compared by examination of Table 14. Comparison of the properties of billets 231 and 240, which were made with coke, with those of billet 232 F, which was made with flour, suggests that the use of coke increases the creep rate somewhat, possibly decreases the WG flexural strength slightly, decreases the AG flexural strength significantly, by approximately 40%, decreases the WG thermal expansion coefficient significantly, by approximately 15%, increases the WG electrical conductivity and lowers the AG electrical conductivity. Again, the ratio of conductivity to strength appears to increase somewhat using coke, as is evident by examination of Figures 22 and 23. The densities of the flour billets were 1 to 2% lower than those of the coke billets.

(CRD) The use of graphitized coke does not appear to have any significant effect on the properties of needle coke composite. The averaged data is shown in Table 15.

(U) The effects of the use of coke from the 2 different sources is compared in Table 16. No major difference on composite properties is evident although source B coke appears to yield a somewhat weaker product. It would appear that the higher impurity level in lot A1 is not detrimental to composite properties.

(CRD) The effects of coke particle size variation on 75-25 billet properties are summarized in Table 17. The use of fines, -325 mesh particles, increases the creep rate by approximately 50%. The WG expansion coefficient is not affected appreciably except for a possible decrease resulting from use of the coarsest coke, the -60 +325 mesh fraction. The flexural strength appears to increase significantly when the finer coke is used. For example, use of -150 +325 mesh materially increases the strength in both grain directions compared with the coarser -100 +325 mesh fraction. The corresponding increase in electrical conductivity is not nearly as great. Therefore, the ratio of electrical conductivity to flexural strength shows a definite decrease as the coke size is decreased. Density tended to increase as the coke became finer. The observations on the effects of coke particle size were all made on composite fabricated by the old compaction technique. The experiments should be repeated using the new processing method.

EFFECTS OF PROCESSING (U)

(CRD) The major processing variations studied were a) the temperature for full application of pressure during die compaction, b) the heating and cooling rate, and c) the effects of post pressing.

(CRD) The effects of varying the temperature at which full pressure is applied during fabrication of 75-25 needle coke billets are summarized in Table 16. In the "old" process, full pressure was applied at 2500° C or less. In the "new" process full pressure was applied at 2900° C. The actual temperature at the time of pressure application for the individual billets is given in Tables 3 and 4. The following conclusions are based on the summary data in Table 16 for billets that were not post pressed. The new processing tends to decrease the creep rate somewhat. The WG expansion coefficient is increased significantly by 5 to 10%. The flexural strength is increased significantly, from below 10,000 psi in the WG direction and from below 5000 psi in the AG direction to approximately 14,000 and 7000 psi, respectively. The electrical conductivity is similarly increased by the new processing. Density is increased slightly, possibly by as much as 1%. In summary, it may be stated that properties were improved dramatically by the new processing.

(CRD) Only one coke billet having the 65-35 composition was die compacted by the old technique. This was billet 180. It was post pressed at 2700° C and 2000 psi. Its properties are summarized in Table 14. Flexural strength and electrical conductivity were clearly lower using the old processing. The creep rate was not affected significantly, but a lower expansion coefficient was obtained.

(CRD) Variations in the heating and cooling rates were found to have no major effect on the individual properties of 75/25 needle coke billets.

(CRD) An inverse relationship between AG flexural strength and WG expansion coefficient was discovered as shown in the empirical plot of Figure 33. The use of the slow cycle may tend to decrease the WG coefficient of expansion, particularly as it relates to the AG

room temperature flexural strength as shown in Figure 33. The 4 billets to the left of the $7.2 \times 10^{-6}/^{\circ}\text{C}$ expansion coefficient abscissa were fabricated by the old processing whereas those to the right of this line were fabricated by the new processing. Only one billet, No. 224, was fabricated by rapid heating and also by the new processing. Although the temperature of application of full pressure is the more important processing variable, heating rate is also significant in that rapid heating tends to lower the flexural strength somewhat, particularly in the AG direction, and to increase the WG expansion coefficient somewhat.

(U) The effects of post pressing are analyzed in a later section.

EFFECT OF BILLET SIZE AND MORPHOLOGY (U)

(CRD) Since the larger billets were more prone to cracking as a result of applying the die compaction pressure at too low a temperature, it could be expected that there might be a property-billet size relationship using the old billet fabrication technique. Such was found to be the case in that large billet 225, to which full pressure was applied at 2500°C during its die compaction, had an exceptionally low flexural strength in both grain directions. There is no indication of a billet size effect in billets made using the new die compaction technique. No billet size effect had been found when fabricating 75-25 graphite billets⁴⁾.

(U) The majority of billets fabricated in this program exhibited good homogeneity as revealed by radiographic and metallographic examination. Those few that did exhibit some heterogeneity, as shown in Tables 3, 4, and 5, yielded properties that were not inferior compared with homogeneous billets.

EFFECT OF POST PRESSING (C-RD)

(CRD) The effects of post pressing, for 1 hour at 2700°C and 2000 psi, on the properties of 75-25 needle coke billets may be ascertained by examination of the data in Table 16.

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No significant effect of post pressing is evident. This conclusion is supported by the observation that the averaged properties on post pressed 75-25 needle coke production billets, as shown in Table 20, are very similar to the properties obtained on unpost pressed billets in this experimental program using the new die compaction process.

(CRD) The effects of post pressing, for 2 hours at 2500° C and 3000 psi, on the properties of 65-35 needle coke billets may be ascertained by comparison in Table 14 of the data for the post pressed half of billet 231 with that of the other half billet 231 and of billet 240 which were not post pressed. It would appear that the only significant property change was an increase in the WG expansion coefficient. This surprising phenomenon must be checked with more billets in order to be conclusive. A reduction in creep rate as a result of the post pressing operation was expected. Examination of the billet fabrication data in Table 5 reveals a strain of only 1.2% in the post pressing of billet 231. This small strain offers an explanation why a larger effect on creep property was not obtained by the post pressing operation on this particular billet.

(U) Billet 193, which had been die compacted by the old technique, was post pressed at 2500° C and 3000 psi for 2 hours. Its creep rate was considerably increased, to 6.7%. This unexpected very high creep rate can only be attributed to the fact that the material was inferior, as die-compacted. Its expansion coefficient and AG flexural strength were both low. Billet 197 which had also been die compacted by the old technique, was post pressed for 1 hour at 2700° C and 3000 psi instead of 2000 psi. Although the post pressing strain was quite high, 16%, no significant property change was observed compared with unpost pressed billets.

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EFFECT OF COMPOSITION (U)

(CRD) The effects of composition on the properties of NbC-graphite composite material made with graphite flour and also with needle coke is summarized in Table 18. Data on one 50/50 flour billet, one 60/40 flour billet and one 10/90 flour billet, which were fabricated and tested at WANL under another program are included. The flour size is -150 +325 mesh in every case. All coke billets were fabricated by the new processing technique using -100 +325 mesh coke. None of the billets were post pressed.

(CRD) Lowering the composition of needle coke billets from 75/25 to 65/35 lowers the WG expansion coefficient by approximately 20%, lowers the WG flexural strength slightly, by perhaps 10%, lowers the AG flexural strength by approximately 25% and increases the void volume by 1 to 2%. The apparent increase in creep rate is somewhat surprising and more billets must be evaluated before a final conclusion can be reached.

(CRD) Lowering the composition of flour billets from 75-25 to 65-35 lowers the creep rate by approximately 20%, does not effect WG flexural strength, lowers the AG flexural strength by approximately 20%, lowers the WG expansion coefficient by 5%, and has no effect on density. These conclusions are tentative since they are based on data from a small number of 65-35 billets. Altering the composition from 75-25 to 65-35 definitely lowers the electrical conductivity a greater amount than the flexural strength as is evident from the tables as well as by examination of Figures 22 and 23. Lowering the composition to below 65 w/o NbC lowers the WG expansion coefficient, the flexural strength, and the density, but has no further effect on the creep rate.

(CRD) Examination of Table 14 reveals that replacement of half, by weight, of the NbC in 65-35 composite with TaC, to form the 35-35-30 composition, lowers the creep rate somewhat, on the average. The other properties were not significantly affected.

PROPERTY VARIATION WITHIN THE BILLET (U)

(C-RD) Examination of Figures 25 through 30 reveals that the WG expansion coefficient tends to be lower at the ends of the billet than in the center. Further, the creep tends to be lower at the center than at the ends. The WG flexural strength also appears generally to be higher at the center than at the ends. Examination of Figures 26 and 28 specifically, reveals that both billets 242 and 250 have a low CTE, a high creep rate, and a low flexural strength at their tops. Examination of the longitudinal density profile in Figure 31 gives no conclusive insight into the causes of these phenomena. It may be associated with a longitudinal temperature gradient during die compaction.

(U) The plots of flexural strength as a function of electrical conductivity reveals a definite relationship between those 2 types of measurement and suggests the possibility of developing an electrical resistance technique as a non-destructive testing technique for quality control of hardware items.

IX. EVALUATION OF PRODUCTION BILLETS (U)

(CRD) During the course of the experimental program described in this report, a series of post pressed production billets of 75/25 needle coke composite, were fabricated by an outside vendor. The first 48 billets were evaluated, under the cognizance of the WANL Quality Control Department, with respect to density, creep rate, flexural strength, and coefficient of thermal expansion. The fabrication procedure and the raw data generated are detailed in Appendix B. Electrical conductivity profile measurements were made by the Materials Department on the flexural strength bars from the first 29 billets and this data is also given in Appendix B.

(U) The property data is summarized in Table 20. The 48 billets evaluated were made in 5 different periods of time and, on this basis, were divided into 5 lots. Examination of Table 20 reveals a wide scatter in flexural strength and electrical conductivity in the first 3 lots and a gradual deterioration in these properties. A total of 7 billets were rejected in the first 3 lots due to low flexural strength. Only 3 billets from the first 3 lots had average creep rates above 6% so that creep strength was not a major problem.

(CRD) Lots 4 and 5 were definitely superior in that the creep rate was lower and the flexural strengths were higher than previous lots with less scatter in the data. Averaged data from lots 4 and 5 show a 1 hour 2500° C, 4000 psi creep of 3.3%, a WG flexural strength of 13,200 psi, an AG flexural strength of 6,700 psi, and a WG expansion coefficient of $7.2 \times 10^{-6}/^{\circ}\text{C}$. This is in good agreement with data, shown in Tables 15 and 16, on WANL 75/25 needle coke billets made by the new die compaction method, whether they were post pressed or not. This agreement then confirms the WANL finding that post pressing at 2700° C has no significant effect on properties.

(CRD) The process log sheets submitted by the vendor for each billet were examined in detail in an attempt to correlate the pressure-temperature-time cycle with billet properties. No correlation could be found except that when half or full pressure was applied at or above 2800° C, a good billet was always produced. If pressure was applied below 2800° C, the billet was good or poor.

(U) Billet density and specimen density data are summarized in Table 21. The average billet density was 96.2% of theoretical. The average densities of the creep specimens indicate a density gradient along the length of the billet with the high density at the top. One explanation of the low density at the bottom is the possibility of a lower temperature at the bottom of the billet during pressing. The density gradients could not be confirmed with the AG flexural specimens since top, middle, and bottom strengths were determined on a single specimen.

(CRD) A summary of creep, AG flexural strength and electrical conductivity quality control data as a function of specimen location within the billet is given in Table 22. The creep rate showed no consistent variation between the top and the bottom of the billet. However, the AG flexural strength measurements did reveal gradients along the length of the billet. Lot 3 billets were definitely stronger in the middle than at the ends. This is particularly apparent in billets 7919, 7920, and 7922 (see Appendix B) which were rejected. The raw data in Appendix A also shows the ends of these billets to be low in electrical conductivity. Lot 5 billets showed a definite gradient in AG flexural strength with strength decreasing from top to bottom. A statistical analysis was made of the flexural strengths, in both grain directions, of the 14 billets in lot 5. This was the first lot of sufficient size and data consistency to merit such an evaluation. The results showed a coefficient of variation, within the billet, of 5.0% and 9.4%, in the WG and AG directions, respectively. The over-all coefficient of variation, including the billet to billet variation, was 7.2% and 13.3%, in the WG and AG directions, respectively.

(U) The relationship between the flexural strength and the electrical conductivity on the first 29 billets is shown, by the plots in Figures 1B and 2B in Appendix B, to be fairly consistent, having a coefficient of variation in either grain direction of approximately 10%. The greater portion of the variation is probably in the flexural strength since it normally shows more scatter than the electrical conductivity in the same specimen. This data supports the suggestion for development of a non-destructive electrical conductivity, or eddy current, test as a quality control tool.

(CRD) The results of a study to determine property variation within the billet is also reported in Appendix B. The first production billet, No. 7870, was found to be somewhat heterogeneous with respect to creep rate and flexural strength whereas the fifth billet, No. 7874, was fairly homogeneous with respect to these properties. No consistent effect of longitudinal or diametral specimen location on strength properties could be found in either billet. On the other hand, both billets were found to have a significantly higher WG coefficient of thermal expansion near the middle than near the ends of the billet. A logical explanation for this is that the coke particles at the ends are better oriented since the powder at the ends moves a greater distance during the initial pressing operation than does the powder at the longitudinal middle of the billet.

DISCUSSION (U)

(CRD) The effects of composition and graphite type, needle coke or graphite flour, are summarized in the "Major Conclusions" section of this report. In general, the data reveals that substantial reductions in WG expansion coefficient can be achieved by lowering the NbC content and by substituting needle coke for graphite flour. However, a penalty does exist in that the low temperature AG flexural strength is lowered. Specifically, a 65/35 needle coke composite has a 20% lower WG expansion coefficient and approximately one-half the AG flexural strength compared with 75/25 flour composite.

(CRD) The lowering of the WG expansion coefficient as well as the gross lowering of the AG room temperature flexural strength by substitution of needle coke for graphite flour is supported by the most recent data generated by IITRI⁶⁾. When comparing the IITRI data, it is important to compare materials made using the same size of carbon source particle.

(CRD) Post pressing at 2700° C has been found to have no significant effect on the properties of 75 w/o NbC composite, whether made with needle coke or graphite flour. Post pressing 65/35 billets at 2500° C lowers the creep rate at 2500° C as expected. A similar lowering of the creep rate of 75/25 billets was also effected by a similar treatment. However, other properties, including the expansion coefficient, were not affected significantly by post pressing. The WANL sponsored study⁷⁾ at Carborundum had revealed that post pressing does lower the WG expansion coefficient significantly. No explanation for this discrepancy can be made, excepting poor process control.

(U) Two other properties, which were not studied in this program, are of extreme significance in reactor design. These are, of course, the corrosion resistance and the thermal shock resistance.

(CRD) The corrosion resistance had been found²⁾ to be adequate for 65/35 composite made with -60 +150 mesh, or coarser, carbon. The use of finer carbon would tend to increase the continuity of the carbon phase and might thereby decrease the corrosion resistance somewhat.

(CRD) Data on the thermal conductivity, yield strength, thermal expansion and elastic modulus is inadequate for the accurate calculation of the thermal shock parameter $k_s/\alpha E$ for needle coke and flour composites. However, the available data does allow rough estimates for the individual properties to be made. Relative property values are so estimated in Table 19. The resulting thermal shock parameters are thought to give, at least, a fair qualitative comparison of flour and needle coke composite at 2 composition levels, 75/25

and 65/35. Decreasing the carbide content, or using needle coke instead of graphite flour, or both, increases the thermal shock resistance in the WG direction. However, neither variable has a strong effect on the thermal shock resistance in the AG direction. Hence, a thermal shock anisotropy is introduced. The over-all effect is a multi-axial, multi-stress mode thermal stress design problem which is beyond the scope of this report. However, the principle temperature gradients in the final hot end support application are WG and it is intuitively felt that the use of coke or a lower NbC content would allow a net improvement in thermal shock resistance.

(CRD) The accurate analytical treatment of the thermal shock problem must await the procurement of more accurate data on the four properties indicated above at all temperatures from 25 to 2500° C. In addition, the various composites should be compared experimentally in thermal shock using a specimen configuration and test procedure that at least simulates the stress failure mode expected in the hardware application.

(CRD) The mechanism of expansion coefficient lowering in the WG direction cannot be considered in a quantitative way due to lack of data concerning the interphase bond strength as well as the shear strength of the graphite particles. It can be stated qualitatively that the lowering is caused by a mechanical alignment of the elongated graphite particles and is further enhanced by any orientation of the hexagonal network within the graphite particle. The coke particles are definitely more acicular or needle like than are the graphite flour particles. Quite good alignment is effected in the WG plane during the hot die compaction. Assuming that a bond exists between the phases, the thermal expansion mismatch between the phases effects an anisotropy in the composite with respect to strength and expansion coefficient. This mechanism does not require any property anisotropy within the coke particles, but can be strictly a function of coke particle shape. The shear micro-stresses are maximized in the WG direction. The continuous carbide matrix of the cooled

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billet is in tension in the WG direction and in lower tension or possibly in compression in the AG direction. Conversely, the graphite particles, which are mainly isolated from each other, are in compression in both grain directions or possibly in tension in the AG direction.

(CRD) In actual fact, the anisotropic characteristics of the composites are no doubt further influenced, perhaps quite strongly, by crystallographic alignment within the graphite particles. The raw needle coke owes its acicularity to the fact that the c axes of the hexagonal crystallites, or crystal seed areas, are distributed in a partially random manner in a plane perpendicular to the long axis of the coke particle. This results in a preferred orientation of the a-b axes, which is the low thermal expansion plane, in a direction perpendicular to the length of the needle, which is in turn aligned in the WG direction in the billet. Similarly, the a-b plane of flat, plate-like particles become oriented in the WG billet direction.

(CRD) If interphase bonding is good and the compressive strength of the graphite particles is sufficiently high, tensile rupture of the carbide matrix can occur. This has been observed as micro-cracking in the AG direction as well as shear micro-cracking around the larger carbon particles. Micro-cracking in the AG direction has also been observed in the graphite particles in the composite. When using needle coke, carbide matrix micro-cracking usually occurs when the pressure during die compaction is applied at too low a temperature. This is thought to be associated with the release of sulfur from the coke and it may be hypothesized that the early application of pressure retains the sulfur through a critical period wherein either the strength of the coke particle or the interphase bond is improved. Either mechanism could conceivably cause a rupture of the carbide matrix. Matrix cracking was entirely absent in all cases when fine, minus 325 mesh, needle coke was used. Theory supports this observation in that the matrix residual tensile localized

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microstress is a direct function of graphite particle size. Unfortunately, the use of fine carbon particles yields a material with a high creep deformation rate and a higher elastic modulus⁵⁾. Proper control of bonding, particle strength, and/or particle size could conceivably yield optimized properties. Niobium sulfide powder has been procured as an aid in such a program. This material exists as a molten compound around 2500° C.

(CRD) X-ray diffraction studies have shown that the degree of crystallization as well as the crystal orientation is greater in the graphite contained in composite material made with needle coke than with graphite flour. IITRI⁶⁾ makes a similar observation by stating that the hot pressed composite contains a "well ordered graphite phase ---- regardless of the carbon source". It must then be assumed that any subsequent heat treatment, whether or not accompanied by a compressive force, would not yield any further significant degree of recrystallization. Further mechanical alignment of the graphite particles would also not be great. It is, then, understandable that the post pressing operation did not significantly affect the coefficient of thermal expansion.

(CRD) In order to satisfy classical formulae used to calculate the expansion coefficients of composites when applied to NbC-needle coke composites, it is necessary to assume a WG Young's modulus for the coke that is considerably higher than the WG Young's modulus of hot pressed ZTA graphite. Also, IITRI⁶⁾ have determined, from thermal conductivity measurements made on needle coke composites of various compositions, that the WG thermal conductivity of the pure needle coke phase, extrapolated to zero NbC content, is greater than the WG thermal conductivity of ZTA graphite. Both of these observations indicate that the graphite crystallite orientation in NbC-needle coke hot pressed composites is greater than that present in ZTA graphite. This suggests a very high degree of pre-ordering in the raw coke, or an ordering during hot die compaction that is influenced by the presence of NbC. It is suggested that ordering in the graphite phase might be still further enhanced by the addition of a small quantity of molybdenum metal to the powdered mixture. It is

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known that graphite recrystallization occurs in the vicinity of graphite joints brazed with molybdenum. Prior to the end of hot die compaction, the molybdenum would be taken up in solid solution in the NbC phase.

(CRD) The use of flat graphite particles such as natural flake graphite or ground pyrolytic graphite should yield WG properties in a composite similar to needle coke, since the a-b planes of the graphite crystallites are preferentially aligned in the transverse billet plane or WG plane. However, the longitudinal billet direction or AG direction would contain only c-axes in the case of flake whereas it contains a mixture of a- and c- axes in the case of needle coke. Hence, the anisotropy would be much greater with flake with a very low tensile strength and a very high expansion coefficient in the AG direction. Some of the calcined petroleum coke needles are flat whereas others are acicular indicating a c-axis orientation in a billet part way between fully in the AG direction and random between the AG and WG direction. One billet was fabricated at WANL using a coarse, minus 30 mesh, natural flake graphite. The properties, which are not described in this report, did indicate a mica-like fracture with very low AG flexural strength. Further, the creep rate of this 75/25 material was excessively high, perhaps due to a shear slip in the WG direction. The extreme weakness of flake composites casts doubt in their use feasibility. IITRI⁶⁾ has been able to lower the WG expansion coefficient to below that obtained with needle coke using fine Ceylon graphite, which is a highly oriented natural graphite with a morphology similar to pyrographite. The properties of a 75 w/o NbC composite made with this material are given in Table 2. The high creep deformation rate is probably mainly due to the relatively high iron content, 0.7%, of the graphite. It was planned to repeat this study at WANL using a purified flake type graphite particle with variations in particle size and fabrication processing variables.

(U) The room temperature flexural strength of a carbide-graphite composite with a continuous carbide matrix is, logically, controlled by the strength of the carbide matrix which is definitely the stronger of the two phases. Since the carbide phase is isotropic, the strength anisotropy must then be controlled by the graphite particle shape and morphology. The strength level in each direction is controlled by the residual stresses and microcracking in the carbide phase and hence is related to the coefficient of thermal expansion and the electrical conductivity. Any increase in residual stress would lower both the WG flexural strength and the WG expansion coefficient. Microcracking would lower the strength in both directions, lower the WG thermal expansion coefficient and lower the electrical conductivity in both directions.

(CRD) The use of fine needle coke increases the room temperature strength as evidenced by the IITRI study⁶⁾ as well as the findings in this program. This, combined with the absence of carbide microcracking when fine carbon is used, supports the residual carbide microstress hypothesis. Regardless of grain direction, the ratio of flexural strength to electrical conductivity is, according to the findings of this program, decreased by increasing the graphite particle size or by using needle coke instead of flour. This can be at least partially explained on the assumption that residual stresses in the carbide matrix affect strength but not electrical conductivity.

(U) It is recommended that a detailed theoretical analysis of the microstress problem be made using certain property assumptions for the individual phases. This should lead to a better understanding of the mechanisms controlling composite properties and suggest possible avenues of approach for property optimization.

(U) The exact compressive creep deformation mechanism for carbide-graphite composites is unknown. However, since the carbide phase is known to be continuous, it is most likely mainly controlled by that phase. It is difficult to control the various processing parameters and raw materials variations so that their individual effects on creep rate can be determined.

(CRD) Compressive creep deformation data obtained in this program and hot flexural strength and flexural creep data obtained at IITRI⁶⁾ reveals that NbC-graphite composite material made with fine carbon particles has inferior creep strength. This suggests that the creep mechanism is interphase slip. However, an alternate explanation of this particle size effect can be reached by treating the graphite particles as voids with the finer voids exerting a greater effect on the creep rate.

(CRD) It is now quite well confirmed that the addition of graphite to an NbC matrix reduces the creep strength up to the 50 v/o graphite level. Again, this phenomenon can be satisfied by either an interphase slip or a void creep mechanism or both.

(CRD) The generally accepted⁸⁾ hot creep mechanism in metals is dislocation climb which in turn depends on the rate at which vacancies can diffuse to edge dislocations. This requires mass transport by diffusion and is a thermally activated process. The cubic NbC crystal may be considered as a cubic niobium metal lattice with a cubic carbon lattice superimposed interstitially upon it. It appears possible that the same thermally activated mechanism applies to the hot creep of pure carbide as well as to its composites with graphite. This is supported by the observation that the creep activation energy of NbC-graphite composite is comparable with the activation energy for self-diffusion in NbC⁹⁾. Further, impurities such as iron lower the creep strength of carbide-graphite composite and are also known to lower the sintering temperature of the pure carbide by several hundred degrees. In addition, the use of TaC, instead of NbC, which melts at a higher temperature and self-diffuses more slowly than NbC, is known to increase the creep strength. More recently, it has been found that the replacement of a portion of the NbC with TaC to form a solid solution lowers the creep rate of the carbide-graphite composite somewhat.

(CRD) The use of extra high purity NbC which had been fabricated at WANL from carbon felt and niobium pentachloride produced a carbide-graphite composite with approximately

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one-half of the creep rate obtainable with NbC of commercial purity⁶⁾. However, the carbide particle size of the high purity NbC was several times greater than that of the commercial material. It also had a more elongated shape. The advantage is thought, however, to have been realized through improved purity. However, before a definite conclusion can be reached regarding whether the controlling parameter was carbide grain size or purity, further work is required.

(U) Studies⁸⁾ with pure metals have cast doubt as to whether grain size has any real effect on creep rate. Instead it may be controlled by other associated properties such as grain boundary angle. Any future study of the effects of particle size and purity on creep rate should include a careful study of the composite microstructures.

(CRD) In addition to the known methods of improving creep strength, which are: a) billet deformation at the creep temperature⁴⁾, b) use of high purity NbC²⁾, or increased NbC grain size²⁾, and c) substitution of all or part of the NbC with TaC, there is the possibility of incorporating a finely dispersed, stable particulate phase into the carbide phase, to form a structure similar to that of TD nickel. This has been done with TiC using B₄C as the dispersed phase¹⁰⁾. The stability of any dispersed phase at the hot pressing temperature, 3100° C, is doubtful. However, its precipitation from solid solution at 2500 to 3000° C by heat treating a billet is conceivable.

(CRD) X-ray diffraction analysis as well as microstructural examination of the heterogeneous NbC-TaC-graphite billets produced in this laboratory showed that carbide solid solution was not complete. It is expected that attainment of solid solution by lengthy heat treatment would further improve the creep strength of the composite over that of NbC-graphite material.

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(CRD) The feasibility of utilizing 65/35 needle coke composite in a component such as a fuel element tip where a low WG expansion coefficient offers the advantage of reducing the residual stresses at the joint due to contraction mismatch can now be examined with more conviction. Of course, any exact analysis must necessarily be largely controlled by design. However, certain generalities can be made. The other important requirements are adequate AG compressive creep strength, adequate thermal shock resistance, and adequate corrosion resistance. The creep strength appears to be comparable with that of 75/25 flour composite. It appears that thermal shock is improved over 75/25 flour composite, at least in the WG direction described above. Limited corrosion testing at WANL of coated specimens of 65/35 coke composite made with -60 + 150 mesh coke had indicated this material to be roughly equivalent in corrosion resistance to 75/25 flour composite.

(CRD) Although incomplete, the data on 65/35 needle coke composite suggests that it offers a safe advantage over 75/25 flour composite. However, before its use in a reactor can be recommended, it is suggested that corrosion testing of coated components be tested under simulated thermal cyclic conditions. It is conceivable that the coating-substrate bond could be weakened under these conditions due to a combination of graphite leach out near the coating interface and the fluctuating thermal shear stress.

(U) It is apparent that certain properties of composite material can be controlled but not always independently of all other properties. Better property control is expected to emerge as a better basic understanding of component materials and fabrication processing is achieved. It must be emphasized that the choice of optimum properties and of processing will be dictated more and more precisely by design requirements as the materials technology matures.

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Table 1

Typical Data for 75w/o NbC Composite Material
Made with Graphite Flour (CRD)

	<u>WANL Material</u> (avg. of 10 billets)	<u>Material From 4 Vendors</u> (avg. of 8 billets)
Density, % theor. ¹⁾	95.5	95.6
AG Creep, % ²⁾	3.4	3.6
WG Flexural Strength, psi	14, 600	15, 500
AG Flexural Strength, psi	10, 700	10, 500
WG Expansion Coefficient /°C (25 to 2500°C)	7.6×10^{-6}	7.6×10^{-6}
WG Electrical Conductivity, ohm ⁻¹ cm ⁻¹	11, 000	11, 300
AG Electrical Conductivity, ohm ⁻¹ cm ⁻¹	8, 700	8, 800

1) Theoretical density 4.83 g/c. c.

2) Compressive creep in 1 hour at 2500°C and 4000 psi.

Table 2

IITRI Data on 75w/o NbC Composite
(CRD)

<u>Carbon Type</u>	<u>Carbon Mesh Size</u>	<u>Grain Direction</u>	<u>Flexural Strength, psi</u>		<u>Elastic Modulus, MSI</u>	<u>CTE/^oC</u>
			<u>RT</u>	<u>2500^o C</u>		
M2 Graphite Flour	-200	WG	16,800	14,900	8.7	8.3
	+325	AG	9,200	8,800	7.0	10.7
Calcined Petroleum Coke	-325	WG	15,400	11,500	12.0	7.1
		AG	7,400	6,500	5.1	11.1
Ceylon Graphite	-325	WG	16,600	8,900	9.7	6.6 ¹⁾
		AG	5,100	3,500	4.6	---

1) This value from Reference 6. All other data in table from Reference 5.

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Table 3
Billet Fabrication and Characterization Data (75 w/o NbC bal. C) (CRD)

Billet No	Coke Lot	Coke Mesh Size	Blend Time Hrs.	Pressing Cycle	Full Pressure on at X°C	NbC Lot No.	Billet Size, In.		Billet Density % Theor.	Billet Homogeneity	
							D	L		Banding	Carbide Agglomerates
Flour	Flour	150/325	0.5	Fast	2300		2 1/4	2 1/4	95.5	Absent	Absent
132	Al	150/325	0.5	Fast	2300	5	2 1/4	2 3/4	97.8	Light	Present
156	Bl	150/325	2	Fast	2400	10	2 1/4	2 3/4	97.3	Medium	Absent
182	Al	100/325	4	Slow	2350	5	2 1/4	2 3/4	97.3	Medium	Vague
222	Al	100/325	18	Slow	2900	11	3 1/4	2 1/2	96.7	Absent	Absent
223	Al	100/325	17	Slow	2900	11	3 3/4	4	97.0	Absent	Absent
224	Al	100/325	14	Fast	2900	11	3 1/4	2 3/4	98.7	Medium	Absent
225	Al	100/325	14	Fast	2500	11	3 1/4	2	97.2	Light	Absent
227	Al ¹⁾	100/325	18	Slow ²⁾	2900	11	3 1/4	2	97.8	Very Light	Absent
229	Al	100/325	16	Slow	2900	11	4	4	98.3	Light	Absent
234	Al	100/325	16	Slow	2900	11	3 3/4	3 3/4	97.9	Light	Absent
239	Al ¹⁾	100/325	16	Slow	2900	11	3 1/4	2 1/2	97.4	Light	Absent
249	B2	100/325	16	Slow	2900	13	3 3/4	3 3/4	98.0	Absent	Absent
252	B2	100/325	16	Slow ²⁾	2900	13	3 3/4	3 3/4	97.6	Absent	Absent
Average									97.4		

1) Coke graphitized prior to blending with NbC

2) One-hour hold at 2900°C without pressure during heat-up.

Table 4

Post Pressed Billet Fabrication and Characterization Data (75 w/o NbC Bal. C)
(CRD)

Billet No.	Coke Lot	Coke Mesh Size	NbC Lot No.	Billet Size In.		Blend Time Hrs.	Die Hot Pressing Cycle Pon at X°C		% Post Press Strain 2700°C 2 ksi 1 hr.	Billet Density % Theor.		Billet Homogeneity Carbide Banding / Agglomerates	
				D	L					Before PP	After PP		
145	BI	60/325	10	2 1/4	2 3/4	2	Fast	1950	4.7	96.6	95.1	Medium	Absent
146	AI	60/325	10	2 1/4	2 3/4	2	Fast	2150	5.6	94.6	94.6	Heavy	Vague
149	Flour	150/325	10	2 1/4	2 3/4	2	Fast	2300	4.6	94.2	94.8	Absent	Absent
153	BI	150/325	10	2 1/4	2 3/4	2	Fast	2400	9.5	97.3	95.2	Absent	Absent
157	BI	100/325	10	2 1/4	2 3/4	2	Fast	2150	10.7	97.0	94.4	Light	Absent
174	AI	100/325	5	2 1/4	2 3/4	4	Slow	2200	5.8	96.8	95.5	Medium	Vague
192	BI	-325	11	2 1/4	2 3/4	4	Slow	2400	8.0	98.5	97.7	Absent	Absent
193	BI	150/325	11	2 1/4	2 3/4	4	Slow	2300	4.7 ^{a)}	97.7	98.1	Absent	Absent
197	BI	150/325	11	2 1/4	2 1/2	15	Fast	2500	16.1 ^{b)}	100.6	98.7	Absent	Absent
212	A2	150/325	11	2 1/4	2 1/2	16	Slow	2250	5.1	96.9	97.2		
222	AI	100/325	11	1 1/2	2 1/4 ^{c)}	18	Slow	2900	2.0	96.7	96.3	Absent	Absent
250	B2	100/325	13	3 3/4	3 1/2	16	Slow	2900	3.4	98.0	97.9	Absent	Absent
251	B2	100/325	13	3 3/4	3 3/4	16	Slow	2600	3.4	97.9	96.0	Absent	Absent
Average										97.1	96.4		

a) Post pressed 2 hours at 2500°C & 3 KSI

b) Post pressed 1 hour at 2700°C & 3 KSI

c) Small billet machined from original large billet 222

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Table 5

Billet Fabrication and Characterization Data (65 w/o NbC bal. C and 35 w/o NbC 35 w/o TaC bal. C)
(CRD)

Billet No.	Carbon Type	Carbon Mesh Size	NbC Lot No.	Billet Size		In. L	Blend Time Hrs.	Post Press	Billet Density % Theor.		% Post Press Strain			Billet Homogeneity Carbide	
				D	L				Before PP	After PP	2500°C	3 ksi	2 hrs.	Banding	Agglomerates
<u>65 NbC 35C</u>															
180	Bl Coke	100/325	5	2 1/4	x 2 3/4	4	Yes ^{b)}	97.8	96.9			2.3 ^{b)}	Medium	Vague	
231	Al Coke	100/325	11	3 1/4	x 2 1/2	16	No	96.4	----			----	Light	Absent	
231 ^{a)}	Al Coke	100/325	11	1 1/2	x 2 1/4	16	Yes	96.4	97.4			1.2	Light	Absent	
232	Flour	150/325	11	3 1/4	x 2 1/2	16	No	95.5	----			----	Absent	Absent	
233	Flour	150/325	11	3 1/4	x 2 1/2	16	Yes	95.2	95.2			1.4	Medium	Absent	
240	Al Coke	100/325	13	3 3/4	x 3 3/4	16	No	<u>96.5</u>	----			----	Light	Absent	
							Avg. Coke	96.8	97.2						
							Avg. Flour	95.4	95.2						
<u>35 NbC 35 TaC 30 C</u>															
238	Flour	150/325	11	3 3/4	x 4	16	No	94.5	----			----	Heavy	Present	
242	Al Coke	100/325	13	3 3/4	x 3 1/2	16	No	96.5	----			----	Heavy	Present	
256	B2 Coke	100/325	13	3 3/4	x 4	16	No	96.4	----			----	Heavy	Present	

a) Billet halved longitudinally and a smaller cylindrical billet machined from one half was subjected to post pressing.

b) Post pressed 1 hour at 2700°C and 2000 psi. Primary hot pressing made with fast cycle and P on at 2400°C.

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Table 6

Analysis of Carbide Powders
(CRD)

	<u>NbC Lot 5</u>	<u>NbC Lot 10</u>	<u>NbC Lot 11</u>	<u>NbC Lot 13</u>	<u>TaC Lot SP-26721</u>
Comb. C	10.92w/o	10.91w/o	10.82w/o	11.02w/o	6.00w/o
Free C	0.12	0.22	0.12	0.21	0.02
Al	0.001	0.001	0.002	0.002	0.017
Fe	0.019	0.023	0.005	0.019	0.006
Ni	<0.001	<0.001	0.002	0.001	<0.001
Co	0.001	0.001	<0.001	0.001	<0.001
Cr.	0.002	0.009	0.006	0.008	0.005
Nb	-----	-----	-----	-----	0.012
Ti	0.004	0.027	0.010	0.010	0.001
Mo	0.047	0.008	0.007	0.010	<0.001
Si	0.001	<0.001	0.002	0.001	0.008
W	0.06	0.10	0.05	0.02	<0.03
Ta	0.06	0.16	0.11	0.14	-----
Fisher Sub sieve Size, Microns	5.2	4.9	5.2	4.4	6.7
Bulk Density, g/c. c.	2.9	2.9	2.9	2.7	4.6
Tap Density, g/c. c.	4.7	4.5	4.6	4.5	7.3

Table 7

Chemical Analysis of Calcined Petroleum Coke (U)

	<u>Lot A1</u>	<u>Lot A2</u>	<u>Lot B1</u>	<u>Lot B2</u>
Ash	0.31 w/o	0.12 w/o	0.05 w/o	0.09 w/o
Sulfur	0.85	1.02	1.17	1.37
Al	0.017	0.004	0.002	0.005
B	0.001	<0.001	<0.001	0.0006
Ca	0.016	0.002	0.002	0.006
Co	<0.001	<0.001	<0.001	<0.001
Cr	0.004	<0.001	<0.001	<0.001
Fe	0.018	0.007	<0.001	0.006
Mg	0.003	<0.003	<0.003	0.001
Na	0.005	0.004	0.001	0.007
Ni	0.020	0.007	<0.001	<0.001
Li	0.019	0.006	0.002	0.008
Ti	<0.003	<0.001	<0.001	<0.001
V	0.019	<0.001	<0.001	<0.001
W	0.013		<0.001	<0.003

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Table 8

Screen Analysis of As Received Carbon Powder (U)

<u>Mesh Size</u>	<u>CPC Lot A1</u>	<u>CPC Lot B1</u>	<u>CPC Lot B2</u>	<u>Graphite Flour</u>
+60	13 w/o	0	2	0
-60 + 100	22	23	23	9
-100 + 150	15	24	26	18
-150 + 200	13	23	24	16
-200 + 270	6	14	13	8
-270 + 325	7	12	6	10
-325	24	4	6	39

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Table 9

Secant Thermal Expansion Coefficient Data -
With Grain $\bar{\alpha}$ = Microinches/inch/ $^{\circ}$ C
(U)

<u>Billet No.</u>	<u>Specimen No.</u>	<u>Cycle</u>	<u>2000°C</u>	<u>2300°C</u>	<u>2500°C</u>	<u>Max Temp. °C</u>	<u>Permanent Deformation%</u>	<u>Specimen Size</u>	
Baseline					7.6				
75-25 Flour Billets									
132	IE ¹⁾	1	6.4	6.5		2330	.08	Small	
		2	6.0	6.1		2350	-.11		
		3	6.6	6.6		2370	-.02		
145	IE	1	6.2	6.5		2310	-.02	Small	
		2	6.5	6.7	6.7	2530	.07		
		3	6.2	6.2	6.3	2530	-.04		
	3C ²⁾	1	7.1	7.3		2300	.07		
		2	7.1	7.2	7.0	2530	-.01		
		3	6.9	6.8	6.9	2520	.00		
	146	IE	1	7.0	7.3		2310	-.09	Small
			2	7.2	7.4	7.3	2520	-.03	
			3	6.9	6.8	6.8	2490	.03	
2C		1	7.2	7.1		2240	-.06		
		2	7.0	7.0	6.9	2580	-.02		
		3	6.9	6.8	6.8	2620	.00		
3C		1	6.8	7.0		2300	-.08		
		2	7.1	7.2	7.1	2520	-.07		
		3	6.9	6.8	6.8	2480	.01		
4E		1	6.5	6.5	6.5	2500	-.11		
		2	6.8	6.8	6.7	2580	.02		
		3	6.4	6.3	6.4	2620	-.03		

1) E - Specimen taken near end of billet. 2) C - Specimen taken near center of billet.

Table 9 (con't)

<u>Billet No.</u>	<u>Specimen No.</u>	<u>Cycle</u>	<u>2000°C</u>	<u>2300°C</u>	<u>2500°C</u>	<u>Max Temp. °C</u>	<u>Perm. Deform. %</u>	<u>Specimen Size</u>
149 (flour)	IE	1	7.4	7.6		2300	.00	Small
		2	8.2	8.4	8.4	2450	.11	
		3	7.8	7.8	7.8	2500	.05	
	3C	1	7.5	7.5		2300	-.02	
		2	8.2	8.3	8.4	2450	.12	
		3	7.4	7.4	7.7	2500	.04	
153	IE	1	7.2	7.1	6.9	2450	-.06	Small
		2	7.3	7.2	7.0	2480	-.05	
		3	7.2	6.9	6.8	2470	-.03	
	2C	1	7.3	7.1	6.9	2450	-.06	
		2	7.7	7.5	7.4	2480	-.05	
		3	7.5	7.4	7.3	2470	-.03	
156	IE	1	7.4	7.4	7.4	2450	.03	Small
		2	7.1	6.9	6.9	2480	.00	
		3	6.9	6.9	6.9	2460	-.03	
	2C	1	6.8	6.8	6.7	2460	-.12	
		2	7.2	7.3		2250	.01	
		3	7.3	7.2	7.1	2500	-.01	
157	IE	1	7.3	7.2	7.0	2450	-.14	Small
		2	7.6	7.5	7.3	2480	.03	
		3	7.0	7.0	7.1	2450	-.04	
174 ³⁾	IE	1	---	---	---	2380	.14	Small
		2	6.9	7.0	6.7	2490	-.16	
180	IE	1	5.9	5.7	5.7	2450	-.05	Small
		2	5.9	5.8	5.8	2410	-.01	
		3	5.8	5.7	5.7	2480	-.03	

3) First cycle was a heat treatment without strain measurement

Table 9 (con't)

<u>Billet No.</u>	<u>Specimen No.</u>	<u>Cycle</u>	<u>2000°C</u>	<u>2300°C</u>	<u>2500°C</u>	<u>Max Temp. °C</u>	<u>Perm. Deform. %</u>	<u>Specimen Size</u>
	2C	1	5.8	5.8	5.6	2460	-.04	
		2	5.9	5.8	5.7	2410	.01	
		3	5.8	5.8	5.7	2480	-.01	
182	IE	1		test system malfunction				Small
		2	6.9	6.9	6.6	2480	-.03	
		3	7.1	7.0	6.8	2500	-.05	
	2C	1	7.1	7.0		2300	.02	
		2	6.9	6.9	6.6	2480	-.07	
		3	7.2	7.1	6.8	2500	-.03	
192	IE	1	---	---	---	2380	.09	Small
		2	7.3	7.0	6.7	2490	-.02	
193	IE	1	---	---	---	2380	-.05	Small
		2	6.5	6.3	6.1	2490	-.12	
197	IE	1	---	---	---	2380	-.08	Small
		2	7.0	6.9	6.7	2480	-.05	
212	2C	1	7.4	7.3	7.0	2460	-.07	Small
		2	7.4	7.3	7.1	2430	-.02	
		3	7.4	7.4	7.1	2480	-.01	
222	IC	1	7.5	7.5	7.3	2500	.01	Small
		2	7.5	7.5	7.2	2450	.02	
		3	7.4	7.4	7.3	2500	.01	

Table 9 (con't)

<u>Billet No.</u>	<u>Specimen No.</u>	<u>Cycle</u>	<u>2000°C</u>	<u>2300°C</u>	<u>2500°C</u>	<u>Max. Temp. °C</u>	<u>Perm. Deform. %</u>	<u>Specimen Size</u>
222D	IE	1	7.6	7.5	7.4	2480	.01	Small
		2	7.5	7.4	7.3	2450	.01	
		3	7.4	7.4	7.3	2520	-.01	
223	IC	1	7.2	7.2		2310	-.03	Large
		2	7.4	7.4		2280	-.01	
		3	7.4	7.4		2320	.05	
224	2C	1	7.5	7.4		2320	-.02	Large
		2	7.6	7.6		2290	-.01	
		3	7.6	7.6		2320	.02	
225	2C	1	6.7	6.7		2320	-.08	Large
		2	6.8	6.9		2280	-.03	
		3	6.8	7.0		2320	.00	
229	IE	1	7.3	7.1		2310	-.02	Large
		2	7.4	7.4		2270	-.01	
		3	7.4	7.3		2300	.02	
231	IE	1	6.3	6.2	6.1	2500	.00	Large
		2	6.3	6.2	6.0	2450	.02	
		3	6.2	6.1	5.9	2500	.02	
231D	IE	1	6.6	6.5	6.3	2480	-.09	Small
		2	6.7	6.6	6.5	2450	-.01	
		3	6.6	6.6	6.5	2520	-.02	
232	IE	1	7.2	7.3		2290	-.01	Large
		2	7.3	7.4		2300	.00	
		3	7.4	7.5		2300	.01	

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Table 9 (con't)

<u>Billet No.</u>	<u>Specimen No.</u>	<u>Cycle</u>	<u>2000°C</u>	<u>2300°C</u>	<u>2500°C</u>	<u>Max. Temp. °C</u>	<u>Perm. Deform. %</u>	<u>Specimen Size</u>
	2C	1	7.1	7.2		2390	-.04	Large
		2	7.4	7.5		2300	.00	
		3	7.3	7.5		2300	.02	
		4	7.4	7.4		2300	.00	
233	1	1	7.1	7.0		2280	-.03	Large
		2	7.2	7.2		2290	.00	
		3	7.0	7.1		2290	-.02	
	2	1	7.0	6.9		2290	-.04	
		2	7.1	7.1		2290	-.02	
		3	7.1	7.1		2290	.01	
		4	7.2	7.1		2290	.00	
234	1C	1	7.2	7.2		2320	.00	Large
		2	7.3	7.3		2290	-.01	
		3	7.3	7.3		2320	.04	
238	1E	1	6.8	6.9		2250	-.01	Large
		2	7.0	7.1		2260	.00	
		3	6.9	7.0		2250	.00	
	3C	1	7.0	7.1		2260	.00	
		2	7.0	7.2		2270	.00	
		3	7.0	7.2		2260	.01	
	5C	1	6.9	7.1		2280	-.05	
		2	7.1	7.1		2300	.01	
		3	7.0	7.2		2290	.01	
	7C	1	7.1	7.1		2280	.00	
		2	7.1	7.1		2300	.01	
		3	7.1	7.3		2290	.00	

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Table 9 (con't)

<u>Billet No.</u>	<u>Specimen No.</u>	<u>Cycle</u>	<u>2000°C</u>	<u>2300°C</u>	<u>2500°C</u>	<u>Max. Temp. °C</u>	<u>Perm. Deform. %</u>	<u>Specimen Size</u>
238	9E	1	7.0	7.1		2260	-.02	Large
		2	7.1	7.2		2270	.01	
		3	7.2	7.2		2280	.03	
239	1E	1	7.2	7.3		2270	-.09	Large
		2	7.6	7.8		2290	.00	
		3	7.7	7.8		2290	.01	
		4	7.8	7.8		2290	-.01	
	2C	1	7.4	7.5		2270	-.02	Large
		2	7.5	7.4		2300	.00	
		3	7.4	7.5		2270	-.01	
240	1C	1	5.9	5.9		2320	-.04	Large
		2	6.0	6.1		2300	-.05	
		3	6.3	6.2		2320	-.03	
242	1E	1	6.4	6.3		2260	-.01	Large
		2	6.4	6.4		2270	-.01	
		3	6.4	6.5		2280	.01	
	4C	1	6.5	6.5		2280	-.04	
		2	6.8	6.7		2290	.01	
		3	6.7	6.7		2290	.01	
	6C	1	6.4	6.4		2270	-.06	
		2	6.8	6.7		2300	.01	
		3	6.7	6.7		2290	.00	
	8C	1	6.3	6.3		2270	.02	
		2	6.3	6.2		2290	.02	
		3	6.3	6.2		2290	.01	
249	1	1	7.0	7.1		2280	-.01	
		2	7.0	7.0		2320	.01	
		3	6.8	6.9		2280	-.02	

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Table 9 (con't)

<u>Billet No.</u>	<u>Specimen No.</u>	<u>Cycle</u>	<u>2200°C</u>	<u>2300°C</u>	<u>2500°C</u>	<u>Max. Temp. °C</u>	<u>Perm. Deform. %</u>	<u>Specimen Size</u>
250	3	1	7.3	7.3		2280	-.04	Large
		2	7.4	7.3		2310	.02	
		3	7.1	7.2		2280	-.05	
	5	1	7.2	7.2		2220	-.08	
		2	7.6	7.6		2220	.00	
		3	7.6	7.6		2210	.01	
	7	1	7.3	7.3		2220	-.04	
		2	7.4	7.5		2220	.01	
		3	7.4	7.5		2220	.02	
	9	1	7.2	7.2		2220	.12	Large
		2	6.6	6.8		2220	-.09	
		3	7.1	7.2		2220	.00	
	1	1	6.2	6.5		2280	-.18	
		2	7.0	6.8		2320	.14	
		3	6.0	6.3		2270	-.08	
		4	6.6	6.7		2300	.00	
		5	6.7	6.7		2250	.02	
		6	6.4	6.6		2350	-.01	
	3	1	7.2	7.3		2300	.01	Large
		2	7.1	7.1		2330	.01	
		3	6.8	7.0		2300	-.09	
	5	1	7.3	7.3		2300	-.05	
		2	7.6	7.4		2330	-.01	
		3	7.4	7.5		2300	-.02	
	7	1	7.3	7.3		2300	-.03	
		2	7.5	7.3		2330	.02	
		3	7.1	7.2		2300	-.03	

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Table 9 (con't)

<u>Billet No.</u>	<u>Specimen No.</u>	<u>Cycle</u>	<u>2200°C</u>	<u>2300°C</u>	<u>2500°C</u>	<u>Max. Temp. °C</u>	<u>Perm. Deform. %</u>	<u>Specimen Size</u>
251	9	1	7.0	7.1		2300	-.07	Large
		2	7.3	7.3		2330	.08	
		3	6.8	7.0		2300	-.08	
	1	1	7.0	6.8		2220	.03	
		2	6.5	6.7		2220	.04	
		3	6.3			2050	.12	
	3	1	6.6	6.7		2230	.00	
		2	6.5	6.6		2220	.01	
		3	6.4			2050	.11	
	5	1	7.0	7.0		2250	.00	
		2	6.9	7.0		2230	-.02	
		3	7.0			2060	.06	
	7	1	7.2	7.2		2240	-.02	
		2	7.1	7.2		2240	-.03	
		3	7.3			2060	.05	
	9	1	7.5	7.6		2230	-.01	Large
		2	7.4	7.5		2230	-.03	
		3	7.7			2060	.06	
252	1	1	7.7	7.7		2290	-.05	Large
		2	8.0	7.8		2250	.01	
		3	7.8	7.8		2360	.00	

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Table 9

<u>Billet No.</u>	<u>Specimen No.</u>	<u>Cycle</u>	<u>2200°C</u>	<u>2300°C</u>	<u>2500°C</u>	<u>Max. Temp. °C</u>	<u>Perm. Deform. %</u>	<u>Specimen Size</u>
252	3	1	7.7	7.7		2290	-.03	Large
		2	7.9	7.9		2250	.01	
		3	7.7	7.8		2360	.02	
	5	1	7.6	7.6		2290	-.07	
		2	8.0	7.9		2250	.01	
		3	7.8	7.8		2360	.01	
	7	1	7.7	7.7		2290	-.04	
		2	7.9	7.9		2250	.01	
		3	7.8	7.9		2360	.00	
	9	1	7.7	7.7		2290	-.02	
		2	7.9	7.9		2250	.02	
		3	7.8	7.8		2360	.00	
256	1	1	7.1	7.1		2260	-.01	
		2	7.2	7.1		2260	.02	
		3	7.3	7.2		2240	.01	
	2	1	6.3	6.2		2250	-.03	
		2	6.4	6.4		2230	.02	
		3	6.4	6.3		2250	.02	

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Table 10

Creep Deformation Data - across grain
2500°C 4000 psi (U)

Billet No.	Specimen No.		% Deformation ³⁾		ϵ ²⁾
			1 hr.	1.5 hrs.	
Baseline 75 w/o NbC bal. graphite flour			3.3	4.3	0.67
132	1		3.4	---	0.65
	2		3.6	---	0.64
		Avg.	<u>3.5</u>		<u>0.65</u>
145	1		5.1	---	0.80
	2		4.4	---	0.76
	3		3.9	5.2	0.69
	4		3.5	4.7	0.70
		Avg.	<u>4.2</u>		<u>0.74</u>
146	1		4.2	---	0.66
	2		4.2	5.6	0.69
	3		3.3	4.5	0.70
	4		3.4	4.6	0.75
		Avg.	<u>3.8</u>	<u>4.9</u>	<u>0.70</u>
149	1		3.5	4.5	0.60
	2		4.4	5.8	0.68
	3		2.7	3.5	0.70
	4		2.9	3.6	0.67
		Avg.	<u>3.4</u>	<u>4.4</u>	<u>0.66</u>
153	1 T ¹⁾		3.1	4.3	0.71
	4 B		4.6	6.4	0.77
		Avg.	<u>3.9</u>	<u>5.4</u>	<u>0.74</u>
156	1 T		5.4	8.1	0.76
	2 T		6.0	8.5	0.84
	3 B		5.2	7.4	0.85
	4 B		4.9	7.3	0.79
		Avg.	<u>5.4</u>	<u>7.8</u>	<u>0.81</u>

1) T = top of billet
B = bottom of billet

2) $\epsilon = At^n$

3) by micrometer measurement
before and after the run.

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Table 10 (con't)

Billet No.	Specimen No.	% Deformation		n
		1 hr.	1.5 hrs.	
157	1 T	4.4	6.0	0.74
	2 T	5.2	7.2	0.77
	4 B	4.3	6.0	0.73
	Avg.	<u>4.6</u>	<u>6.4</u>	<u>0.75</u>
174	1 T	5.4	6.9	0.66
	2 T	5.6	7.7	0.74
	3 B	4.8	6.2	0.74
	4 B	3.8	5.1	0.69
	Avg.	<u>4.9</u>	<u>6.5</u>	<u>0.71</u>
180	1 T	4.4	6.0	0.78
	4 B	3.9	5.1	0.66
	Avg.	<u>4.2</u>	<u>5.6</u>	<u>0.72</u>
182	1 T	5.0	7.1	0.83
	2 T	3.8	5.3	0.82
	3 B	4.0	5.7	0.82
	4 B	4.8	6.8	0.82
	Avg.	<u>4.4</u>	<u>6.2</u>	<u>0.82</u>
192	1 T	5.1	7.0	0.75
	4 B	7.0	9.2	0.68
	Avg.	<u>6.1</u>	<u>8.1</u>	<u>0.72</u>
193	2 T	7.2	12.5	0.89
	3 B	6.3	10.2	0.88
	Avg.	<u>6.7</u>	<u>11.5</u>	<u>0.88</u>
197	1 T	3.7	5.3	0.74
	2 T	4.2	6.1	0.67
	3 B	5.7	8.3	0.73
	Avg.	<u>4.5</u>	<u>6.8</u>	<u>0.71</u>
212	1 T	3.2	4.1	----
	4 B	4.8	6.6	0.79
	Avg.	<u>4.0</u>	<u>5.3</u>	<u>0.79</u>

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Table 10 (con't)

Billet No.	Specimen No.	% Deformation		n
		1 hr.	1.5 hrs.	
222	1 T	3.1	3.7	0.65
	2 B	4.1	5.7	0.68
	3 T	3.9	5.2	0.71
	4 B	3.2	4.3	0.71
	Avg.	<u>3.6</u>	<u>4.7</u>	<u>0.69</u>
Post 222 Pressed	1	4.1	5.8	0.77
	2	3.4	4.6	0.67
	3	4.6	6.3	0.72
	4	3.0	4.2	0.64
	Avg.	<u>3.8</u>	<u>5.2</u>	<u>0.70</u>
223	1 T 1)	3.5	4.7	0.72
	2 M	3.1	4.1	0.69
	Avg.	<u>3.3</u>	<u>4.5</u>	<u>0.71</u>
224	1 T	3.6	5.1	0.71
	4 B	3.4	4.6	0.73
	Avg.	<u>3.5</u>	<u>4.8</u>	<u>0.72</u>
225	1 T	4.1	5.4	0.65
	Avg.	---	---	----
229	1 T	3.9	5.3	0.76
	2 M	2.8	3.8	0.71
	3 B	2.6	3.4	0.69
	Avg.	<u>3.1</u>	<u>4.2</u>	<u>0.72</u>
231	1 T	3.7	4.8	0.66
	2 B	4.2	5.6	0.68
	3 T	3.5	4.5	0.58
	4 B	3.6	4.7	0.61
	Avg.	<u>3.8</u>	<u>4.9</u>	<u>0.63</u>
Post 231 Pressed	1	3.6	5.1	0.75
	2	3.5	4.9	0.75
	3	4.8	6.7	0.73

1) M = near longitudinal center of billet

Table 10 (con't)

Billet No.	Specimen No.		% Deformation		<u>n</u>
			<u>1 hr.</u>	<u>1.5 hrs.</u>	
232	4		2.9	3.9	0.69
		Avg.	<u>3.7</u>	<u>5.2</u>	<u>0.73</u>
	1 T		2.8	3.5	0.48
233	4 B		2.6	3.2	0.47
		Avg.	<u>2.7</u>	<u>3.3</u>	<u>0.48</u>
	1 T		2.3	3.0	0.53
234	2		2.3	3.0	0.50
	3		1.9	2.5	0.45
	4		1.8	2.3	0.47
238		Avg.	<u>2.1</u>	<u>2.7</u>	<u>0.49</u>
	1 T		2.4	3.6	0.70
	3 B		2.6	3.2	0.62
239		Avg.	<u>2.5</u>	<u>3.3</u>	<u>0.66</u>
	1 T		1.9	2.4	0.48
	2 M		2.2	2.7	0.44
240	3 B		2.2	2.9	0.46
	4 T		2.2	2.7	0.44
	5 M		1.9	2.3	0.40
242	6 B		2.1	2.6	0.44
		Avg.	<u>2.1</u>	<u>2.6</u>	<u>0.45</u>
	1 B		3.5	5.0	0.77
243	4 T		2.9	3.7	0.61
		Avg.	<u>3.2</u>	<u>4.3</u>	<u>0.69</u>
	1 T		5.6	7.6	0.64
244	2 M		3.8	5.0	0.61
	3 B		3.0	4.0	0.65
		Avg.	<u>4.1</u>	<u>5.5</u>	<u>0.63</u>
245	1 T		3.1	4.0	0.52
	2 M		1.8	2.3	0.53
	3 B		2.4	2.9	0.50

Table 10 (con't)

<u>Billet No.</u>	<u>Specimen No.</u>	<u>% Deformation</u>		<u>n</u>
		<u>1 hr.</u>	<u>1.5 hr.</u>	
249	4 T	2.9	3.7	0.53
	5 M	2.2	2.8	0.49
	6 B	2.0	2.5	0.43
	Avg.	<u>2.4</u>	<u>3.0</u>	<u>0.50</u>
	1 T	4.9	7.0	0.68
	2 M	3.6	5.1	0.67
	3 B	5.8	8.7	0.74
	4 T	6.0	8.4	0.72
	5 M	4.7	7.0	0.72
	6 B	5.2	7.7	0.76
	Avg.	<u>5.0</u>	<u>7.3</u>	<u>0.71</u>
	1 T	4.6	6.5	0.77
	2 M	3.7	5.2	0.74
	3 B	4.1	5.9	0.78
250	4 T	6.2	8.7	0.77
	5 M	3.8	5.3	0.73
	6 B	5.4	7.6	0.87
	Avg.	<u>4.6</u>	<u>6.6</u>	<u>0.78</u>
	1 T	4.4	6.0	0.66
	2 M	3.6	5.1	0.67
	3 B	3.6	4.8	0.63
	4 T	4.3	5.9	0.65
	5 M	3.8	5.4	0.68
	6 B	4.1	5.6	0.62
	Avg.	<u>4.0</u>	<u>5.5</u>	<u>0.65</u>
	1 T	5.0	7.0	0.66
	2 M	4.4	6.5	0.67
	3 B	2.9	3.9	0.63
251	4 T	4.5	5.7	0.69
	5 M	4.0	4.9	0.66
	252			

Table 10 (con't)

<u>Billet No.</u>	<u>Specimen No.</u>		<u>% Deformation</u>		<u>n</u>
			<u>1 hr.</u>	<u>1.5 hrs.</u>	
256	6 B		4.0	5.4	0.64
		Avg.	<u>3.9</u>	<u>5.6</u>	<u>0.66</u>
	1 T		4.4	5.7	0.68
	3 B		2.4	3.3	0.57
		Avg.	<u>3.4</u>	<u>4.5</u>	<u>0.62</u>

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Table 11A

Flexural Strength and Electrical Conductivity
(U)

Billet No.	Specimen No.	With Grain		Across Grain		
		Flex. Strength KSI	Elec. Cond. $\text{ohm}^{-1} \text{cm}^{-1}$	Specimen No.	Flex. Strength KSI	Elec. Cond. $\text{ohm}^{-1} \text{cm}^{-1}$
132	1	12.3	10100	1P	7.3	7200
	2 E ²⁾	8.8	8600	2	8.6	8200
	3	10.6	8700	3	5.4	5200
	4	9.6	10000			
	Avg.	<u>10.3</u>	<u>9400</u>		<u>7.1</u>	<u>6900</u>
145	1	6.2 ¹⁾	6500	1P ³⁾	3.6	4700
	2 E	7.4	8300	2	3.6 ¹⁾	4500
	3	5.7	7900	3	3.7	4700
	4	5.7	7500	4	4.0	5100
	Avg.	<u>6.2</u>	<u>7600</u>		<u>3.7</u>	<u>4700</u>
146	2 E	5.6	8100	1P	1.6 ¹⁾	6500
	3	9.7	12200	2	4.5	5900
	4	7.9	9700	4	3.5	5800
	Avg.	<u>7.7</u>	<u>10000</u>		<u>3.2</u>	<u>6100</u>
149	1	15.1	11000	1P	11.0	8800
	2 E	13.6	10400	2	10.4	8900
	3	13.3	10800	3	10.8	8600
	4	12.3	10500	4	10.0	8900
	Avg.	<u>13.6</u>	<u>10700</u>		<u>10.6</u>	<u>8800</u>
153	1 E	12.0	9900			
	2	14.6	11000	1P	4.4	5700
	3	14.1	11400	3	4.8	5900
	4 E	13.3	10700	4	4.9	5700
	Avg.	<u>13.5</u>	<u>10700</u>		<u>4.7</u>	<u>5800</u>

1) Specimen broke outside of inner span.

2) WG Specimen taken from end of billet - denoted by E

3) AG Specimen taken from periphery of billet - denoted by P

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Table 11A (con't)

Billet No.	With Grain			Across Grain		
	Specimen No.	Flex. Strength KSI	Elec. Cond. ohm ⁻¹ cm ⁻¹	Specimen No.	Flex. Strength KSI	Elec. Cond. ohm ⁻¹ cm ⁻¹
156	1 E	12.9	10200	1P	6.4	6900
	2	12.9	10500	2	5.7	5800
	3	13.8	11100	3	6.1	6000
	4 E	13.7	12100	4	6.2	6500
	Avg.	13.3	11000		6.1	6300
157	1 E	10.7	7400	1P	1.9	4600
	2	7.3	6600	2	2.5	4700
	3	7.0	8200	3	3.9	4800
	4 E	8.5	6500	4	3.9	4900
	Avg.	8.4	7200		3.0	4800
174	1 E	4.3	5300	1P	5.5	5700
	2	6.6	8300	2	4.9	5100
	3	9.4	9800	3	4.5	5500
	4 E	8.2	9400	4	5.5	6800
	Avg.	7.1	8200		5.1	5800
180	1 E	9.7	7700	1P	3.5	3300
	2	8.5	7500	2	3.3	2900
	3	5.3	7500	3	2.2	3400
	4 E	8.3	7800	4	3.1	3400
	Avg.	8.0	7600		3.0	3100
182	1 E	8.4	7500	1P	2.7	3600
	2	9.0	8500	2	3.9	4400
	3	10.7	9200	3	3.9	4600
	4 E	11.2	9300	4	2.1	3500
	Avg.	9.8	8600		3.1	4000
192	1 E	13.0	9700	1P	6.9	5.800
	2	11.2	9700	2	6.2	5.700
	3	10.3	10100	3	6.3	5.300
	4 E	15.0	10000	4	6.5	6000
	Avg.	12.4	9900		6.5	5500

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Table 11A (con't)

Billet No.	Specimen No.	With Grain		Specimen No.	Across Grain			
		Flex. Strength KSI	Elec. Cond. ohm ⁻¹ cm ⁻¹		Flex. Strength KSI	Elec. Cond. ohm ⁻¹ cm ⁻¹		
193	1 E	16.1	12900	1 P	5.2	6000		
	2	12.6	11500	2	5.4	5900		
	3	14.6	12000	3	5.5	5700		
	4 E	11.6	11600	4	5.7	5900		
	Avg.	13.7	12000		5.5	5900		
197	1 E	15.6	13200	1 P	5.0	6100		
	2	10.7	12000	2	5.0	5700		
	3	12.9	12300	3	5.1	5700		
	4 E	15.2	13600	4	5.1	5500		
	Avg.	13.6	12800		5.0	5800		
212	1 E	9.3	10100	1 P	4.9	6100		
	2	8.3	10400	2	4.8	5600		
	3	9.7	10300	3	5.1	5500		
	4 E	10.9	10800					
	Avg.	9.5	10400		5.0	5700		
222	1	12.0	10400	1 P	6.9	7400		
	2	12.3	10400	2	7.0	6600		
	3	14.3	11900	3	7.6	7600		
	4 E	12.9	11300	4 P	6.8	7900		
	Avg.	12.9	11000		7.1	7400		
222P ⁴⁾	1	11.1	10400	1P	7.7	7000		
	2	12.1	10600	2	6.2	7300		
	3	14.8	12000	3	6.4	7700		
	4 E	11.2	10400	4P	6.7	7700		
	Avg.	12.3	11800		6.8	7400		
223	1 E	13.8	12000	1 P	Top 7.4	Bottom 5.0	Top 7300	Bottom 6900
	2	14.1	12000	2 P	6.6	7.1	7300	7500
	3	13.7	12000	3 P	7.7	6.9	7300	7400

4) Post pressed one half of billet 222.

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Table 11A (con't)

With Grain				Across Grain					
Billet No.	Specimen No.	Flex. Strength KSI	Elec. Cond. ohm ⁻¹ cm ⁻¹	Specimen No.		Flex. Strength KSI	Elec. Cond. ohm ⁻¹ cm ⁻¹		
	4	13.0	12000	4 P	7.3	6.5	7400	7500	
	5	13.6	12000	5	7.6	6.6	7800	6300	
	6 E	13.7	12000	6	7.2	6.9	7400	7200	
	Avg.	13.7	12000		7.3	6.5	7400	7100	
				Billet Avg. - 6.9				7200	
224	1 a	15.8		1		6.1	7500		
	1 b	14.1		2		5.6	7300		
	2 a	15.0		3		6.3	7000		
	2 b	16.0		4		5.9	7200		
	3 a	15.1							
	3 b	15.1							
	4 a E	15.3							
	4 b E	14.8							
	Avg.	15.1				6.0	7300		
225	1 a	2.0		1		1.2	1400		
	1 b	2.1		2		1.3	1700		
	2 a	2.9		3		1.9	2800		
	2 b	2.5		4		1.5	5000		
	3 a	3.1							
	3 b	3.2							
	4 a E	2.3							
	4 b E	2.1							
	Avg.	2.5				1.5	2700		
						Top	Bottom	Top	Bottom
229	1 E	18.4	13200	1 P	8.4	7.6	8100	7400	
	2	17.2	13200	2 P	8.4	6.0	7200	7100	
	3	17.5	13200	3 P	7.5	7.2	7700	6800	
	4	17.1	13200	4 P	7.4	6.8	7700	7100	
	5	17.3	13200	5	5.1	5.5	6800	6600	
	6 E	17.9	13200	6	6.4	6.2	7000	6600	
	Avg.	17.6	13200		7.2	6.5	7400	6900	
				Billet Avg. - 6.9				7200	

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Table 11A (con't)

Billet No.	Specimen No.	With Grain		Specimen No.	Across Grain	
		Flex. Strength KSI	Elec. Cond., ohm ⁻¹ cm ⁻¹		Flex. Strength KSI	Elec. Cond., ohm ⁻¹ cm ⁻¹
231	1	13.4	8800	1 P	5.5	5000
	2	12.8	8100	2	4.6	4100
	3	12.4	8900	3	5.1	4100
	4 E	10.0	7300	4 P	6.1	5000
	Avg.	12.2	8300		5.3	4600
231 P ⁴⁾	1	----		1 P	---	
	2	12.1	8500	2	4.8	3900
	3	12.8	8700	3	4.8	3700
	4 E	10.5	7200	4 P	5.6	4200
	Avg.	11.8	8100		5.1	4000
232	1 a	13.7	7600	1	8.6	5600
	1 b	12.2	7100	2	8.6	5700
	2 a	14.1	7600	3	7.8	5500
	2 b	14.4	7400	4	9.0	5500
	3 a	14.7	7700			
	3 b	16.0	8000			
	4 a E	13.5	7300			
	4 b E	13.4	7700			
	Avg.	14.0	7600		8.5	5600
233	1 a	14.4	7700	1	9.1	5500
	1 b	14.6	7700	2	7.2	5600
	2 a	14.1	7700	3	7.8	5500
	2 b	15.1	8000	4	7.1	5400
	3 a	16.3	8400			
	3 b	15.8	8300			
	4 a E	14.6	8200			
	4 b E	16.1	8200			
	Avg.	15.1	8000		7.8	5500

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Table 11A (con't)

With Grain				Across Grain			
Billet No.	Specimen No.	Flex. Strength	Elec. Cond.	Specimen No.	Flex. Strength	Elec. Cond.	
		KSI	$\text{ohm}^{-1} \text{cm}^{-1}$		KSI	$\text{ohm}^{-1} \text{cm}^{-1}$	
					Top	Bottom	Top Bottom
234	1 E	14.4	11300	1 P	7.0	7.5	8200 8600
	3	12.9	11000	2 P	7.2	7.8	7500 7600
	4	13.9	11600	3 P	5.9	6.5	8400 8100
	5	13.7	11600	5	5.9	6.3	7000 7600
	6 E	15.2	12000	6	5.6	5.8	6900 7000
	Avg.	13.9	11500		6.3	6.8	7600 7800
				Billet Avg. - 6.5			7700
239	1 a	15.2	12700				
	1 b	14.3	12800	1	5.7		6300
	2 a	16.0	12500	2	6.2		6300
	2 b	14.6	12700	4	5.6		6400
	3 a	17.4	13200				
	3 b	16.4	13600				
	4 a E	14.5	12300				
	4 b E	13.7	12100				
	Avg.	15.3	12700		5.8		6300
					Top	Bottom	Top Bottom
240	1 E	13.9	9200	1 P	4.8	4.4	3500 3100
	2	13.7	8600	2 P	4.4	4.6	4400 3900
	3	12.2	8600	3 P	6.1	4.7	4900 3900
	4	12.8	8700	4 P	4.4	4.5	4000 3400
	5	13.2	8200	5	3.4	5.0	4400 4100
	6 E	12.6	8400	6	4.3	5.4	4500 4100
	Avg.	13.1	8600		4.6	4.8	4300 3800
				Billet Avg. - 4.7			4000

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Table 11 B

Flexural Strength and Electrical Conductivity
(U)

With Grain								Across Grain						
Billet No.	Specimen No.	Flex. Strength KSI			El. Cond. ohm ⁻¹ cm ⁻¹			Spec. No.	Flex. Strength KSI			El. Cond. ohm ⁻¹ cm ⁻¹		
		Left	Mid	Right	Left	Mid	Right		Top	Mid	Bottom	Top	Mid	Bottom
238	1	14.1	12.4	11.8	7800	7600	7200	1	5.1	7.1	6.2	4700	5200	4900
	2	Specimen Broken			----	----	----	2	5.3	5.6	5.9	4700	5200	5100
	3	15.1	15.1	14.7	8400	8400	8400	3	6.5	5.3	---	5000	5200	5100
	4	14.7	13.9	14.2	8300	8400	7900	4	6.4	6.4	7.3	5100	5300	5000
	5	14.4	14.6	12.8	8200	7700	7400	5	6.6	---	7.7	5100	5200	4900
	6	13.1	12.5	----	7800	7300	6900							
	7	10.8	14.4	14.3	6600	7700	7700							
	8	12.9	13.7	13.3	7700	7700	7700							
	9	13.1	12.9	12.4	7700	7500	7300							
	Avg.	13.4	13.6	13.4	7200	7200	7500		6.0	6.1	6.8	4900	5300	5000
Billet Avg.		13.5			7200				6.3			5100		
242	1	10.6	10.8	8.3	7600	7800	6400	1	5.5	4.8	5.1	4100	4000	4000
	2	11.9	12.1	12.3	8100	8200	8500	2	4.9	4.2	5.2	3800	3700	4600
	3	11.7	11.6	12.8	7900	8200	8600	3	4.9	4.6	---	4500	3500	3500
	4	11.8	13.6	13.0	8600	8900	8700	4	---	---	---	----	----	----
	5	12.9	13.1	12.9	8400	9200	8500	5	---	5.1	4.1	4200	3900	4000
	6	14.6	14.0	14.2	10000	9700	10000	6	4.7	4.3	4.8	3900	3600	4500
	7	14.8	14.3	14.7	9800	9700	10100							
	8	14.3	14.3	13.7	10000	9800	9000							
	9	12.8	13.2	12.4	9300	9400	9800							
	Avg.	12.8	13.0	12.7	8800	9000	8800		5.0	4.6	4.8	4100	3700	4100
Billet Avg.		12.8			8900				4.8			4000		

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Table 11 B

Flexural Strength and Electrical Conductivity
(U)

With Grain									Across Grain					
Billet No.	Specimen No.	Flex.Strength KSI			El. Cond. ohm ⁻¹ cm ⁻¹			Spec. No.	Flex. Strength KSI			El. Cond. ohm ⁻¹ cm ⁻¹		
		Left	Mid	Right	Left	Mid	Right		Top	Mid	Bottom	Top	Mid	Bottom
249	1	Specimen Broken			---	---	----	1	Specimen Broken			----	---	----
	2	13.8	14.7	14.9	11800	12200	12000	2	6.2	6.0	6.2	6100	6800	7300
	3	14.7	15.4	15.0	12100	11900	12000	3	6.3	5.8	6.3	5900	5800	7000
	4	11.8	14.7	13.7	11400	12300	12300	4	5.9	5.5	6.2	5700	5800	6700
	5	14.3	15.0	13.7	12500	12700	12300	5	5.8	6.0	6.6	6000	6200	7300
	6	13.8	14.7	13.6	12600	12600	11900	6	Specimen Broken			----	----	----
	7	14.8	14.3	12.8	13500	12300	11500							
	8	13.8	----	11.6	11700	11800	10300							
	9	15.9	13.7	13.4	12400	11400	11200							
	Avg.	14.1	14.7	13.6	12200	12100	11800		6.1	5.8	6.3	5900	6100	7100
	Billet Avg.	14.1			12000				6.1			6400		
250	1	4.3	6.4	----	5100	8300	5300	1	---	6.1	5.9	4200	5900	6500
	2	9.2	13.1	9.4	8600	11300	7900	2	5.9	5.6	---	6500	5300	4900
	3	14.9	13.8	13.7	11300	11900	11000	3	5.9	5.9	---	6200	5500	5000
	4	13.7	15.2	15.1	12000	12600	12100	4	---	5.0	3.8	6200	5400	4700
	5	15.9	14.4	13.9	13000	12400	12200	5	5.8	5.4	---	6800	5700	4700
	6	13.2	13.5	14.1	12300	11600	12200	6	5.9	5.5	---	6300	6000	3700
	7	14.1	13.6	15.0	12400	12000	13000							
	8	13.5	14.1	13.8	11600	11500	12300							
	9	----	9.2	8.1	7100	10100	8800							
	Avg.	12.4	12.6	12.9	10400	11300	10500		5.9	5.6	4.9	6000	5600	4900
	Billet Avg.	12.6			10700				5.5			5500		

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Table 11 B

Flexural Strength and Electrical Conductivity
(U)

With Grain								Across Grain						
Billet No.	Specimen No.	Flex. Strength KSI			El. cond. $\text{ohm}^{-1}\text{cm}^{-1}$			Spec. No.	Flex. Strength KSI			El. Cond. $\text{ohm}^{-1}\text{cm}^{-1}$		
		Left	Mid	Right	Left	Mid	Right		Top	Mid	Bottom	Top	Mid	Bottom
251	1	4.9	6.1	6.4	6100	7100	7200	1	4.8	4.0	4.3	5700	5300	5100
	2	4.8	6.5	5.6	5300	7300	6400	2	5.2	3.9	3.0	5400	4800	4200
	3	5.5	4.6	5.2	5900	5200	6200	3	4.9	---	---	5300	4700	3400
	4	7.1	6.3	8.3	8200	7500	9000	4	4.8	3.7	---	5200	4700	3400
	5	8.7	9.6	9.3	10900	9700	10300	5	Specimen Broken					
	6	11.3	11.5	11.3	10900	11200	11100	6	4.1	4.9	4.6	4900	5000	5300
	7	11.8	12.1	10.9	10700	11200	10900							
	8	11.6	12.6	11.6	10900	10400	10600							
	9	11.1	12.2	13.1	11500	11600	11700							
	Avg.	8.5	9.1	9.1	9200	9000	9300		4.8	4.1	4.0	5300	4900	4300
Billet Avg.			8.9			9200				4.3			4800	
252	1	14.1	13.5	14.0	11700	9900	11200	1	6.9	6.2	6.2	6400	6000	6800
	2	13.9	12.9	12.7	11800	11000	10000	2	6.8	6.4	6.0	6400	5800	6400
	3	15.0	15.3	16.2	12300	12700	13000	3	6.9	6.3	6.4	6700	5800	6400
	4	13.8	15.4	14.1	11600	12700	11700	4	6.6	6.3	6.3	6200	5900	6200
	5	15.5	16.0	13.7	13000	13000	11500	5	6.2	5.2	6.2	6700	5800	6500
	6	14.3	16.5	16.3	12900	13300	13100	6	6.4	5.9	6.1	7100	6500	6800
	7	14.9	16.2	15.1	11600	13100	12600							
	8	12.5	14.7	14.2	11700	12500	12300							
	9	14.1	14.7	13.7	12600	12700	12400							
	Avg.	14.4	15.0	14.4	12200	12300	12000		6.6	6.1	6.1	6600	6000	6600
Billet Avg.			14.6			12200				6.3			6400	

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Table 11 B

Flexural Strength and Electrical Conductivity
(U)

With Grain								Across Grain						
Billet No.	Specimen No.	Flex. Strength KSI			El. Cond.ohm ⁻¹ cm ⁻¹			Spec. No.	Flex. Strength KSI			El. Cond. ohm ⁻¹ cm ⁻¹		
		Left	Mid	Right	Left	Mid	Right		Top	Mid	Bottom	Top	Mid	Bottom
256	1	11.8	10.5	11.5	7400	7100	7400	1	3.8	4.5	4.3	3400	3200	3200
	2	11.6	12.5	12.6	8100	8100	8100	2	3.5	3.7	4.5	3300	3300	3300
	3	13.2	13.3	12.3	8700	8800	8300	3	4.4	4.1	3.8	3400	3300	3400
	4	14.2	13.8	14.4	9400	9000	9400	4	3.4	3.8	5.0	3700	3600	4100
	Avg.	13.0	12.5	12.6	8400	8200	8300		3.8	4.0	4.4	3400	3400	3500
Billet Avg.			12.7			8300			4.1			3400		

Table 12
Data Summary - 75/25 Calcined Petroleum Coke Billets
 (C-RD)

Billet No.	1 Hour Creep		$\times 10^{-6}/^{\circ}\text{C}$	Temp. $^{\circ}\text{C}$	C T E		Flexural Strength, KSI		Electrical Conductivity $\text{ohm}^{-1} \text{cm}^{-1}$	
	%	n			Permanent Deformation, %		WG	AG	WG	AG
Baseline (graphite flour)	3.5	0.67	7.6	2500	.02	.02	15.0	10.6	11200	8800
132	3.5	0.65	6.6	2370	.08	-.05	10.3	7.1	9400	6900
156	5.4	0.81	7.0	2480	-.05	-.02	13.3	6.1	11000	6300
182	4.4	0.82	6.8	2500	.02	-.08	9.8	3.1	8600	4000
222	3.6	0.69	7.3	2500	.01	.05	12.9	7.1	11000	7400
223	3.3	0.71	7.4	2320	-.03	.01	13.7	6.9	12000	7200
224	3.5	0.72	7.7	2320	-.02	-.01	15.1	6.0	13200	7300
225	4.1	0.65	7.0	2320	-.08	-.11	2.5	1.5	1700	2700
227	5.0	0.79	7.6	2320	.01	.01	15.6	7.3	12900	7900
229	3.1	0.72	7.3	2300	-.02	-.01	17.6	7.2	13200	6900
234	2.5	0.66	7.3	2320	.00	.03	13.9	6.6	11500	7800
239	3.2	0.69	7.5	2280	-.05	-.06	15.3	5.8	12700	6300
249	5.0	0.71	7.3	2280	-.01	-.01	14.1	6.1	12000	6400
252	3.9	0.66	7.8	2320	-.04	-.02	14.6	6.3	12200	6600

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Table 13

Data Summary - 75/25 Calcined Petroleum Coke Billets - Post Pressed Material
(Billet Averages)
(CRD)

Billet No.	1 Hour Creep		C T E				Flexural Strength, KSI		Electrical Conductivity ohm ⁻¹ cm ⁻¹	
	%	n	X10 ⁻⁶ /°C	Temp. °C	Permanent 1st cycle	Deformation, % 3 cycle total	WG	AG	WG	AG
145	4.2	0.74	6.6	2520	.03	.03	6.2	3.7	7600	4700
146	3.8	0.70	6.7	2550	-.09	-.12	7.7	3.2	10000	6100
149	3.4	0.66	7.7	2500	-.01	.15	13.6	10.6	10700	8800
153	3.9	0.74	7.0	2470	-.06	-.14	13.5	4.7	10700	5800
157	4.4	0.74	7.1	2450	-.14	-.15	8.4	3.0	7200	4800
174	4.6	0.68	6.7	2490	.14	-.02	7.1	5.1	8200	5800
192	6.1	0.72	6.8	2490	.09	.07	12.4	6.5	9900	5500
193			6.6	2490	-.05	-.17	13.7	5.5	12000	5900
197	4.5	0.71	6.7	2480	-.08	-.13	13.6	5.0	12800	5800
212	4.0	0.79	7.1	2480	-.07	-.10	12.9	7.1	11000	7400
222	3.8	0.70	7.3	2520	.01	.01	12.3	6.8	11800	7400
250	4.6	0.78	7.1	2300	-.04	-.02	12.6	5.5	10700	4900
251	4.0	0.65	7.0	2250	.00	.02	8.9	4.3	9200	4800

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Table 14

Data Summary - 65 w/o NbC Bal. C and 35 w/o NbC 35 w/o TaC Bal. C Composite
 (Billet Averages)
 (CRD)

Billet No.	1 Hour Creep %	Creep n	C T E		Permanent Deformation, %		Flexural Strength KSI		Electrical Conductivity	
			X10 ⁻⁶ /°C	Temp. °C	1st cycle	3 cycle total	WG	AG	WG	AG
65 NbC 35C										
231	3.8	0.64	5.9	2500	.00	.02	12.2	5.3	8300	4600
232F*	2.7	0.48	7.4	2300	-.02	-.01	14.0	8.5	7600	5600
240	4.1	0.63	6.2	2320	-.04	-.12	13.1	4.7	8600	4000
65 NbC 35C (Post Pressed 2 Hrs. at 2500°C and 3 KSI)										
180 ⁺	4.2	0.72	5.7	2480	-.05	-.07	8.0	3.0	7600	3100
231	3.7	0.70	6.5	2520	-.09	-.12	11.8	5.1	8100	4000
233F*	2.1	0.49	7.1	2300	-.03	-.01	15.1	7.8	8000	5500
35 NbC 35 TaC 30C										
238F*	2.1	0.45	7.2	2300	-.02	.00	13.5	6.3	7200	5100
242	2.4	0.50	6.5	2300	-.02	.00	12.8	4.8	8900	4000
256	3.4	0.62	6.9	2250	-.02	.00	12.7	4.1	8300	3400

*Billet made with graphite flour. All others made with calcined petroleum coke.

+Billet 180 die compact formed by the old technique - all other billets formed by the new technique - billet 180 was post pressed 1 hour at 2700 $^{\circ}\text{C}$ and 2 KSI.

Table 15

Comparison of 75 - 25 Composite Made With Calcined Petroleum Coke vs. Graphite Flour (C-RD)

Composition	Carbon	Data Source (Billet Nos.)	Density % Theor.	1 Hr. Creep % n		CTE $10^{-6}/^{\circ}\text{C}$	1st Cycle Perm. Deform.	Flex. Strength KSI		Elec. Conductivity $\text{ohm}^{-1} \text{cm}^{-1}$	
								WG	AG	WG	AG
75 - 25	Coke	222, 223, 229, 234, 249, 252, Scatter	97.6	3.7	.69	7.4	-.02	15.4	6.8	12400	7100
			96.7/	2.5/	.66/	7.1/	-.04/	12.9/	6.1/	11000/	6400/
			98.3	5.0	.72	7.8	.01	17.6	7.2	13200	7800
75 - 25	Flour	10 WANL ⁴⁾ Billets Scatter	95.5	3.4	.63	7.5	.00	14.7	10.7	11000	8700
			95.1/	2.7/	.57/	7.3/	-.05/	12.8/	8.9/	10200/	8200/
			96.0	4.0	.67	7.7	.03	15.6	13.5	11700	9100
75 - 25	Flour	8 Billets ³⁾ from 4 alternate vendors Scatter	95.6	3.6	.66	7.6	.02	15.5	10.5	11300	8800
			94.7/	2.3/	.59/	7.4/	-.01/	12.6/	8.8/	10300/	8000/
			96.5	5.8	.78	7.7	.05	17.4	14.4	12200	9800
75-25	Graphi- tized Coke	227, 239 Scatter	97.6	4.1	.74	7.5	.02	15.4	6.5	12800	7100
			97.4/	3.2/	0.69/	7.5/	.01/	15.3/	5.8/	12700/	6300/
			97.8	5.0	0.79	7.6	-.05	15.6	7.3	12900	7900

4) See Bibliography

3) See Bibliography

Table 16

Comparison of 75 - 25 Composite Made With Old vs. New Processing (C-RD)

Coke Source	Compacting Technique	Post Pressed	Data Source (Billet No.)	Density % Theor.	1 Hr. Creep %		CTE $10^{-6}/^{\circ}\text{C}$	1st Cycle Perm. Deform.	Flex. Strength KSI		Elec. Cond. $\text{ohm}^{-1} \text{cm}^{-1}$	
									WG	AG	WG	AG
A	Old ¹⁾	No ³⁾	132, 182, 225	97.4	4.2	0.71	6.8	.00	7.5	3.9	6700	4500
A	Old	Yes ³⁾	146, 174, 212	<u>96.2</u>	<u>4.1</u>	<u>0.72</u>	<u>6.8</u>	<u>-.01</u>	<u>9.2</u>	<u>5.1</u>	<u>9800</u>	<u>7400</u>
			Avg.	96.8	4.2	0.72	6.8	.00	8.4	4.5	8300	6000
A	New ²⁾	No	222, 223, 229	97.7	3.1	0.70	7.3	-.01	14.5	7.0	12200	7300
			234									
A	New	Yes ³⁾	222	96.3	3.8	0.70	7.3	.01	12.3	6.8	11800	7400
			Avg.	<u>97.1</u>	<u>3.5</u>	<u>0.70</u>	<u>7.3</u>	<u>-.00</u>	<u>13.5</u>	<u>6.8</u>	<u>12000</u>	<u>7300</u>
B	Old	No ³⁾	156	97.3	5.4	0.81	7.0	-.05	13.3	6.0	11000	6300
B	Old	Yes ³⁾	145, 153, 157, 251	<u>97.0</u>	<u>4.2</u>	<u>0.74</u>	<u>6.9</u>	<u>-.06</u>	<u>9.2</u>	<u>3.9</u>	<u>8700</u>	<u>5000</u>
			Avg.	97.1	4.8	0.77	6.9	-.06	11.2	5.0	9800	5700
B	New	No ³⁾	249, 252	97.8	4.4	0.69	7.5	-.04	14.4	6.2	12100	6500
B	New	Yes ³⁾	250	<u>97.0</u>	<u>4.1</u>	<u>0.76</u>	<u>7.2</u>	<u>-.04</u>	<u>12.6</u>	<u>5.5</u>	<u>10700</u>	<u>4900</u>
				97.4	4.3	0.72	7.4	-.04	13.5	5.9	11400	5700
			Avg. New Processing	97.2	3.9	0.71	7.3	-.02	13.5	6.4	11700	6500
			Avg. Old Processing	97.0	4.5	0.74	6.8	-.03	9.8	4.7	9000	5900
			Avg. sans Post Pressing	97.4	4.3	0.74	7.1	-.02	12.4	5.8	10500	6100
			Avg. with Post Pressing	96.9	4.1	0.73	7.0	-.03	10.8	5.3	10300	6200

- 1) Full pressure on at 2500°C or less
 2) Full pressure on at 2900°C
 3) Post pressed 1 hour at 2700°C and 2000 KSI

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Table 17

Effects of Coke Particle Size Variation on Properties of 75 - 25 Composite (C-RD)

(Old compacting technique - full pressure on at 2500°C or less)

82	Coke Size, (Mesh)	Post Pressed	Data Source (Billet Nos.)	Density %Theor.	1 Hr. Creep		CTE $10^{-6}/^{\circ}\text{C}$	1st Cycle/Flex. Str. Perm. Deform.	KSI		Elec. Cond.		$\times 10^3$
					%	n			WG	AG	WG	AG	
	60/325	Yes	145, 146	95.6	4.0	0.72	6.6	-.03	7.0	3.5	8.8	5.4	
	100/325	No	182, 225	97.3	4.5	0.74	6.9	.04	6.2	2.3	5.2	3.4	
	100/325	Yes	157, 174	96.9	4.5	0.71	7.1	-.14	7.7	4.0	7.7	5.3	
	150/325	No	132, 156	97.6	4.5	0.73	6.8	.01	11.8	6.6	10.2	6.6	
	150/325	Yes	153, 212	97.3	4.0	0.77	7.0	-.06	13.2	5.9	10.9	6.6	
	-325	Yes	192	97.7	6.1	0.72	6.8	.09	12.4	6.5	9.9	5.5	

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Table 18

Effects of Composition and Graphite Type on Billet Properties

(U)

Ratio w/o NbC Graphite	75/25	65/35	60/40	50/50	10/90	75/25	65/35
Graphite Type	Flour	Flour	Flour	Flour	Flour	Coke	Coke
% Theoretical Density	95.5	95.5		92.6		97.6	96.5
WG CTE/ $^{\circ}\text{C} \times 10^{-6}$	7.6	7.3	6.6	5.4	4.7	7.4	6.2
AG% Compressive Creep 1 hr. 2500 $^{\circ}\text{C}$ 4 KSI	3.5	2.7	2.7	2.7		3.7	3.9
WG Flex. Strength, psi	15,000	14,500	11,500	11,400		15,400	12,400
AG Flex. Strength, psi	10,600	8,200	7,800	6,100		6,800	5,000
Ratio WG/AG Flex.	1.42	1.77	1.48	1.87		2.27	2.48
WG Elec. Conductivity $\text{ohm}^{-1} \text{cm}^{-1}$	11,200	7,800				12,400	8,500
AG Elec. Conductivity $\text{ohm}^{-1} \text{cm}^{-1}$	8,800	5,600				7,100	4,300
Ratio WG/AG Elec. Cond.	1.27	1.39				1.75	1.98

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Table 19

Relative Data on Thermal Shock Parameter

2000°C

(U)

Flour Composite

Calcined Petroleum Coke Composite

	<u>75/25</u>		<u>65/35</u>		<u>75/25</u>		<u>65/35</u>	
	<u>WG</u>	<u>AG</u>	<u>WG</u>	<u>AG</u>	<u>WG</u>	<u>AG</u>	<u>WG</u>	<u>AG</u>
k	100	70	110	70	120	50	130	50
α	7.6	8	7.2	9	7.2	9	6.1	11
S	15	10	14	8	15	7	13	5
E	10	6	8	3.2	10	4	8	2
$\frac{k s}{\alpha E}$	20	15	29	13	25	10	40	11

k - thermal conductivity

α - coefficient of thermal expansion

S - strength

E - Young's modulus of elasticity

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Table 20

Summary of Production Billet Evaluation - 75/25 Calcined Petroleum Coke Composite (C-RD)

<u>Lot No.</u>	<u>No. of Billets</u>	<u>% Creep 2500°C 4 KSI, 1 hr.</u>	<u>Flex. Strength, KSI WG</u>	<u>AG</u>	<u>Elec. Conductivity, ohm⁻¹cm⁻¹ WG</u>	<u>AG</u>	<u>Coefficient of Thermal Expansion in/in/°C - WG</u>	
1	8 Average Range ¹⁾	4.2 2.9-8.2	13.7 11.6-15.4	6.5 4.1-7.8	12300 11400-12600	7500 5900-8600	7.1 6.8-7.4	(billets - 1st thermal cycle only)
2	13 Average Range ¹⁾	4.1 2.7-7.2	11.6 6.8-15.4	5.8 5.1-7.2	11300 7200-13300	7100 6600-7800	6.9	(1 billet) ²⁾
3	8 Average Range ¹⁾	3.5 2.8-4.4	11.1 5.1-13.4	4.9 2.9-6.5	9400 5200-12100	6100 4600-7500	7.3 7.0-7.6	(2 billets)
4	5 Average Range ¹⁾	2.6 2.1-2.9	13.6 13.1-14.1	7.0 6.5-7.4			7.2 7.1-7.3	(3 billets)
5	14 Average Range ¹⁾	3.6 2.7-4.4	13.1 11.9-14.6	6.6 5.9-7.8			7.3 7.2-7.5	(3 billets)
Overall Average -		3.7	12.5	6.1	11,000	6900	7.2	
Average of lots 4 and 5 -		3.3	13.2	6.7			7.2	

1) Range of Billet Averages

2) This billet was rejected by Quality Control due to low flexural strength

Note: All billets were post pressed for 1 hour at 2700 °C and 2000 psi.

Table 21

Summary of Production Material Densities - 75/25 Calcined Petroleum Coke Composite
(% Theoretical) (CRD)

Lot No.	No. of Billets	Billet	Creep Specimens			WG Flexural Specimens (bottom of billet)	Side of Billet opposite creep specimens
			Top	Bottom	Avg.		
1	8 Range ¹⁾	95.6 94.5/96.2	95.5 93.8/96.1	94.4 92.2/96.1	95.0 93.0/96.1	95.7 94.5/96.3	94.5 92.6/95.3
2	13 Range ¹⁾	96.1 95.7/96.7	96.0 95.5/96.7	95.3 94.2/96.5	95.7 95.0/96.6	95.4 93.6/96.7	95.1 93.8/95.8
3	8 Range ¹⁾	96.6 95.5/96.8	95.2 92.6/96.3	94.4 92.2/96.1	94.8 93.0/96.1	95.5 92.2/96.7	95.3 94.2/96.3
Avg. 3 lots		96.1	95.6	94.7	95.2	95.5	95.0
4	5 Range ¹⁾	96.2 96.0/96.3					
5	14 Range ¹⁾	96.4 95.8/97.3					
Avg. of 5 lots		96.2					

¹⁾ Range of individual billets or specimens

Table 22

Effect of Location Within the Billet on Properties of
75/25 Calcined Petroleum Coke Production Billets-
Q. C. Data (C-RD)

<u>Lot No.</u>	<u>No. of Billets</u>	<u>% Creep</u>		<u>AG Flex. Strength</u>			<u>AG El. Conductivity</u>		
		<u>Top</u>	<u>Bottom</u>	<u>Top</u>	<u>Mid</u>	<u>Bottom</u>	<u>Top</u>	<u>Mid</u>	<u>Bottom</u>
1	8	3.7	4.7	6.6	6.4	6.5	7400	7500	7500
2	13	4.5	3.7	5.8	6.0	5.5	7000	7300	6900
3	8	3.6	3.3	4.6	5.5	4.7	6100	6700	5600
4	5	2.4	2.6	7.4	6.7	6.8			
5	14	3.6	3.4	7.1	6.5	6.1			
Avg.		3.7	3.6	6.3	6.1	5.9	6900	7200	6700

Niobium Carbide

Lot 5

Tantalum Carbide

Lot SP 26721

2

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Figure 2. Charged Graphite Die with Carbon
Felt Insulation

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Figure 3. Graphite Die in Coil Prior
to Pressing

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Figure 4. Hot Press and Temperature Indicator

Figure 5. Etched Microstructure of 75-25 Billet
225 Showing Cracked Carbide Matrix
Due to Improper Hot Pressing Cycle

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Billet 182 - Medium Banding and Vague Carbide Segregation.

Billet 224 - Medium Banding. No Segregation.

Billet 227 - Very Light Banding - No Carbide Segregation.

Billet 238 - Heavy Banding.

Figure 6. Positive Prints of Billet Radiographs - Magn. 1X.

Billet 145 - BI Coke Magn. 10X

Billet 146 - AI Coke Magn. 10X

Billet 145 - BI Coke Magn. 250X

Billet 146 - AI Coke Magn. 250X

Figure 7. Microstructures of Post Pressed 75-25 Billets
Made With -60 + 325 Mesh Coke From Two
Sources.

Billet 149 - Graphite Flour

Magn. 10X

Billet 153 - BI Coke

Magn. 10X

Billet 149 - Graphite Flour

Magn. 250X

Billet 153 - 153 BI Coke

Magn. 250X

Figure 8. Microstructures of Post Pressed 75-25 Billets Made with -150+325 Mesh Coke and Flour, Respectively.

Billet 157 - 100 + 325 Mesh

Billet 153 - 150 + 325 Mesh

Billet 192 - 325 Mesh

Figure 9. Microstructures of Post Pressed 75-25 Billets made with Different Mesh Sizes of BI Coke. Magn. 50X

Billet 182 - Not Post Pressed

Billet 174 - Post Pressed

Figure 10. Microstructures of 75-25 Billets made with -100
+325 Mesh Al Coke - with and without Post
Pressing.

Magn. 50X

Billet 174 - Al Coke Magn. 10X

Billet 157 - Bl Coke Magn. 10X

Billet 174 - Al Coke Magn. 250X

Billet 157 - Bl Coke Magn. 250X

Figure 11. Microstructures of Post Pressed 75-25 Billets made with
-100 + 325 Mesh Coke From Two Sources.

Billet 182 - Raw Coke Magn. 10X

Billet 187 - Graphitized Coke Magn. 10X

Billet 182 - Raw Coke Magn. 250X

Billet 187 - Graphitized Coke Magh. 250X

Figure 12. Microstructures of 75-25 Billets Made with -100 + 325 Mesh Al Coke with and without Pregraphitization of the Coke.

Billet 187 - Graphitized Coke
Magn. 50X

Billet 222 - Raw Coke
Magn. 50X

Figure 13. Microstructures of 75-25 Billets Made with -100 + 325 Mesh Al Coke with and without Pregraphitization of the Coke.

Billet 222 - Al Coke -100+325 Mesh Opti-
mized Processing. Magn. 10X

Billet 192 - Bl Coke -325 Mesh Billet Post
Pressed. Magn. 10X

Billet 222 - Al Coke -100+325 Mesh Opti-
mized Processing. Magn. 250X

Billet 192 - Bl Coke -325 Mesh Billet Post
Pressed. Magn. 250X

Figure 14. Microstructures of 75-25 Billets.

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Magn. 1.5X

Magn. 10X

Magn. 50X

Magn. 250X

Figure 15. Microstructure of Post Pressed 75-25 Billet 250 made with -100 +325 Mesh B2 Coke.

Billet 180 - BI Coke - Billet Post Pressed.
Magn. 10X

Billet 231 - AI Coke - Billet Not Post Pressed.
Magn. 10X

Billet 180 - BI Coke - Billet Post Pressed.
Magn. 250X

Billet 231 - AI Coke - Billet Not Post Pressed.
Magn. 250X

Figure 16. Microstructures of 65-35 Billets Made From -100+325 Mesh Coke
From Two Different Sources.

Billet 233 - Post Pressed - 150+325 Mesh Graphite Flour

Billet 180 - Post Pressed - 100+325 Mesh
BI Coke.

Billet 231 - Not Post Pressed - 100+325
Mesh AI Coke.

Figure 17. Microstructures of 65-35 Billets. Magn. 50X.

Magn. 1X

Magn. 10X

Magn. 250X

Figure 18. Microstructure of Post Pressed 65-35 Billet 233
Made with - 150+325 Mesh Graphite Flour.

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Billet 238 - -150+325 Mesh Graphite Flour.
Magn. 50X

Magn. 1X

Billet 242 - -100+325 Mesh Al Coke.
Magn. 50X

Magn. 1X

Figure 19. Microstructures and Macrostructures
of 35 TaC 35 NbC 30 Carbon Billets

Billet 238 -150 + 325 Mesh Graphite Flour.
Magn. 10X.

Billet 242 -100+325 Mesh Al Coke.
Magn. 10X

Billet 238 -150+325 Mesh Graphite Flour.
Magn. 250X

Billet 242 -100+325 Mesh Al Coke.
Magn. 250X

Figure 20. Microstructures of 35 TaC 35 NbC 30 Carbon
Billets.

Figure 21. Thermal Expansion Data -75/25 Coke Billet

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Figure 22. Relationship Between Flexural Strength and Electrical Conductivity - Composite Material - With Grain Direction.

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Figure 23. Relationship Between Flexural Strength and Electrical Conductivity - Composite Material - Across Grain Direction.

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Figure 24. Specimen Sampling Plan for Property Variation within the Billet.

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Figure 25. Property Variation Within Billet 238-A 35-35-30 Flour Composite.

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Figure 26. Property Variation Within Billet 242-A
35-35-30 Needle Coke Composite.

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Figure 27. Property Variation Within Billet 249-A
75-25 Needle Coke Composite.

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Figure 28. Property Variation within Post Pressed Billet 250 - A 75-25 Needle Coke Composite.

Figure 29. Property Variation within Post Pressed Billet 251-A 75-25 Needle Coke Composite Fabricated by the Old Die Compaction Process.

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Figure 30. Property Variation Within Billet 252-A
 75-25 Needle Coke Composite.

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Figure 31. Longitudinal Density Profiles

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Figure 32. Diametral Density Profiles (CRD)

Figure 33. Relationship Between AG Flexural Strength and WG Coefficient of Expansion of 75/25 Composite made with -100 + 325 Mesh Needle Coke.

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XII. APPENDIX A

WANL-LASL COMPARATIVE CREEP TESTING (U)

(CRD) It has been shown in this laboratory ⁴⁾ that the compressive creep deformation rate is a sensitive function of both test temperature and stress. For example, at 2500° C and 4000 psi the creep rate of a 75-25 flour composite material can be increased by approximately 25% by increasing the temperature by 40° C or by increasing the stress by 10%. In order to demonstrate accurate environmental control during creep testing at WANL and at LASL, an exchange program was agreed upon at a composite coordination meeting attended by LASL, SNPO-C, and WANL personnel in March, 1968. LASL was to supply specimens of 40 w/o NbC 40 w/o TaC 20 w/o graphite flour composite and WANL was to supply specimens of 75 w/o NbC 25 w/o graphite flour composite. The 40-40-20 material was creep tested in both laboratories during the second calendar quarter of 1968 and the 75-25 material was tested in both laboratories during the third calendar quarter of 1968.

(U) The creep specimens, which were 0.5 inch in diameter by 1 inch long, were held at 2500° C under a compressive stress of 4000 psi. The creep deformation represents the difference between specimen cold length measurement made before and after the test. Half of the tests in each laboratory were made with a 1-hour holding time and the other half were made with a 2-hour holding time. Each laboratory calibrated the optical temperature control system using the eutectic melting point, 2486° C, of the rhenium-carbon system. One difference in technique was discovered. LASL removed the load at the end of the holding period whereas WANL maintained the load during the cooling period. This maintenance of the load during cooling prevents the possibility of strain due to relaxation during cooling. However, a slight amount of extra creep may be incurred. No modification of the WANL creep apparatus or technique was made during this period.

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(U) Four creep specimens were machined by LASL from each of eight billets of 40-40-20 composite material. The billets were fabricated by LASL using -150 +325 mesh graphite flour. Two specimens, one from the top and one from the bottom, from each billet were submitted to WANL for creep measurement using a testing plan prescribed by Mr. R. E. Riley of LASL. The raw data obtained by WANL is listed in Table 1A. The density of each specimen was determined by weighing and measuring.

(U) Radiographs of the 16 specimens of 40-40-20 revealed that half of the billets contained a carbide agglomerate type of "spotty" heterogeneity. The other 4 billets were almost perfectly homogeneous as had been earlier billets of 40-40-20 composite submitted by LASL.

(U) The continuous monitoring of ram movement during the tests at WANL allowed the 1 hour cold type measurement to be estimated from the 2-hour runs. This estimated data is combined with the 8 actual 1 hour data points to obtain the averages for all 16 specimens and the data is summarized in Table 2A. The homogeneous billets deformed less (2.4% average in 1 hour) than did the inhomogeneous billets (4.0% average in 1 hour). The homogeneous billets were on the average, nearly 1% more dense than the inhomogeneous billets. Further, the average density was 1% higher for the top half of the billets than on the bottom half.

(U) The data received from LASL on their 40-40-20 composite material is given in Table 3A. Inspection of the table shows that the inhomogeneous billets, No.'s 1796, 1797, 1798, and 1799, crept more than the homogeneous billets. This is in agreement with the findings at WANL. Comparison of Tables 1A and 3A reveals that the 2 laboratories did not agree well with WANL obtaining nearly twice the creep rate as that obtained by LASL.

(CRD) All of the 75-25 creep specimens were machined by WANL from WANL billet 176. This billet, which was 3.5 inches in diameter by 3.25 inches long, was made from a mixture

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of Kawecki lot 293-1 NbC powder and -150 +325 mesh graphite flour and was pressed at 3080° C. The billet density was 4.62 grams per cubic centimeter or 95.5% of theoretical. Billet homogeneity was fair with some very light banding and a few rather vague carbide agglomerates near the bottom of the billet as observed in the transverse radiograph.

(CRD) Twelve specimens of 75-25 flour composite were creep tested by each laboratory, 6 for 1 hour and 6 for 2 hours, under a compressive stress of 4000 psi at 2500° C. The raw data for each laboratory are given in Tables 4A and 5A respectively. The average creep figures from the six 1-hour runs were 2.75% and 3.00% for LASL and WANL respectively. The average figures for the six 2-hour runs were 4.13 and 4.30% for LASL and WANL respectively. The LASL data is more consistent showing a 3 sigma deviation of 0.5% compared with 1.0% by WANL, both for the 1-hour data. Analysis of the data with respect to specimen location within the billet revealed no consistent trend except that the material near the central axis creeps, on the average, somewhat less, approximately 0.2%, than the over-all average. The density appears to be somewhat lower near the central axis of the billet.

(CRD) All of the creep data is averaged and summarized in Table 6A for comparative purposes. Correlation between the 2 laboratories can be seen to be poor with the 40-40-20 material but excellent for the 75-25 material. Testing on the latter material was the more recent.

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TABLE 1A
WANL CREEP DEFORMATION DATA ON LASL
40-40-20 COMPOSITE MATERIAL (U)
(2500° C 4000 psi)

1 HOUR DATA

<u>BILLET NO.</u>	<u>RADIOGRAPHY</u>	<u>BOTTOM LEFT¹⁾</u>		<u>TOP RIGHT¹⁾</u>	
		<u>% CREEP</u>	<u>% THEOR. DENSITY²⁾</u>	<u>% CREEP</u>	<u>% THEOR. DENSITY</u>
1792	Clean	3)	93.1	2.33	93.9
1793	Clean	2.34	93.2	2.29	94.1
1796	Spotty	4.40	92.4	3.75	94.0
1797	Spotty	<u>3.71</u>	<u>91.8</u>	<u>3.02</u>	<u>93.5</u>
Average		3.15	92.6	2.85	93.9

2 HOUR DATA

1794	Clean	4.18	93.1	3.54	93.4
1795	Clean	4.02	93.1	3.50	93.4
1798	Spotty	6.85	92.1	6.24	93.4
1799	Spotty	<u>6.72</u>	<u>91.9</u>	<u>6.52</u>	<u>92.4</u>
Average		5.44	92.6	4.95	93.2

Estimated 1 hour average
in 2 hour runs (by ram
movement)

3.47

3.21

- 1) Location within the billet
2) Based on a theoretical density of 5.98 g/cc
3) Poor temperature control - data invalid

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TABLE 2A

SUMMARY OF WANL CREEP DATA ON LASL
40-40-20 COMPOSITE MATERIAL (U)

(2500° C 4000 psi 1 hour)

	% CREEP		% THEOR. DENSITY	
	<u>AVG.</u>	<u>RANGE</u>	<u>AVG.</u>	<u>RANGE</u>
Clean	2.37	2.27 - 2.59	93.4	93.1 - 94.1
Spotty	3.99	3.02 - 4.44	92.7	91.8 - 94.0
Top of Billet	3.03	2.27 - 4.12	93.6	92.4 - 94.1
Bottom of Billet	3.31	2.34 - 4.44	92.6	91.8 - 93.2
Over-all Average	3.17		93.1	

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TABLE 3A

LASL CREEP DATA ON LASL 40-40-20 COMPOSITE MATERIAL (U)

(2500° C 4000 psi)

1 HOUR DATA

<u>BILLET NO.</u>	<u>BOTTOM RIGHT¹⁾</u>	<u>TOP LEFT¹⁾</u>
1792	1.7%	1.3%
1793	1.5	1.4
1796	3.3	2.9
1797	<u>2.1</u>	<u>1.9</u>
Average	1.65	1.88

2 HOUR DATA

1794	2.1	1.7
1795	2.2	1.4
1798	3.7	3.4
1799	<u>3.2</u>	<u>2.6</u>
Average	2.80	2.28

1) Location within the billet

TABLE 4A

WANL CREEP DATA ON WANL 75-25 FLOUR COMPOSITE BILLET 176 (CRD)
(2500° C 4000 psi)

<u>SPECIMEN NO.</u>	<u>BILLET LOCATION</u>	<u>1 HOUR CREEP</u>	<u>2 HOUR CREEP</u>	<u>% THEOR. SPECIMEN DENSITY</u>
1T	Top - peripheral	3.38%		96.2
2T	Top - mid radius	3.39		96.4
7T	Top - peripheral	2.50		96.5
9T	Top - center	2.59		94.8
10T	Top - peripheral	3.31		95.1
9M	Middle - center (centroid)	2.35*	3.37%	95.1
10M	Middle - peripheral	3.15*	5.01	95.1
3B	Bottom - center	2.72*	4.13	95.6
4B	Bottom - mid radius	2.79*	4.14	96.0
5B	Bottom - peripheral	3.22*	5.14	96.0
9B	Bottom - center	2.56*	3.98	95.7
10B	Bottom - peripheral	<u>2.83</u>	<u> </u>	<u>95.7</u>
	Average	2.90	4.30	95.7
	Range	2.50-3.39	3.37-5.01	94.8-96.5
	Standard Deviation	0.35	0.60	0.5
	Average excluding the estimated data	3.00		

* - 1 hour data point estimated by monitoring ram movement during the run.

TABLE 5A

LASL CREEP DATA ON WANL 75-25 COMPOSITE BILLET 176 (CRD)

(2500° C 4000 psi)

<u>SPECIMEN NO.</u>	<u>BILLET LOCATION</u>	<u>1 HOUR CREEP</u>	<u>2 HOUR CREEP</u>
3T	Top - center	2.57	
4T	Top - mid radius		3.60
8T	Top - peripheral	2.90	
1M	Middle - peripheral	2.65	
2M	Middle - mid radius		4.15
3M	Middle - center (Centroid)		4.20
4M	Middle - mid radius	2.52	
7M	Middle - peripheral		3.85
1B	Bottom - peripheral		4.15
2B	Bottom - mid radius	2.85	
7B	Bottom - peripheral	3.00	
8B	Bottom - peripheral		<u>4.90</u>
	Average	2.75	4.13
	Range	2.52-3.00	3.60-4.90
	Standard Deviation	0.17	0.25

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TABLE 6A

COMPARISON OF LASL-WANL CREEP DATA (CRD)

40-40-20 Composite

	<u>1 Hour</u>	<u>2 Hours</u>
LASL	1.76	2.54
WANL	3.00	5.20
Ratio WANL/LASL	1.70	2.05

75-25 Composite

LASL	2.75	4.13
WANL	3.00	4.30
Ratio WANL/LASL	1.09	1.04

XIII. APPENDIX B

EVALUATION OF PRODUCTION BILLETS (C-RD)

(CRD) During the course of the experimental program described in the main body of this report, a series of production billets of 75/25 needle coke composite were fabricated by an outside vendor for Pewee 2 hot end support hardware. They were fabricated in accordance with WANL specification PDS 30106-3 Revision E, which requires a post pressing operation. The die compaction was made at 3100° C under a stress of 2800 psi for 15 minutes. The pressure-temperature-time cycle during heat up was not specified except that the time to reach temperature was not to exceed 4 hours. The heat up rate was necessarily slow, 2 to 3 hours, because the vendor heated the die by radiation. The vendor was not advised to apply the pressure at any particular temperature. The post pressing operation consisted of holding the billet, after removal of the graphite die, under an axial compressive load of 2000 psi for 1 hour at 2700° C. NbC powder of sufficient purity so as to not degrade the properties was used. The calcined petroleum (needle) coke was screened and a 50:50 mixture of the 2 fractions -100 +140 and -140 +325 mesh was used. Dry blending in a V blender was used exclusively.

(U) As a quality control procedure, the billets were evaluated on the basis of billet density, composition, radiography, AG compressive creep deformation in 1 hour at 2500° C and 4000 psi for 1 hour, WG coefficient of thermal expansion to 2500° C, and room temperature flexural strength in both grain directions.

(U) Two creep specimens were taken from each billet, one at the top and one at the bottom on the same angular location in the billet and near the periphery. Two AG flexural strength specimens were taken near the periphery across the diameter from the creep specimens. They extended the full length of the billet and were broken in 3 places so

as to reflect strength at the top, middle, and bottom of the billet. Triplicate WG flexural specimens and a single WG thermal expansion specimen were taken from a slab cut from the bottom of the billet.

(U) Billet density, creep, and flexural strength data for the first 48 billets submitted by the vendor are listed in Table 1B. These billets were made during 5 different periods of time and, on this basis, are divided into 5 lots. Electrical conductivity measurements were made on the first 29 billets, by the WANL Materials Department, on the flexural strength specimens.

(U) The quality control thermal expansion data is shown in Table 2B. The first 5 billets, the fifteenth, the twenty-fifth, the twenty-ninth, and 6 of the last 20 billets were checked for secant expansion coefficient to 2500° C.

(U) Examination of Table 1B reveals a wide scatter in properties in the first 3 lots with a gradual deterioration of flexural strength and electrical conductivity in lot 1 through 3. A total of 7 billets were rejected in the first 3 lots due to low flexural strength. Only 3 of the billets had average creep rates above 6%. Flexural strength was, then, clearly the major cause for rejection. Lots 4 and 5 were definitely superior in that the creep rate was lower and the flexural strengths were higher than the previous lots with less scatter in the data.

(CRD) Table 1B further reveals no consistent difference in creep rate between the top and the bottom of the billet. However, gradients in AG flexural strength, and AG electrical conductivity along the length of the billet are apparent. Maxima occur in the centers of lot 3 billets and a decrease in AG flexural strength from top to bottom is apparent in lot 5.

(U) Radiographic examination of the billets revealed that the majority of them were banded, in varying degree. An arbitrary classification of the billets in accordance with their degree of banding is given in Table 3B. Although this classification is only semi-quantitative, it was thought that it might reveal a major trend. The seven reject billets in the first 3 lots were found to be more heavily banded than the average for these lots. However, lot 4 contains no reject billets although it is the most heavily banded lot. Further, no reject billets were found in lot 5 although banding was heavy. In the final analysis, it must be concluded that no consistent correlation was found between banding and billet rejection. No correlation was found between banding and creep rate.

(CRD) The thermal expansion data, in Table 2B, on lots 1 and 2 represent one thermal cycle only. When more than 1 cycle was made, the data for the last cycle was arbitrarily accepted as correct. On this basis, the average for 6 billets in lots 4 and 5 is $7.2 \times 10^{-6}/^{\circ}\text{C}$. Permanent deformation, after thermal cycling, was not great, being generally less than 0.05%. The low expansion coefficient, $6.3 \times 10^{-6}/^{\circ}\text{C}$, reported in lot 2 was taken from a billet which had been rejected because of low flexural strength. This supports the findings of the WANL experimental program in which low flexural strengths and low CTE's were obtained on certain billets which had been made in accordance with the old in-house processing.

(U) The billet densities and the specimen densities are listed in Table 4B. Only one of the 7 rejected billets, No. 7876, had a low density. The majority of the WG flexural strength specimens that gave cause for billet rejection were found to have low densities. For example, specimen 1 from billet 7884 had a strength of 3900 psi and a specimen density of 93.6% of theoretical, as observed in Tables 1B and 4B. This strongly suggests an inhomogeneity which was apparent, primarily as banding, in the billet radiographs.

(CRD) A density gradient, decreasing from top to bottom of the billet, is evident from the densities of the creep specimens. The value of measuring the densities of all specimens is questionable. However, measurement of the density of the quality control slab cut from the end of the billet has merit since it reflects possible property gradients which in turn reflects process control.

(U) The electrical conductivity measurements were made on the flexural strength specimens in the specimen length direction over a 1/2 inch span. A profile was run on the AG specimens by measuring the electrical conductivity in 3/8 inch increments. The results are listed in Table 5B.

(U) The electrical conductivity figures listed in Table 1B represent the total specimen volume under the 1/2 inch maximum tensile stress span in the flexural strength test. The expected correlation between the flexural strength and the electrical conductivity is illustrated in the plots shown in Figures 1B and 2B. Each point corresponds to an individual flexural break. Examination of the data in Table 1B reveals a wider scatter, within the billet, in the flexural strength data than in the electrical conductivity data. This is to be expected since the former is a function of only one side, the tension side, of the 1/2 inch long span prism whereas the latter is a function of the entire volume within this same prism. It may then be inferred that the scatter in Figures 1B and 2B are primarily caused by weak areas within the 1/2 inch maximum tension span and near the tension surface.

(U) It is interesting to note that the lines drawn through the flexural strength - electrical conductivity data points in Figures 1B and 2B superimpose fairly closely in the 2 grain directions. This suggests that the flexural strength is controlled primarily by the carbide phase in accordance with the following reasoning. The electrical conductivity is known to be controlled by the carbide matrix since it is 10 times more conductive than the graphite. The anisotropy in flexural strength and electrical conductivity would then, seem to be controlled by the shape of the interlacing carbide matrix which is in turn controlled by the shape of the carbon particles. Furthermore, cracks in the carbide matrix would be expected to lower both the electrical conductivity and the flexural strength rather drastically.

(U) A limited study was completed on 2 of the billets to determine the effects of location within the billet on properties. Specifically, the effects of longitudinal and diametral location on creep rate, expansion coefficient, and room temperature flexural strength was determined on the first billet, No. 7870, and the fifth billet, No. 7874. Specimens were taken from each billet at those locations specified in Figure 3B. The coefficient of expansion and the flexural strength were determined in both grain directions whereas the creep deformation rate was determined in the across grain direction only. The resulting data is shown in Tables 6B through 9B and in Figures 4B and 5B.

(U) Examination of the creep deformation data reveals that billet 7874 is homogeneous with respect to this parameter whereas billet 7870 is heterogeneous. No conclusions on the effect of specific location within billet 7870 can be made due to inconsistencies in the data.

(CRD) The thermal expansion data reveals that both billets have a greater WG coefficient of thermal expansion in the middle than at the ends. A logical explanation for this is that the coke particles at the ends are better oriented since the powder at the ends moves a further distance during the initial pressing operation. The higher values of the expansion coefficient in the AG direction indicates a fair degree of anisotropy. The AG coefficient began to rise rapidly at approximately 2000° C. The data represents a maximum temperature of only 2300° C. A 2500° C AG coefficient would be expected to be considerably higher. However, the WG coefficient would be affected only very little by a similar temperature increase, as indicated by the data in Table 9 in the main body of this report.

(U) Examination of the WG flexural strength data again indicates that billet 7870 is heterogeneous whereas billet 7874 is reasonably homogeneous. No consistent effect of billet location could be found except that the strength at the top of billet 7870 is lower than the average. However, even this observation is inconclusive due to lack of consistent data.

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(U) The AG flexural strength data shows that billet 7874 is more homogeneous than billet 7870. No statistically significant effect of longitudinal location is evident. There is possibly a weak area in the left hand portion of billet 7870 Section G.

(U) Billet 7874 shows an over-all superiority with 20% and 10% greater flexural strength in the AG and WG directions, respectively, 40% smaller creep rate, and 3% higher CTE than billet 7870.

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TABLE 1B

RAW PROPERTY DATA ON 75-25 NEEDLE COKE PRODUCTION BILLETS (CRD)

Billet No.	Density % Theor.	% Creep, 2500° C 4 KSI, 1 hour		WG Flex. Strength KSI			AG Flex. Strength KSI			WG El. Conductivity ohm ⁻¹ cm ⁻¹ x 10 ⁻²			AG El. Conductivity ohm ⁻¹ cm ⁻¹ x 10 ⁻²		
		Top	Bottom	1	2	3	Top	Mid.	Bottom	1	2	3	Top	Mid.	Bottom
LOT 1															
7870	95.8	3.5	5.1	14.7	14.9	14.2	5.6	6.2	4.4	----	----	----	64	75	70
Avg.		4.3			14.6		5.6	6.4	7.0				66	73	79
								5.9						71	
7871	95.8	4.2	3.8	14.1	16.5	15.5	6.6	6.8	6.7	127	126	123	76	78	84
Avg.		4.0			15.4		7.2	7.1	7.5				79	80	82
								7.0			125			80	
7872	95.5	3.8	4.6	13.9	14.7	14.4	8.2	7.2	8.7	131	120	127	84	85	92
Avg.		4.2			14.3		7.7	7.0	7.8				84	86	84
								7.8			126			86	
7873	95.7	2.3	3.4	14.2	14.1	9.9	6.7	6.6	7.0	123	121	124	71	79	76
Avg.		2.9			12.7		7.6	6.8	6.5				79	76	78
								6.9			123			77	
7874	95.2	3.6	3.4	14.2	15.0	11.7	8.2	6.1	7.7	124	123	120	86	79	92
Avg.		3.5			13.6		7.5	6.5	8.3				91	77	86
								7.4			122			85	
7875	96.2	3.2	2.8	13.1	15.1	14.4	7.0	5.8	5.4	131	128	116	75	70	65
Avg.		3.0			14.2		6.2	5.5	5.3				73	67	63
								5.9			125			69	

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Table 1B
Page 2

Billet No.	Density % Theor.	% Creep, 2500° C 4 KSI, 1 hour		WG Flex. Strength KSI			AG Flex. Strength KSI			WG El. Conductivity ohm ⁻¹ cm ⁻¹ x 10 ⁻²			AG El. Conductivity ohm ⁻¹ cm ⁻¹ x 10 ⁻²				
		Top	Bottom	1	2	3	Top	Mid.	Bottom	1	2	3	Top	Mid.	Bottom		
7876R	94.5	5.5	10.8	10.4	13.3	11.1	5.0	4.4	2.4	98	128	116	66	63	46		
Avg.		8.2			11.6		2.7	4.4	5.6				50	63	68		
								4.1			114			59			
7877	95.8	3.8	3.4	14.2	12.9	13.0	5.7	6.8	6.0	131	123	123	69	75	69		
Avg.		3.6			13.4		4.7	6.2	6.4				69	71	68		
								6.0			126			70			
LOT 1 Location Averages																	
		3.7	4.7				6.6	6.4	6.5				74	75	75		
LOT 1 Average																	
	95.6	4.2			13.7			6.5			123			75			
COEFFICIENT OF VARIATION (AG Flex. and Conductivity) (Billet Averages)								17%								11%	
<u>LOT 2</u>																	
7878	96.2	7.4	2.4	15.5	15.4	15.3	6.2	6.5	4.9	136	131	131	67	74	67		
Avg.		4.9			15.4		6.2	6.6	5.6		133		68	71	68		
								6.0						69			
7879	95.8	3.0	3.4	14.1	12.4	13.2	5.1	5.3	6.0	123	123	123	76	74	77		
							5.6	5.8	5.2		123		76	81	75		
		3.2			13.1			5.5						77			

R = Reject Billet

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Table 1B
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Billet No.	Density % Theor.	% Creep, 2500° C 4 KSI, 1 hour		WG Flex. Strength KSI			AG Flex. Strength KSI			WG El. Conductivity ohm ⁻¹ cm ⁻¹ x 10 ⁻²			AG El. Conductivity ohm ⁻¹ cm ⁻¹ x 10 ⁻²		
		Top	Bottom	1	2	3	Top	Mid.	Bottom	1	2	3	Top	Mid.	Bottom
7880	95.9	6.9	7.4	13.9	14.2	13.7	6.2	5.6	5.1	128	126	124	73	80	68
Avg.		7.2			13.9		5.9	5.9	7.5		126		66	77	69
					6.0									72	
7881	95.9	8.9	3.7	14.2	14.0	12.4	8.0	7.7	5.7	124	122	119	79	89	67
Avg.		6.3			13.5		8.0	8.0	5.7				79	87	67
								7.2			121			78	
7882	95.9	5.5	4.3	12.1	13.3	14.0	5.1	5.7	5.4	123	123	129	69	71	66
Avg.		4.9			13.1		5.3	5.9	6.0				70	71	64
								5.6			125			69	
7883	95.7	3.8	3.8	9.2	10.0	13.2	5.5	5.9	5.6	97	110	114	63	67	66
Avg.		3.8			10.8		5.6	6.1	5.7				70	68	64
								5.7			107			66	
7884R	95.7	3.5	3.8	3.9	5.6	11.7	5.6	6.0	4.3	57	74	113	66	72	65
Avg.		3.7			7.0		5.0	5.5	5.2				67	67	63
								5.3			81			67	
7885	95.9	3.4	3.6	11.1	14.2	14.3	5.4	6.1	5.5	123	129	120	73	68	66
Avg.		3.5			13.2		5.6	5.5	4.9		124		68	66	64
								5.5						68	
7886	96.7	2.8	3.2	12.1	15.1	13.1	7.0	5.9	6.5	126	124	125	76	72	80
Avg.		3.0			13.4		6.9	6.2	6.2		125		76	69	77
								6.5						75	
7887	96.7	3.0	2.8	12.6	11.8	13.6	5.1	3.5	6.0	123	123	123	68	66	72
Avg.		2.9			12.7		4.6	5.5	6.0				67	68	76
								5.1			123			70	

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Table 1B
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Billet No.	Density % Theor.	% Creep, 2500° C 4 KSI, 1 hour		WG Flex. Strength KSI			AG Flex. Strength KSI			WG El. Conductivity ohm ⁻¹ cm ⁻¹ x 10 ⁻²			AG El. Conductivity ohm ⁻¹ cm ⁻¹ x 10 ⁻²			
		Top	Bottom	1	2	3	Top	Mid.	Bottom	1	2	3	Top	Mid.	Bottom	
7888R	95.9	4.3	3.8	4.6	4.9	11.3	4.1	6.1	5.4	64	89	111	73	76	72	
							6.0	6.2	5.1		89		70	77	72	
Avg.		4.1			6.9			5.5						73		
7889	96.5	3.1	2.3	11.3	11.8	11.6	6.1	5.5	5.0	124	123	120	67	72	68	
							6.1	5.1	6.2				67	69	72	
Avg.		2.7			11.6			5.7			122			70		
7890R	96.2	3.0	3.5	3.0	10.9	6.5	5.7	6.5	3.5	37	102	76	65	72	70	
							3.9	6.6	4.6				70	74	66	
Avg.		3.3			6.8			5.1			72			71		
LOT 2 Location Averages																
		4.5	3.7				5.8	6.0	5.5				70	73	69	
LOT 2 Average																
	96.1	4.1			11.6			5.8			113			71		
COEFFICIENT OF VARIATION (AG Flex. and Conductivity - billet averages)								10%			5%					
<u>LOT 3</u>																
7915	96.5	4.0	3.2	11.6	13.5	12.8	6.2	5.5	5.8	115	113	114	67	64	60	
							5.8	6.1	4.7				67	64	59	
Avg.		3.6			12.6			5.7			114			64		

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Table 1B
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Billet No.	Density % Theor.	% Creep, 2500° C 4 KSI, 1 hour		WG Flex. Strength KSI			AG Flex. Strength KSI			WG El. Conductivity ohm ⁻¹ cm ⁻¹ x 10 ⁻²			AG El. Conductivity ohm ⁻¹ cm ⁻¹ x 10 ⁻²		
		Top	Bottom	1	2	3	Top	Mid.	Bottom	1	2	3	Top	Mid.	Bottom
7916	96.5	2.9	3.1	12.7	13.8	13.8	6.5	5.1	4.9	118	116	121	70	62	58
							4.4	5.9	7.2				62	66	68
Avg.		3.0			13.4			5.7			121			64	
7917	96.5	3.6	2.8	13.2	14.4	12.9	6.4	6.0	6.8	117	125	115	73	78	77
							6.2	6.9	6.9				68	76	77
Avg.		3.2			13.4			6.5			119			75	
7918	96.7	2.8	3.3	12.0	13.0	12.5	6.4	6.7	7.1	115	117	113	76	74	70
							6.4	5.9	6.4				79	66	70
Avg.		3.1			12.5			6.5			115			73	
7919R	96.8	4.2	3.5	2.2	3.0	10.6	3.0	4.2	2.3	16	41	100	41	60	37
							2.3	3.9	1.9				43	56	47
Avg.		3.9			5.3			2.9			52			46	
7920R	95.5	4.2	3.5	3.1	8.3	4.0	2.0	4.8	2.0	38	82	55	46	65	30
							1.9	3.9	1.9				43	64	29
Avg.		3.9			5.1			2.8			58			46	
7921	96.8	3.2	3.9	10.9	13.3	14.0	5.8	5.6	5.3	107	116	118	68	68	65
							5.5	5.3	6.3				65	72	70
Avg.		3.6			12.7			5.6			114			68	
7922R	96.5	3.6	3.4	2.8	9.3	3.2	2.6	5.8	2.8	26	95	51	51	67	44
							2.4	6.0	2.6				56	66	43
Avg.		3.5			5.1			3.7			57			55	

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Billet No.	Density % Theor.	% Creep, 2500° C 4 KSI, 1 hour		WG Flex. Strength KSI			AG Flex. Strength KSI			WG El. Conductivity ohm ⁻¹ cm ⁻¹ x 10 ⁻²			AG El. Conductivity ohm ⁻¹ cm ⁻¹ x 10 ⁻²		
		Top	Bottom	1	2	3	Top	Mid.	Bottom	1	2	3	Top	Mid.	Bottom
LOT 3 Location Averages		3.6	3.3				4.6	5.5	4.7				61	67	56
LOT 3 Average		96.6	3.5		11.1			4.9			94			61	
COEFFICIENT OF VARIATION (AG Flex. and Conductivity - Billet Averages)								29%					17%		
<u>LOT 4</u>															
11401	96.3	2.2	2.0	13.6	12.9	15.7	6.7 8.2	7.7 7.0	7.5 7.2	----	----	----	--	--	--
Avg.		2.1			14.1		7.4								
11402	96.0	2.6	2.5	12.5	14.0	13.4	8.0 7.0	7.7 6.1	6.7 6.4	No electrical conductivity data from here on					
Avg.		2.6			13.3		7.0								
11403	96.3	2.3	2.7	14.9	12.3	12.0	6.8 7.0	6.4 6.1	6.0 6.4						
Avg.		2.5			13.1		6.5								
11404	96.0	2.4	3.0	12.3	12.1	15.4	7.5 8.0	7.1 6.3	7.1 6.7						
Avg.		2.7			13.3		7.1								
11405	96.3	2.7	3.0	14.7	14.6	12.9	7.4 7.3	6.7 7.0	7.1 7.2						
Avg.		2.8			14.1		7.1								
LOT 4 Location Averages		2.4	2.6				7.4	6.7	6.8						

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Table 1B
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Billet No.	Density % Theor.	% Creep, 2500° C 4 KSI, 1 hour		WG Flex. Strength KSI			AG Flex. Strength KSI			WG El. Conductivity ohm ⁻¹ cm ⁻¹ x 10 ⁻²			AG El. Conductivity ohm ⁻¹ cm ⁻¹ x 10 ⁻²		
		Top	Bottom	1	2	3	Top	Mid.	Bottom	1	2	3	Top	Mid.	Bottom
LOT 4 Average															
	96.2	2.5		13.6			7.0								
COEFFICIENT OF VARIATION (AG Flex - Billet Averages)									4%						
<u>LOT 5</u>															
11073	97.3	2.7	2.6	13.6	13.0	11.3	7.3	6.6	4.9						
							7.4	6.3	6.0						
Avg.		2.7		12.7			6.4								
11074	96.7	2.7	3.1	13.0	12.5	12.7	7.0	5.6	5.5						
							6.3	5.1	5.6						
Avg.		2.9		12.8			5.9								
11075	97.1	3.5	4.4	13.7	14.0	13.4	5.2	6.4	5.8						
							6.6	6.2	5.8						
Avg.		4.0		13.7			6.0								
11076	97.3	4.0	2.6	14.0	13.3	13.8	6.9	6.7	6.5						
							6.9	6.7	6.3						
Avg.		3.3		13.7			6.7								
11077	96.7	3.6	3.4	12.6	12.6	13.2	7.0	4.8	5.7						
							6.5	5.6	5.9						
Avg.		3.5		12.8			5.9								
11078	96.3	4.4	3.9	13.1	12.6	12.4	7.0	6.5	5.9						
							6.9	6.3	5.5						
Avg.		4.2		12.7			6.4								

Table 1B
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Billet No.	Density % Theor.	% Creep, 2500° C 4 KSI, 1 hour		WG Flex. Strength KSI			AG Flex. Strength KSI			WG El. Conductivity ohm ⁻¹ cm ⁻¹ x 10 ⁻²			AG El. Conductivity ohm ⁻¹ cm ⁻¹ x 10 ⁻²		
		Top	Bottom	1	2	3	Top	Mid.	Bottom	1	2	3	Top	Mid.	Bottom
11079	96.0	4.4	2.8	13.1	12.2	12.3	6.7	5.6	6.4						
Avg.		3.6			12.5		7.1	6.1	6.0						
11080	96.3		4.2	13.0	12.8	12.4	7.2	5.9	6.1						
Avg.					12.7		7.0	6.0	6.0						
11081	96.0	3.8	2.5	12.6	11.9	11.2	7.2	6.2	6.5						
Avg.		3.2			11.9		6.6	6.1	6.1						
11082	95.8	2.7	4.9	12.8	13.1	11.6	6.9	5.5	5.9						
Avg.		3.8			12.5		6.8	5.9	6.1						
11083	96.3	3.7	2.7	11.8	13.0	14.1	7.7	6.3	6.8						
Avg.		3.2			13.0		7.5	6.3	6.4						
11084	96.3	2.9	5.2	14.0	13.4	12.8	8.2	7.8	7.2						
Avg.		4.2			13.4		8.1	8.5	7.1						
11085	96.5		2.6	14.3	14.4	15.2	8.1	7.0	6.4						
Avg.					14.6		8.3	7.4	6.1						
11086	96.5	4.2	4.6	14.6	13.5	15.2	7.2	8.2	7.4						
Avg.		4.4			14.4		6.9	7.2	7.4						

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Table 1B
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Billet No.	Density % Theor.	% Creep, 2500° C 4 KSI, 1 hour		WG Flex. Strength KSI			AG Flex. Strength KSI			WG El. Conductivity ohm ⁻¹ cm ⁻¹ x 10 ⁻²			AG El. Conductivity ohm ⁻¹ cm ⁻¹ x 10 ⁻²		
		Top	Bottom	1	2	3	Top	Mid.	Bottom	1	2	3	Top	Mid.	Bottom
LOT 5 Location Avg.															
		3.6	3.4				7.1	6.5	6.1						
LOT 5 Avg.															
	96.4	3.5			13.1			6.6							
COEFFICIENT OF VARIATION (AG Flex. - Billet Averages)								8%							
OVERALL LOCATION AVERAGES															
		3.7	3.6				6.3	6.1	5.9				69	72	67
OVERALL AVERAGES															
	96.2	3.7			12.5			6.1			110		69		
OVERALL RANGES OF BILLET AVERAGES															
	94.5-96.8	2.1-8.2			5.1-15.4			2.9-7.8			52-113		46-86		

R - Reject Billet

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TABLE 2B

SECANT THERMAL EXPANSION COEFFICIENTS ON 75-25

NEEDLE COKE PRODUCTION BILLETS - WITH GRAIN (CRD)

$\bar{\alpha}$ = Microinches/inch/° C

Billet No.	Cycle ¹⁾	2000° C	2300° C	2500° C	Max. Temp. ° C	Permanent Deformation, %
7870	1	6.9	7.0	7.0	2450	-.12
7871	1	7.2	7.2	7.2	2450	-.09
7872	1	7.2	7.2	7.0	2450	-.09
7873	1	7.2	7.3	7.4	2450	-.10
7874	1	<u>6.9</u>	<u>6.9</u>	<u>6.8</u>	2450	<u>-.12</u>
Avg. Lot 1		7.1	7.1	7.1		-.10
LOT 2						
7884R	1	6.4	6.3	6.3	2470	-.15
LOT 3						
7918	1	7.7	7.6	7.5	2500	.01
	2	7.8	7.8	7.6	2520	-.04
7922	1	5.7	5.8	5.7	2500	-.28
	2	7.2	7.2	7.0	2500	-.09
Avg. Lot 3		7.5	7.5	7.3		
LOT 4						
11401	1	7.4	7.3	7.0	2450	-.06
	2	7.5	7.4	7.3	2420	-.01
	3	7.6	7.6	7.3	2500	.01
11403	1	7.3	7.3	7.2	2450	.00
	2	7.3	7.3	7.2	2420	-.09
	3	7.3	7.3	7.2	2500	-.02
11404	1	7.3	7.3	7.2	2450	-.03
	2	7.3	7.4	7.2	2420	-.01
	3	7.3	7.4	7.1	2500	-.03
Avg. Lot 4		7.4	7.4	7.2		
LOT 5						
11073	1	7.2	7.1	7.0	2480	-.04
	2	7.5	7.4	7.2	2330	.01
	3	7.4	7.5	7.2	2500	-.02

1) All billets heat treated 1 hour at 2500° C

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Table 2B
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<u>Billet No.</u>	<u>Cycle¹⁾</u>	<u>2000° C</u>	<u>2300° C</u>	<u>2500° C</u>	<u>Max. Temp. ° C</u>	<u>Permanent Deformation, %</u>
11076	1	7.3	7.2	7.1	2480	-.03
	2	7.5	7.4	7.2	2430	.00
	3	7.5	7.4	7.3	2500	.03
11086	1	7.4	7.4	7.1	2490	-.04
	2	7.6	7.5	7.3	2430	-.02
	3	7.6	7.6	7.4	2480	.00
Avg. Lot 5		7.5	7.5	7.3		

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TABLE 3B

COMPARATIVE RADIOGRAPHIC EXAMINATION OF 75-25 NEEDLE

COKE PRODUCTION BILLETS (CRD)

	<u>No Banding</u>	<u>Light Banding</u>	<u>Medium Banding</u>	<u>Heavy Banding</u>
LOT 1				
Billet 7870			X	
7871	X			
7872	X			
7873		X		
7874			X	
7875			X	
7876R			X	
7877		X		
	2	2	4	0
LOT 2				
Billet 7878		X		
7879			X	
7880		X		
7881			X	
7882			X	
7883			X	
7884R			X	
7885		X		
7886			X	
7887				X
7888R				X
7889		X		
7890R				X
TOTALS	0	4	6	3

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	<u>No Banding</u>	<u>Light Banding</u>	<u>Medium Banding</u>	<u>Heavy Banding</u>
LOT 3				
Billet 7915				X
7915			X	
7917			X	
7918	X			
7919R				X
7920R				X
7921		X		
7922R				X
TOTALS	$\bar{1}$	$\bar{1}$	$\bar{2}$	$\bar{4}$
LOT 4				
Billet 11401				X
11402			X	
11403				X
11404				X
11405				X
TOTALS	$\bar{0}$	$\bar{0}$	$\bar{1}$	$\bar{4}$
LOT 5				
Billet 11072				X
11073				X
11074			X	
11075				X
11076			X	
11077				X
11078			X	
11079			X	
11080		X		
11081	X			
11082			X	
11083				X
11084			X	
11085		X		
11086				X
TOTALS	$\bar{1}$	$\bar{2}$	$\bar{6}$	$\bar{6}$
GRAND TOTALS	4	9	19	17

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R = Reject Billets

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TABLE 4B

DENSITY OF PROPERTY SPECIMENS FROM 75-25 NEEDLE COKE

PRODUCTION BILLETS (% THEORETICAL) (CRD)

Billet No.	Creep		Flexural Strength							
	Top	Bot.	With Grain				Across Grain			
LOT 1			Avg.	1	2	3	Avg.	a	b	Avg.
7870	95.5	93.9	94.7					95.3	95.3	95.3
7871	96.0	94.5	95.4	96.3	96.1	95.9	96.1	94.5	94.7	94.6
7872	95.5	93.8	94.7	95.7	95.8	95.7	95.8	94.5	94.6	94.6
7873	95.8	94.5	95.2	95.1	95.1	95.8	95.5	94.5	94.6	94.6
7874	96.1	94.5	95.3	95.8	95.8	95.8	95.8	94.2	94.0	94.1
7875	96.1	96.1	96.1	96.1	95.7	95.5	95.8	95.1	94.2	94.7
7876	93.8	92.2	93.0	94.5	95.8	95.5	95.3	92.8	92.6	92.7
7877	95.7	95.7	95.7	96.1	95.7	94.6	95.5	95.2	95.2	95.2
Avg.	95.5	94.4	95.0	95.7	95.7	95.8	95.7	94.5	94.4	94.5
LOT 2										
7878	96.1	96.5	96.3	96.5	95.8	95.7	96.0	95.1	95.1	95.1
7879	96.1	96.1	96.1	94.5	94.5	95.2	94.7	93.8	94.5	94.2
7880	95.8	95.2	95.5	96.1	95.7	96.5	96.1	95.5	95.7	95.6
7881	95.5	95.5	95.5	95.1	95.2	95.2	95.2	95.1	95.2	95.2
7882	95.8	94.2	95.0	95.5	95.5	95.2	95.4	95.1	95.1	95.1
7883	95.7	94.6	95.2	95.1	96.1	95.2	95.5	94.6	94.6	94.6
7884	96.3	95.2	95.8	93.6	95.5	96.5	95.2	95.2	95.1	95.2
7885	96.1	94.6	95.4	95.8	94.8	95.8	95.5	95.2	95.2	95.2
7886	96.5	94.8	95.7	96.5	94.8	97.1	96.1	94.8	95.8	95.8
7887	96.1	95.5	95.8	94.2	95.7	95.1	95.0	95.7	95.7	95.7
7888	95.5	94.6	95.1	94.8	94.8	95.7	95.1	94.8	94.8	94.8
7889	96.7	96.5	96.6	96.1	96.3	96.7	96.4	95.2	95.1	95.2
7890	95.5	96.1	95.8	94.2	94.8	93.6	94.2	95.2	95.7	95.5
Avg.	96.0	95.3	95.7	95.2	95.3	95.6	95.4	95.1	95.2	95.1
LOT 3										
7915	96.3	94.6	95.5	96.5	95.7	95.1	95.8	95.8	96.1	96.0
7916	96.1	94.6	95.4	95.8	95.8	96.1	95.9	96.1	95.8	96.0

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Billet										
No.	Top	Bot.	Avg.	1	2	3	Avg.	a	b	Avg.
7917	95.8	96.3	96.1	96.3	96.1	95.7	96.0	94.6	94.6	94.6
7918	96.3	94.6	95.5	95.7	95.8	95.8	95.8	95.8	96.3	96.1
7919	92.6	93.4	93.0	94.2	94.2	96.7	95.0	95.1	95.1	95.1
7920	94.0	92.2	93.1	95.1	95.5	94.8	95.1	95.1	94.8	95.0
7921	96.1	94.8	95.5	95.7	95.5	95.8	95.7	95.1	94.8	95.0
7922	94.8	94.6	94.7	92.2	96.1	95.1	94.5	94.2	94.5	94.4
Avg.	95.2	94.4	94.8	95.3	95.6	95.6	95.5	95.2	95.3	95.3
Overall										
Avg.	95.7	94.8	95.2	95.4	95.4	95.7	95.5	95.0	95.0	95.0

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TABLE 5B

ELECTRICAL CONDUCTIVITY PROFILES ON AG FLEXURAL
STRENGTH SPECIMENS FROM 75-25 NEEDLE COKE PRODUCTION BILLETS (CRD)

Billet No.	1/2" span 3/8" increments $\text{ohm}^{-1} \text{cm}^{-1} \times 10^{-2}$ (Measurement Locations 1 through 10)										Billet Avg.
	1	2	3	4	5	6	7	8	9	10	
LB 7870											
1A	73	64	70	68	75	75	75	69	70	75	7140
1B	86	66	74	68	72	73	80	84	79	75	7570 7400
LB7871											
2A	76	76	77	72	76	79	75	84	84	86	7860
2B	79	79	73	72	78	82	79	82	82	81	7870 7900
LB7872											
3A	86	84	82	81	86	84	90	90	92	86	8610
3B	88	84	84	82	84	88	81	84	84	88	8470 8500
LB 7873											
4A	73	71	72	79	79	78	78	72	76	79	7570
4B	79	79	78	78	78	73	71	78	68	71	7530 7600
LB 7874											
5A	88	86	88	75	79	79	92	92	92	86	8570
5B	88	91	88	70	79	75	81	86	86	84	8280 8400
LB7875											
6A	71	75	72	72	71	67	79	79	64	67	7180
6B	72	73	75	72	68	66	66	64	63	64	6830 7000
LB7876											
7A	68	66	65	63	64	62	60	55	47	45	5940
7B	45	50	58	59	62	63	65	65	68	69	6040 6000
LB7877											
8A	67	69	68	72	76	73	69	69	69	68	7000
8B	67	69	69	69	69	73	73	68	68	67	6920 7000

LOT 1 Avg.

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Table 5B
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<u>Billet</u> <u>No.</u>	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>	<u>6</u>	<u>7</u>	<u>8</u>	<u>9</u>	<u>10</u>	<u>Avg.</u>	<u>Billet</u> <u>Avg.</u>
LB7878												
9A	71	67	72	73	76	71	69	67	67	68	7010	
9B	68	68	69	71	73	69	69	71	68	68	6940	6970
LB7879												
10A	71	76	75	75	76	82	79	79	77	73	7630	
10B	75	76	75	76	79	82	79	75	75	73	7650	7600
LB7880												
11A	68	73	76	79	82	77	72	66	68	67	7300	
11B	68	66	64	66	72	82	76	79	79	70	7200	7300
LB7881												
12A	74	79	82	82	88	90	76	68	67	69	7800	
12B	70	79	84	86	90	84	72	65	67	68	7700	7700
LB7882												
13A	73	69	70	76	72	70	68	65	66	67	6900	
13B	70	70	72	70	72	69	68	65	64	66	6900	6900
LB7883												
14A	57	63	65	64	66	68	67	68	66	46	6300	
14B	64	70	68	67	67	69	68	68	64	48	6500	6400
LB7884												
15A	54	66	69	70	72	72	68	64	65	53	6600	
15B	56	67	73	72	67	66	66	66	63	54	6500	6600
LB7885												
16A	73	73	71	69	68	68	66	66	66	69	6700	
16B	66	68	69	68	66	65	64	62	64	69	6600	6600
LB7886												
17A	72	76	77	77	74	70	73	77	80	76	7500	
17B	73	76	77	74	69	69	76	73	77	76	7400	7500

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Billet No.	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>	<u>6</u>	<u>7</u>	<u>8</u>	<u>9</u>	<u>10</u>	<u>Avg.</u>	Billet Avg.
LA7887												
18A	68	68	65	68	65	67	70	74	72	74	6900	
18B	73	67	67	69	67	69	72	76	76	70	7000	7000
LA7888												
19A	72	73	73	72	74	77	79	79	72	66	7400	
19B	72	70	72	72	74	79	77	73	72	67	7300	7400
LB7889												
20A	67	67	70	73	74	69	69	70	68	66	7000	
20B	69	67	67	68	68	69	72	72	72	68	6900	7000
LB7890												
21A	56	65	70	68	70	74	76	73	70	49	7000	
21B	42	70	76	74	77	70	68	69	66	61	7000	<u>7000</u>
LOT 2 Avg.												7100
LB7915												
22A	66	67	63	63	65	63	63	61	60	61	6300	
22B	65	67	66	65	67	61	57	58	59	61	6300	6300
LB7916												
23A	70	70	66	64	64	60	64	59	58	60	6400	
23B	63	62	61	64	68	63	63	64	68	69	6500	6400
LB7917												
24A	71	73	73	70	72	84	77	76	77	72	7500	
24B	66	68	72	72	70	82	80	76	77	76	7400	7500
LB7918												
25A	72	76	77	77	76	72	70	70	70	70	7300	
25B	70	79	67	66	66	65	68	68	70	69	7800	7600
LB7919												
26A	23	41	55	64	64	56	52	46	34	25	4600	
26B	20	43	56	60	58	54	51	46	34	23	4400	4500

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Table 5B

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<u>Billet</u> <u>No.</u>	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>	<u>6</u>	<u>7</u>	<u>8</u>	<u>9</u>	<u>10</u>	<u>Avg.</u>	<u>Billet</u> <u>Avg.</u>
LB7920												
27A	32	46	56	68	69	61	51	41	30	20	4700	
27B	30	43	56	67	67	60	49	42	29	19	4600	4700
LB7921												
28A	68	68	66	66	66	69	67	65	65	64	6600	
28B	63	65	66	64	72	72	69	69	70	69	6800	6700
LB7922												
29A	32	51	63	64	68	66	59	51	44	31	5300	
29B	41	56	66	64	66	66	61	53	43	28	5400	<u>5400</u>
LOT 3 Avg.												6200

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TABLE 6B

CREEP DEFORMATION vs. LOCATION WITHIN THE BILLET (CRD)

75-25 NEEDLE COKE PRODUCTION MATERIAL (CRD)
(2500° C 4000 psi 1 hour)

<u>Specimen No.</u>	<u>Diametral Location</u>		<u>Longitudinal Location</u>		
	<u>Central</u>	<u>Peripheral</u>	<u>Top</u>	<u>Middle</u>	<u>Bottom</u>
<u>Billet 7870</u>					
B1*		3.5%	3.5%		
B2*		5.1			5.1
G8	5.8%		5.8		
G9	5.5			5.5	
G10	4.6				4.6
G17					
J7	Specimen broken during machining.				
J8	Specimen broken during machining.				
J9	5.8				5.8
Averages	<u>5.4%</u>	<u>4.3%</u>	<u>4.7%</u>	<u>5.5%</u>	<u>5.2%</u>

Billet 7870 Avg. 5.0%

Billet 7874

B1*		3.6	3.6		
B2*		3.4			3.4
G8	3.6		3.6		
G9	3.3			3.3	
G10	Specimen broken during machining				
G17		3.6		3.6	
J7	3.3		3.3		
J8	3.6			3.6	
J9	4.2				4.2
Average	<u>3.6</u>	<u>3.5</u>	<u>3.5</u>	<u>3.5</u>	<u>3.8</u>

Billet 7874 Average 3.6%

* Quality Control data

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TABLE 7B

THERMAL EXPANSION vs. LOCATION WITHIN THE BILLET

75-25 NEEDLE COKE PRODUCTION MATERIAL (CRD)

Secant α = Microinches/inch/° C

Specimen No.	Grain Direction	Billet Location	Cycle ¹⁾	2000° C	2300° C	Max. Temp. ° C	Permanent Deformation, %
<u>Billet 7870</u>							
C4	WG	Bottom	1	6.9	7.0	2450	-.12
H1	WG	Top	1	7.3	7.2	2200	.00
			2	7.3	7.2	2280	-.03
H9	WG	Middle	1	7.3	7.3	2210	-.07
			2	7.5	7.5	2290	-.03
H17	WG	Bottom	1	7.1	7.1	2210	-.03
			2	7.2	7.2	2290	-.04
	Avg. (WG)				<u>7.2</u>		<u>-.05</u>
G7	AG	Central	1	8.1	8.4	2220	.11
			2	7.7	8.2	2330	.00
J13	AG	Peripheral	1	8.1	8.4	2220	.06
			2	7.9	8.3	2330	.03
	Avg. (AG)				<u>8.3</u>		<u>.05</u>
<u>Billet 7874</u>							
C4	WG	Bottom	1	6.9	6.9	2450	-.12
H1	WG	Top	1	7.3	7.3	2320	-.04
			2	7.4	7.3	2290	.01
			3	7.5	7.3	2320	.00
H9	WG	Middle	1	7.3	7.3	2320	-.10
			2	7.6	7.7	2280	-.02
			3	7.6	7.9	2320	.00
H17	WG	Bottom	1	7.2	7.2	2340	-.09
			2	7.4	7.5	2290	.01
			3	7.5	7.5	2340	-.01
	Avg. (WG)				<u>7.4</u>		<u>-.03</u>
G7	AG	Central	1	8.6	9.3	2340	-.02
			2	8.2	9.0	2290	.00
			3	8.2	9.0	2340	
J13	Specimen broken during machining						
	Avg. (AG)				9.0		-.01

1) Billets heat treated 1 hour at 2500° C

2) Quality Control Data

TABLE 8B

FLEXURAL STRENGTH vs. LOCATION WITHIN THE BILLET

75-25 NEEDLE COKE PRODUCTION MATERIAL, KSI (CRD)

(With Grain)

<u>Specimen No.</u>	<u>Diametral Location</u>		<u>Longitudinal Location</u>		
	<u>Left</u>	<u>Right</u>	<u>Top</u>	<u>Middle</u>	<u>Bottom</u>
<u>Billet 7870</u>					
C1*					14.7
C2*					14.8
C3*					14.2
H2	12.4	11.0	12.4		
			11.0		
H4	11.6	3.8	11.6		
			3.8		
H7	13.0	14.7		13.0	
				14.7	
H10	13.4	8.0		13.4	
				8.0	
H13	13.0	13.9			13.0
					13.9
H15	12.6	10.8			12.6
					10.8
Averages	<u>12.8</u>	<u>10.6</u>	<u>9.7</u>	<u>12.5</u>	<u>13.6</u>
Averages (omitting values under 10 KSI)	12.8	12.9	11.7	13.7	13.6
Billet 7870 Avg. 12.3					

Billet 7874

C1*					14.2
C2*					15.0
C3*					11.7
H2	13.6	12.9	13.6		
			12.9		
H4	Broken	13.7	13.7		
H7	Broken	14.1		14.1	
H10	13.4	13.4		13.4	
H13	12.7	14.1			12.7
					14.1

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<u>Specimen No.</u>	<u>Diametral Location</u>		<u>Longitudinal Location</u>		
	<u>Left</u>	<u>Right</u>	<u>Top</u>	<u>Middle</u>	<u>Bottom</u>
H15	15.0	13.8			15.0 13.8
Averages	<u>12.8</u>	<u>13.7</u>	<u>13.4</u>	<u>13.6</u>	<u>13.8</u>
Billet 7874 Average	13.7				

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TABLE 9B

FLEXURAL STRENGTH vs. LOCATION WITHIN THE BILLET

75-25 NEEDLE COKE PRODUCTION MATERIAL, KSI (CRD)

(Across Grain)

<u>Specimen No.</u>	<u>Top</u>	<u>Middle</u>	<u>Bottom</u>
<u>Billet 7870</u>			
A1*	5.6	6.2	4.4
A4*	5.6	6.4	7.0
G1	---	3.6	1.7
G4	4.3	---	---
G11	7.1	5.1	5.5
G14	6.4	5.5	5.4
J1	---	3.6	5.4
J4	4.8	5.3	5.0
J10	7.1	4.4	5.5
Averages	<u>5.8</u>	<u>5.0</u>	<u>5.0</u>

Billet 7870 Average 5.2

<u>Billet 7874</u>			
A1*	8.2	6.1	7.7
A4*	7.5	6.5	8.3
G1	4.9	5.6	5.7
G4	Specimen broken during machining		
G11	5.9	5.7	6.0
G14	6.0	5.7	5.9
J1	6.6	5.7	6.4
J4	5.9	6.4	5.9
J10	Specimen broken during machining		
Averages	6.4	6.0	6.6

Billet 7874 Average 6.3

* Quality Control Data

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613138-1B
Figure 1B - With-Grain Strength vs. Electrical Conductivity Relationship for Production
Billets of 75/25 Needle Coke Composite-Individual Specimens (C-RD)

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Figure 2B - Across-Grain Strength vs. Electrical Conductivity Relationship for
Production Billets of 75-25 Needle Coke Composites - Individual Specimens (CRD)

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613138-3

Figure 3B - Specimen Sampling Plan for Property Variation within 75-25 Needle

Coke Production Billets

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H13	13.0	13.9	FLEX
H15	12.6	10.8	FLEX
H17	7.2		CTE
C1	14.7	14.8	FLEX
C2	14.2		
C3		7.0	CTE

J-5	5.4	3.6	—	FLEX
J-4	5.0	5.3	4.8	FLEX
J-10	5.5	4.4	7.1	FLEX
J-13			8.3	CTE

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AG CREEP DEFORMATION, %
2500°C, 4000 PSI, 1 HR

TOP

WG FLEX STRENGTH, PSI $\times 10^3$
& WG EXP COEFF $\times 10^{-6}/^{\circ}\text{C}$

AG FLEX STRENGTH & AG EXP COEFF

TOP

613138-5B

Figure 5B - Property Variation within 75-25 Needle Coke production Billet 7874 (CRD)

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XIV. APPENDIX C

ELECTRICAL CONDUCTIVITY DATA ON BILLETS USED FOR HOMOGENEITY STUDY (U)

(U) The raw data is shown in Tables 1C and 2C.

(CRD) Plots of electrical conductivity versus flexural strength using data specimens taken from Billets 238, 242, 249, 250, 251, and 252 are shown in Figures 1C and 2C. The raw data was taken from Table 11 in the main body of this report. The lines for the 75 w/o NbC composition show reasonably good agreement with similar data on production billets as described in Appendix B and shown in Figures 1B and 2B.

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APPENDIX C

TABLE 1C

ELECTRICAL CONDUCTIVITY PROFILE DATA - WITH GRAIN (U)

0.5 inch span 0.25 inch increments

Longitudinal Test Location Numbers Starting From the Left
(See Figure 24 Section BB for specimen location)

Specimen No.	1	2	3	4	5	6	7	8	9	10	11	12
Billet 238 (35-35-30)												
1	7700	7830	8010	7930	7770	7560	7270	7040	6910	7180	7580	----
3	8230	8380	8530	8530	8530	8380	8350	8380	8490	8400	8070	----
4	8190	8310	8660	8850	8790	8780	8580	8350	8170	7990	7850	----
5	8140	8160	8190	8210	8210	8070	7910	7710	7360	7280	7420	
6	7900	7880	7820	7710	7680	7640	7480	7270	6840	6720	6940	----
7	6590	7180	7550	7560	7450	7580	7650	7710	7740	7770	7700	----
8	7710	7740	7710	7730	7740	7740	7770	7820	7740	7640	7650	----
9	7740	7730	7590	7590	7580	7500	7450	7430	7430	7450	7340	----

Billet 242 (35-35-30)

1	7640	7560	7680	7710	7800	7880	7650	7220	7060	6500	6400	----
2	8060	8020	7980	7820	7860	8210	8190	8190	8310	8530	8420	----
3	7900	7940	8208	8160	8040	8170	8020	7900	8170	8570	8810	----
4	8060	8570	9310	9380	9340	9160	8950	8910	8930	9000	8970	8680
5	8420	9000	9250	9520	9590	9430	9180	9160	8890	8440	8530	8620
6	10100	10310	10000	9950	9830	9560	9700	9840	9800	9630	10030	10030
7	10150	10340	9750	9430	9660	9610	9700	9830	10050	10050	10080	10130
8	9970	10100	10100	10080	10000	9800	9830	9900	9610	8950	8950	9610
9	9430	9310	9400	9540	9380	9490	9450	9400	9180	9520	9800	9780

Billet 249 (75-25)

1	Specimen Broken											
2	11225	11320	11830	12240	12310	12240	12270	12310	12010	11690	10940	
3	11940	11980	12090	12160	11980	11940	11940	12090	12050	11980	11730	
4	10650	11260	11450	11870	12240	12310	12750	12710	12310	11900	11260	
5	12240	12390	12550	12710	12710	12710	12670	12390	12310	11940	11830	
6	11490	12200	12630	12590	12670	12630	12510	12390	11900	11730	12200	
7	12630	13000	13450	13490	12880	12350	12350	11900	11520	11450	11350	
8	12240	12120	11730	11870	12240	11800	10910	10680	10340	10050	10340	
9	12200	12550	12430	12270	11980	11420	10940	10970	11160	11590	11730	

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APPENDIX C
Table 1C
Page 2

Specimen No.	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>	<u>6</u>	<u>7</u>	<u>8</u>	<u>9</u>	<u>10</u>	<u>11</u>	<u>12</u>
Billet 250 (75-25)												
1	5060	6320	7730	8330	8280	7940	7990	7430	5340	5270	5810	4740
2	8580	9340	9610	10100	10910	11320	11190	10970	10560	9490	8600	7930
3	11290	11900	12010	12050	12120	11900	12270	12240	11620	11490	11040	10450
4	11870	11940	12010	12160	12390	12630	12310	12240	12270	12200	12120	11980
5	13090	12960	12960	12830	12790	12710	12590	12390	12390	12050	12050	12200
6	12240	12310	12270	12240	11730	11450	11590	12310	12390	12310	12590	12240
7	12310	12350	12390	12590	12590	12430	12240	12010	12200	12270	12390	13050
8	12240	12200	11590	11830	11980	11800	11490	11690	12430	12710	12590	12270
9	7120	8700	10210	10420	10970	10710	10100	10340	10590	10400	10000	8760
Billet 251 (75-25)												
1	5060	5400	6130	6500	6780	7140	7000	6860	7180	7180	6500	----
2	6040	5530	5260	5810	6700	7300	7060	6600	6380	6270	6630	----
3	8010	7190	5850	5340	5280	5210	5210	5460	6180	6700	7490	----
4	9180	9040	8240	7550	7310	7530	7960	8550	8970	9160	9210	----
5	10000	10400	10880	10530	10000	9660	9870	10080	10310	10480	10940	----
6	10590	10590	10910	11010	11010	11160	10970	11040	11100	10880	10560	----
7	10370	10680	10670	10650	11040	11190	10940	10940	10910	10650	10970	----
8	10970	10940	10880	10680	10450	10370	10510	10590	10620	10530	10640	----
9	11730	11490	11520	11550	11450	11550	11490	11550	11660	11490	11290	
Billet 242 (75-25)												
1	11550	12010	11730	11040	10480	9920	9780	10180	11160	11660	11690	----
2	11940	11870	11590	10940	10680	11010	11730	12200	11940	10650	10050	----
3	12590	12390	12350	12470	12710	12710	12790	13130	12960	13000	12880	----
4	12350	11980	11690	11940	12350	12710	12790	12920	13090	12710	11660	----
5	12670	12710	13000	13090	13220	13180	13050	12790	12670	12010	11520	----
6	12468	12880	13130	13220	13310	13490	13590	13540	13310	13220	13220	13090
7	11620	12390	12960	13090	13090	13130	13220	13180	13130	12750	12630	12390
8	11320	11350	11690	12310	12670	12670	12510	12470	12670	12550	12270	10970
9	12160	12390	12630	12750	12790	12710	12750	12710	12590	12390	12350	12470

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TABLE 2C

ELECTRICAL CONDUCTIVITY PROFILE DATA - ACROSS GRAIN (CRD)

0.5 inch span 0.25 inch increments

	Diametral Test Location Numbers Starting From the Top (See Figure 4 Section CC for specimen location)											
Specimen No.	1	2	3	4	5	6	7	8	9	10	11	12
Billet 238 (35-35-30)												
1	5380	5040	4700	5120	5690	5690	5200	4910	4870	4860	4950	5190
2	5180	4990	4690	4930	5550	5740	5250	5010	4960	4970	5070	5210
3	5030	5051	5010	4980	5200	5430	5230	5030	4950	5100	5250	5320
4	5490	5120	5100	5190	5340	5460	5310	5060	4930	4950	4950	4930
5	5410	5120	5080	5030	5050	5180	5240	5150	4940	4860	4910	4900
Billet 242 (35-35-30)												
1	4130	4490	4370	4240	4160	3980	3860	3750	3830	4020	4030	----
2	3830	3750	3720	3720	3790	3720	3900	4360	4580	4630	4040	----
3	3790	4530	4850	4740	4160	3490	3490	3460	3490	3610	3650	----
5	4480	4380	4230	4240	4530	4240	3860	4060	4010	3900	4000	----
6	3940	3830	3750	3720	3580	3790	4240	4230	4140	4280	4520	----
Billet 249 (75-25)												
1	Specimen Broken											
2	6270	5980	6150	6330	6350	6760	6560	6350	7300	8010	7410	----
3	6090	5840	5850	5730	5790	5850	5800	6150	6960	7710	8440	----
4	6060	5910	5720	5710	5790	5790	5970	6190	6680	7430	7530	----
5	6100	6130	6050	6340	6430	6350	6180	6460	7270	7460	7050	----
6	Specimen Broken											
Billet 250 (75-25 post pressed)												
1	4250	5430	5970	6040	5850	5720	5940	5920	5810	6490	6880	----
2	6820	6480	5970	5800	5530	5330	5490	5850	5790	5540	4920	----
3	6950	6470	6180	5810	5460	5240	5450	5630	5710	5760	5050	----
4	7560	7310	6170	5530	5540	5430	5460	5760	5800	5330	4700	----
5	6480	6840	5990	5600	5810	5700	5690	6150	6000	5630	4730	----
6	5830	6354	6260	6180	6470	6010	5730	6140	6090	5110	3720	----

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Table 2C

Page 2

Specimen No.	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>	<u>6</u>	<u>7</u>	<u>8</u>	<u>9</u>	<u>10</u>	<u>11</u>	<u>12</u>
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Billet 251 (75-25 Post Pressed) (Die Compacted by the old process)

1	5500	5340	5710	5680	5350	5270	5460	5320	5120	5130	5140	----
2	5540	5530	5420	5160	4970	4810	4910	4690	4250	4160	4290	----
3	5480	5460	5270	4990	4820	4740	4370	3750	3430	3550	4050	----
4	5470	5470	5210	4940	4910	4720	4580	4050	3370	3180	3260	----
5	Specimen Broken											
6	4640	5050	4930	5030	5060	5030	4990	5180	5320	5600	5710	----

Billet 252 (75-25)

1	7040	6540	6350	6460	6380	6040	5960	6210	6630	6660	6630	6790
2	7060	6440	6330	6270	6120	5830	5810	5880	6040	6390	6790	6630
3	7280	6680	6180	6070	5970	5700	5770	5790	5890	6340	6400	6320
4	6800	6230	6060	6070	6980	6870	5900	5860	5850	6190	6400	6150
5	7040	6740	6460	6300	6030	5830	5890	5890	6010	6600	6800	6450
6	7880	7620	7060	7140	7150	6470	6080	6190	6420	6750	7010	6840

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613138-6B

Figure 1C - With-Grain Strength vs. Electrical Conductivity Plot for Property Homogeneity Billets (U)

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613138-7B

Figure 2C - Across-Grain Strength vs. Electrical Conductivity Plot for Property Homogeneity Billets (U)