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PHYSICAL PROPERTIES
OF REFRACTORY MATERIALS

S. A. Bortz
IIT Research Institute

TECHNICAL REPORT NO. AFML-TR-66-223
August 1966

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Air Force Materials Laboratory
Research and Technology Division
Air Force Systems Command
Wright-Patterson Air Force Base, Ohio

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Air Force Materials Laboratory
Research and Technology Division
Air Force Systems Command
Wright-Patterson Air Force Base, Ohio

FOREWORD

This report was prepared by the Ceramics Research Division of IIT Research Institute, Chicago, Illinois, under Contract No. AF 33(615)-3028. The contract was initiated under Project No. 7381, "Materials Application," Task No. 738106, "Design Information Development." The work was administered under the direction of Marvin Knight, of the Materials Information Branch (MAAM), Research and Technology Division, Air Force Materials Laboratory, Wright-Patterson Air Force Base, Ohio.

This report covers work conducted from 1 May 1965 to 1 May 1966.

S. A. Bortz, Senior Engineer, Ceramics Research, was project engineer. Others who cooperated in the research were C. Connors, Assistant Engineer; J. Hedge, Research Engineer; V. Hill, P. Herman and P. Janson.

Detailed data for the program are recorded in IITRI Logbooks Nos. C 16489 and C 16815. This report has been given the internal designation IITRI-G-6001-12.

Manuscript of this report was released by the author in May 1966 for publication as an RTD Technical Report.

This technical report has been reviewed and is approved.



D. A. SHINN
Chief, Materials Information Branch
Materials Applications Division
Air Force Materials Laboratory

ABSTRACT

The purpose of this program is to characterize and correlate the physical and mechanical properties of JTA graphite and, from these data, determine its critical engineering properties.

Three JTA billets were studied, two of 7 in. diameter by 6 1/2 in. long, and one 14 in. diameter by 7 in. long. These were examined by Carbon Products Division of Union Carbide Corporation for gross flaws and differences in density, by radiographic methods. The material was found to be acceptable within the limitations, as provided in the procurement specifications. The densities of the three billets are above 3.00 g/cc. Metallographic, x-ray, and microprobe analyses confirm the presence of graphite, ZrB_2 , and βSiC . Metallographic examination indicates only a slight variation in distribution of the three principal elements of JTA graphite. The ZrB_2 and βSiC content of the edges of the billet appear to be somewhat less than that in the center.

Mechanical measurements indicate that JTA graphite is anisotropic; the "with" grain orientation is about twice as strong as the "across" grain orientation. Size effect studies indicate that a minimum 1/4 in. cross section dimension is adequate for determining average mechanical properties. Studies were made using both flexure and tension measurements. The mechanical strength of the three billets appears to be the same.

The specific heat of the three billets was found to be the same.

Temperature and oxygen pressure have a great effect on the oxidation resistance of JTA graphite. The material appears to be more resistant at the higher temperatures.

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I. INTRODUCTION

This report consists of a summary of work accomplished during the period of May 1965 to May 1966, under Contract No. AF 33(615)-3028, which is directed toward obtaining property data for JTA graphite. Delays in obtaining material, due to manufacturing problems, have caused the program to proceed at a very low rate during this contract period. The work reported on is approximately equivalent to that originally planned for the first quarter of the program.

Nose cone, rocket nozzle, and leading edge applications under extreme environmental conditions require materials which will withstand severe thermal and mechanical conditions. Attention has been focused on the refractory composite materials because of their potential high strength at elevated temperatures, thermal insulation, and oxidation resistance. However, most of these materials often exhibit a wide variability in properties used in the design of structures required to resist extreme environments. This investigative program is designed to correlate the physical and mechanical parameters of composite refractory oxidation-resistant material, JTA graphite.

Considerable emphasis has been placed on determining the uniformity of JTA from billet to billet and from one size billet to another. Data have been obtained from two 7 in. diameter by 6 1/2 in. long JTA billets and one 14 in. diameter by 7 in. long billet. The information developed during this program is being used to analyze and compare data obtained from previous programs.

II. SUMMARY

Not enough data have been obtained to draw any definite conclusions; however, the following statements appear to be valid from the limited data available:

1. There are no gross flaws or density differences through the billets. The material under study consists of graphite, ZrB_2 , and βSiC .
2. The three billets appear to have roughly the same composition with only a slight microstructural variability throughout the bulk material.
3. The densities are above the specified 3.00 g/cc and appear to be uniform throughout the bulk material.

4. A 1/4 in. major cross-sectional dimension is adequate for determining the average JTA graphite mechanical properties.
5. The JTA graphite is highly anisotropic, and the failure modes for the "with" grain and "across" grain orientations appear to be very dissimilar. The former has a statistical failure range similar to oxide ceramics, while the latter is "deterministic" and can be treated as a metal.
6. The temperature and oxygen pressure have a profound effect on the oxidation resistance of JTA. The material appears to be more resistant at the higher temperatures.
7. The enthalpies of material from the three billets measured are the same.

Table I provides a summary of the mechanical property data presently available.⁽¹⁾ These data can be compared to those shown in Table II. The room-temperature data obtained to date appears comparable to some of the data previously obtained.

Experimental work is being continued to obtain the remainder of the data as outlined in Section III, Material Characterization.

III. MATERIAL CHARACTERIZATION

JTA graphite is manufactured by Carbon Products Division of Union Carbide Corporation. This grade is a multiple-phase composite material⁽²⁾ containing 48 wt% carbon 35 wt% zirconium, 8 wt% boron, and 9 wt% silicon, which is hot-pressed in an induction-heated graphite mold. Carbon Products Division designed this material for good oxidation resistance and mechanical strength at high temperatures. Material characterization makes use of metallographic techniques, electron microprobe analyses, and x-ray analyses, and relates these findings with the following properties:

- | | |
|---|-----------------------------|
| 1. Density | 8. Creep |
| 2. Oxidation resistance | 9. Thermal shock resistance |
| 3. Porosity | 10. Thermal expansion |
| 4. Tensile strength | 11. Emittance |
| 5. Compressive strength | 12. Specific heat |
| 6. Flexural strength | 13. Thermal diffusivity |
| 7. Elastic properties (Young's modulus and Poisson's ratio) | |

Table I
PREVIOUS DATA ON JTA GRAPHITE (1)
(Room temperature, 1/4 in. diameter billets)

| Property | With Grain | | | Across Grain | | |
|--|--------------|------------------|--------------|--------------|--------------|-------------------|
| | No. of Tests | Avg. | Error (v), % | Billet No. | No. of Tests | Avg. Error (v), % |
| Bulk density, g/cc | 22 21 | 2.966 2.854 | 23 3.5 | 1 2 | 8 | 3.028 1.1 |
| Coefficient of expansion, in/in/°C: | | | | | | |
| RT to 1500°C | 2 | 5.6 | | 1,2 | 2 | 6.0 |
| 1500°-2000°C | 2 | 5.6 | | 1,2 | 2 | 11.0 |
| Tensile strength, psi | 4 | 10,010 | 7.0 | 1 | | |
| Tensile elastic modulus, 10 ⁶ psi (from Ref. 4) | | 5.6 | | | | 3.6 |
| Flexure strength, psi | 5 5 | 17,750 12,784 | 4.7 5.6 | 1 2 | 5 | 9,592 5.2 |
| Flexure elastic modulus, 10 ⁶ psi | 5 5 | 9.1 7.1 | | 1 2 | 5 | 4.95 |
| Compressive strength, psi | 3 5 | 15,330 12,592 | 2.6 11.2 | 1 2 | 5 | 22,160 8.7 |
| Compressive elastic modulus, 10 ⁶ psi | 5 | 2.9 | | 1 | 5 | 3.6 |

Table II
PROPERTIES OF JTA GRAPHITE (ROOM TEMPERATURE)

| Property | With Grain | | | Across Grain | | |
|---|--------------------|--------|--------------------------|--------------------|------|--------------------------|
| | No. of Tests | Avg. | Error (ν), % | No. of Tests | Avg. | Error (ν), % |
| Bulk density, g/cc | | | | | | |
| Billet 7-F-12 | 20 | 3.091 | 0.68 | | | |
| Billet 7-F-14 | 20 | 3.077 | 0.74 | | | |
| Billet 14-G-1 | 20 | 3.065 | 1.60 | | | |
| Tensile strength, psi | 5 | 11,970 | 7.35 | 5 | 6000 | 3.80 |
| Flexure strength, psi | 60 | 17,850 | 14.87 | 60 | 8940 | 9.73 |
| Flexure elastic modulus, 10 ⁶ psi | 60 | 11.0 | 21.14 | 60 | 4.5 | 26.06 |

The interrelationships of these measurements are analyzed-- for example, mechanical and thermal properties with thermal-shock resistance, or oxidation resistance with mechanical and thermal properties.

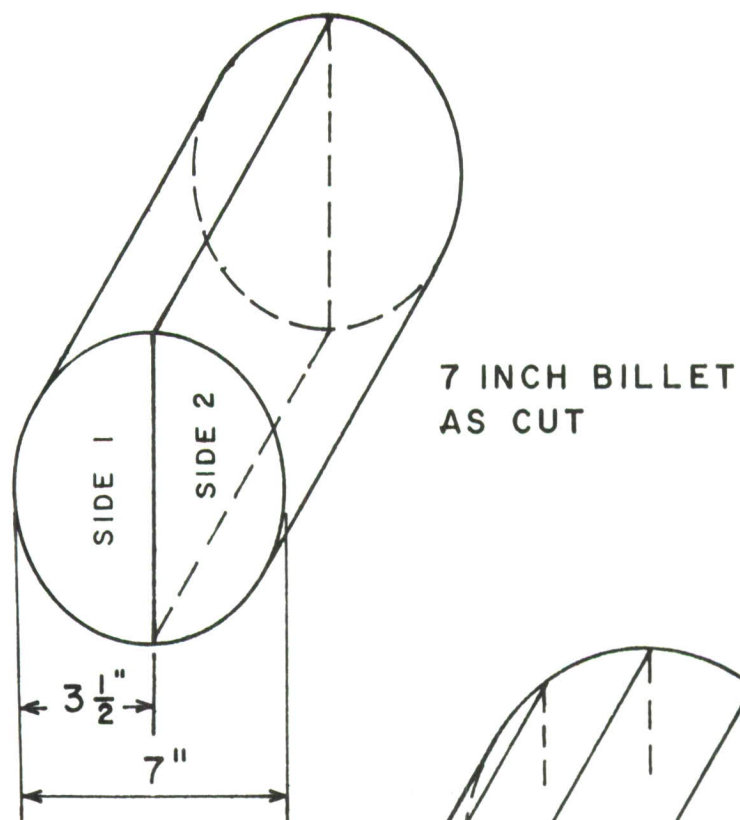
The billets were sectioned and radiographed for cracks and voids in excess of 0.03 in. in major dimensions by Carbon Products before they were sent to IITRI for use on this program. Each billet was identified as follows: 7-F-12, 7-F-14, and 14-G-1. The first number identifies the diameter of the billet, the letter is the code for the pressing series, and the last number identifies the billet for a particular pressing series.

Figure 1 shows how the billets were cut for radiographic examination. Each piece was given three exposures: 4 min at 265 kv, 3 1/2 min at 240 kv, and 2 1/2 min at 200 kv. A medium speed film, type AA, was used for this work. The exposures are shown in Figs. 2, 3, 4, 5, and 6. Examination of these negatives reveals that the billets appear to be a heterogeneous mixture. No major flaw areas can be distinguished, nor can large differences in density be detected.

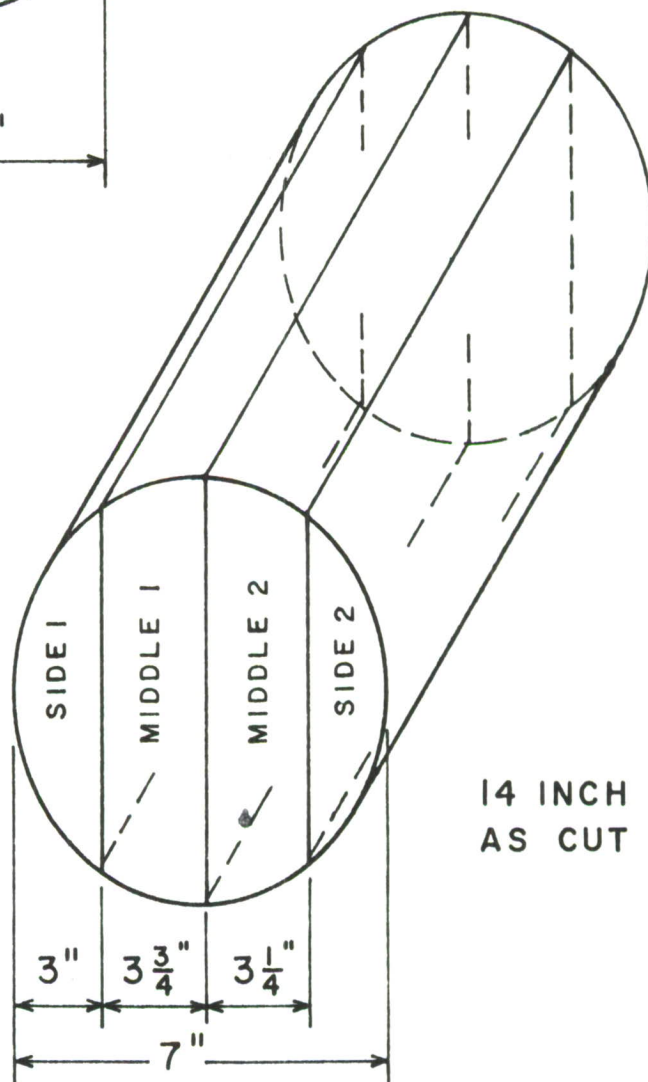
Samples were cut from each billet as shown in Fig. 7 for the 7 in. billets and Fig. 8 for the 14 in. billet. The material cut from these areas was used for metallographic examination, microprobe, and x-ray analyses.

A. Powder X-Ray Analysis

Powder x-ray patterns have been completed for billet 7-F-12. The results of this analysis are listed in Table III. The major constituents were found to be graphite, zirconium diboride (ZrB_2), and traces of β silicon carbide (βSiC). Carbon Products⁽²⁾ reports that graphite, zirconium diboride, and α silicon carbide (αSiC) should be present in JTA graphite. The difference in the SiC structure is probably due to changes in fabrication procedures between the early experimental material and the material used for this program. The βSiC is the low-temperature cubic phase of this compound which begins to convert to the hexagonal phase at 2150°C. Assuming that the raw material consisted of powders of carbon, zirconium diboride, and silicon, βSiC will begin to form at temperatures above 2150°C and will be completely transformed to αSiC between 2150° and 2300°C.⁽³⁾ If the material is very slowly cooled from 2300°C to room temperature, the α will revert back to βSiC . However, this process usually will not occur under normal cooling procedures. The inference is that the JTA graphite under study in this program was hot-pressed at a temperature below 2200°C.

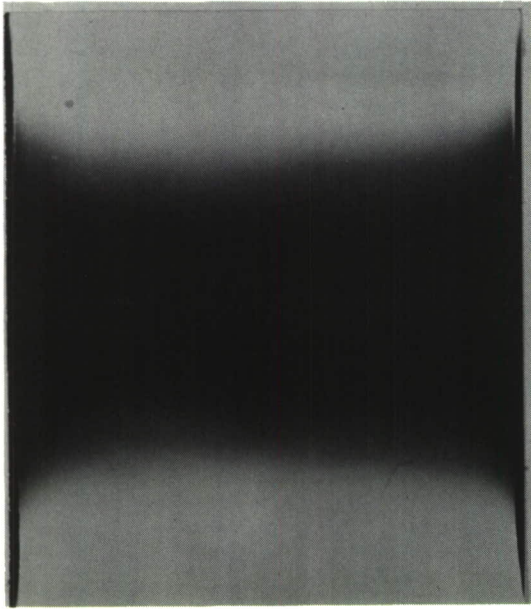


7 INCH BILLET
AS CUT

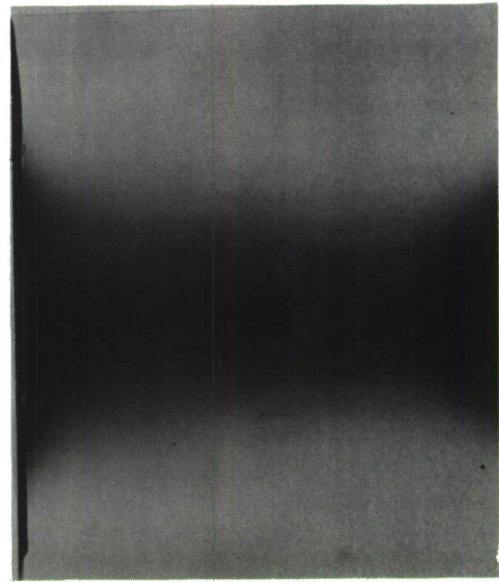


14 INCH BILLET
AS CUT

Fig. 1 - SECTIONING OF JTA BILLETS
FOR RADIOGRAPHIC ANALYSIS



4 MIN. 265 KV
EXPOSURE

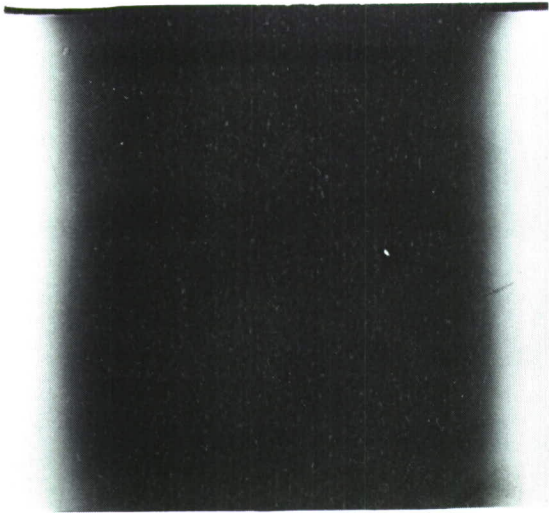


3 1/2 MIN. 240 KV
EXPOSURE

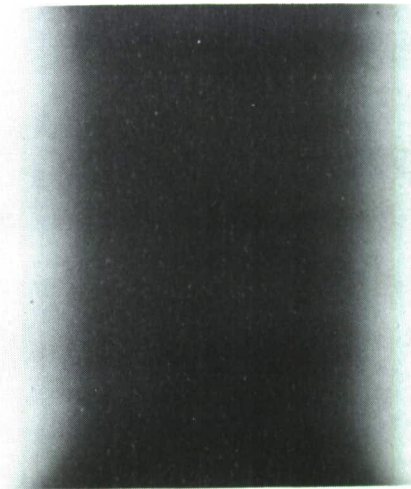


3 1/2 MIN, 240 KV
EXPOSURE

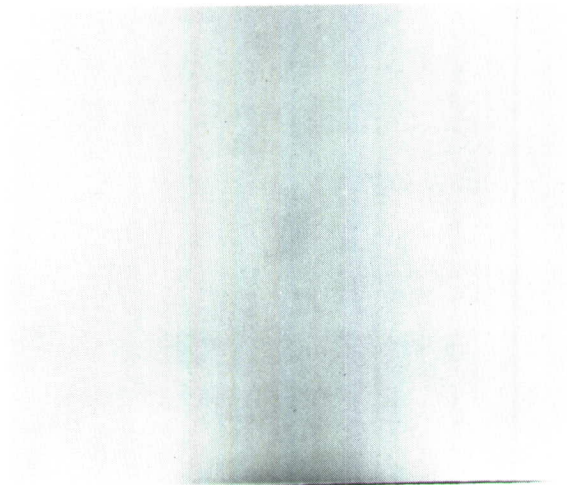
Fig. 2 - RADIOGRAPHS OF BILLET 7-F-12 (SIDE 1)



4 MIN, 265 KV
EXPOSURE

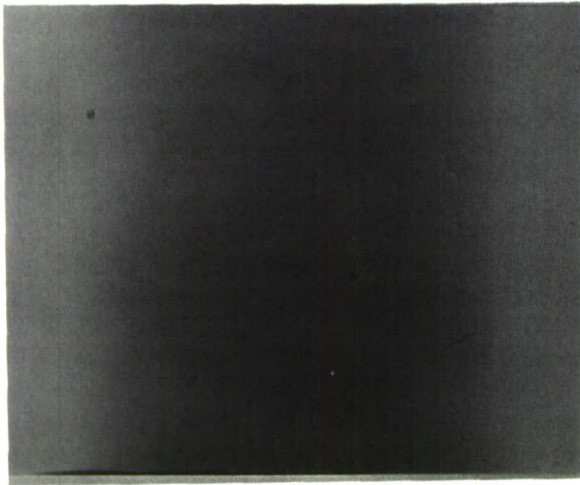


3 1/2 MIN, 240 KV
EXPOSURE

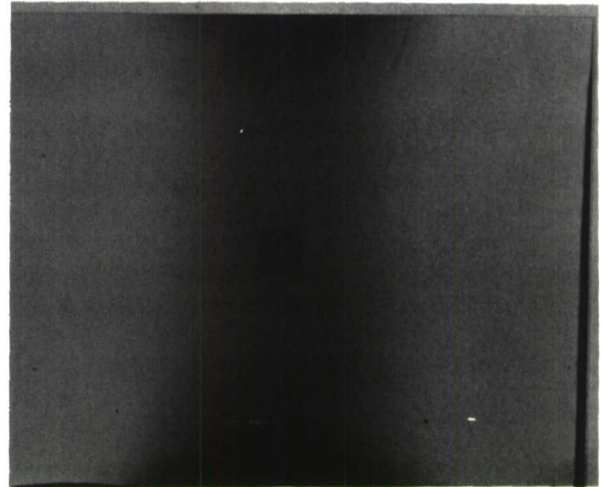


2 1/2 MIN, 200 KV
EXPOSURE

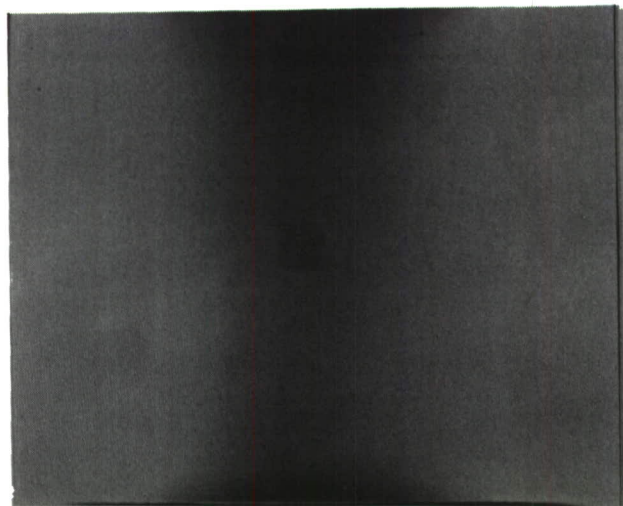
Fig. 3 - RADIOGRAPHS OF BILLET 7-F-12 (SIDE 2)



4 MIN, 265 KV
EXPOSURE

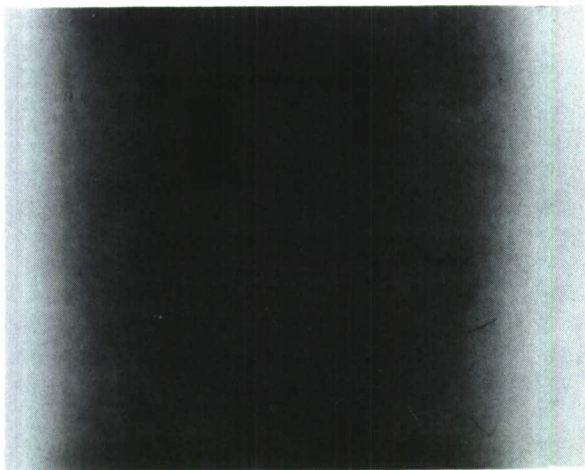


3 1/2 MIN, 240 KV
EXPOSURE

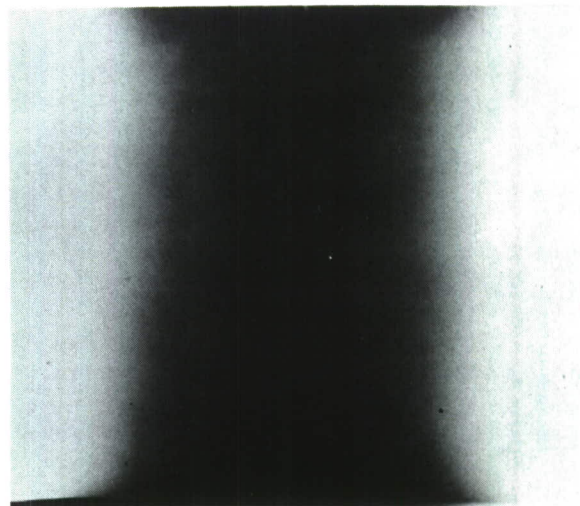


2 1/2 MIN, 200 KV
EXPOSURE

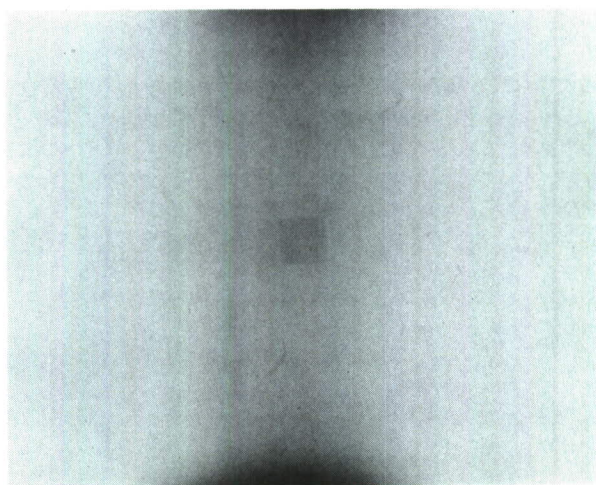
Fig. 4 - RADIOGRAPHS OF BILLET 7-F-14 (SIDE 1)



2 1/2 MIN, 200 KV
EXPOSURE

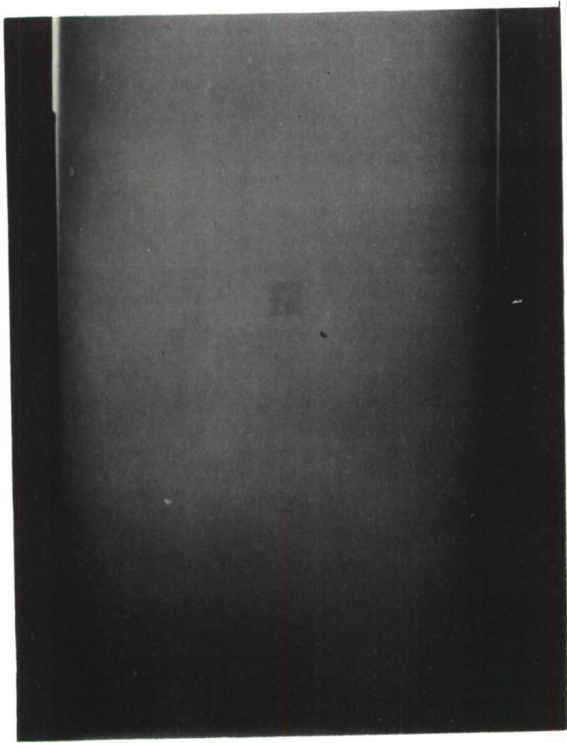


3 1/2 MIN, 240 KV
EXPOSURE



4 MIN, 265 KV
EXPOSURE

Fig. 5 - RADIOGRAPHS OF BILLET 7-F-14 (SIDE 2)



4 MIN, 265 KV
EXPOSURE



3 1/2 MIN, 240 KV
EXPOSURE



2 1/2 MIN, 200 KV
EXPOSURE

Fig. 6 - RADIOGRAPHS OF BILLET 14-G-1

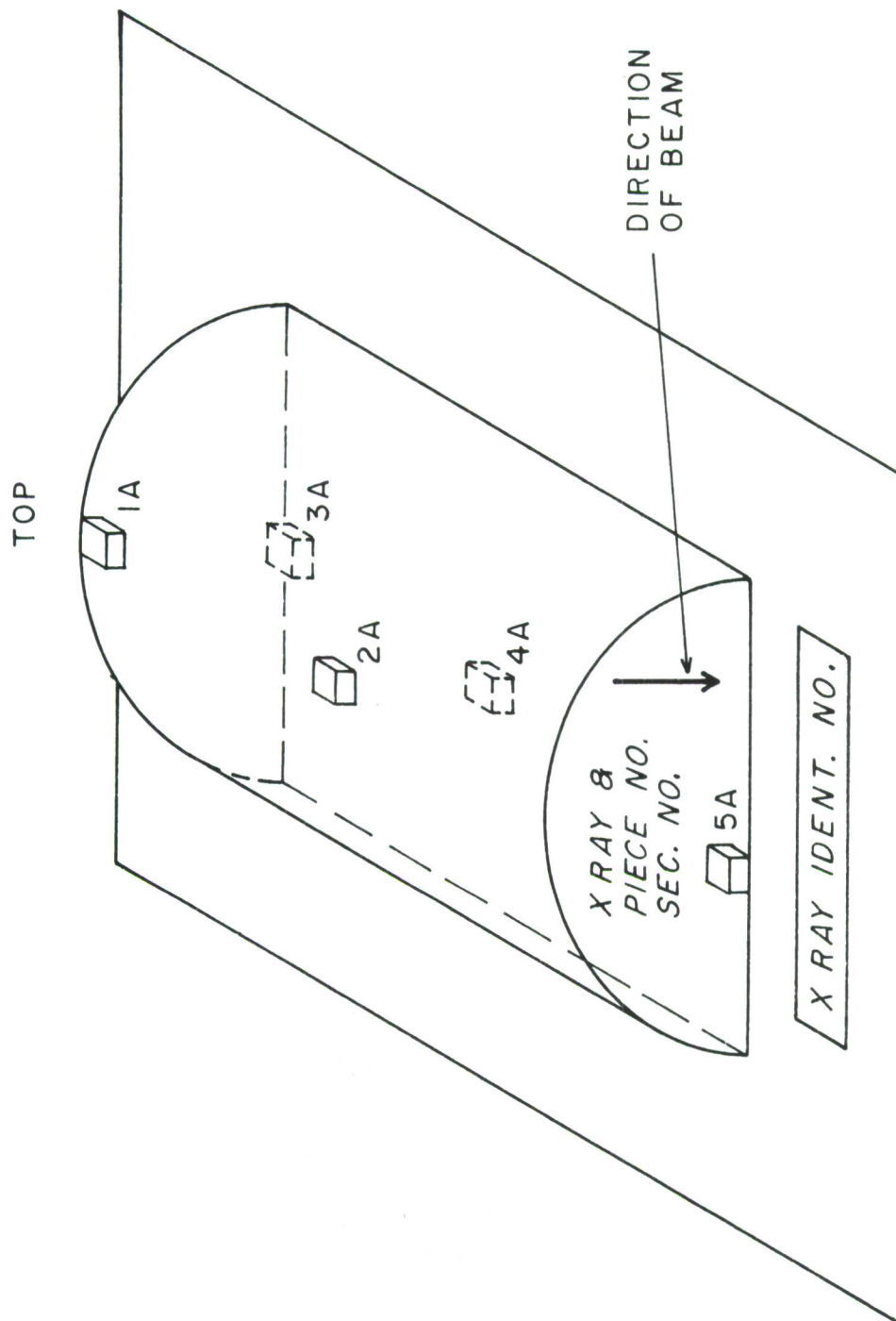


Fig. 7 - SECTION OF 7 IN. DIAMETER BY 6 1/2 IN. LONG BILLET FOR RADIOGRAPHIC EXAMINATION INDICATING LOCATIONS FROM WHICH SPECIMENS FOR MICROPROBE, X-RAY, AND METALLOGRAPHIC ANALYSES WERE OBTAINED

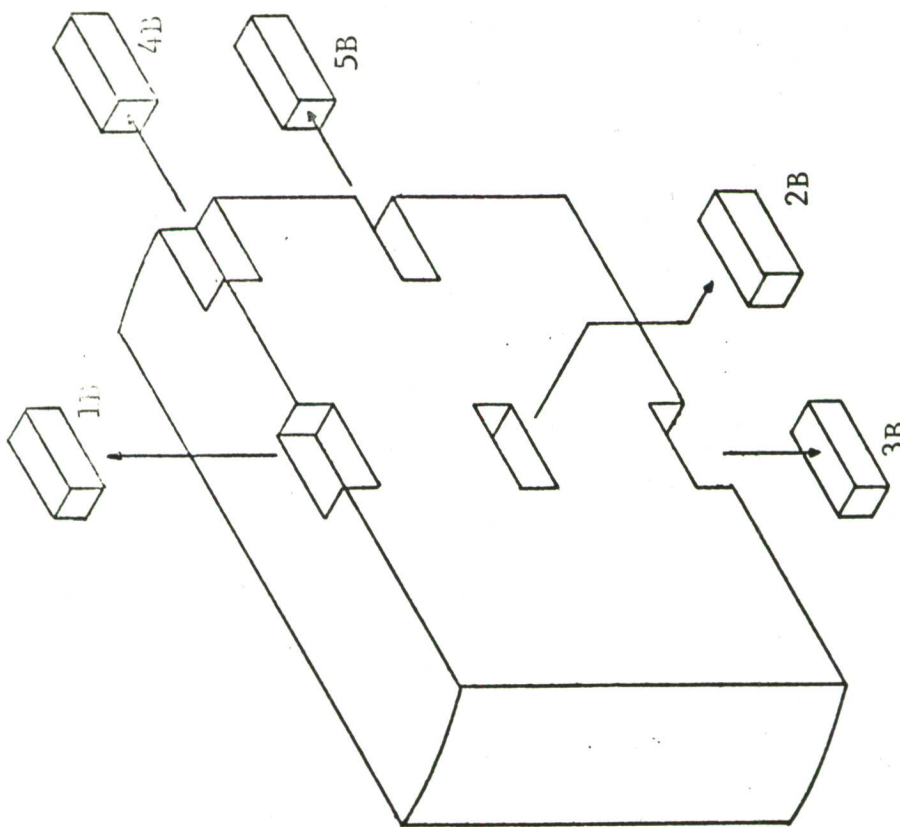


Fig 8 - SECTION OF 14 IN. DIAMETER BY 7 IN. LONG BILLET
FOR RADIOGRAPHIC EXAMINATION INDICATING LOCATIONS
FROM WHICH SPECIMENS FOR MICROPROBE, X-RAY, AND
METALLOGRAPHIC ANALYSES WERE OBTAINED

Table III
CHARACTERIZATION OF JTA GRAPHITE
FROM X-RAY ANALYSIS

| Location (see Fig. 7) | Composition |
|--------------------------|---|
| 1A | Graphite + ZrB_2 + βSiC |
| 2A | Graphite + ZrB_2 + (Trace) βSiC |
| 3A | Graphite + ZrB_2 + (Trace) βSiC |
| 4A | Graphite + ZrB_2 + βSiC |
| 5A | Graphite + ZrB_2 + βSiC |

B. Spectrographic Analysis

Since x-ray analysis is effective only to about a 5% material content, other impurities and the formation of other carbides or silicides of less than 5% content were determined through a spectrographic analysis, Table IV. This work was performed by the Chicago Spectro-Service Laboratory, Inc.

The assumption that these impurities were volatilized even though the base material was not, is only true for Al, Fe, Ba, Cu, Co, Mn, and Ca, and probably is not true for Ti, Mo, V, and Cr. Thus the analysis holds for the lighter elements and those which do not form refractory carbides.

Examination of Table IV shows considerable impurity variation between billets for Al, Fe, Mo, V, Mn, and Cr. Since samples of the original powders from which these billets were formed have not as yet been made available, no analysis can be made for the source of these impurities. The maximum amount of impurities found was less than the 1.5% limit in the material specification.

C. Metallographic Analysis

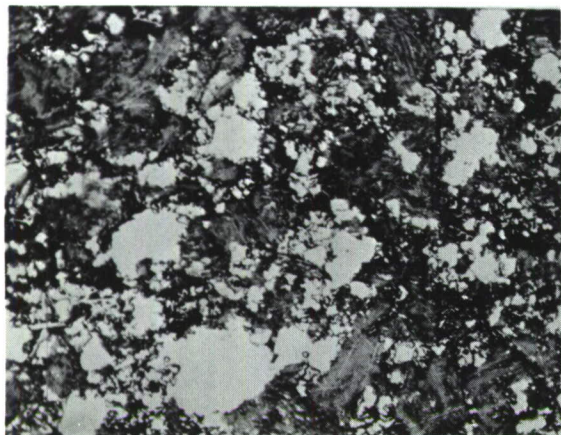
Figures 9, 10, and 11 show the structure of the JTA graphite billets. The light areas are zirconium diboride, the dark gray areas are graphite, and the light gray areas are silicon carbide. In general, the graphite is uniformly distributed in the billets while the amount of zirconium diboride and silicon carbide varies. There appears to be a slight difference in the quantity of these materials and size of grains between the interior of the billets and the outside edges. The average size of the graphite grain appears to be $120 \pm 30\mu \times 70 \pm 15\mu$, the size of the zirconium diboride varies from about 100μ to less than 1μ , and the silicon carbide varies from 50μ to less than 1μ . Billets 7-F-12 and 7-F-14 are similar in structure in that material from the center of the billet shows random distribution of the various phases while the edge material appears to have a higher concentration of small grains of zirconium diboride with silicon carbide predominating at the graphite grain boundaries. Also, there appears to be somewhat less zirconium diboride in the edge material. An exception to the above generalization is the view from the top center of billet 7-F-14, which shows a structure similar to the edge material. Other work⁽⁴⁾ in hot-pressing graphite-carbide composites has shown that low-melting phases will be squeezed out of the structure and diffused into the mold during hot-pressing and the remaining material will concentrate along the grain boundaries. The 7 in. diameter JTA graphite composite appears to be reacting in a similar manner. The 14 in. material exhibits a much greater homogeneity of

Table IV
SEMIQUANTITATIVE ANALYSES (WT%)
OF JTA GRAPHITE

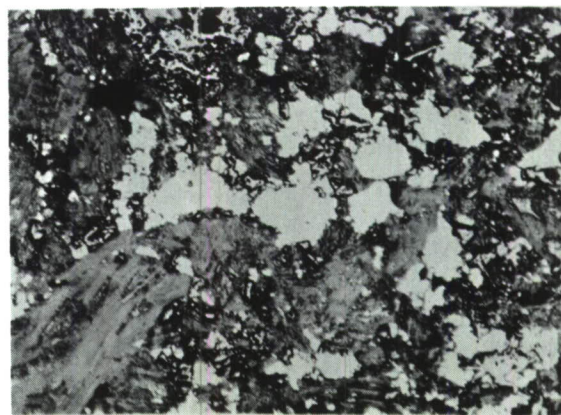
| Sample No. | 7-F-12 | 7-F-14 | 14-G-1 |
|------------|--------|--------|--------|
| Zirconium | ** | ** | ** |
| Boron | ** | ** | ** |
| Silicon | ** | ** | ** |
| Aluminum | 0.05 | 0.2 | 0.2 |
| Iron | 0.2 | 0.08 | 0.05 |
| Titanium | 0.1 | 0.1 | 0.1 |
| Molybdenum | 0.1 | <0.01* | <0.01* |
| Barium | 0.03 | 0.03 | 0.03 |
| Copper | 0.02 | 0.03 | 0.03 |
| Vanadium | 0.02 | 0.005 | 0.005 |
| Cobalt | 0.01 | 0.01 | 0.01 |
| Manganese | 0.01 | 0.005 | 0.005 |
| Chromium | 0.01 | 0.001 | 0.002 |
| Calcium | 0.005 | 0.005 | 0.005 |
| Magnesium | 0.0002 | 0.0004 | 0.0002 |

*Trace

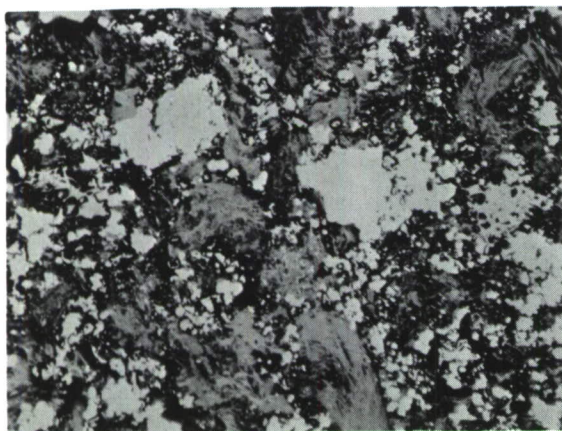
**The major portion of the base material present in these samples was not volatilized in the D.C. arc discharge; therefore, we cannot give any quantitative results for the principal elements. The figures shown are based on the assumption that the impurities were volatilized even though the base material was not.



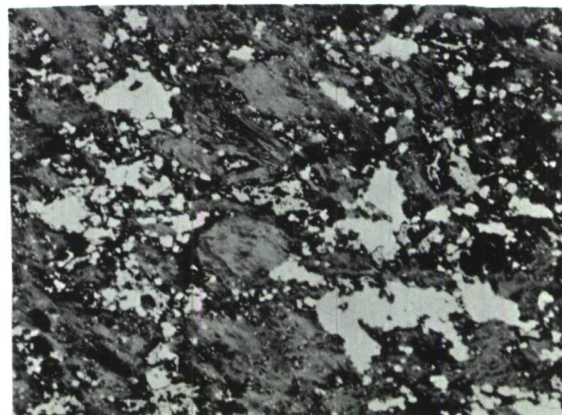
1A Location



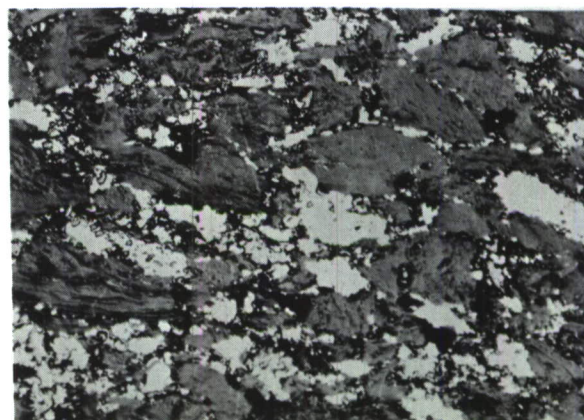
3A Location



2A Location



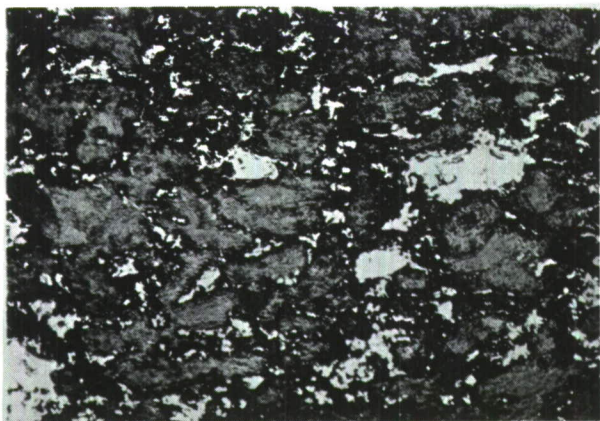
4A Location



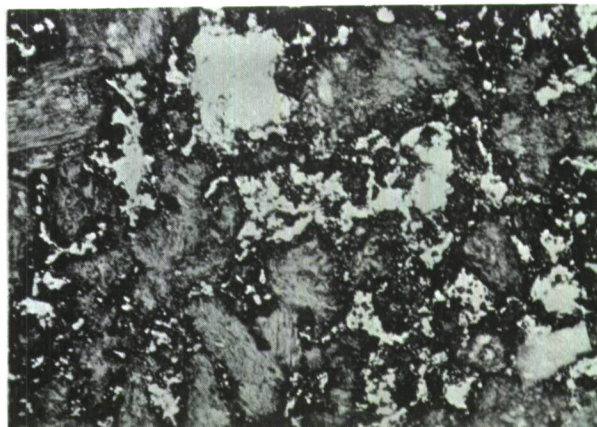
5A Location

50μ

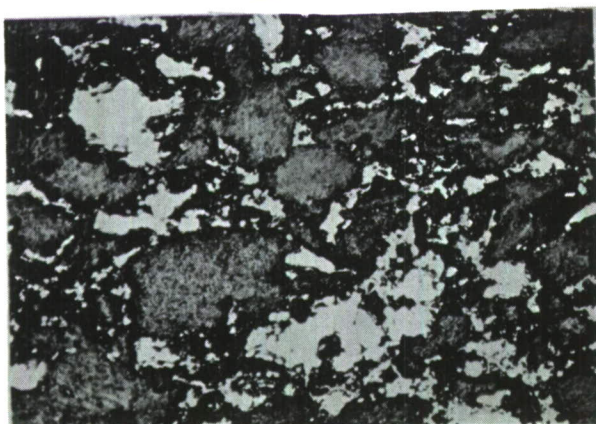
Fig. 9 - PHOTOMICROGRAPHS FROM JTA BILLET 7-F-12



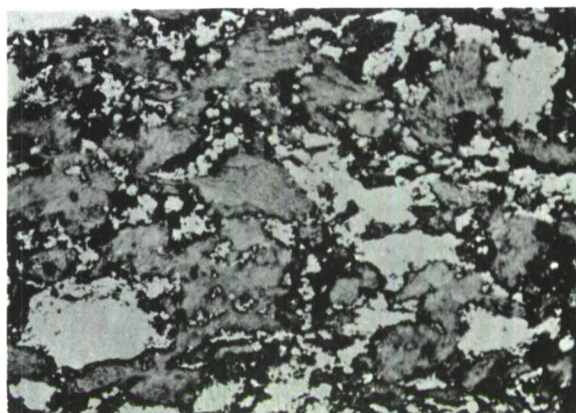
1A Location



3A Location

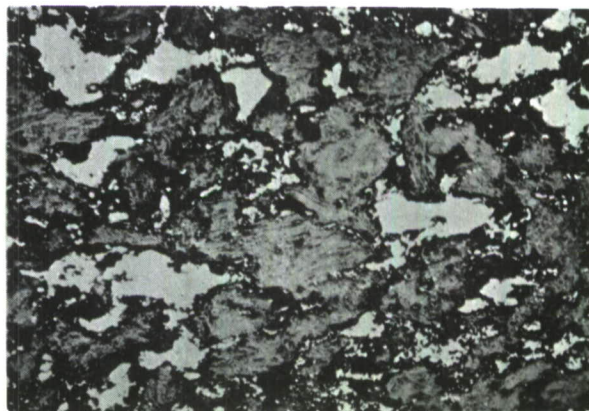


2A Location



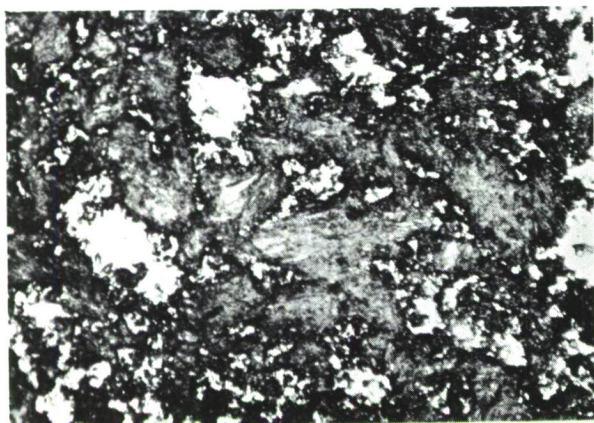
4A Location

$\overline{50\mu}$

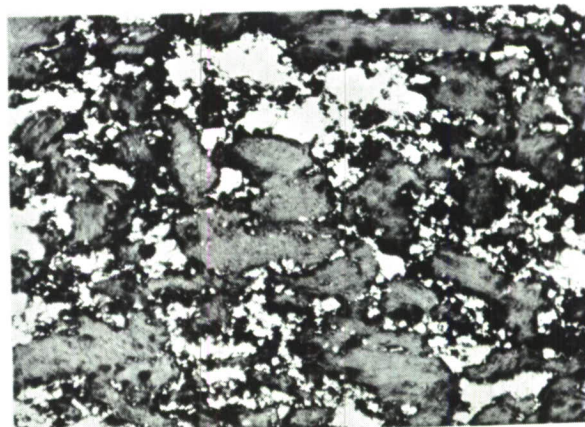


5A Location

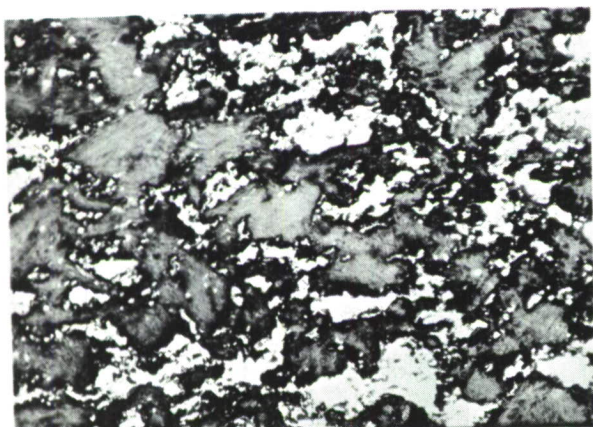
Fig. 10 - PHOTOMICROGRAPHS FROM JTA BILLET 7-F-14



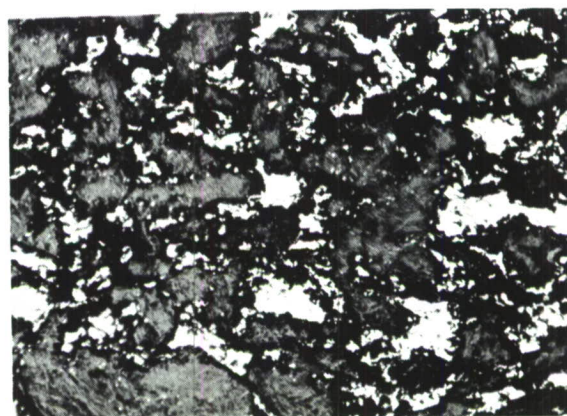
1A Location



3A Location

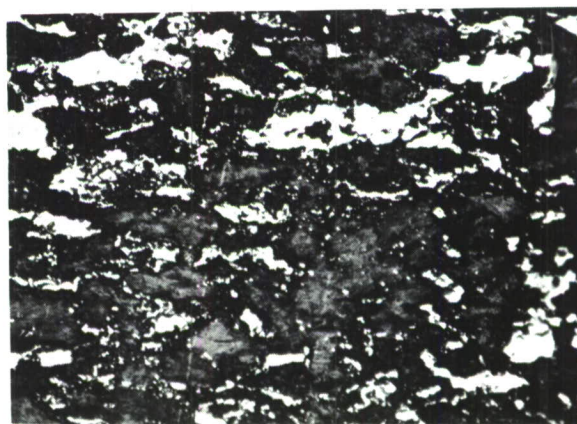


2A. Location



4A Location


 50 μ



5A Location

Fig. 11 - PHOTOMICROGRAPHS FROM JTA BILLET 14-G-1

structure than the 7 in. material, although the trend of grain boundary segregation of small grains of second and third phase materials is still present. The billets are hot-pressed in the form of truncated cones and are machined to a cylindrical shape. Since the larger billet probably has more material cut away than the smaller billets, the evidence of edge segregation would be less for the larger billets than the smaller sizes. The effect of structural variability will be observed through a correlation with mechanical strength.

Close examination of the samples under the microscope at about 1000 magnification shows what appear to be microcracks in the zirconium diboride. Once located, they can also be seen in the lower magnification (320X) photomicrographs shown in the report. No extensive analysis of these microcracks has as yet been made. However, they have also been seen by L. Marcus (6) in a study at Bell Aerosystems Company.

D. Electron Microprobe Analysis

Figure 12 provides the results of microprobe analysis taken at position 3A of billet 7-F-12. The equipment at INTEL can detect an amount of material above 5 wt% with an error of ± 5 wt%. Penetration depth is 5 wt% for C, Si, and B, and 1 wt% for Zr.

While the microprobe confirms the x-ray work that graphite, zirconium, boron, and silicon are present, the results of the analysis are inconclusive as to the amounts and location of these elements with respect to x-ray analysis and the literature.

Theoretically, in the zirconium-rich area there should be 81 wt% Zr and 19 wt% B and in the silicon-rich area there should be 70 wt% Si and 30 wt% C. Actual analysis (Fig. 10) does not conform to the theoretical results. In the carbon-rich area less than 5 wt% Si, Zr, and B are present. In the zirconium-rich area, 46 wt% Zr, 22 wt% B, and less than 5 wt% C and 1 wt% Si were found; in the silicon-rich area, 58 wt% Si and less than 5 wt% C, Zr, and B were found.

The conclusion drawn from the above data is that free silicon and little SiC are present, and this is very unlikely. This phase of the program is still under study. The microprobe sees only a very small area; when more data are available, it should be possible to make better correlations with the other available information.

If the literature values for component materials are assumed to be correct, the actual weight per cent of the three major components of JTA graphite should be 43 wt% ZrB_2 , 13 wt% SiC, and 44 wt% C.



BEAM SCAN



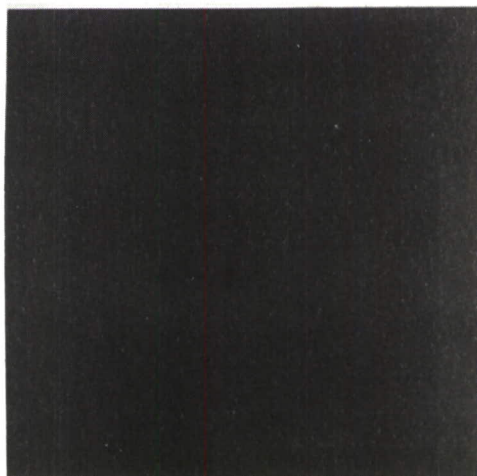
CARBON



ZIRCONIUM



SILICON



BORON

Fig. 12 - MICROPROBE ANALYSIS OF BILLET 7-F-12,
SECTION 3A

E. Density

Specification requirements are that the bulk density of the material to be delivered to IITRI be greater than 3.00 g/cc. Bulk density measurements confirm the uniformity shown by the radiographic and metallographic analyses. Random measurements of 20 samples from each billet show very little variation through the centers of each billet; 7-F-12 averages 3.063 g/cc, 7-F-14 averages 3.077 g/cc, and 14-G-1 averages 3.091 g/cc. See Table V for complete data.

IV. MECHANICAL PROPERTIES

A series of preliminary studies were made to: (1) determine reasonable sample sizes and shapes, and (2) determine the effect of oxidation on strength, under various pressures and times of exposure. The size effect experiments were performed to show if a specimen cross-section with a minimum dimension of 1/4 in. will have strengths which are typical of bulk material.

A. Size Effect Study

Experiments were performed at room temperature. The data were fitted to a statistical size effect analysis which provided the necessary information on reliability of results from specimens with three different cross-sectional areas.

1. Flexure Tests

Table VI lists the specimen size and the total number tested for this phase of the program. All flexure testing was done using a 4-point loading system with the load points at the end of the specimen and at the third points. The distance between the points was maintained at a 10:1 ratio with regard to the depth of the cross section. Surface finishes for these specimens averaged 20-50 rms for both grain orientations. In order to procure as much data as possible from these tests, sonic modulus data were obtained for all specimens tested. In addition, the deflection of each specimen under load was measured (Fig. 13) and the static Young's elastic modulus calculated. Eighteen of the 1/2 x 1/2 x 6 in. specimens were instrumented with strain gages, and both the longitudinal and lateral strains recorded under load for "with" and "across" grain orientations. Young's elastic modulus and Poisson's ratio can be calculated from this information. The data for each property measurement were correlated with the other properties for each specimen. The bending fixture is rigidly fastened to the moving head, and the specimen is supported on knife edges which are free to rotate in two directions; Fig. 14 shows this test setup.

Table V
SAMPLE DENSITIES (g/cc) THROUGH CENTER
OF JTA GRAPHITE BILLETS

| 14-G-1 | 7-F-14 | 7-F-12 |
|--------------------------|----------------|----------------|
| 3.093 | 3.040 | 3.082 |
| 3.065 | 3.071 | 3.112 |
| 3.067 | 3.118 | 3.082 |
| 3.103 | 3.052 | 3.082 |
| 3.102 | 3.063 | 3.082 |
| 3.073 | 3.048 | 3.107 |
| 3.116 | 3.053 | 3.112 |
| 3.088 | 3.042 | 3.076 |
| 3.141 | 3.103 | 3.082 |
| 3.114 | 3.085 | 3.132 |
| 3.074 | 3.101 | 2.982 |
| 3.067 | 3.072 | 3.032 |
| 3.116 | 3.103 | 3.047 |
| 3.083 | 3.083 | 3.077 |
| 3.086 | 3.115 | 2.954 |
| 3.072 | 3.085 | 2.984 |
| 3.091 | 3.057 | 3.041 |
| 3.065 | 3.068 | 3.087 |
| 3.117 | 3.095 | 3.079 |
| 3.088 | 3.082 | 3.090 |
| 3.091 Avg | 3.077 | 3.065 |
| <u>±</u> 0.021 Std. Dev. | <u>±</u> 0.023 | <u>±</u> 0.049 |

Table VI

DETERMINATION OF SIZE EFFECT OF JTA GRAPHITE

SPECIMENS AT ROOM TEMPERATURE

(Flexure Testing)

| Specimen Length, in. | Cross-Section Dimensions, in. | <u>No. of Specimens</u> | |
|----------------------------|-------------------------------------|-------------------------|-----------------|
| | | With Grain | Across Grain |
| 1 | 1/16 x 1/16 | 30 | 30 |
| 3 | 1/4 x 1/4 | 30 | 30 |
| 6 | 1/2 x 1/2 | 30 | 30 |

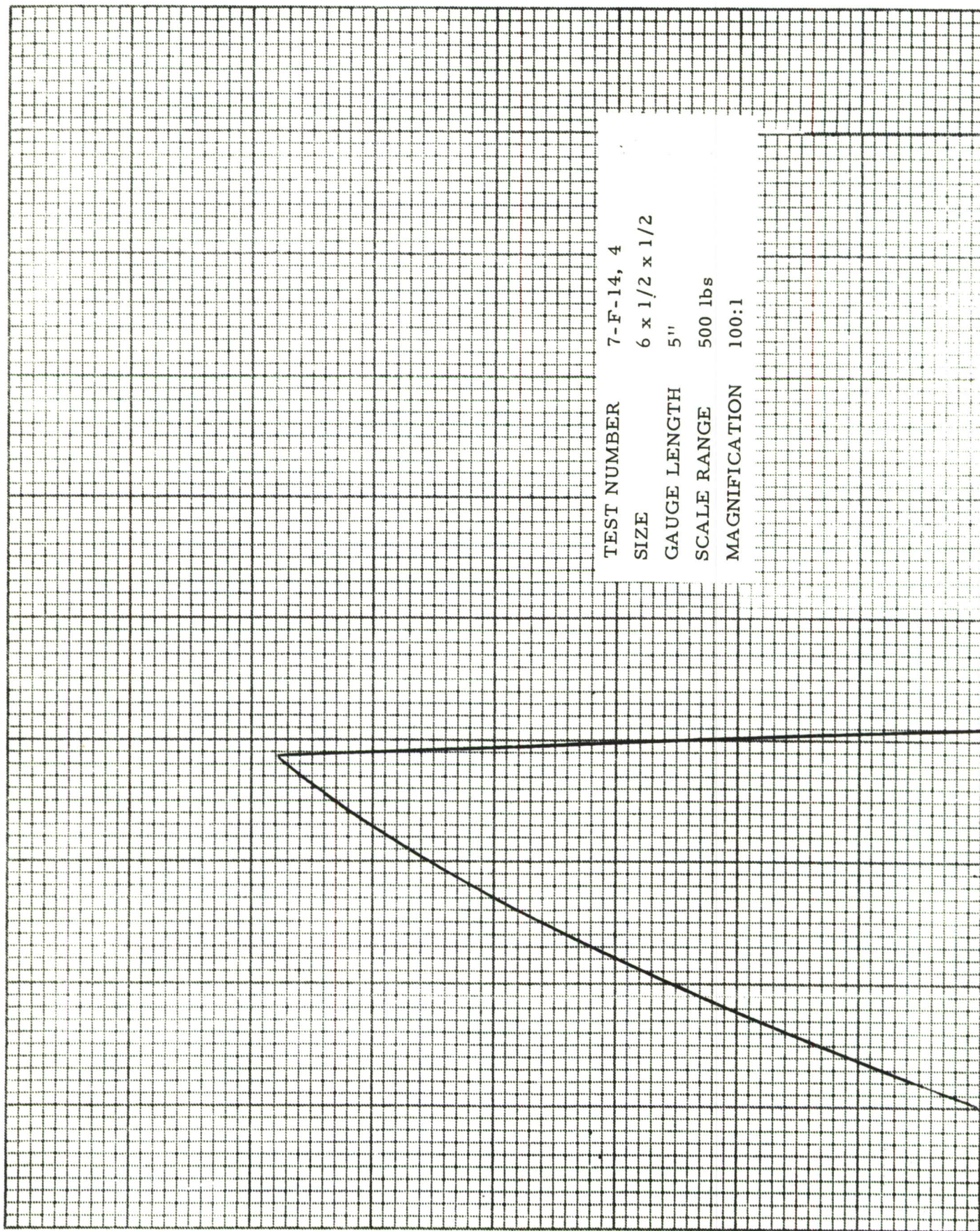


Fig. 13 - LOAD-DEFLECTION CURVE

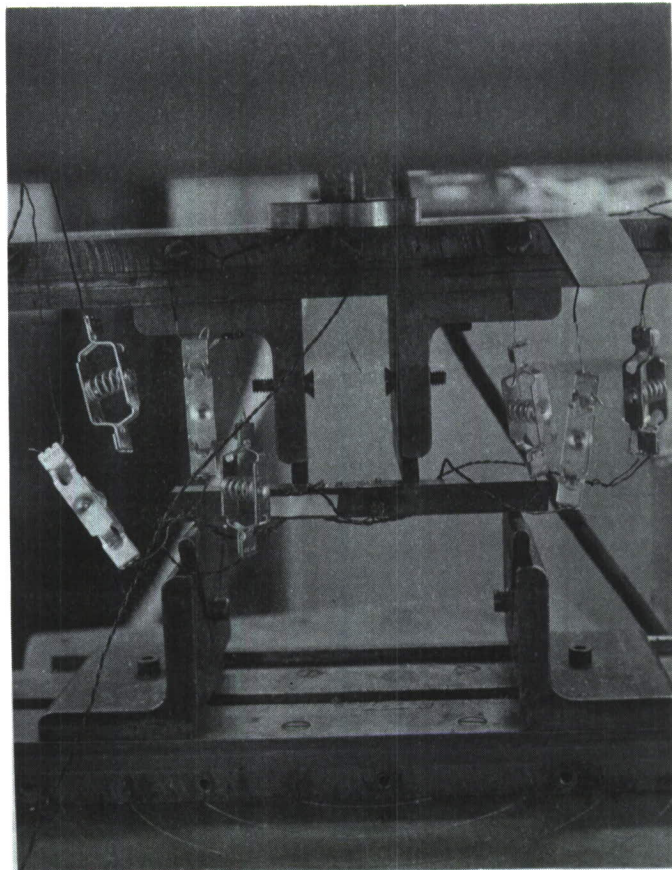


Fig. 14 - TEST SETUP FOR OBTAINING POISSON'S
RATIO WITH SR-4 STRAIN GAGES

Tables VII, VIII, and IX list the results obtained during the size effect experiments. In all cases, there is an apparent increase in strength with decreasing cross section except for the $1/16 \times 1/16 \times 1$ in. "across" grain specimens. These either exhibit about the same strength, or show a loss in strength compared to the $1/4 \times 1/4 \times 3$ in. or the $1/2 \times 1/2 \times 6$ in. specimens. In all cases, the $1/16 \times 1/16 \times 1$ in. specimen exhibited a large increase in variability. As previously shown, the graphite grain size averages about $120 \pm 30\mu$ (0.00485 in.) long and $70 \pm 15\mu$ (0.0027 in.) wide. The $1/16$ in. cross section reduces to 0.0625 in., which is approximately 13 times the average grain size. Thus, these experiments apparently verify the rule-of-thumb relationship that the depth of the beam must be greater than ten times the largest dimension of the grain in the cross section. Since the grain dimensions given are averages, it is easy to account for the large variability exhibited by the "across" grain specimens of $1/16$ in. cross-sectional area. Table X and Fig. 15 provide a summary analysis of the size effect data.

2. Tension Tests

Tensile strength is being measured using direct techniques. Preliminary experiments were made to determine whether the pin-type specimen (Fig. 16) offers any advantages over a collet grip specimen (Fig. 17). The latter test grips (Fig. 18) were developed on another IITRI program. Since JTA is a high-cost material, the preliminary experiments were made using a less costly graphite, POCO EP 1924. Table XI lists data obtained from these experiments. The literature strength values for the tensile strength of EP 1924 is 6000 psi. These experiments, while limited in scope, indicate that the collet specimens appear to provide more reliable data than the pin-specimen. On the basis of these tests the collet specimen was chosen for use in this program.

In order to study the critical size range and eliminate the problem due to fabricating small $1/16$ in. diameter gage sections, cross-section diameters of $1/8$, $3/16$ and $1/4$ in. were studied. Figure 19 is a plot of these data. The "with" grain plot exhibits a certain amount of inconsistency, and it is conceivable that the dotted line is a better average plot of their values than the solid curve line because of the small population groups used. The conclusion to be drawn from these data is that a $1/4$ in. cross section will provide an accurate representation of the JTA graphite strength in tension as well as flexure.

Table VII
EXPERIMENTAL DATA ON SIZE EFFECT FOR BILLET 7-F-14

| Specimen Cross-Section,* in. | Flexural Strength | | Tangent Modulus | | Sonic Modulus | |
|------------------------------------|-------------------|----------------------|-----------------|----------------------|---------------|----------------------|
| | Avg, psi | Coef. of Error, % | Avg, psi | Coef. of Error, % | Avg, psi | Coef. of Error, % |
| <u>With Grain</u> | | | | | | |
| 6 x 1/2 x 1/2 | 14,972 | 9.89 | 8.36 | 10.10 | 18.00 | 5.94 |
| 3 x 1/4 x 1/4 | 18,352 | 6.73 | 10.42 | 11.13 | 19.36 | 1.86 |
| 1 x 1/16 x 1/16 | 20,428 | 7.49 | --- | --- | 4.20 | 9.29 |
| <u>Across Grain</u> | | | | | | |
| 6 x 1/2 x 1/2 | 7,100 | 7.91 | 3.26 | 10.61 | 9.78 | 5.62 |
| 3 x 1/4 x 1/4 | 9,306 | 3.88 | 4.56 | 10.09 | 13.57 | 3.54 |
| 1 x 1/16 x 1/16 | 8,704 | 16.29 | --- | --- | 3.65 | 3.56 |

*10 specimens of each cross-section were tested.

Table VIII
EXPERIMENTAL DATA ON SIZE EFFECT FOR BILLET 7-F-12

| Specimen Cross-Section,* in. | Flexural Strength | | Tangent Modulus | | Sonic Modulus | |
|------------------------------------|-------------------|----------------------|-----------------|----------------------|---------------|----------------------|
| | Avg, psi | Coef. of Error, % | Avg, psi | Coef. of Error, % | Avg, psi | Coef. of Error, % |
| <u>With Grain</u> | | | | | | |
| 6 x 1/2 x 1/2 | 14,820 | 22.3 | 9.4 | 21.5 | 14.6 | 9.5 |
| 3 x 1/4 x 1/4 | 17,320 | 21.2 | 13.0 | 21.3 | 17.1 | 2.5 |
| 1 x 1/16 x 1/16 | 23,220 | 19.0 | 7.2 | 29.1 | 5.1 | 21.8 |
| <u>Across Grain</u> | | | | | | |
| 6 x 1/2 x 1/2 | 8,640 | 8.1 | 4.2 | 8.3 | 10.5 | 8.5 |
| 3 x 1/4 x 1/4 | 9,370 | 11.5 | 4.0 | 44.6 | 6.8 | 3.9 |
| 1 x 1/16 x 1/16 | 7,580 | 43.5 | 4.6 | 17.7 | 5.25 | 15.7 |

*10 specimens of each cross-section were tested.

Table IX
EXPERIMENTAL DATA ON SIZE EFFECT FOR BILLET 14-G-1

| Specimen Cross-Section,* in. | Flexural Strength | | Tangent Modulus | | Sonic Modulus | |
|------------------------------------|-------------------|----------------------|-----------------|----------------------|---------------|----------------------|
| | Avg, psi | Coef. of Error, % | Avg, psi | Coef. of Error, % | Avg, psi | Coef. of Error, % |
| <u>With Grain</u> | | | | | | |
| 6 x 1/2 x 1/2 | 15,818 | 15.99 | 9.32 | 14.37 | 15.30 | 9.67 |
| 3 x 1/4 x 1/4 | 17,880 | 13.23 | 9.83 | 13.22 | 18.31 | 4.15 |
| 1 x 1/16 x 1/16 | 24,190 | 11.58 | --- | --- | 4.58 | 10.26 |
| <u>Across Grain</u> | | | | | | |
| 6 x 1/2 x 1/2 | 8,800 | 5.32 | 4.13 | 8.29 | 10.50 | 7.00 |
| 3 x 1/4 x 1/4 | 8,160 | 2.92 | 4.99 | 10.62 | 15.47 | 3.04 |
| 1 x 1/16 x 1/16 | 9,045 | 22.54 | --- | --- | 3.72 | 7.80 |

*10 specimens of each cross-section were tested.

Table X
COMBINED SIZE EFFECT DATA

| Specimen Cross-Section,* in. | Flexural Strength | | Tangent Modulus | | Sonic Modulus | |
|------------------------------------|-------------------|----------------------|-----------------|----------------------|---------------|----------------------|
| | Avg, psi | Coef. of Error, % | Avg, psi | Coef. of Error, % | Avg, psi | Coef. of Error, % |
| <u>With Grain</u> | | | | | | |
| 6 x 1/2 x 1/2 | 15,203 | 14.37 | 9.26 | 16.63 | 15.97 | 12.52 |
| 3 x 1/4 x 1/4 | 17,850 | 14.87 | 11.08 | 21.14 | 18.26 | 5.46 |
| 1 x 1/16 x 1/16 | 22,613 | 15.59 | --- | --- | 4.63 | 17.71 |
| <u>Across Grain</u> | | | | | | |
| 6 x 1/2 x 1/2 | 8,180 | 11.77 | 3.86 | 14.22 | 10.26 | 7.89 |
| 3 x 1/4 x 1/4 | 8,945 | 9.73 | 4.52 | 26.06 | 11.95 | 30.79 |
| 1 x 1/16 x 1/16 | 8,443 | 29.18 | --- | --- | 4.21 | 21.31 |

*Total of 30 specimens for each cross-section were tested.

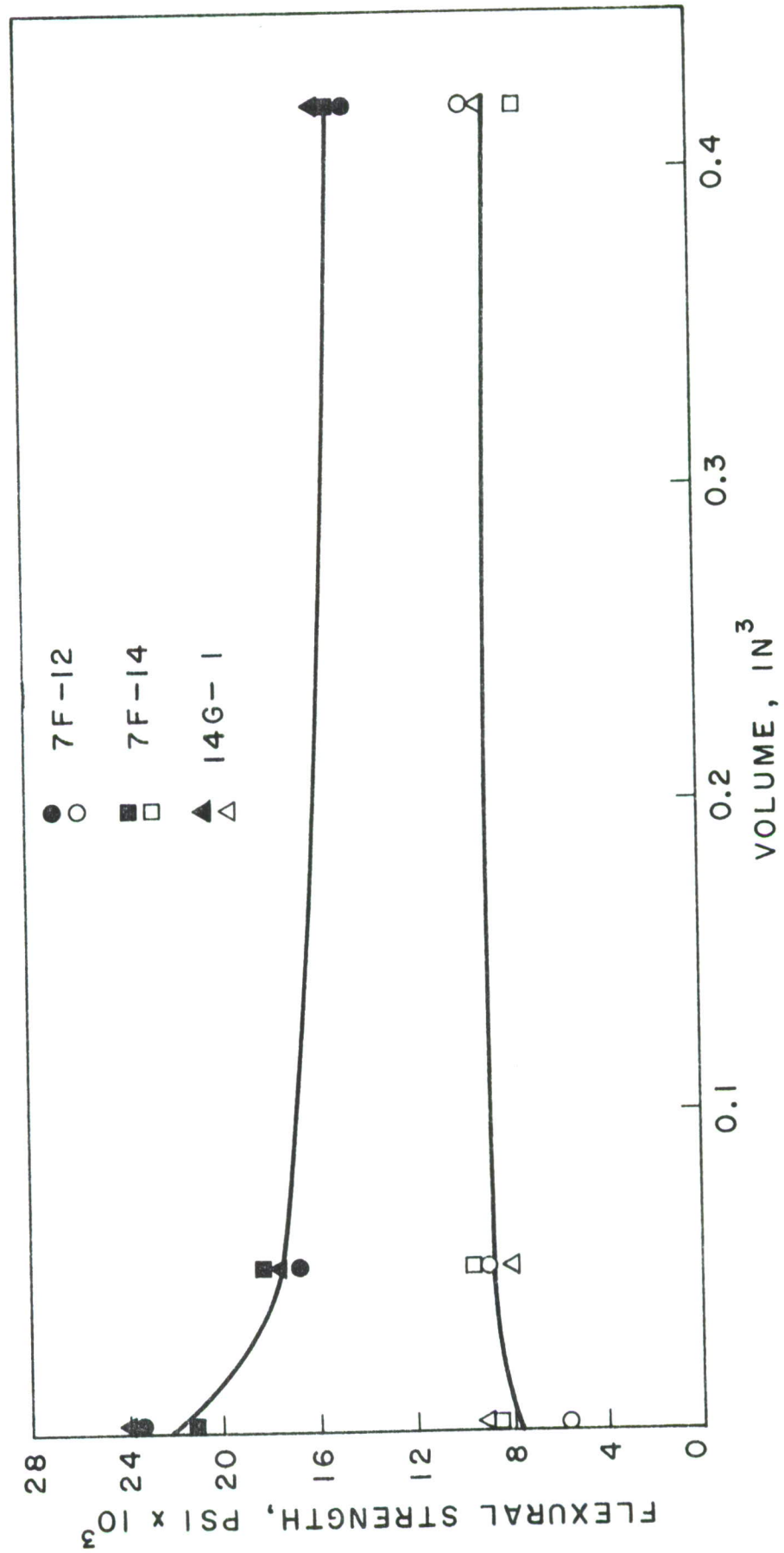


Fig. 15 - SIZE EFFECT ON FLEXURAL STRENGTH

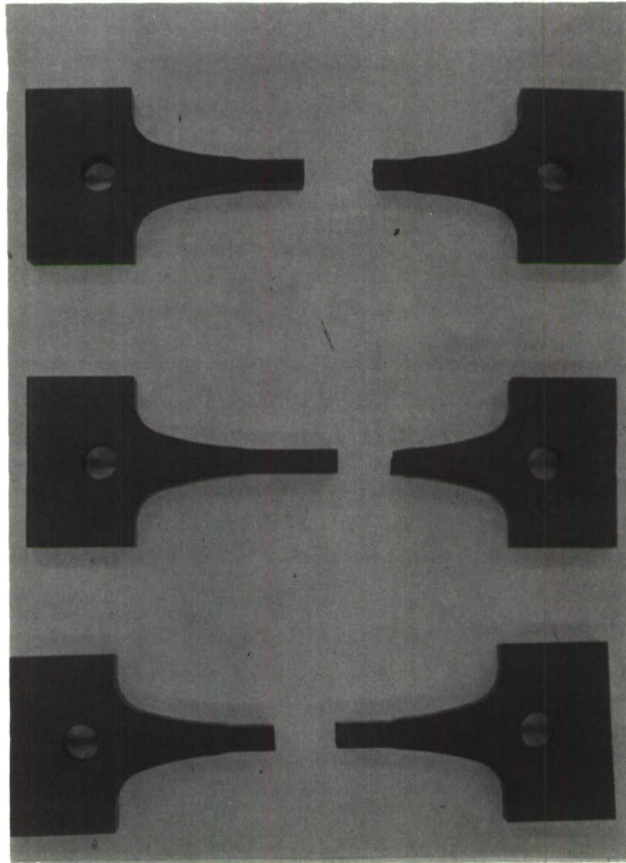


Fig. 16 - PIN-TYPE TENSION SPECIMEN
SHOWN WITH TYPICAL FRACTURES

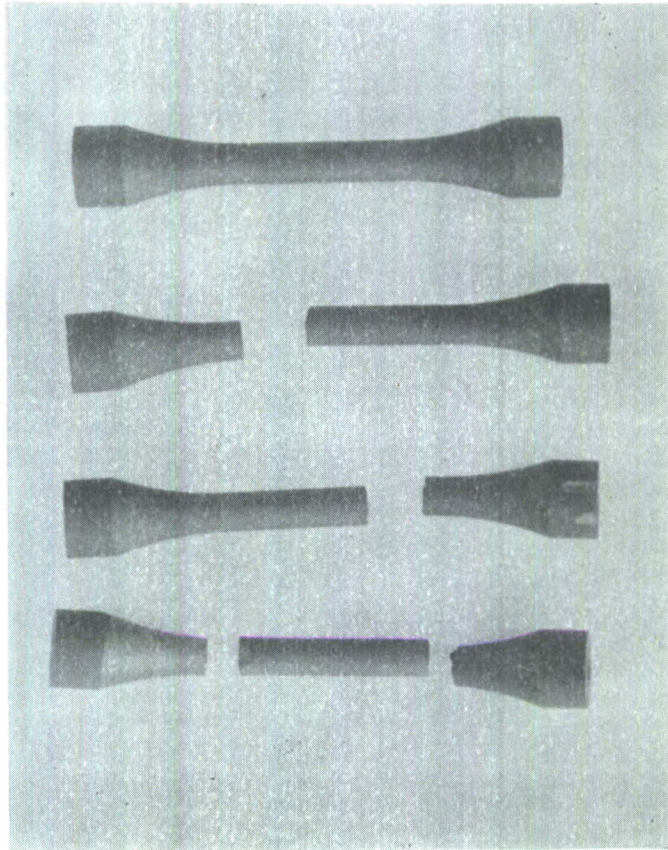


Fig. 17 - COLLET TENSION SPECIMEN
SHOWN WITH TYPICAL FRACTURES

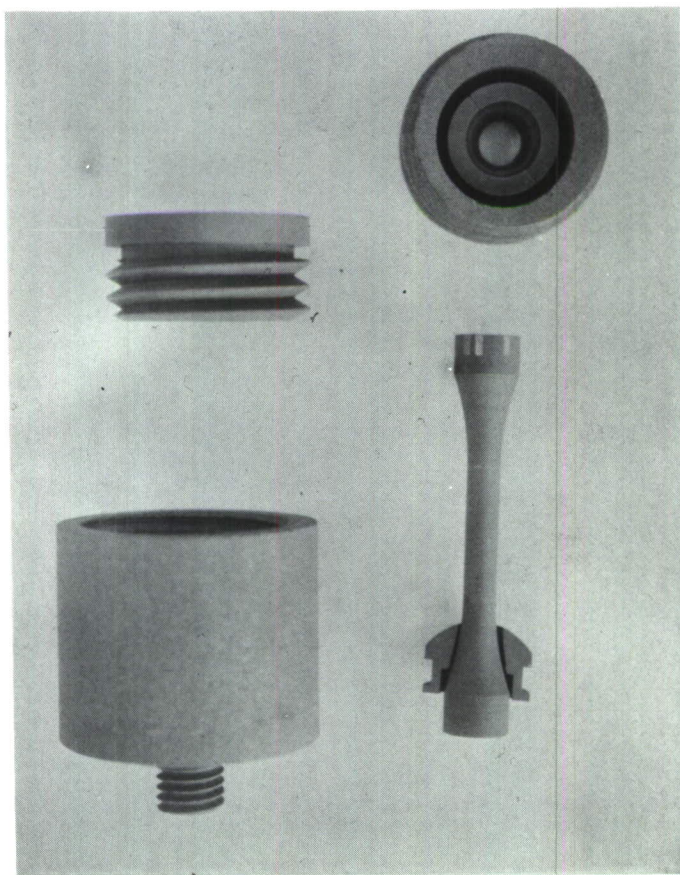


Fig. 18 - TENSION GRIPS DEVELOPED AT IITRI
TO BE USED WITH COLLET SAMPLES

Table XI
TENSILE STRENGTH OF POCC GRAPHITE
EP 1924

| Type of Tension Specimen | No. of Tests | Tensile Strength, psi | Coefficient of Variation, % |
|--------------------------|--------------|-----------------------|-----------------------------|
| Pin-type | 5 | 6480 | 19.5 |
| Collet type | 7 | 6140 | 14.2 |

Table XII
ROOM-TEMPERATURE ELASTIC PROPERTIES
OF JTA GRAPHITE

| Billet No. | Modulus in Flexure 10 ⁶ psi | | Poisson's Ratio in Flexure | |
|---------------------|---|-------------|----------------------------|----------------|
| | Tension | Compression | Tension | Compression |
| <u>With Grain</u> | | | | |
| 7-F-12 | 5.535 | 6.268 | .233* .082** | .309* .125* |
| 7-F-14 | 8.100 | 9.125 | .252 .079 | .321 .135 |
| <u>Across Grain</u> | | | | |
| 7-F-12 | 3.139 | 3.493 | 0.119 | 0.145 |
| 7-F-14 | 3.140 | 4.074 | 0.093 | 0.151 |
| 14-G-1 | 4.397 | 4.279 | 0.110 | 0.116 |

* Poisson's ratio determined in A direction.

** Poisson's ratio determined in C direction.

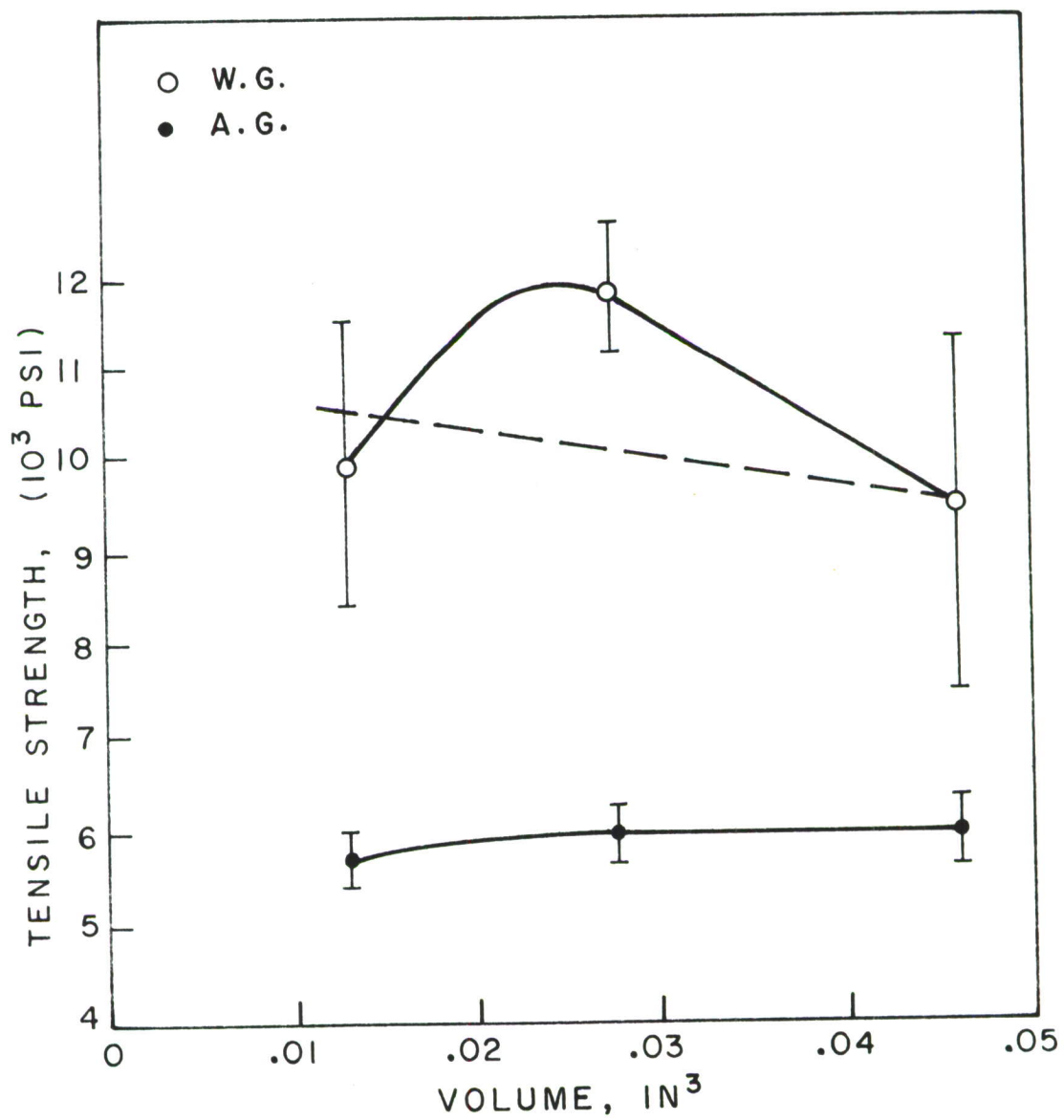


Fig. 19 - SIZE EFFECT ON TENSILE STRENGTH.

B. Elastic Properties

In order to obtain as much data as possible from the size effect experiments, SR-4 electric resistance gages were fastened to 5 specimens from each billet to determine Poisson's ratio. Two gages were applied to each of the four longitudinal faces; one measured the longitudinal strain and one the cross strain. Thus the elastic modulus and Poisson's ratio were measured for two orientations of "with" and "across" grain relationship. The test setup is shown in Fig. 14. Table XII presents the results of these experiments. It is obvious from the data that there are two Poisson's ratios which have to be considered, depending on the microstructure--one for the "A" direction and one for the "C" direction orientation; the first represents the broad face of the graphite crystallite and the second the edge of the crystallite. The "with" grain orientation will exhibit Poisson's ratio for both axes depending on how the sample is oriented for measurement.

In addition, the elastic properties in compression are higher, as expected, than that exhibited in tension. Thus, in order to obtain a true flexure strength the data must be corrected for the true position of the neutral axis of the bars.

Sonic elastic modulus measurements are generally higher than the static elastic modulus obtained from deflection or direct strain measurements. In order to verify the sonic modulus reported on this program, a "with" grain specimen of JTA graphite was obtained from Carbon Products Division of Union Carbide Corporation, and the values obtained using their equipment were compared to those obtained using IITRI equipment. Carbon Products obtained a value of $12.89 + 0.5 \times 10^6$ psi, and IITRI measured the sonic modulus as $13.66 + 0.4 \times 10^6$ psi--a difference of 6%. Considering that different equipment and techniques were used, the agreement between measurements is good and provides confidence in the reliability of the sonic measurements.

C. Evaluation of Flexure Strength Test Data

The failure distribution for the 1/4 in. cross section flexure strength data is plotted in Figs. 20 and 21. An analysis of variance shows that all the strength data come from the same population (see Appendix) at the 95% confidence level. However, examination of the curves reveals several factors of additional interest.

A rank sum test⁽⁶⁾ comparing the three sample populations reveals that while the "with" grain orientation data can be considered from the same material population, the "across" grain populations are not the same. However, the narrow failure distributions of the "across" grain population have average strengths

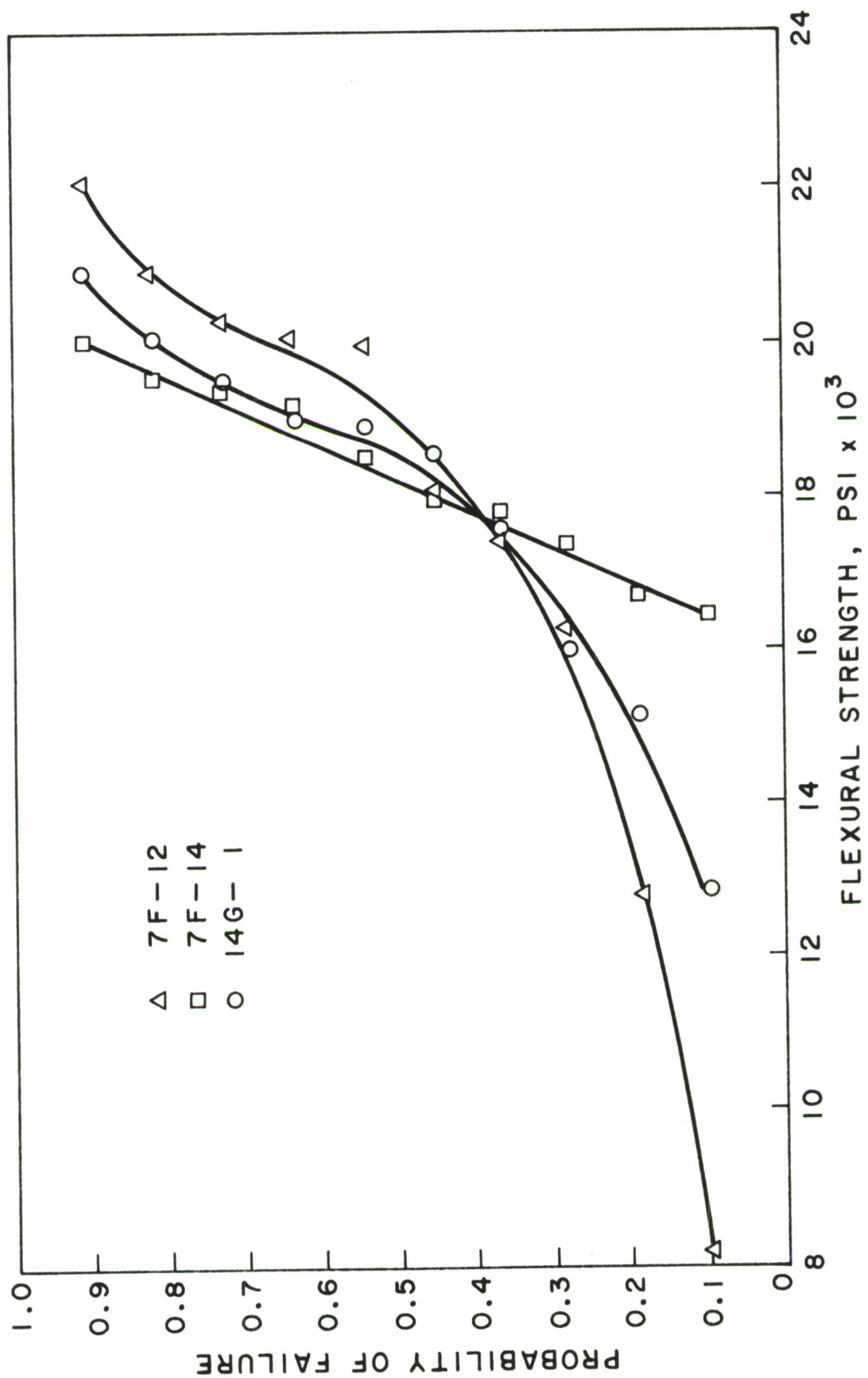


Fig. 20 - FLEXURAL STRENGTH DISTRIBUTION CURVES, "WITH" GRAIN ORIENTATION

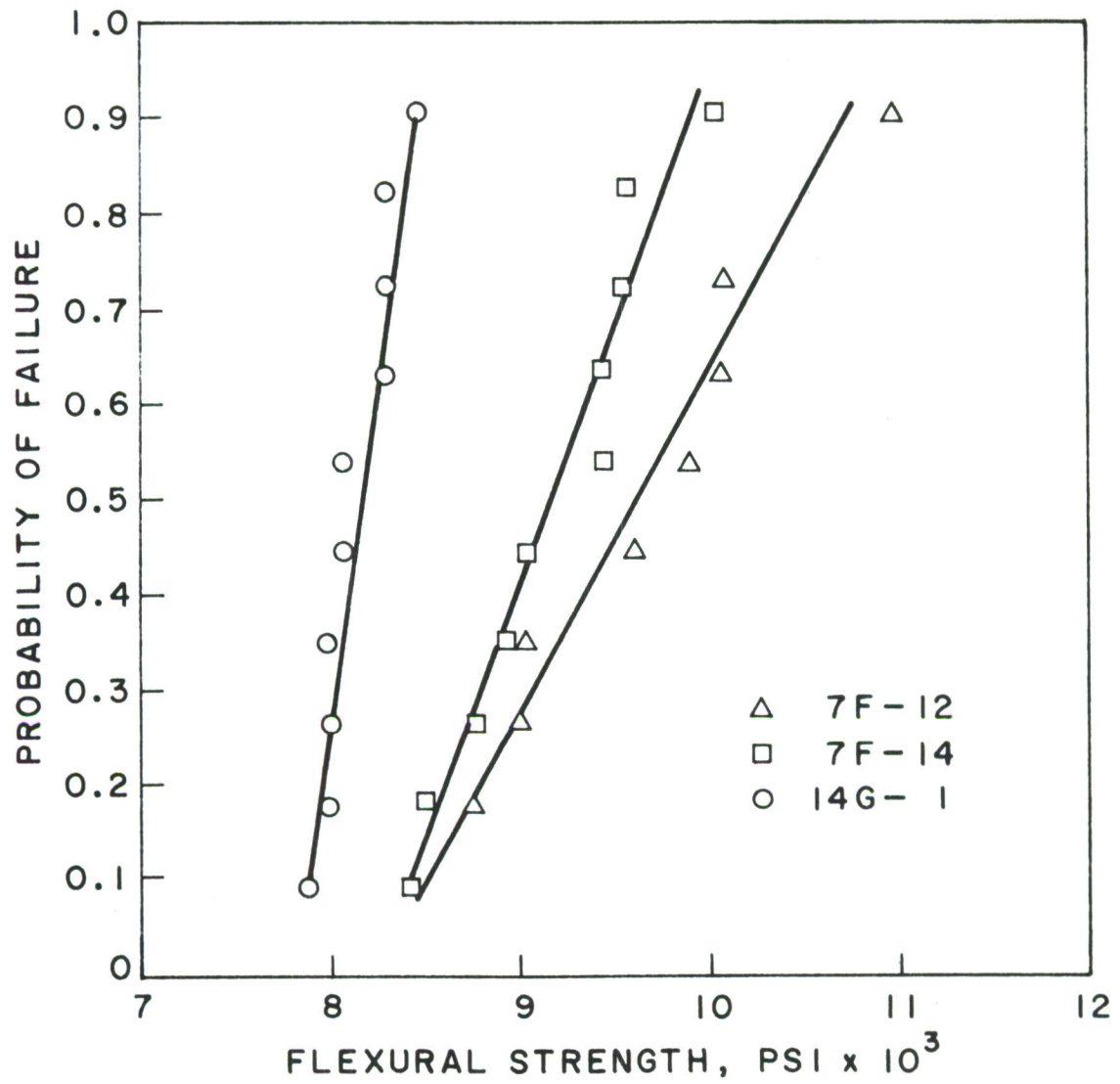


Fig. 21 - FLEXURAL STRENGTH DISTRIBUTION CURVES, "ACROSS" GRAIN

which are very nearly the same. Thus, while the two statistical analyses are partially contradictory, the test data of the three billets are considered as part of the same data population and the strengths of these billets are the same.

The failure modes of the "with" grain and "across" grain orientation appear to be different. The probability-of-fracture curves for "with" grain orientation are typical for brittle materials, in that they are S-shaped while the "across" grains are straight lines. The S-curves for brittle materials normally have a skew, bell-shaped distribution with a wide strength variation biased in the direction of low strength. The straight lines of the "across" grain plots are indicative of a square failure distribution curve of small variability.

If data from a previous program are added for analysis, a similar trend is evident. The anisotropy due to average crystallite orientation not only affects the strength, elastic, and thermal properties, but also affects the failure mode for JTA. A review of the available literature at IITRI has failed to reveal a similar analysis for any of the graphite materials. It may be that all graphites show this phenomenon. This behavior can probably be traced to the different bonding mechanisms of the graphite crystals, weak van der Waals forces for the average "across" grain orientation, and much stronger ionic bonding for the "with" grain orientation.

Combined failure strength curves are shown in Figs. 22 and 23. There is one data point for the "across" grain plot which is out of line with the rest of the data. A statistical analysis indicates that this point can be dropped as nonrepresentative of the material strength. It is conceivable that damage was introduced into this specimen during cutting. A similar analysis for the "with" grain specimen does not show these extraneous data points.

From a design point of view, the "across" grain data appear to be deterministic while the "with" grain data exhibit "statistical" behavior similar to oxide ceramics. As the rest of the mechanical property results become available, it will be interesting to observe if the data continue to group in the manner exhibited by the flexure data.

D. Oxidation Effects

JTA graphite was developed for use at high temperature under oxidizing conditions. Mechanical data obtained by standard techniques of testing in inert atmospheres will be correlated with those performed in air. First, a series of experiments are being conducted to determine the effect of oxidation on the strength of JTA. Flexure tests are being employed similar to the size effect experiments.

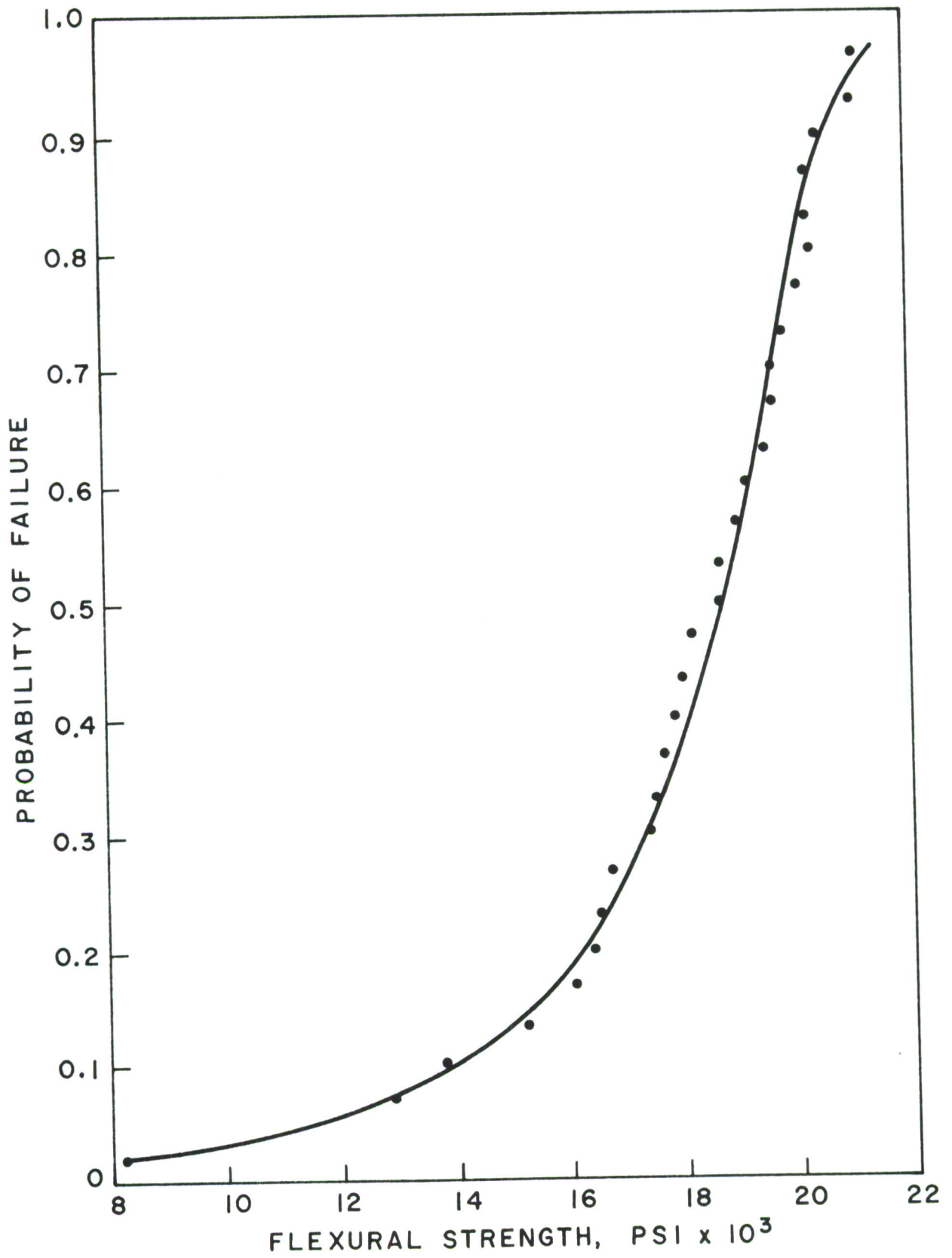


Fig. 22 - COMBINED FLEXURAL STRENGTH DISTRIBUTION CURVE,
WITH GRAIN

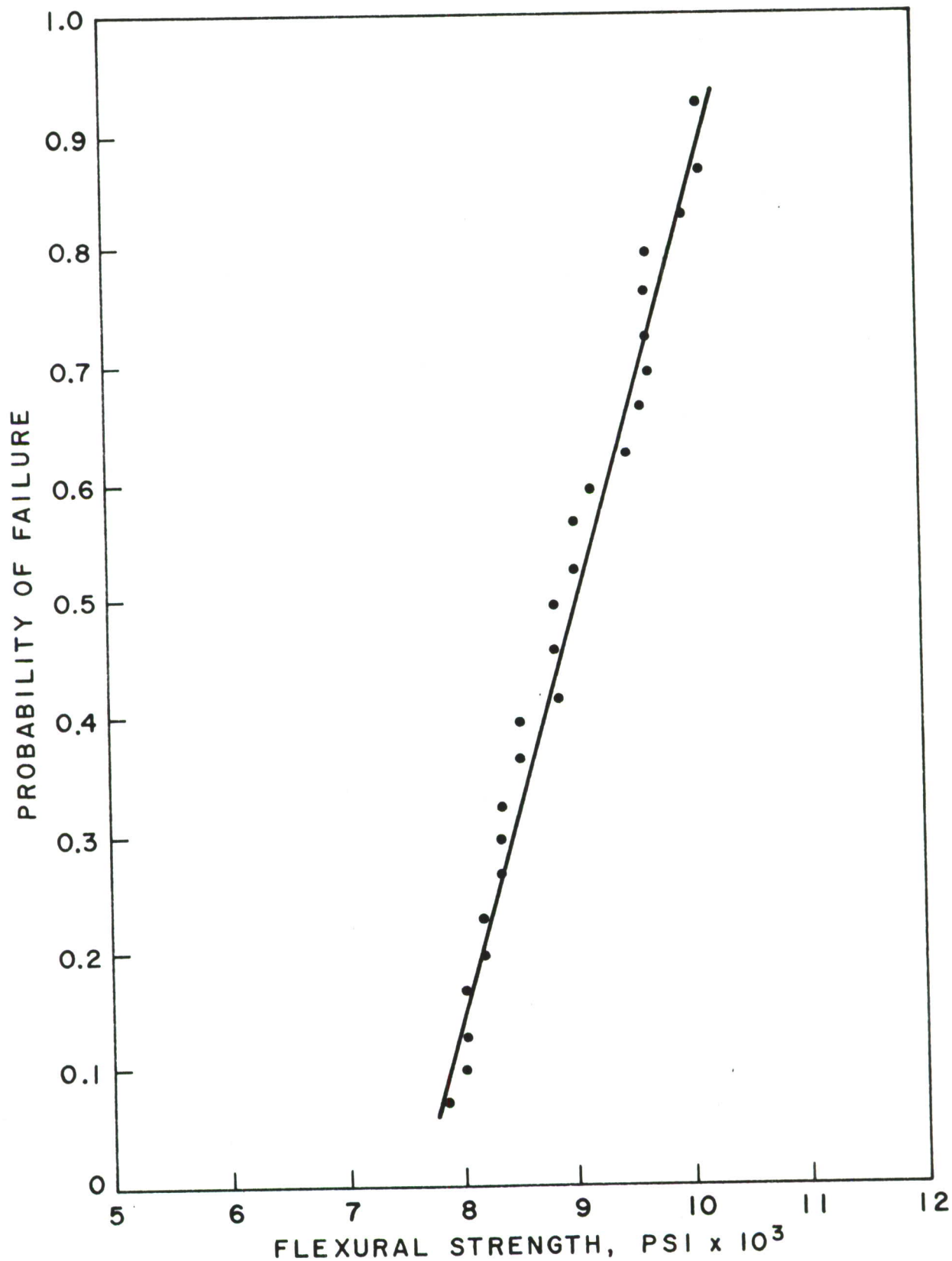


Fig. 23 - COMBINED FLEXURAL STRENGTH DISTRIBUTION CURVE, ACROSS GRAIN.

Specimens have been exposed to three time periods, at temperatures of 1000°, 1750°, and 2000°C for the following pressures: 1×10^{-5} Torr, 1×10^{-1} Torr and atmospheric. Three specimens are tested for each condition of air pressure and temperature.

The data from these experiments will be analyzed with flexural data obtained from tests in an inert atmosphere. After analysis, a similar series of tests will be conducted for tension and compressive strengths under oxidizing conditions (see Table XIII), and the data will be compared to results from inert atmosphere tests. These tests should enable the translation of mechanical property data taken in the inert environment to that from the oxidizing conditions; the depth or amount of oxidation, the nature of the oxidized material, the rate at which oxidation progressed, and the effect on strength will be determined.

Initial oxidation experiments have been initiated. A series of specimens have been exposed to static air at 1000°C, 10^{-5} Torr pressure and atmospheric pressure, and at 2000°C and 10^{-5} Torr pressure. Table XIV lists the strength obtained from room-temperature flexure tests after exposure. The material exposed at 1000°C in static air lost about 20% of its "with" grain strength in the first 20 minutes and retained the remainder through the next 40 minutes.

The "across" grain showed similar behavior; however, the strength loss was somewhat less, about 15%. These values are only tentative since more specimens must be evaluated before any final conclusions can be drawn.

The material tested at 2000°C in a vacuum exhibited little or no strength change: 5% for the "across" and 2% for the "with" grain orientation. This was expected since there was little or no air to effect oxidation changes.

Figures 24, 25, and 26 show weight changes that occurred during these experiments. The material exposed to static air at atmospheric pressure and 1000°C lost weight during the first 20 minutes, gained it back in the second 20 minutes, and then began to lose it again during the last 20 minutes. The curve for weight change at 1000°C at 10^{-5} Torr atmospheric pressure of static air is similar, except that the weight loss is about one order of magnitude less for the same time interval.

The material tested in vacuum at 2000°C only indicates loss of volatiles due to heating which should taper off after a certain amount of exposure. Under highly oxidizing conditions, both the volatiles and graphite are being lost, constituting a

Table XIII
OXIDATION TESTS OF JTA GRAPHITE

| Property | Crystal Orientation | No. of Specimens at Temp. (°C) | | |
|-------------|---------------------|--------------------------------|------|------|
| | | 1000 | 1750 | 2000 |
| Tension | With grain | 10 | 10 | 10 |
| | Against grain | 10 | 10 | 10 |
| Flexure | With grain | 10 | 10 | 10 |
| | Against grain | 10 | 10 | 10 |
| Compression | With grain | 10 | 10 | 10 |
| | Against grain | 10 | 10 | 10 |

Table XIV
OXIDATION EXPERIMENTS ON JTA 7-F-12 BILLET
(1/4 x 1/4 x 3 in. flexural specimens)

| Grain Orientation | Avg Strength (at RT), psi | Time of Exposure, min | Flexure Strength, psi | Strength Change, % |
|--|---------------------------------|-----------------------------|-----------------------------|--------------------------|
| <u>Static Air at 1000°C</u> | | | | |
| Across | 8,945 | 20 | 8,060 | -9.9 |
| | | 40 | 6,400 | -28.2 |
| | | 60 | 7,040 | -21.7 |
| With | 17,850 | 20 | 13,950 | -21.8 |
| | | 40 | 16,900 | -5.3 |
| | | 60 | 14,450 | -19.1 |
| <u>Static Air at 10⁻⁵ Torr and 2000°C</u> | | | | |
| Across | 8,945 | 20 | 8,830 | -1.3 |
| | | 40 | 7,420 | -17.0 |
| | | 60 | 9,220 | +3.0 |
| With | 17,850 | 20 | 17,540 | -1.7 |
| | | 40 | 17,020 | -4.6 |
| | | 60 | 18,820 | -0.2 |

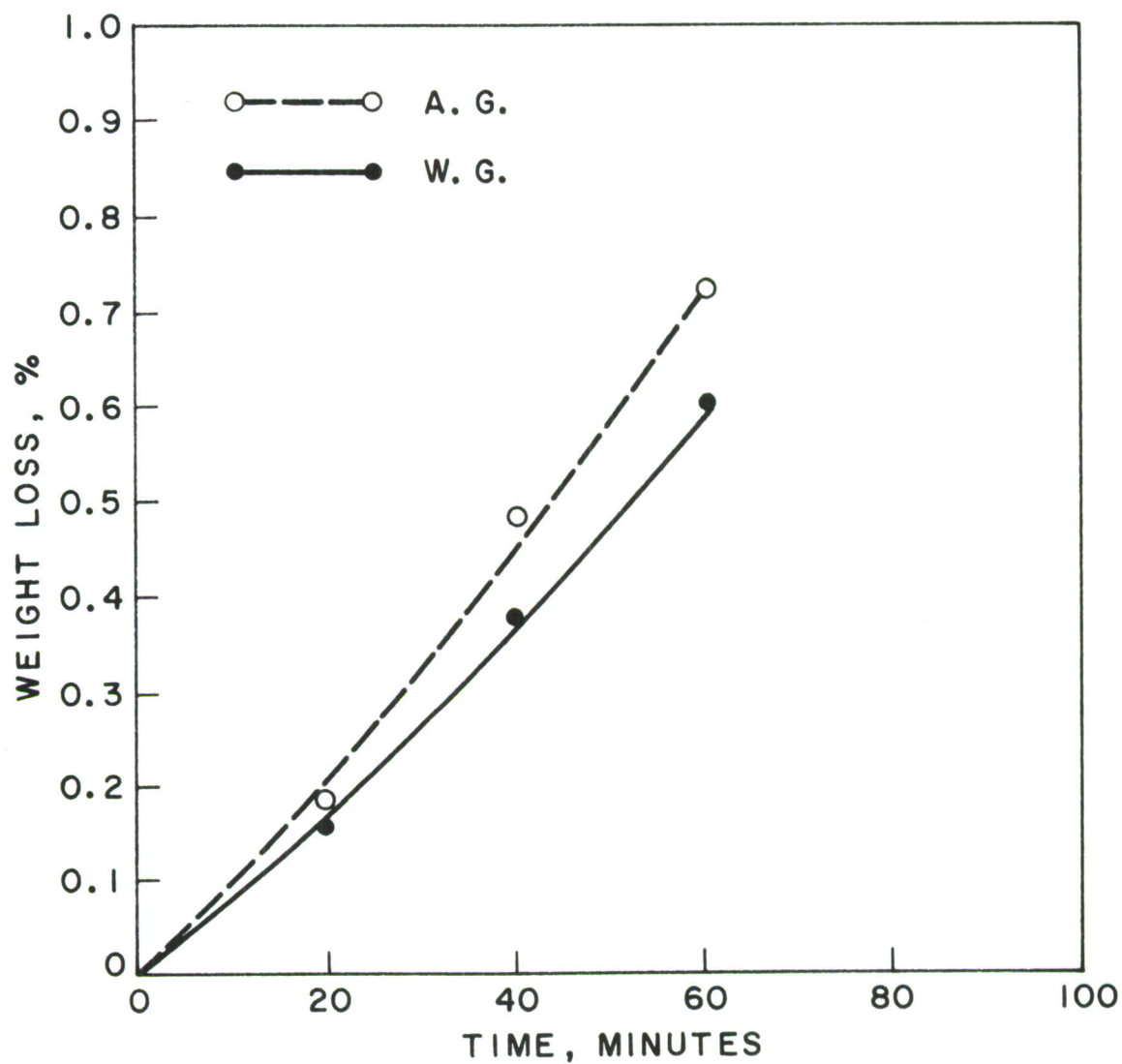


Fig. 24 - WEIGHT CHANGE ON EXPOSURE TO STATIC AIR AT 2000°C AND 10⁻⁵ TORR PRESSURE, BILLET 7-F-12.

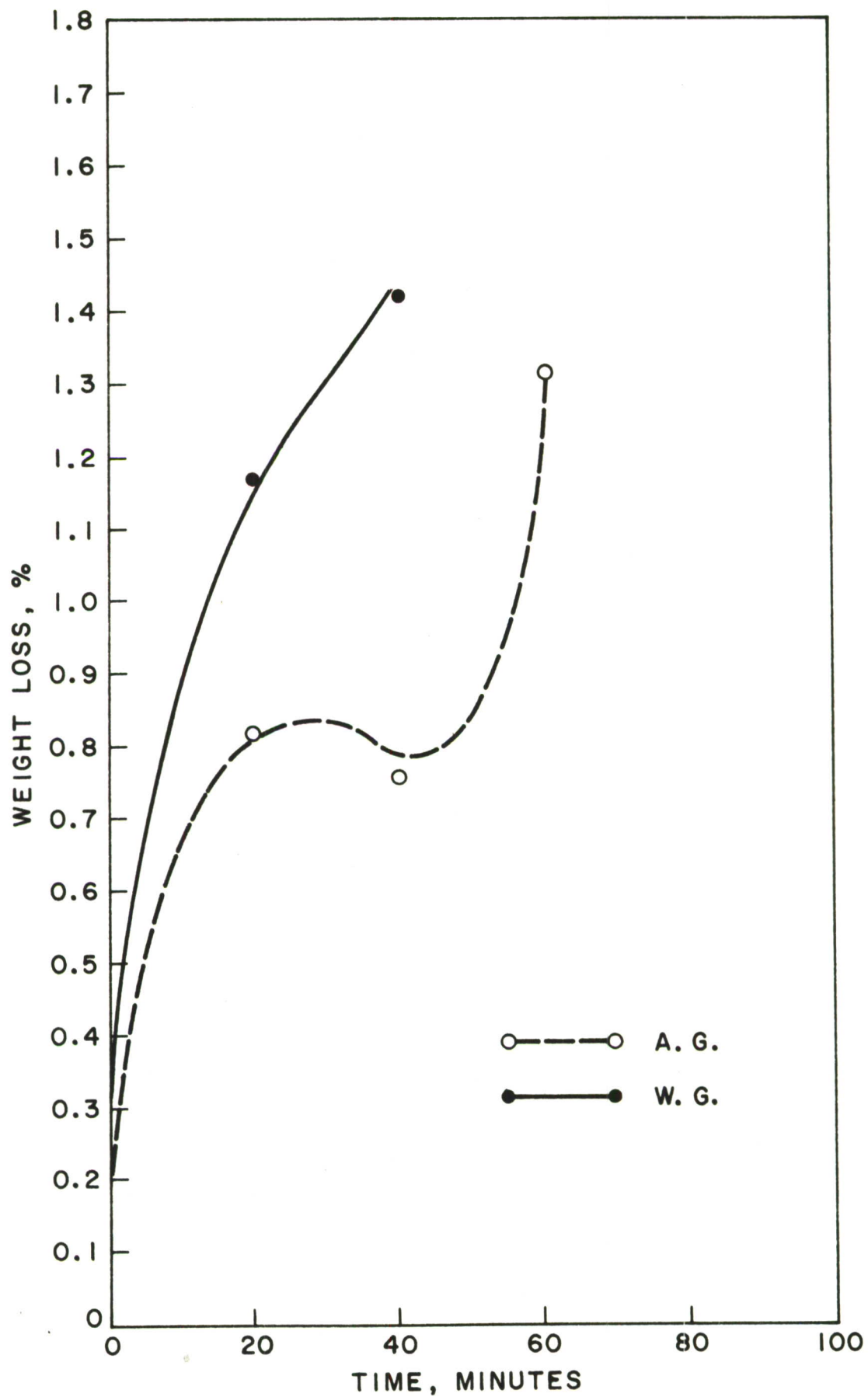


Fig. 25 - WEIGHT CHANGE ON EXPOSURE TO STATIC AIR AT 1000°C AND ATMOSPHERIC PRESSURE, BILLET 7-F-12.

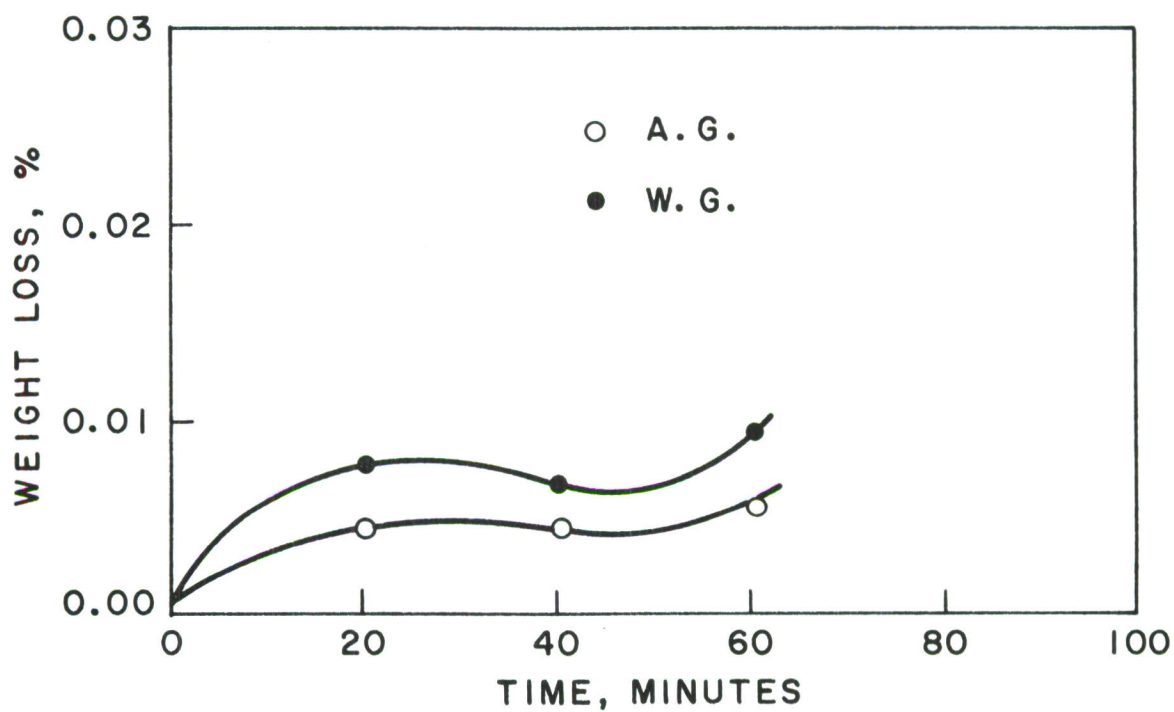


Fig. 26 - WEIGHT CHANGE ON EXPOSURE TO STATIC AIR AT 1000°C AND 10⁻⁵ TORR PRESSURE, BILLET 7-F-12

weight loss; however, as more and more of the zirconium diboride and silicon carbide are exposed, an oxide coating is formed that would reduce the graphite loss and thus result in a net weight increase. Once the coating has formed, a net weight loss would be observed due to diffusion of oxygen through the protective coating and the loss of graphite as CO and CO₂.

Metallographic analysis of the oxidized surface shows a certain amount of surface attack for the air-exposed specimen and less attack for the material tested in a partial vacuum, Figs. 27 and 28. However, the photomicrographs are inconclusive and a chemical analysis is being made to determine what new products are formed at the surface under oxidizing conditions.

V. SPECIFIC HEAT

Enthalpies of the three JTA graphites were measured from room temperature to 4000°F (2225°C) using the drop-calorimeter technique. A sample of each material was heated to a desired temperature in a furnace and then dropped into a modified Parr calorimeter.

Three drops were made at each temperature level for each material in order to assure the accuracy and the reproducibility of the results. The process was repeated at successively higher temperatures at intervals of approximately 200°F (110°C).

The enthalpy of the material was calculated from the experimental measurements using the following equation:

$$\Delta H = \frac{W_e (t_f - t_i)}{M_s}$$

where W_e = water equivalent, receiver, and weight water contained in bucket, in Btu/°F

t_f, t_i = final and initial calorimeter temperatures, respectively, as measured by the calorimeter thermometer, in °F

M_s = mass of sample, in pounds

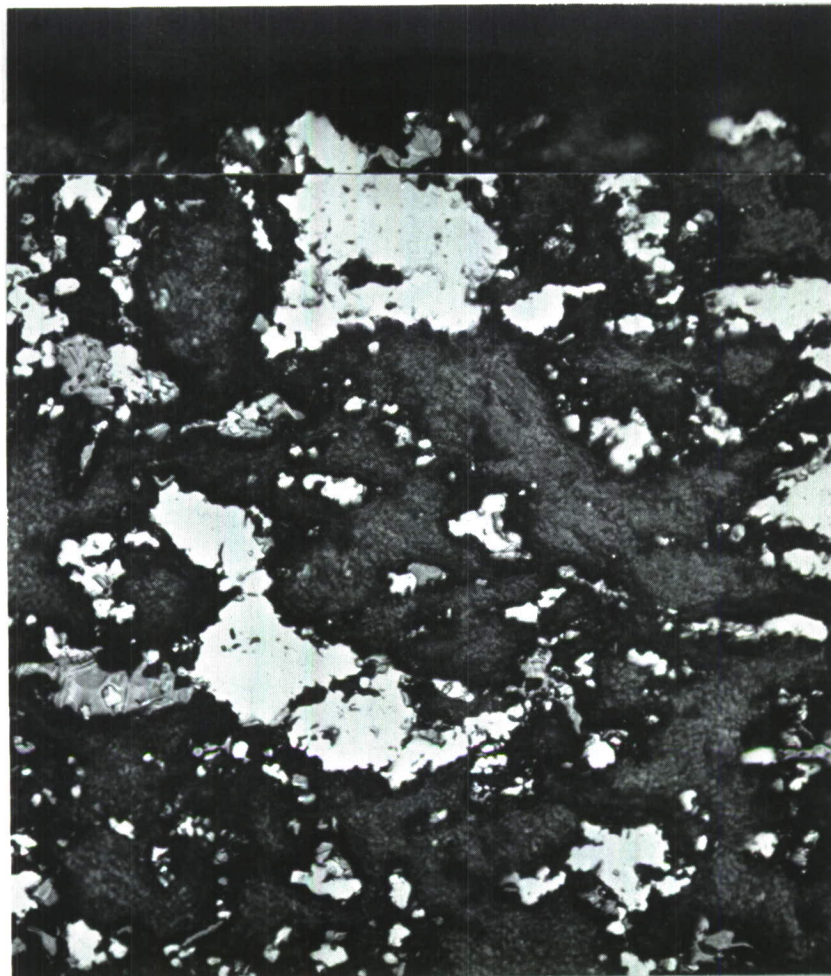
From the enthalpy values, the enthalpy curve vs. temperature was obtained. An equation was fitted to this enthalpy curve, and specific heat was determined by differentiating it with respect to temperature at constant pressure.

$$C_p = \left(\frac{\partial H}{\partial t} \right)$$

where C_p = specific heat at constant pressure

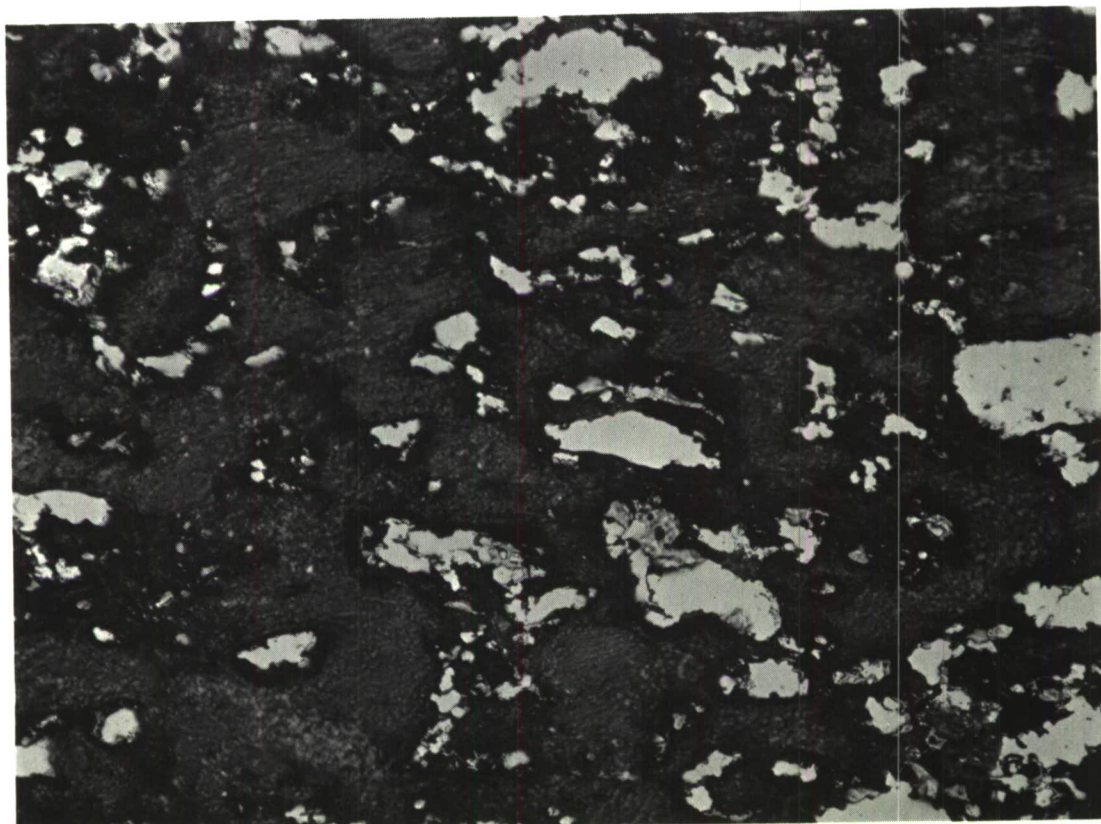
H = enthalpy

t = temperature



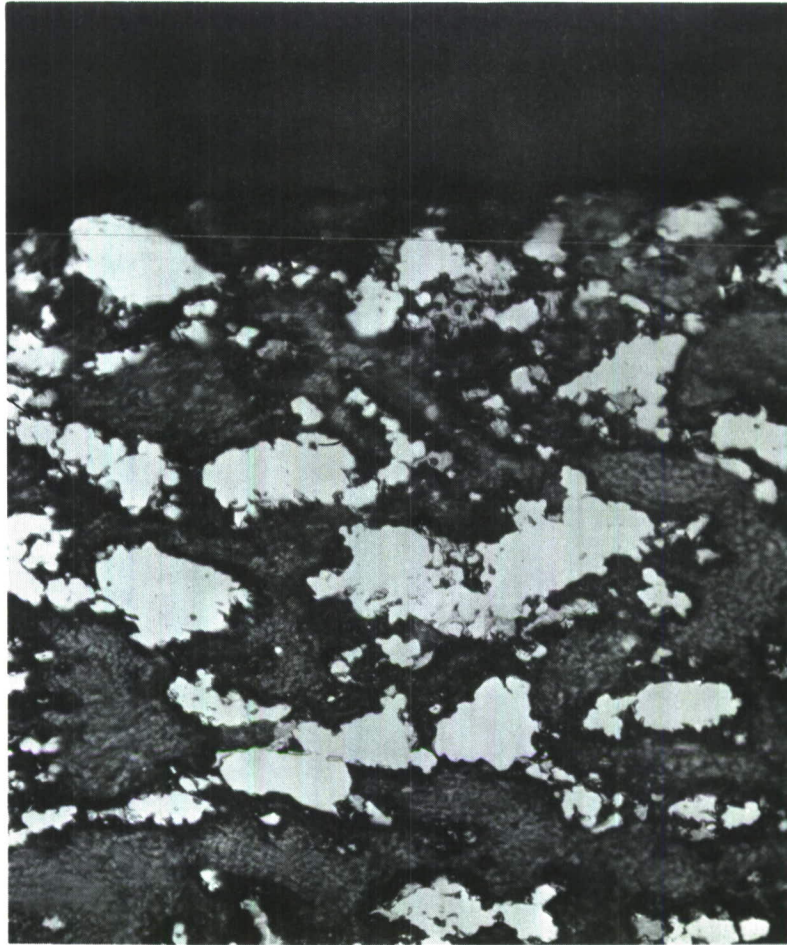
EXTERIOR EDGE

Fig. 27a - PHOTOMICROGRAPHS OF JTA OXIDIZED IN AIR
AT 2000°C AND 5×10^{-5} TORR PRESSURE FOR 1 HR (320X)



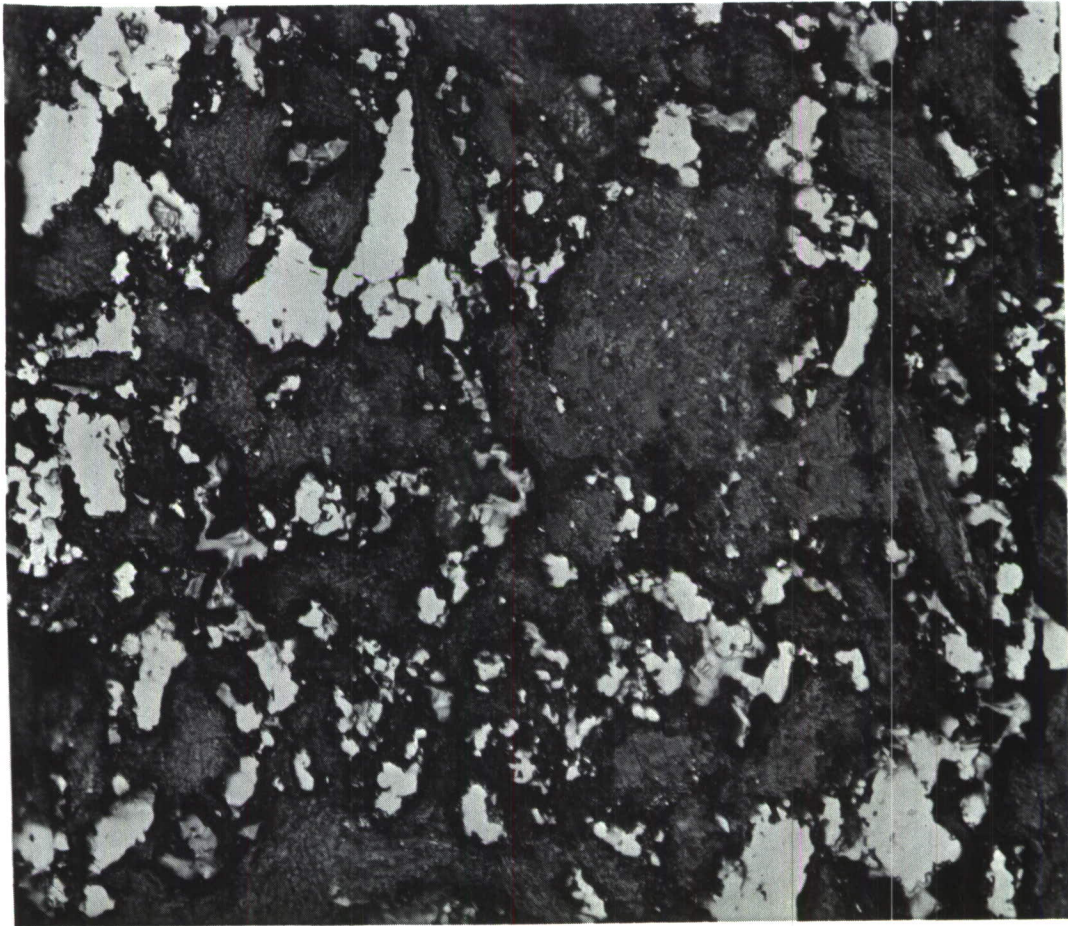
INTERIOR EDGE

Fig. 27b - PHOTOMICROGRAPH OF JTA OXIDIZED IN AIR
AT 2000°C AND 5×10^{-5} TORR PRESSURE FOR 1 HR (320 X)



EXTERIOR EDGE

Fig. 28a - PHOTOMICROGRAPH OF JTA OXIDIZED IN AIR
AT 1000°C AND 1 ATMOSPHERE FOR 1 HR (320X)



INTERIOR EDGE

Fig. 28b - PHOTOMICROGRAPH OF JTA OXIDIZED IN AIR
AT 1000°C AND 1 ATMOSPHERE FOR 1 HR (320X)

The enthalpies of the three materials as functions of temperature are presented in Tables XV through XVII and in Fig. 29.

Since there was no significant difference between enthalpy values of all three JTA graphites, one function for specific heat versus temperature was developed for all three materials. This is shown in Table XVIII and Fig. 30.

VI. REFERENCES

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Table XV
ENTHALPY VALUES FOR JTA GRAPHITE (7-F-14)

| Temperature, °F | ΔH , Btu/lb |
|-----------------|---------------------|
| 80 | 0 |
| 215 | 21 |
| 426 | 60 |
| 630 | 105 |
| 784 | 145 |
| 973 | 196 |
| 1154 | 245 |
| 1342 | 300 |
| 1510 | 350 |
| 1750 | 425 |
| 1912 | 477 |
| 2143 | 560 |
| 2297 | 605 |
| 2510 | 685 |
| 2723 | 774 |
| 2905 | 845 |
| 3120 | 920 |
| 3296 | 1000 |
| 3471 | 1080 |
| 3652 | 1147 |
| 3789 | 1232 |
| 3973 | 1310 |

Table ~~XVI~~
ENTHALPY VALUES FOR JTA GRAPHITE (7-F-12)

| Temperature, °F | ΔH , Btu/lb |
|-----------------|---------------------|
| 80 | 0 |
| 178 | 15 |
| 362 | 48 |
| 583 | 95 |
| 730 | 130 |
| 912 | 180 |
| 1098 | 230 |
| 1306 | 293 |
| 1470 | 338 |
| 1620 | 385 |
| 1835 | 452 |
| 2057 | 528 |
| 2210 | 579 |
| 2445 | 665 |
| 2681 | 750 |
| 2901 | 843 |
| 3124 | 925 |
| 3305 | 1003 |
| 3497 | 1086 |
| 3670 | 1162 |
| 3809 | 1230 |
| 3962 | 1315 |

Table XVII
ENTHALPY VALUES FOR JTA GRAPHITE (14-G-1)

| Temperature, °F | ΔH , Btu/lb |
|-----------------|---------------------|
| 80 | 0 |
| 212 | 20 |
| 415 | 58 |
| 620 | 103 |
| 783 | 143 |
| 963 | 192 |
| 1140 | 241 |
| 1315 | 290 |
| 1508 | 350 |
| 1690 | 404 |
| 1903 | 475 |
| 2160 | 560 |
| 2345 | 627 |
| 2516 | 690 |
| 2749 | 782 |
| 2910 | 841 |
| 3117 | 922 |
| 3315 | 1005 |
| 3491 | 1080 |
| 3663 | 1154 |
| 3796 | 1221 |
| 3971 | 1312 |

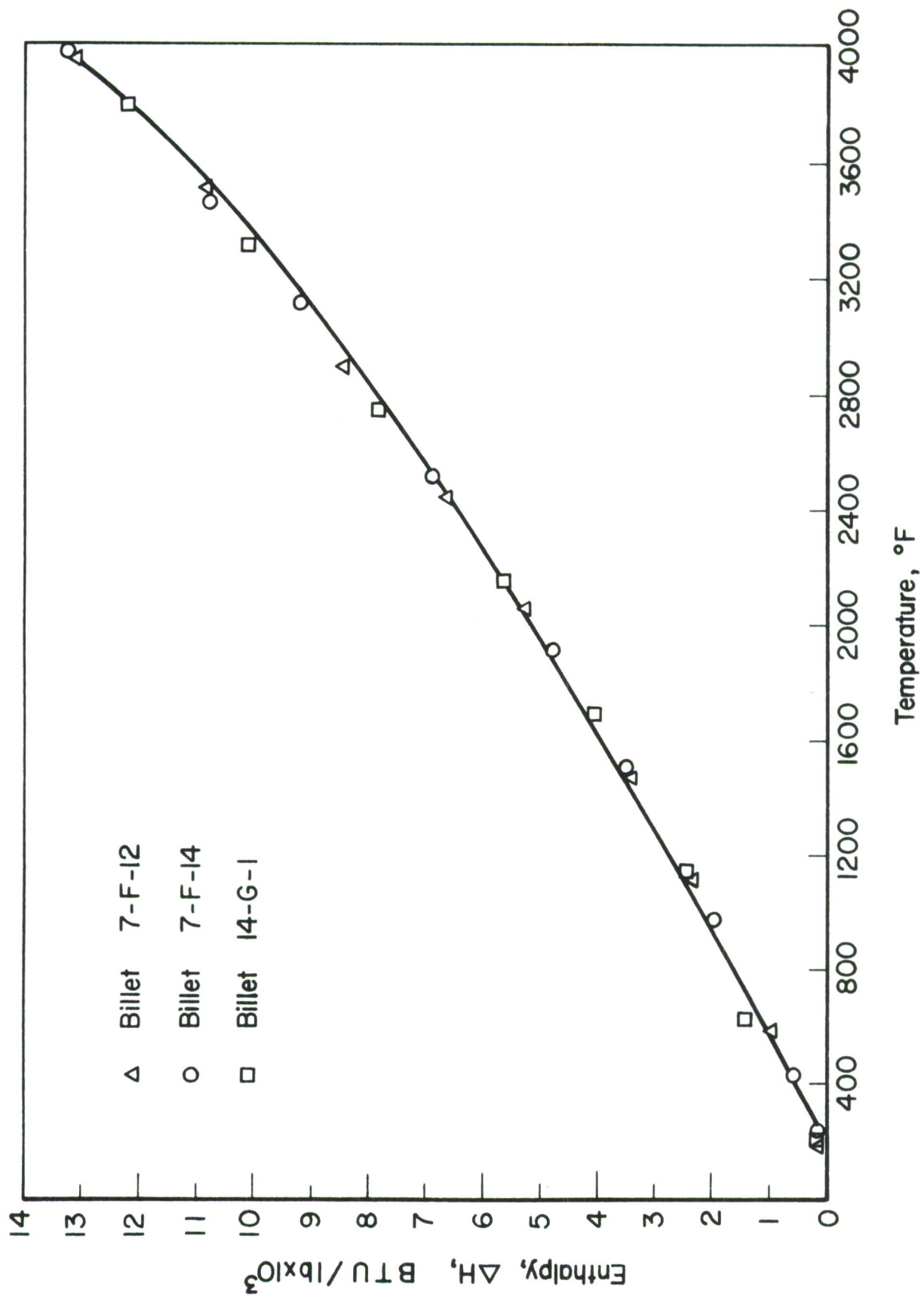


Fig. 29 - ENTHALPY OF JTA VS. TEMPERATURE

Table XVIII
SPECIFIC HEAT OF JTA GRAPHITE SPECIMENS
 (7-F-14, 7-F-12, 14-G-1)

| Temperature, °F | C _p , Btu/lb °F |
|-----------------|----------------------------|
| 500 | 0.220 |
| 1000 | 0.270 |
| 1500 | 0.310 |
| 2000 | 0.325 |
| 2500 | 0.360 |
| 3000 | 0.390 |
| 3500 | 0.430 |
| 4000 | 0.470 |

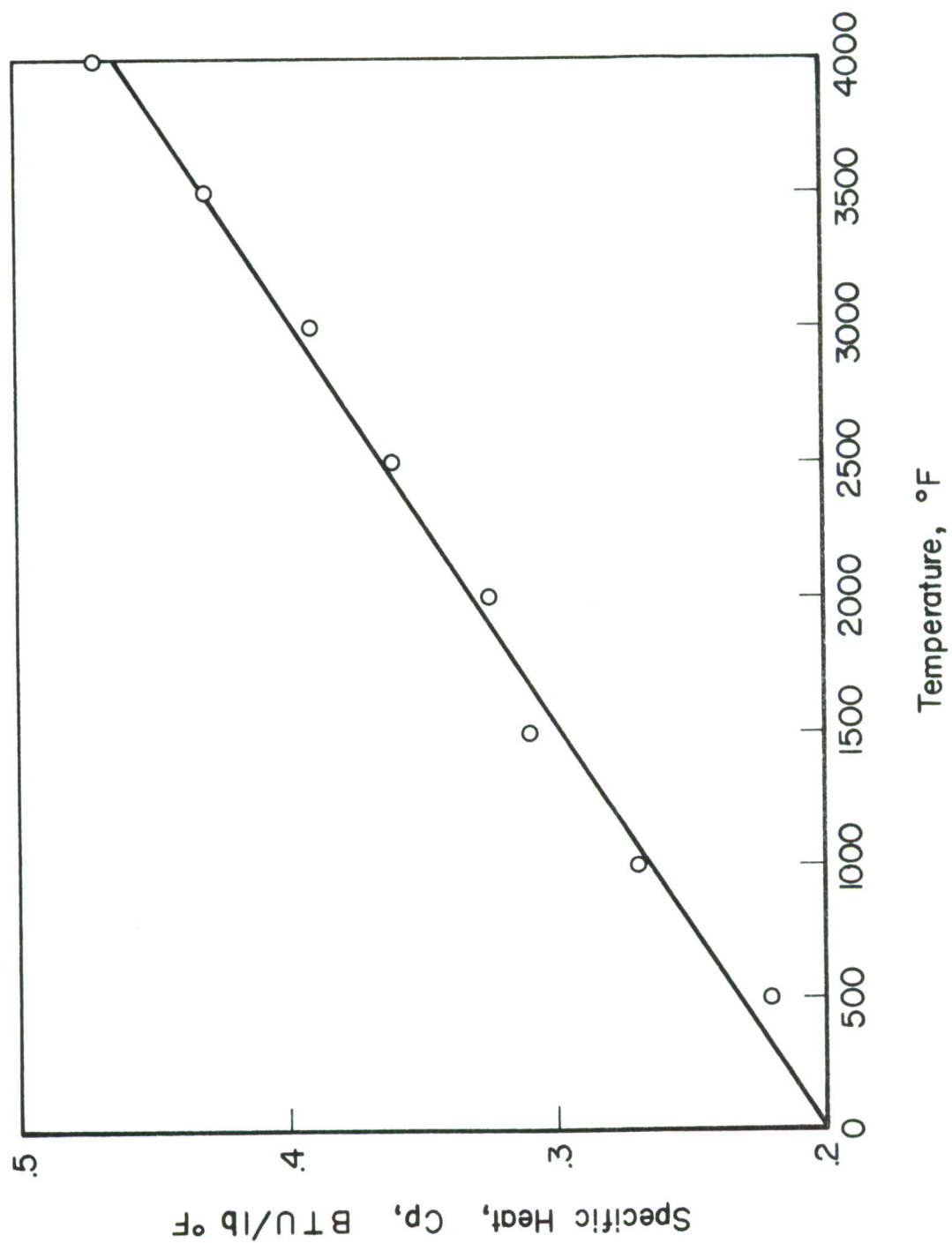


Fig. 30 - SPECIFIC HEAT OF JTA VS. TEMPERATURE

APPENDIX

ANALYSIS OF VARIANCE OF FLEXURAL STRENGTH FOR 7-F-12, 7-F-14, AND 14-G-1

An analysis of variance is a test, whereby one may determine whether or not several samples are members of the same parent population. The test compares the variation of values between members of the samples with the variation between samples. If all of the samples are from the same population, the ratio of these two variations should be small. A large ratio, on the other hand, is an indication that there are differences between samples either in the material or in the test itself.

The following is an outline of the method of calculation used in the analysis:

N = number of items in all samples

T = grand total of all of the values measured

\bar{X} = grand average T/N

n = number of samples

i = number of items in a given sample

\bar{x} = the average of a given sample

To calculate the variance between samples

$$\sum_n (\bar{x}_n - \bar{X})^2 i_n = \text{between-samples sum of squares}$$

$n - 1$ = between-samples degrees of freedom, then

$$\frac{\sum_n (\bar{x}_n - \bar{X})^2 i_n}{n - 1} = \text{between-samples estimate of variance}$$

To calculate the variance with the samples,

$$\sum_{ni} (x_i - \bar{x}_n)^2 = \text{within-sample sum of squares}$$

$$\sum_n (i_n - 1) = \text{within-sample degrees of freedom}$$

$$\text{then } \frac{\sum_i (x_i - \bar{x}_n)^2}{\sum_n (i_n - 1)} = \text{within-sample estimate of variance.}$$

The largest ratio of these two estimates is then compared with tables which indicate the values of the ratio which will be exceeded with various degrees of probability* (i.e., in the case analyzed, the ratio will exceed a value of 19.5 in 5% of the analyses and will exceed 99 in only 1% of them). Normally, a calculated value which exceeds the 1% value from statistical tables is taken as a very strong indication of real differences between samples and a value exceeding the 5% value is taken as a moderate indication of real differences between samples.

As may be seen from Tables XIX to XXI, in the analysis carried out on JTA specimens, all tests except the sonic modulus test give variance ratios well below the 5% level. The high values of the ratio obtained in the case of sonic modulus, when considered in the light of the low values obtained in the flexure and tangent modulus analyses, seem to indicate a variability in the test rather than in the material.

* Moroney, M. J., Facts From Figures, Penguin Books, Baltimore (1954).

Table XIX
ANALYSIS OF VARIANCE FOR FLEXURE STRENGTHS
OF 3 x 1/4 x 1/4 IN. SPECIMENS
FROM BILLETS 7-F-12, 7-F-14, AND 14-G-1

| Source of Variation | Sums of Squares | Degrees of Freedom | Variance Estimate | F* |
|---------------------|-----------------|--------------------|-------------------|------|
| <u>With Grain</u> | | | | |
| Between samples | 5,338,040 | 2 | 2,669,020 | 2.86 |
| Within samples | 206,067,650 | 27 | 7,632,135 | |
| Total | 211,405,690 | 29 | | |
| <u>Across Grain</u> | | | | |
| Between samples | 9,271,710 | 2 | 4,635,855 | 9.3 |
| Within samples | 13,479,100 | 27 | 498,070 | |
| Total | 22,750,810 | 29 | | |

* 1% level of F = 99; 5% level of F = 19.5.

Table XX
ANALYSIS OF VARIANCE FOR TANGENT MODULUS
OF 3 x 1/4 x 1/4 IN. SPECIMENS
FROM BILLETS 7-F-12, 7-F-14, AND 14-G-1

| Source of Variation | Sums of Squares, x10 ¹² | Degrees of Freedom | Variance Estimate x10 ¹² | F* |
|---------------------|---------------------------------------|--------------------|--|------|
| <u>With Grain</u> | | | | |
| Between samples | 53.789 | 2 | 26.8945 | 6.78 |
| Within samples | 107.085 | 27 | 3.966 | |
| Total | 160.874 | 29 | | |
| <u>Across Grain</u> | | | | |
| Between samples | 4.561 | 2 | 2.2805 | 1.67 |
| Within samples | 36.752 | 27 | 1.361 | |
| Total | 41.313 | 29 | | |

* 1% level of F = 99; 5% level of F = 19.5.

Table XXI
ANALYSIS OF VARIANCE FOR SONIC MODULUS
OF 3 x 1/4 x 1/4 IN. SPECIMENS
FROM BILLETS 7-F-12, 7-F-14, AND 14-G-1

| Source of Variation | Sums of Squares x10 ¹² | Degrees of Freedom | Variance Estimate x10 ¹² | F* |
|---------------------|--------------------------------------|--------------------|--|------|
| <u>With Grain</u> | | | | |
| Between samples | 25.585 | 2 | 12.7925 | 38.9 |
| Within samples | 8.904 | 27 | 0.329 | |
| Total | 34.489 | 29 | | |
| <u>Across Grain</u> | | | | |
| Between samples | 415.373 | 2 | 207.6865 | 1070 |
| Within samples | 5.215 | 27 | 0.193 | |
| Total | 420.588 | 29 | | |

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| | | 2b. GROUP None | |
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| 14 KEY WORDS | LINK A | | LINK B | | LINK C | |
|---|--------|----|--------|----|--------|----|
| | ROLE | WT | ROLE | WT | ROLE | WT |
| <p>Characterize and correlate physical and mechanical properties.</p> <p>Two 7" billet; one 14" billet.</p> <p>High temperature testing in inert and oxidizing atmospheres.</p> | | | | | | |

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