214-2E2 (1st Quarterly Report)

First Quarterly Report

INVESTIGATION OF THE REINFORCEMENT OF DUCTILE METALS WITH STRONG, HIGH MODULUS DISCONTINUOUS, BRITTLE FIBERS

by

A. Gatti, J. M. Berry, J. J. Gebhardt, E. Feingold

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

February 1967

CONTRACT NASw-1543

Space Sciences Laboratory
GENERAL ELECTRIC COMPANY
Missile and Space Division

FACILITY FORM 602

N 67-20010
(ACCESSION NUMBER)

(THRU)

34
(PAGES)

1
(CODE)

CR-82998
(NASA CR OR TMX OR AD NUMBER)

17
(CATEGORY)

TABLE OF CONTENTS

LIST OF ILLUSTRATIONS

SUMMARY

This progress report covers the period 1 November 1966 to 31 January 1967; the work is being performed under Contract NASw-1543, with Mr. James J. Gangler of NASA-Headquarters serving as Program Monitor. The purpose of this program is to define and investigate the critical factors affecting the reinforcement of ductile metals with short, brittle fibers. The materials system selected for study is aluminum (or it alloys) reinforced with B_4C fibers or B_4C whiskers. Related tasks in the program include the development of a more economical process for growing B_4C whiskers and characterization of the individual constituents of the final composites. The latter also involves the structural and chemical interactions of the combined elements (fibers, matrix, coatings, etc.).

The results obtained during this period are summarized as follows:

1) The growth of B_4C whiskers by a chemical vapor deposition process was demonstrated. Control over factors such as air leakage, residual gases, and over-saturation, was found to be important, and will be emphasized in future studies.

2) The matrix metal presently used for composite fabrication, commercially pure aluminum (1100), was tested in uniaxial tension. The room temperature true-stress, true-strain characteristics were determined.

3) Individual tungsten-core filaments of boron carbide, (B_4C/W)* were pulled in tension and a statistical study of their strength and elastic modulus was made using gauge lengths of 15" and 1". The 15"-specimens had an average strength of 200,000 psi while the 1"-specimens averaged 330,000 psi. The average modulus was 59.2×10^6 psi. The stability of the aluminum-B_4C/W composites at elevated temperatures was studied using an electron probe technique; no chemical interactions were detected between the fiber and matrix.

---

*Made by the vapor deposition of boron carbide on boron-tungsten filament (Ref. 6)

4) A hot-pressing technique developed under a previous NASA contract (# NASw-1383) was utilized to fabricate single filament composites consisting of B_4C/W filaments imbedded in the pure aluminum.

5) The fracture behavior of single filament composites was studied. It was observed that fracture of the filaments occured between 1 and 1-1/2% strain in the composites. Using these strain values and the elastic modulus of the filaments (59.2×10^6 psi), the tensile strengths of the filaments was computed to be between 590,000 psi and 885,000 psi. It can be tentatively concluded that the strengths of fibers tested within a composite may be considerably different from the strength determined by individually testing the fibers. However, these results are based on the assumption that no slippage occurred between the filament and the matrix, a point that will be checked in future work.

6) Studies of the fracture behavior of single-filament composite specimens were also made at increasing test temperatures so that the fracture mode of the filament could be observed as a function of the decreasing shear strength of the matrix. As would be expected, the apparent critical length (L_c) of the filament increased as a linear function of temperature as the shear strength of the matrix (τ) was decreased.

I. INTRODUCTION

From a reinforcing viewpoint, whiskers (single-crystal filaments) appear to have many desirable characteristics. A number of classes of compounds have been prepared in this form including metals, oxides, nitrides, carbides, and graphite. The maximum strengths observed for these whiskers range from about 0.05 to 0.1 of their elastic moduli, approaching predicted theoretical strengths. Many also have relatively low densities and are stable at high temperatures. Calculations of whisker-reinforced composite properties based on whisker properties, particularly for the brittle whiskers of high modulus materials, show that they have enormous potential compared to more conventional materials on both a strength/density and a modulus/density basis.

The incorporation of whiskers into composites requires the following series of processing steps:

1) Whisker growth.
2) Whisker beneficiation--to separate strong fibers from the growth debris.
3) Whisker classification--to separate according to size.
4) Whisker orientation--to align the whiskers and maximize reinforcement along a specific axis.
5) Whisker coating--to promote wetting and bonding.
6) Whisker impregnation with matrix material--to form a sound. strong composite.

Because of the many processing steps, there is a large number of imposing technical problems to be solved in order to achieve the high potential strengths. Many of these problems have not been solved yet.

In a few isolated cases, involving very small and carefully prepared samples, the predicted strengths of the brittle whisker/ductile matrix composites have been achieved. However, all too frequently, attempts to scale up the composites into even modest size specimens have resulted in strengths that range from about 10 to 30 per cent of the predicted values.

A list of possible reasons for the low composite strengths values is given in Table I. As can be seen, there are many variables to contend with, and many of these are interrelated and difficult to study experimentally.

TABLE I. VARIABLES AFFECTING THE TENSILE STRENGTH OF WHISKER REINFORCED COMPOSITES

A. Whisker Variables

1. Average strength
2. Dispersion of strength values
3. Strength versus whisker diameter and length
4. Strength degradation during handling and fabrication
5. Strength versus temperature
6. Modulus

B. Matrix Variables

7. Yield strength
8. Flow properties
9. Strength versus temperature (particularly shear strength)
10. Matrix embrittlement due to mechanical constraints on new phases formed.

C. Composite Variables

11. Volume fractions of components--fiber & matrix
12. Homogeneity of whisker distribution

A fundamental difficulty in evaluating the performance of whisker composites is the lack of knowledge concerning the whiskers themselves. This is understandable when one realizes that there are about 10^9 to 10^{10} of them per pound, and characterization of even a small fraction becomes a major task. These and other problems have limited the immediate use of B_4C whiskers which were synthesized and characterized in previous studies(1, 2, 3, 4).

An alternate means to gain useful, fundamental knowledge concerning whisker-reinforced composites involves the use of brittle, continuous filaments. Continuous filaments have several advantages over whiskers when investigating the reinforcement of materials; some of these advantages are listed:

1) It is much easier to characterize the relevant and critical parameters listed in Table I.
2) The available continuous filaments are large relative to the whiskers and can be more readily handled and incorporated into composites.
3) The filaments can be cut to uniform desired lengths of symmetrical geometry so that the effects of discontinuous reinforcements can be assessed.

Experimental work of this type has already been done using ductile filaments such as tungsten in a ductile matrix such as copper(5). Although this work has provided a wealth of information regarding the reinforcement of metals, it does not uncover all of the key problems encountered in the brittle fiber/ductile matrix systems which are potentially of great technological importance. The chief difference between the reinforcement of metals with brittle and ductile fibers, is that ductile fibers can deform to accommodate high stress concentrations, whereas brittle fibers cannot do so. Thus it is necessary to carry out further studies and to evaluate the potential and the engineering limitations of metals reinforced with brittle fibers and whiskers.

This program was therefore initiated to investigate in detail the reinforcement of a ductile metal (aluminum) with fibers of B_4C/W (both continuous and chopped), for the purpose of gaining further information on the behavior of metals reinforced with discontinuous, brittle fibers and for providing data which would be pertinent to whisker-reinforced metals. This program is being conducted in two parts: (1) development of a process to grow B_4C whiskers which would be amenable to eventual scale-up, and (2) to investigate the reinforcement of aluminum with B_4C/W filaments. When sufficient quantities of whiskers become available, they will be used in the composite studies also.

II. EXPERIMENTAL PROCEDURES AND RESULTS

A. WHISKER GROWTH STUDIES

Current studies on the growth of boron carbide whiskers are concentrating on the use of chemical reactions in the gas phase to produce the depositing species. It will be recalled that earlier whisker growing studies(1,2) depended on the sublimation of depositing species from solid boron carbide.

The simultaneous reduction of boron tribromide, BBr_3, with hydrogen, and the thermal decomposition of carbon tetrachloride, CCl_4, are being used to generate boron and carbon respectively in the gas phase, in suitable concentrations and at appropriate conditions to permit boron carbide whiskers to be deposited. Parameters of interest in these studies include a) reaction temperature, b) total pressure, c) total gas concentration of reactants, d) stoichiometry of gas species, e) residence time, f) linear velocity, g) substrate temperature, h) nucleation initiators, and i) furnace geometry. Table II lists these factors and the values of each studied thus far in the experimental program. Of equal importance, because of the low total gas concentration required (~ 1% by volume), is the complete absence of impurities in the furnace which can either react with the boron tribromide or alter the equilibria of the reactions. Water and residual hydrogen bromide can be expected to be troublesome, for example.

Initial studies carried out using parameter values shown in Table II have yielded some whisker deposits, but on the whole have not been satisfactory, primarily because of interfering impurities or side reactions. Table III summarizes the results obtained thus far. Figure 1 shows examples of whiskers produced in the first deposition run. The remainder of the runs (runs 2-9) listed in Table III are believed to have not produced whiskers for one or more of the following reasons: Air leakage and formation of boric acid or oxide, reversal of the reduction equilibrium by residual hydrogen bromide, and formation of hard, continuous coatings of boron carbide because of oversaturation.

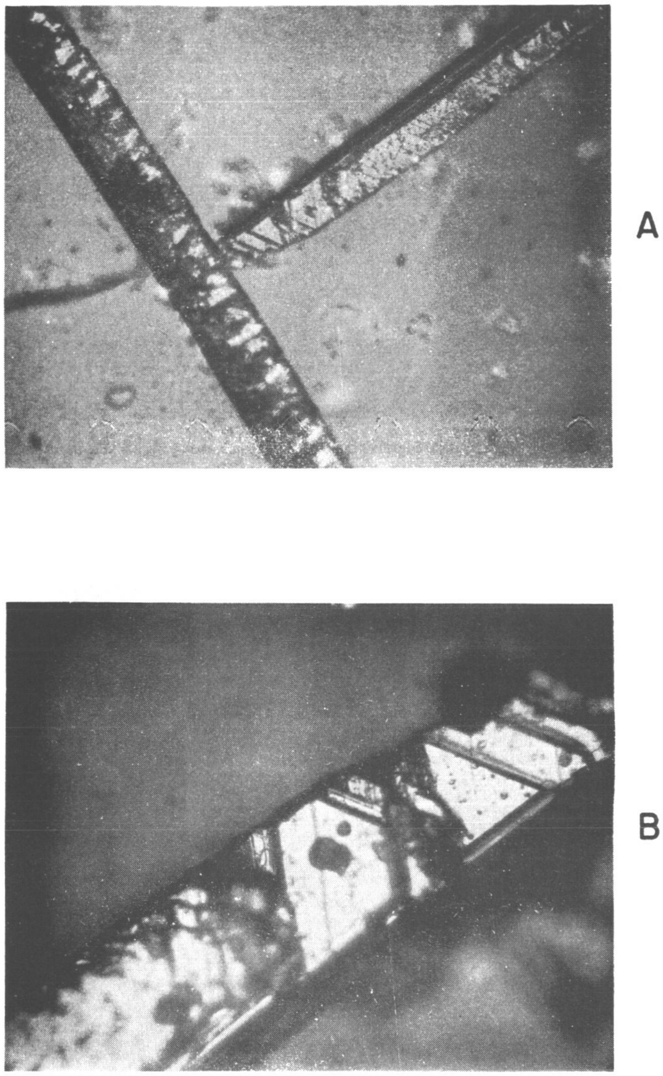


Figure 1. Typical Views of B_4C Deposition Products Formed by the Chemical Vapor Deposition Process
A. Typical Whiskers - 115X
B. Same as A but at 230X

TABLE II. PARAMETERS OF INTEREST IN WHISKER DEPOSITION

Reaction Temperature	1400°C
Total Pressure	760 Torr ± 10 Torr
Total Gas Concentration	0.5 - 1.0%
Stoichiometry	
(a) Liquid Saturator	7 BBr_3: 1 CCl_4
(b) Calculated Gas Feed	4 BBr_3: 1 CCl_4
Residence Time	16.5 - 60 min
Linear Velocity	1.28 - 4.5 cm/min
Substrate Temperature	< 1400°C
Geometry	Open Cylinder 41 mm diam x 760 mm long. In two sections, with connecting plug 8 mm diam.
Nucleation	Vanadium metal

TABLE III. SUMMARY OF WHISKER DEPOSITION RUNS*

Item	Code	Temp (°C)	P_{TOT} (Torr)	Flow (cc/min)	Satur. Temp. (°C)	Gas Conc (% Vol.)	Gas Veloc. (cc/min.)	Results	Comment
1	W1	1400	730-750	15	0	1	1.3	whiskers, dendrites	Slight Over-saturation
2	W2	1400	760	20	0	0.2	1.7	none	Diluted Gas
3	W3	1420	750-770	15	0	1	1.3	none	see #5
4	W4	1400	760	22	0	1	2.5	none slight B_2O_3	see #5
5	W6	1450	760	10	20	10	1.0	large B_2O_3 deposit	Leak in feed line
6	67-2-11D	1400	760	15	0	1	1.2	no deposit	Slight B_2O_3
7	67-3-11D	1420	680-740	23	0	1	1.0	dusty gray deposit	None
8	67-4-11D	1520	680-740	50	23	11	4.2	hard, thick black depos.	Oversaturation
9	67-5-11D	1420	760	54	0	0.5	4.5	no deposit slight B_2O_3	Pump back-streaming

*Notes

Items 1 and 2, substrate used previously for B_4C evaporation study
Item 3 New element; Metallic Vanadium in deposition zone
Item 4 Closed 1" I.D. deposition tube
Item 5 Deliberately oversaturated to locate leak
Item 8 Deliberately oversaturated to obtain deposit
Item 9 Pump was loaded with HBr - may have affected equilibrium

The success attending the first run, and the results of subsequent runs, indicate: a) that boron carbide can be deposited in the furnace being used, and b) it can be deposited in whisker form under conditions close to those listed in Table II, provided that interfering processes can be eliminated and that nucleation can be properly stimulated. Continuing efforts are in progress to accomplish these ends.

B. CHARACTERIZATION OF COMPOSITE MATERIALS

Since this program is concerned with an investigation of the factors which control the mechanical, the physical and the chemical behavior of metal-matrix composites reinforced with brittle, discontinuous fibers, it is highly important that parameters which affect this behavior be well identified and characterized. The approach used evolves from the simple concept of combining well-characterized brittle fibers with a well-characterized matrix metal and with simple composite test configurations.

The characterization of the variables includes such factors as the average strength and strength dispersion of the fibers, fiber aspect ratio (L/d), fiber strength degradation during processing, and so forth. By systematically varying composite parameters and by comparing the results with theory, either the existing theory will be verified or the theory will be modified to account for the experimental observations. Such understanding will delineate the key variables and their relative importance.

1. Matrix Characterization

Commercially pure aluminum (1100 alloy) is being utilized as the matrix material because it has the following advantages:

1) Its properties are well documented
2) It is a ductile metal
3) It can be purchased in almost any form including sheet, tubing, powder, etc.
4) Composite specimens can be fabricated by several techniques.

Tensile specimens 6'' long, containing a reduced section 3'' in length, and 0.028 square inches in cross-section, were cut from the same 1100 alloy aluminum sheet from which single filament composites (to be discussed later) were fabricated. The specimens were tested in uniaxial tension at room temperature. Thus experimental values of the yield strength and the ductility (elongation) of the matrix starting material were obtained. A graph of the true-stress true-strain data is shown in Figure 2.

2. Filament Evaluation

The strength properties of composites containing high modulus, high strength, brittle fibers are primarily dependent on the fiber properties. Therefore, it is essential to measure the strength characteristics of the fibers both before and after fabrication into composites. The use of B_4C/W filaments was selected for this program, because of the following reasons:

1) They are apparently not attacked by molten aluminum, so that composites can be fabricated by an infiltration process.
2) They are much larger in cross-section than whiskers and are therefore much easier to handle, to characterize, to align, and to fabricate into specimens having specified fiber orientation, packing density, etc.
3) They can be can be cut to specific aspect ratios (L/d).
4) They are available in much larger quantities for composite studies.
5) They are available in continuous lengths, so that specimens reinforced with continuous lengths can be used as a standard of comparison for those reinforced with short lengths.
5) They have values of elastic modulus and elastic modulus/ density ratio similar to those of their whisker counterparts.
6) Their properties have been evaluated in a previous study.(6)

Thus far, only the testing of various fiber lengths before fabrication into composites has been done. Testing as a function of gage length yielded information concerning the intrinsic strength of the material as well as the strength characteristics to be expected of the chopped fibers. As can be seen in Figure 3, a variation in strength as a function of gage length seems to exist, and indeed at gage lengths equivalent to observed critical lengths (L_c) may well be in the 600,000 psi range. The average room temperature

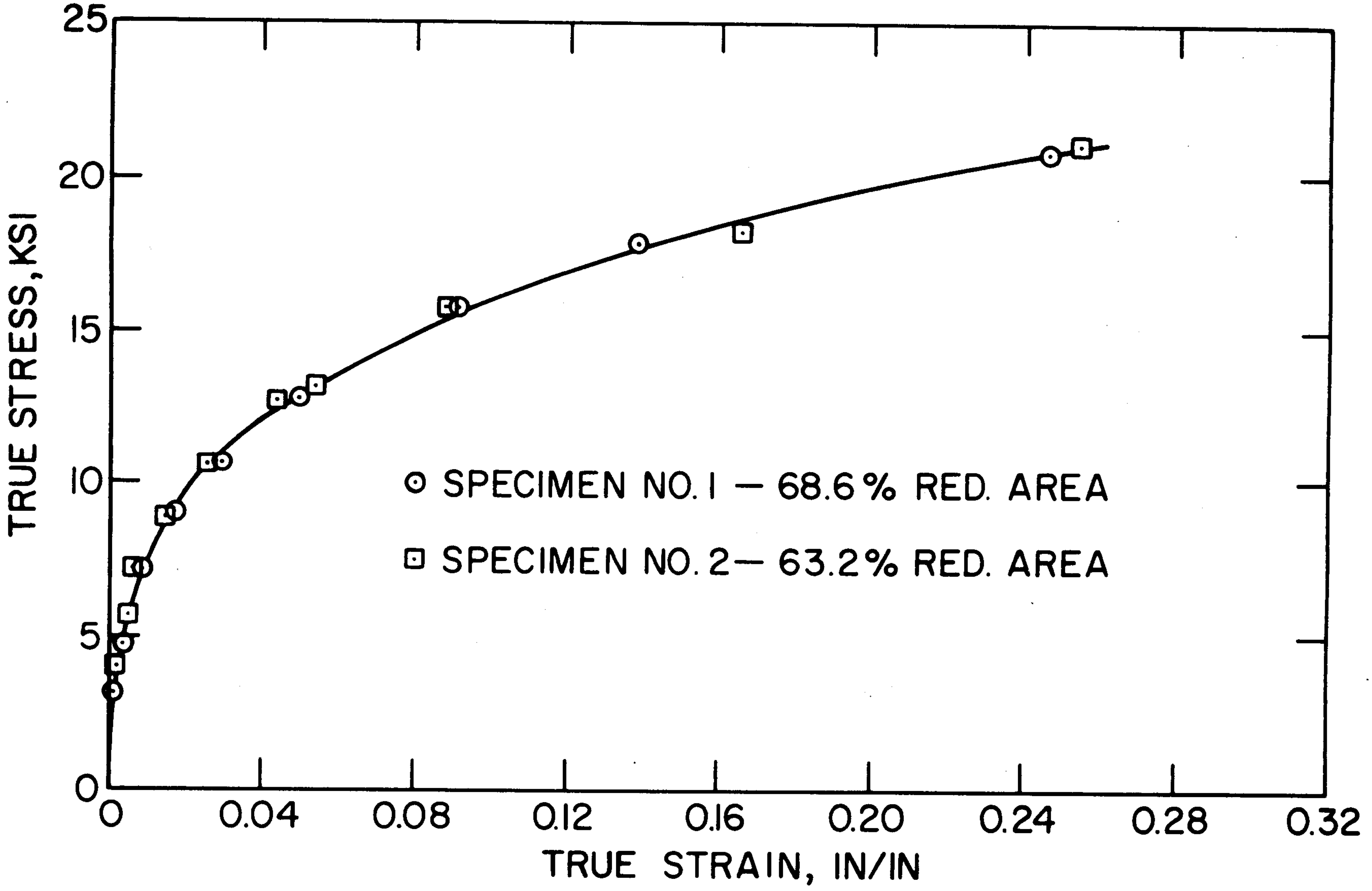


Figure 2. True Stress - True Strain Curve to the Limit of Uniform Elongation for Annealed 1100 Aluminum

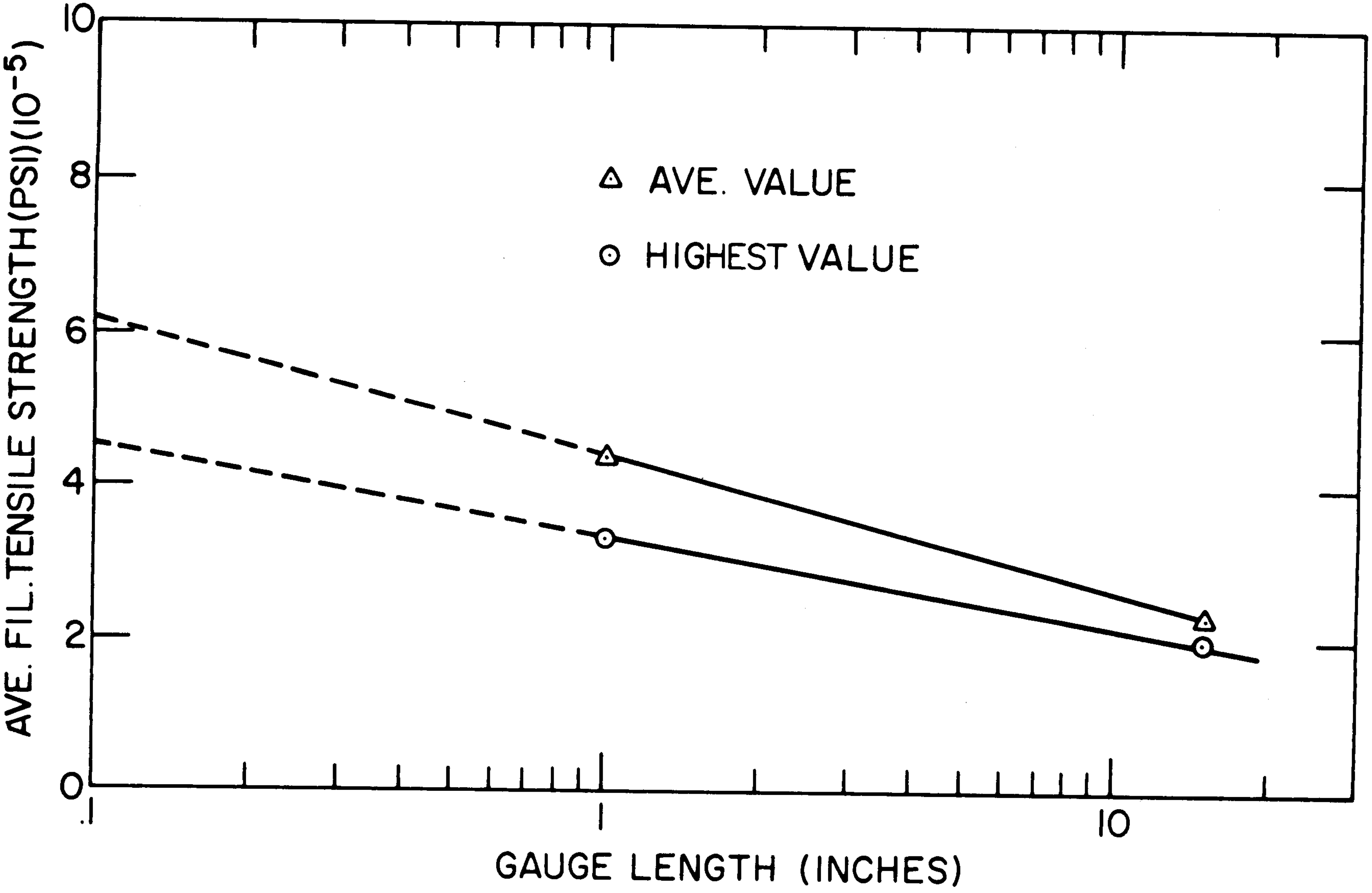


Figure 3. Effect of Specimen Gage Length on the Strength of B_4C/W Filament

modulus of the filaments was 58 x 10^6 psi. Future work will include the testing of very short gauge length specimens to clarify the extrapolated regions of Figure 3.

3. Evaluation of Matrix/Filament Stability

The metal matrix in a fiber reinforced composite serves to transfer the applied mechanical stresses to the reinforcing filaments. In order to achieve optimum stress transfer, it is imperative that an intimate and strong bond exist between matrix and filaments. The filament-matrix bond can be of a mechanical, a chemical, or a Van der Waals type, or more probably, a combination of the three. For any of the bond types to be effective, especially the last two, intimacy of contact between filament and matrix is a prime requirement.

The B_4C/W-aluminum composites evaluated here were made by vacuum infiltrating liquid metal into bundles of oriented B_4C/W filaments. This technique was developed previously under Contract NASw-1383(4).

Unless wetting of the solid filaments by the liquid aluminum phase occurs (contact angle, θ, less than 90 degrees) during fabrication, voids will remain between the filaments and the matrix after matrix solidification. These void regions do not transfer stresses and contribute to the overall weakness of the composite system. The optical photomicrograph in Figure 4 shows that vacuum infiltration resulted in a composite system in which the B_4C/W filaments were indeed in intimate contact with the aluminum matrix.

Even if the intimacy of contact between the filaments and matrix is assured, the resulting composite will be weak if the filaments are not chemically compatible with the liquid matrix. If the chemical reactivity of the filaments with the liquid metal matrix is extensive under the conditions of fabrication (time, temperature), then the filaments will be damaged or degraded and rendered weak and the resulting composites will be weak. Electron probe techniques were used to study the chemical compatibility of these materials.

Electron probe analysis is a technique which permits determination of chemical (elemental) compositions from very small regions of a specimen

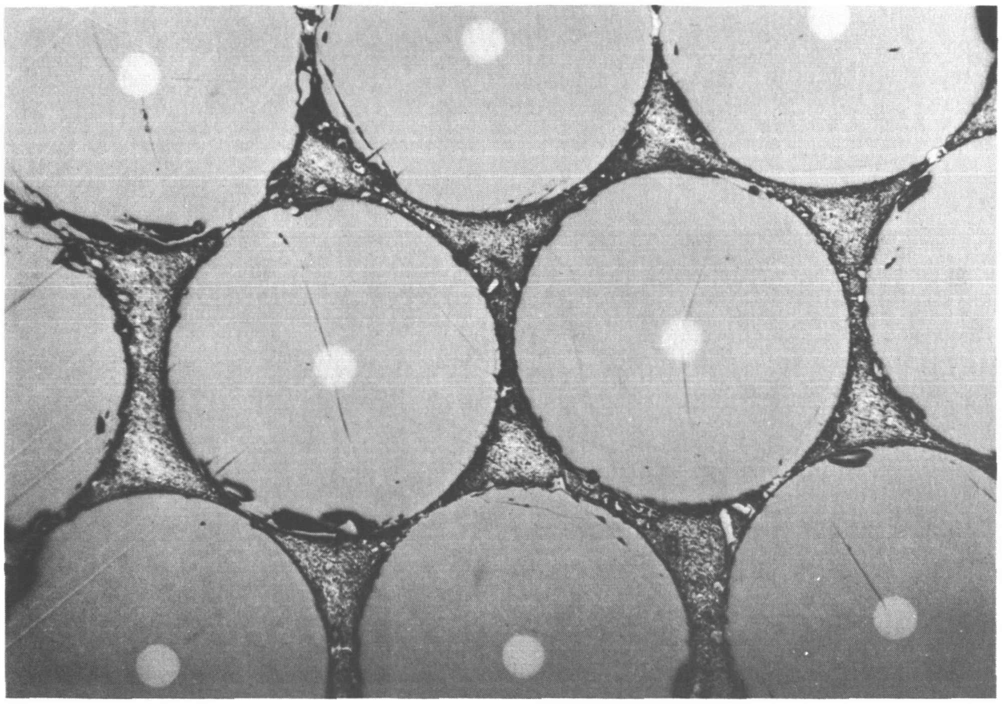


Figure 4. Photomicrograph of a Cross Section of a Vacuum Infiltrated B_4C/W Filament-Aluminum Composite (Mag - 300X)

(a few cubic microns in volume) in a non-destructive manner. In the electron probe, an electron optical system shapes a monoenergetic electron beam into a narrow pencil (ca. 0.5μ diameter) which is caused to impinge on a specimen at any desired position. The interaction of the impinging electrons with the specimen results in the production of x-rays which are characteristic of the composition. The resulting x-rays are analyzed in an x-ray spectrometer and thus the composition of the region of interest may be established.

Modern vacuum x-ray spectrometers, equipped with suitable analyzing crystals and thin window detectors, can be used in the analysis of elements from atomic number 5 (boron) upward in the periodic table of the elements. In this laboratory it has been found that the greatest difficulty in low atomic number x-ray analysis is associated with the stability of thin window detectors. To date, it has not been possible to use commercial thin windows* for more

---

*The thin windows are collodion (about 500A thick) supported on 200 mesh copper or nickel screens than about 15 minutes before they are destroyed. Preliminary electron probe analysis of a B_4C/W aluminum composite was made during this report period using a thin window detector having a useful lifetime of only 15 minutes. Therefore, the analyses are not complete.

Figures 5A, B, C and D are specimen current, boron x-ray, carbon x-ray, and tungsten x-ray scanning electron probe images, respectively. Although these are the results of only a preliminary investigation, they do indicate some pertinent facts: (a) the "boron photograph" shows that the boron(s)-aluminum(ℓ) chemical interaction was not severe during the period that the aluminum was molten, since the dispersion of boron in the solidified aluminum matrix is not extensive; also, the boriding of the tungsten substrate is incomplete; (b) the "carbon photograph" indicates that carbon is dispersed in the aluminum matrix. This should not be taken as conclusive evidence that the dispersed carbon originated from the boron-carbide filaments, since (1) the scanning electron beam results in carbon contamination of the specimen and (2) the carbon-containing metallographic mount could have been smeared over the specimen in the metallographic polishing procedure. The high concentration of carbon in the region of the tungsten substrate can be explained as either resulting (1) from retention of smeared-over mounting medium in the depression caused by polishing the softer (tungsten) core surrounded by the harder boron-carbon deposit and/or (2) from the graphite that is sometimes used in the commercial drawing of fine tungsten wires.

When suitable detector thin windows with realistically long life times become available careful electron probe analyses of the boron-carbon/aluminum interface will be made. These future analyses will provide answers such as oxygen and nitrogen content of filament/matrix interfaces, interdiffusion of boron-carbon and aluminum, etc.

C. COMPOSITE STUDIES

Detailed studies on the B_4C/W-aluminum composite system have been undertaken to obtain experimental strength data which can be related to existing theory. Particular emphasis was placed on measuring

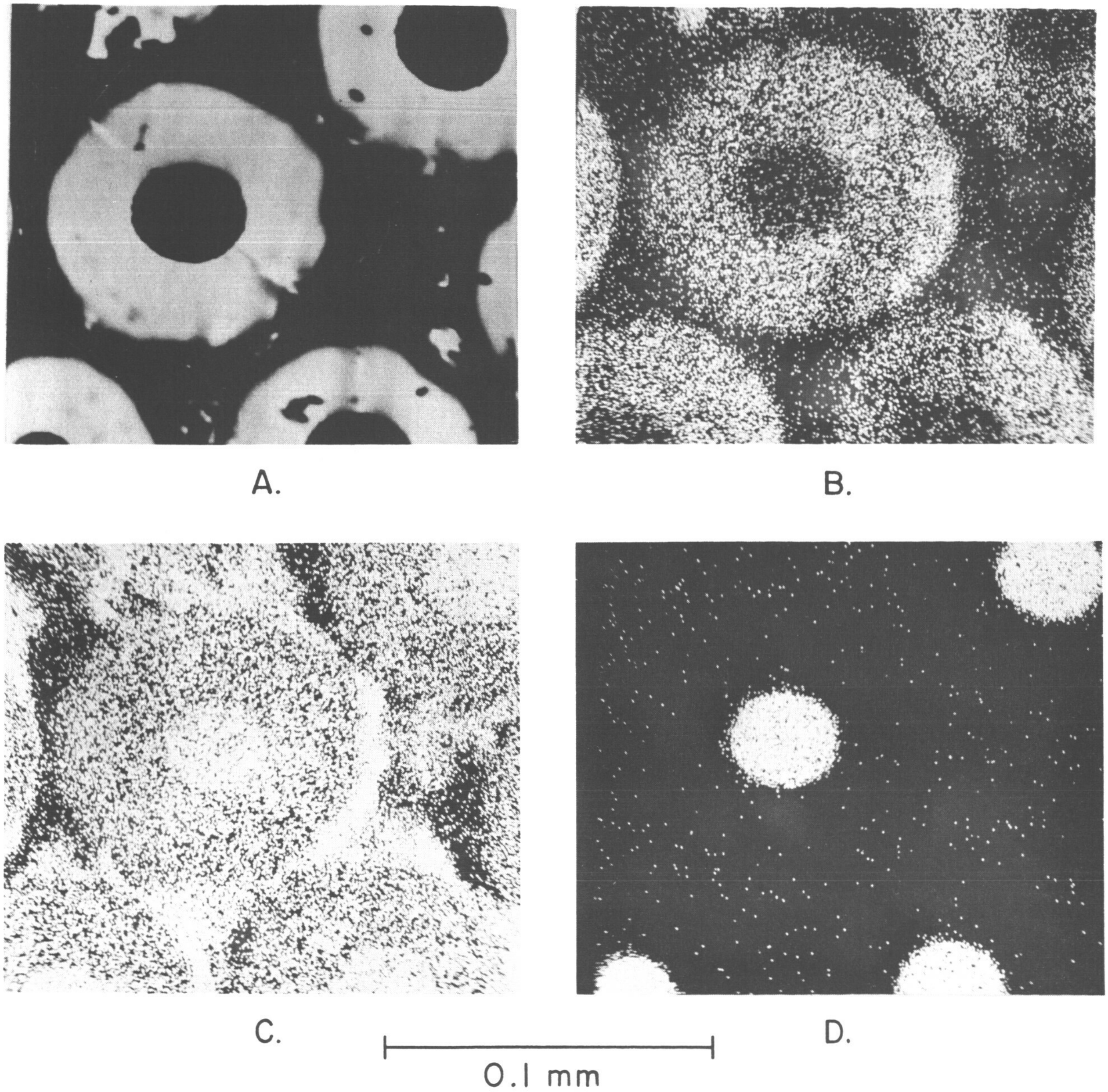


Figure 5. Electron Microprobe Scanning Images of a B_4C/W Filament/ Aluminum Matrix Composite

A. Specimen Current Image
B. Boron X-ray Image
C. Carbon X-ray Image
D. Tungsten X-ray Image

(Mag. = 500X)

and understanding the properties and characteristics of the composite components, viz., the B_4C/W filaments and the aluminum matrix. These components were then carefully combined into various composite configurations to test several of the composite variables deemed important by existing theory.

1. Fabrication Techniques

A hot-pressing technique was used to fabricate the composites studied during this reporting period, and was described previously$^{(4)}$. Briefly, single-filament B_4C/W-aluminum composites were made by laminating two 1/8"-wide x 4"-long by 1/16"-thick 1100 alloy aluminum sheets, between which a 2" long filament was centrally located, and hot pressing it in a graphite die for 10 minutes at 600°C and 3000 psi. A sketch of the fabrication details and final specimen produced, reproduced from a previous report$^{(4)}$ is shown in Figure 6 by way of illustration.

Single filament specimens were used to study the variation of L_c as a function of both strain and testing temperature. These results are discussed in the next section of this report.

2. Tensile Test Results of Single Filament Composites

Previous work had demonstrated the merit of using single-filament aluminum composites (to determine critical length, L_c) and afforded a qualitative measure of the strength of the filament by studying the fractography of the extracted filament fragments from a highly strained composite. After some refinement of the fabrication technique, additional tests were made on composites including several which contained multiple and discontinuous reinforcing filaments. A number of tests were also performed at elevated temperatures. Some pertinent results are shown in Table IV and Figure 7, while Table V presents a more complete summary of the data derived from the elevated temperature test results.

It is evident from Table V that the average length of broken pieces of filament increases with the testing temperature, and this is most likely attributable to the decreasing shear strength of aluminum as the temperature increases. Figure 7 is a plot of the tensile strength at 1-1/2% strain of the

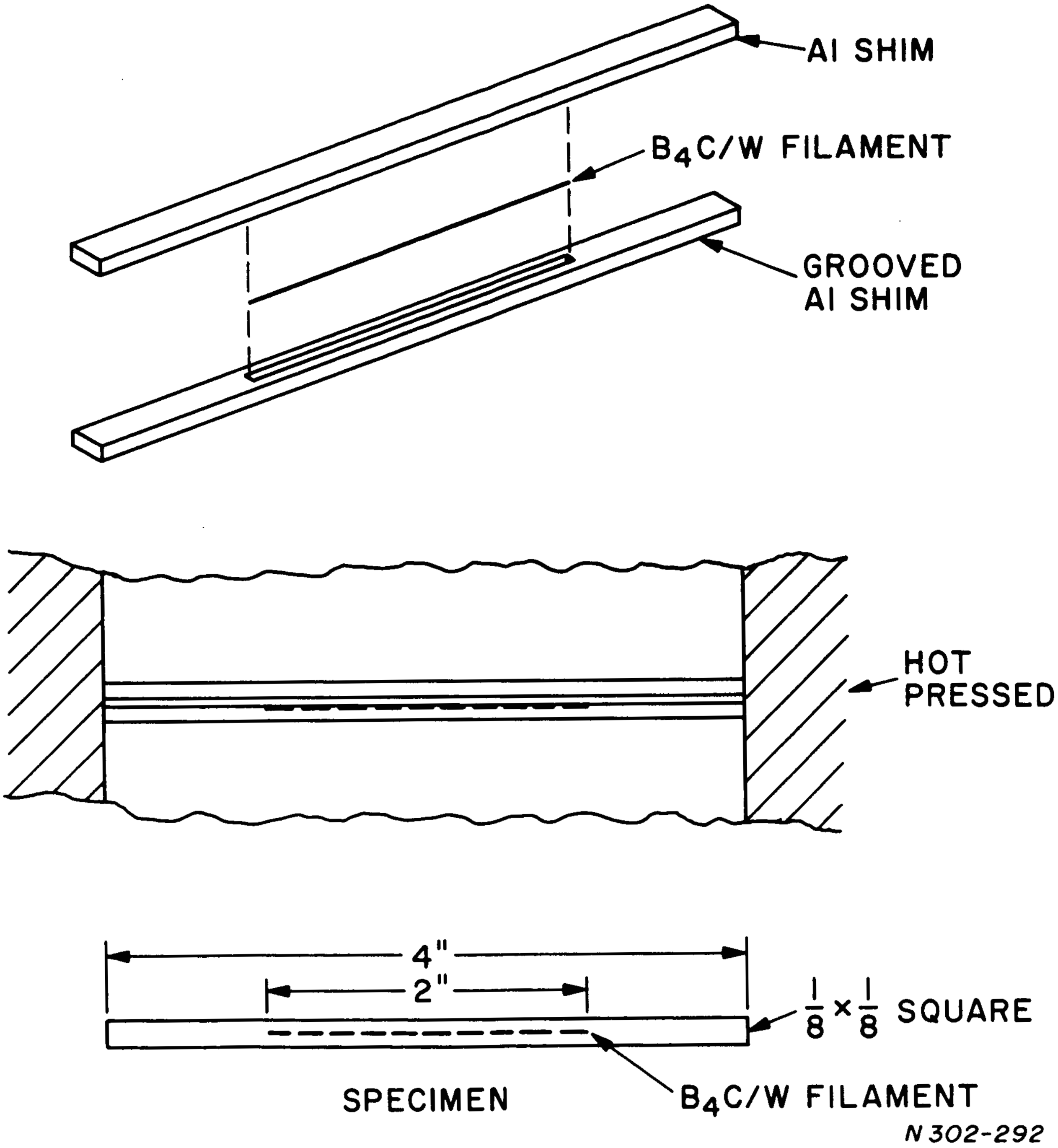


Figure 6. Sketch Showing Processing Details to Produce B_4C/W-Aluminum Composites for Estimating Values of the Filament Critical Length, L_c

TABLE IV. FILAMENT BREAK-UP IN STRAINED UNREINFORCED B_4C-ALUMINUM COMPOSITES

Test No.	No. and Length of Filaments*	Plastic Strain of the Composite (%)	Remarks on Filament Fracture
1	1 2-inch Lengths	≈2	41 Pieces
2	4 1/2-inch Lengths	>15	31 Pieces (see Fig. 6A)
3	5 2-inch Lengths	>15	Large Number of Pieces (see Fig. 6B)
4	1 2-inch Length	>15	Strained at 370°C, 20 Pieces (see Fig. 6C)
5	1 2-inch Length	>15	Strained at 600°C, 7 Pieces
6	1 2-inch Length	0.41	No Breaks
7	1 2-inch Length	0.91	No Breaks
8	1 2-inch Length	1.40	17 Pieces
9	1 2-inch Length	1.85	40 Pieces (see Fig. 6C)

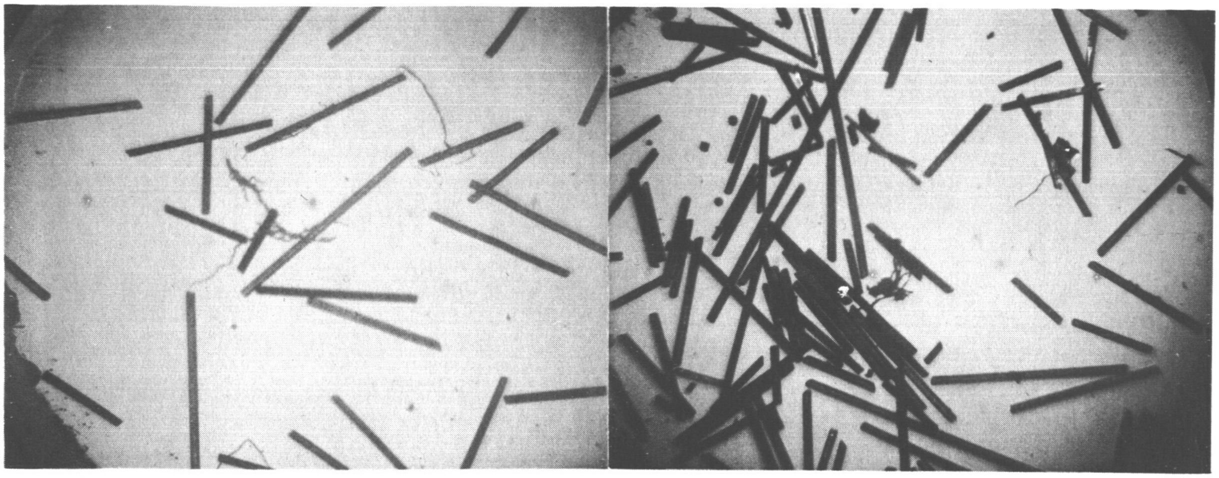
* Prior to testing of composite

TABLE V. RESULTS OF TESTS PERFORMED ON B_4C/W-ALUMINUM COMPOSITES

Specimen Number	Test Temperature (°C)	Composite Tensile Strength (psi)	Tensile Strength (at 1 1/2% Strain) (psi)	Number of Pieces (N)	$\bar{\ell} = \frac{\ell_o}{N}$ (inches)
1	RT	16000	6700	41	0.049
4	370	4750	3480	20	0.109
5	500	1360	1300	7	0.294
-	660	0	0	1	2.00

$\bar{\ell}$ = average fiber length after testing

ℓ_o = original fiber length (2")

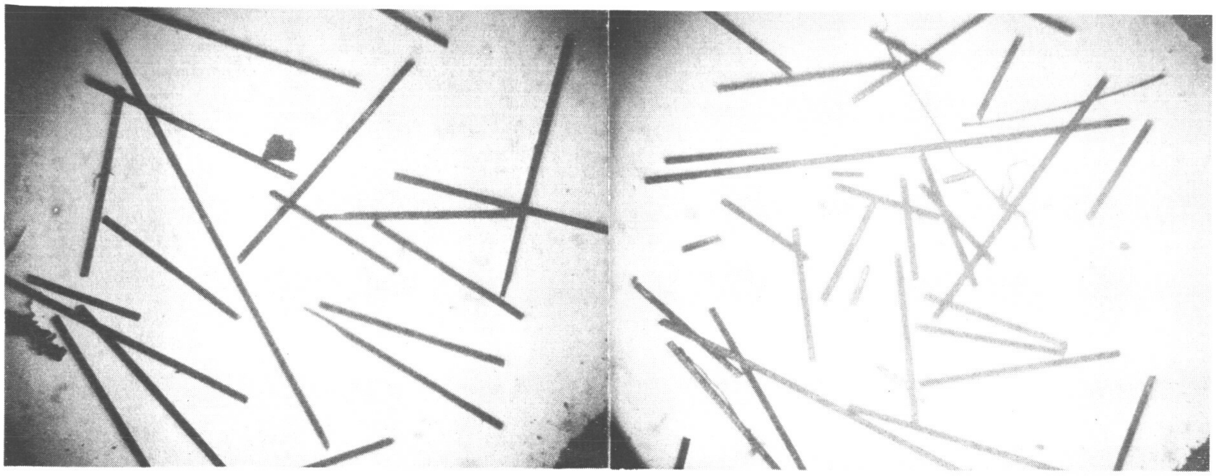


A

TEST 2
DISCONTINUOUS FILAMENTS

B

TEST 3
MULTIPLE FILAMENTS



C

TEST 4
STRAIN AT 370° C

D

TEST 9
TOTAL STRAIN 2%

Figure 7. Examples of Filament Break-up in B_4C/W-Aluminum Composites. The Filaments were Extracted from the Aluminum after the Tests were Performed, (Mag. 17X). For Details see Table IV.

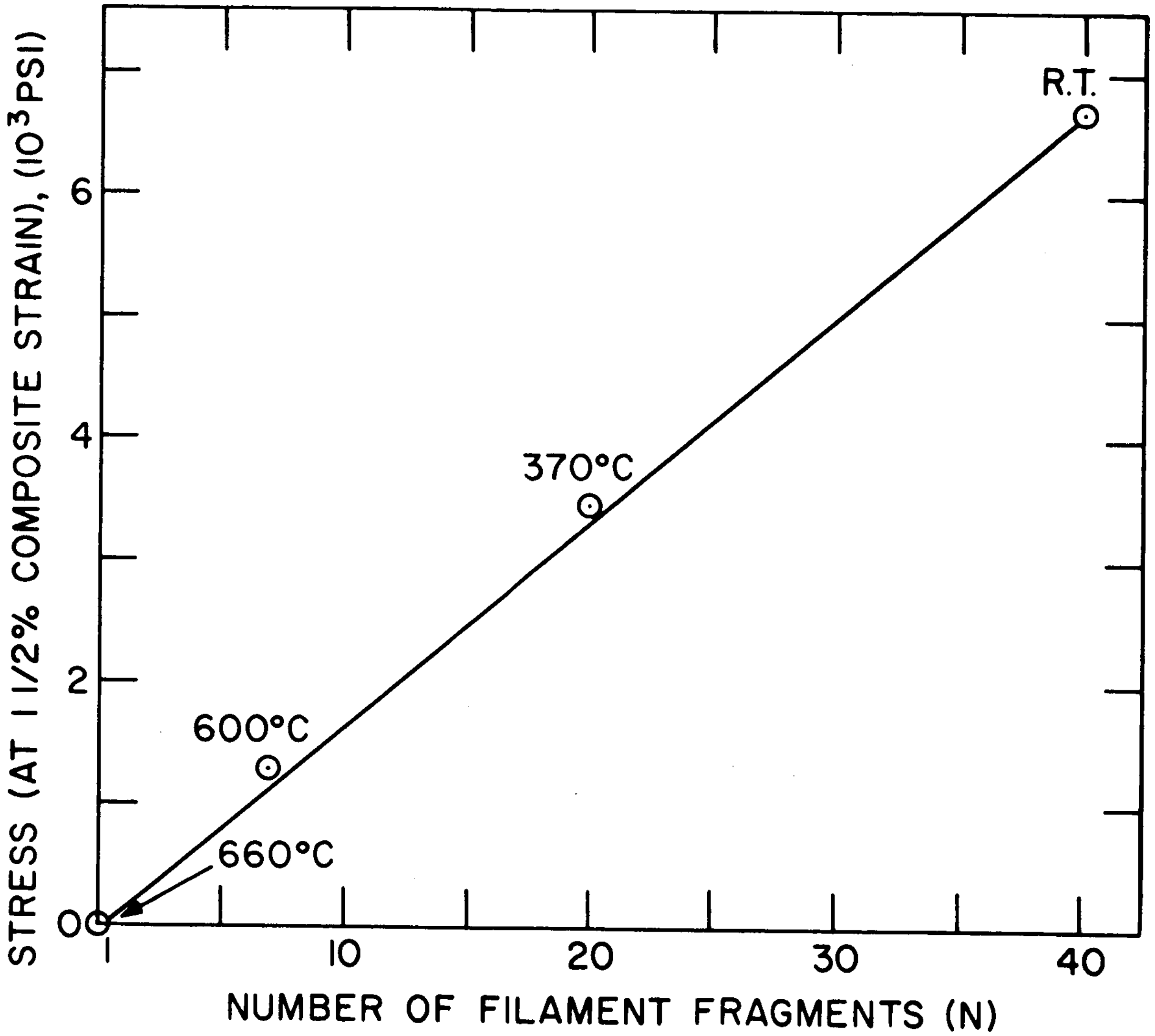


Figure 8. Stress (at 1 1/2% Composite Strain) for a Given Temperature vs. the Number of Pieces of Filaments Extracted from the Composite After Testing

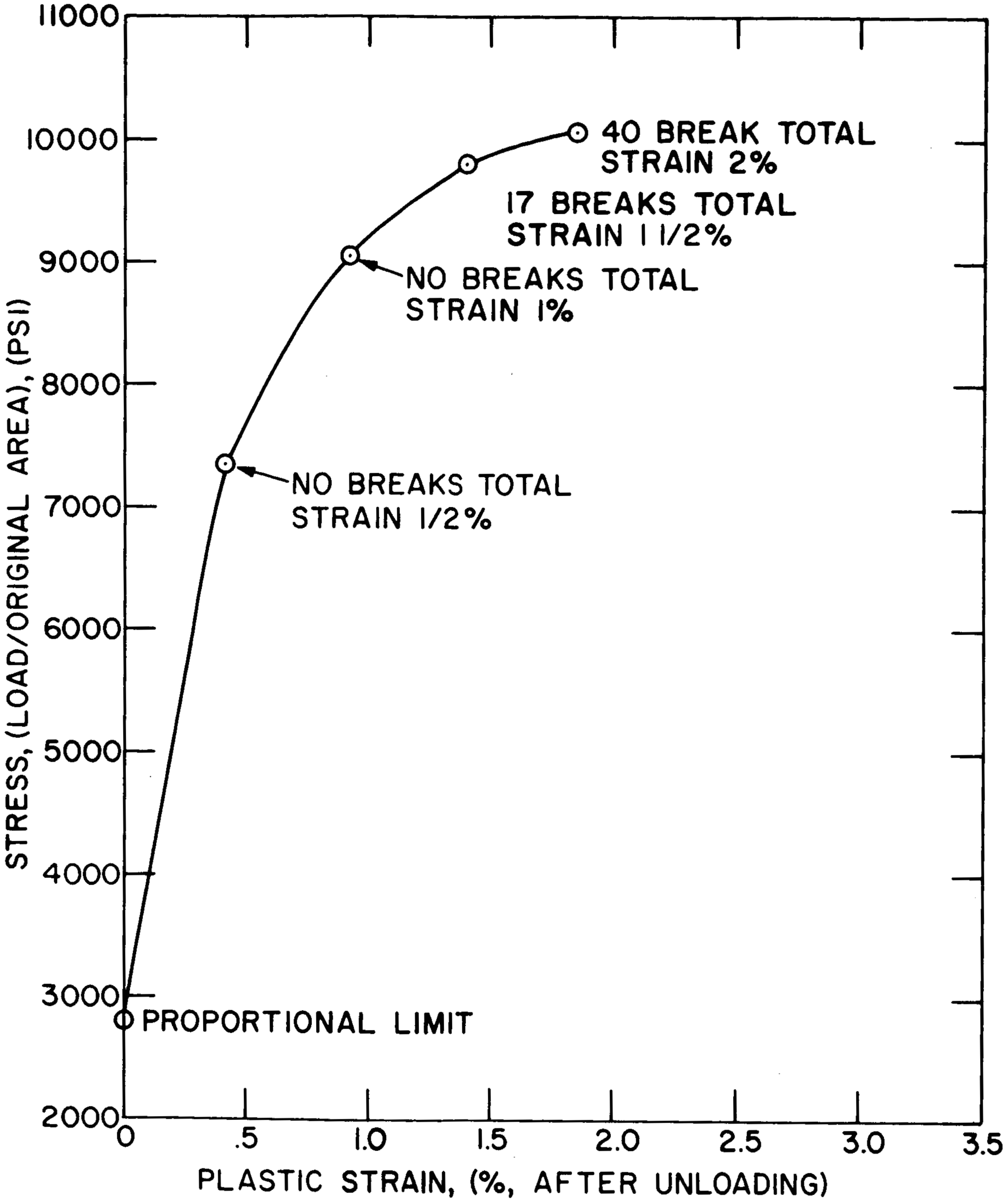


Figure 9. Filament Break-up in Unreinforced B_4C-Al Composites as a Function of Elastic and Plastic Strain

samples tested vs. the number of pieces of filament extracted. Plotted in this manner, the data give a further graphic picture showing the shear strength* of the matrix to be a diminishing function of temperature.

The plastic strain used in most of these experiments was much larger than the expected fracture strain of B_4C/W filaments. The first result obtained at a low value of plastic strain (Test No. 3, Table IV) suggested that, as might be expected, filament break-up is essentially complete at quite small strains. Four tests were made using an extensometer attached to the specimen gage length (tests 7-10), and from these the quantitative relationship between strain and the extent of break-up was established. Figure 8 shows as a single curve the results of the four tests in terms of the stress and plastic strain prior to extracting the broken filaments from the four specimens. Strains of less than 1% (total) did not cause filament breakage, while strains of 2% total resulted in the maximum breakage (~40 pieces). Based on an average strain of 1-1/2% and an elastic modulus of B_4C of 60 x 10^6 psi, this experiment indicates the B_4C filaments ~ 0.1" long had an average tensile strength of 900,000 psi and that, therefore, the shear strength of the strain hardened aluminum adjacent to the filament was ~ 18,000 psi based on the relationship $L_c/d = \frac{\sigma}{2\tau}$. Since both values appear to be somewhat higher than expected, it may be that some strain occured in the aluminum before it started to grip the filament. On the other hand, tensile fracture in such composites was obtained on extremely well aligned "specimens" having very short gage lengths. Since it is commonly observed that the strength of brittle filaments increases with decreasing gage length, strengths of the order of 900,000 psi for 0.1 to 0.2 inch gage-length specimens, may be the rule rather than the exception. Additional work is planned to determine the facts in this matter. Specimens will also

---

* It is assumed that the shear strength of aluminum is directly proportional to its tensile or yield stress.

be fabricated differently using aluminum powder. Filaments will be tested in an epoxy resin of low modulus (to get appreciable strains in the "elastic" region). Finally, conventional tests on short gage length specimens will be made.

III. DISCUSSION AND CONCLUSIONS

1) B_4C whisker growth experiments have been performed using a chemical vapor deposition process which have as their goal the economical production of straight, long, highly perfect whiskers. Data accumulated thus far has shown that the process chosen ($BBr_3 + CCl_4 + H_2$) is feasible. However, all the processing variables have not been optimized because of problems associated with air leakage, residual gases, over-saturation or combinations of these factors.

2) Material characterization studies have confirmed that the commercially pure (1100) aluminum presently used as a composite matrix material has adequate ductility. The B_4C/W continuous filament material presently being used has a 'potential' strength greater than 600,000 psi at lengths of about 0.1 inches. This strength value was arrived at by the extrapolation of the best values obtained during recent testing. It is interesting to note that the calculated strength value of similar filaments, arrived at during composite strength studies as a function of strain, also predict strength values of this magnitude for filaments of similar length.

Electron probe analysis of a cross-section of an infiltrated B_4C/W-aluminum composite which was held molten for 15 minutes at 720°C during fabrication showed no significant interaction between filament and matrix. Thus it would appear that unfavorable interface reactions, which could interfere with or obscure the mechanical behavior of the composites, are not a problem in the B_4C-aluminum system.

3) Previous work using single filament composites for determining an apparent critical fiber length, L_c, afforded a method for qualitatively assessing the strength of the filament by examining the fractured surface of the extracted pieces. Present work, using similar composites, has given additional qualitative data concerning the strength of the filaments used. Strength values in the range 600,000 to 900,000 psi can be calculated for filaments approximately 0.1 inches long, since the data show that filament fracture occurs at about 1-1/2% strain. However, any slippage of the

filament within the composite, due to poor interfacial bonding, would tend to increase the apparent strain in the filament and thus result in spuriously high values. This possibility will be explored further.

4) Single filament - B_4C/W aluminum specimens tested at temperatures as high as 660°C showed variations in the apparent values of L_c with temperature to be of the right order.

IV. FUTURE WORK

Future studies will include continued efforts to grow B_4C whiskers by an economical process. Further studies of single filament composites in both epoxy and aluminum will be pursued to substantiate the present tentative hypothesis of filament failure. Composites containing continuous filaments will also be fabricated to begin a study of the effect of interaction between fibers on the strength behavior of composites. Once the case for continuous filaments is evaluated more fully, greater emphasis will then be placed on discontinuous reinforcements. A study will also be made of short gage length filament specimens (i.e., 0.1" or less) to determine if a gage length vs. strength variation exists to the point where strength values in the 600,000 psi range can be experimentally determined.

REFERENCES

1. A. Gatti, R. Cree, E. Feingold, "Synthesis of Boron Carbide Filaments," NASw-670 Final Report, July 10, 1964.

2. A. Gatti, C. Mancuso, E. Feingold, R. Mehan, R. Cree, "Study of the Growth Parameters Involved in Synthesizing Boron Carbide Filaments," NASA CR-251, July 1965.

3. A. Gatti, C. Mancuso, E. Feingold, R. Jakas, "Study of the Growth Parameters Involved in Synthesizing Boron Carbide Whiskers," NASw-1205, Final Report, March 1, 1966.

4. A. Gatti, E. Feingold, J. J. Gebhardt, J. Berry, V. A. Cordua, "Study of the Grown Parameters Involved in Synthesizing Boron Carbide Whiskers", NASw-1383, Final Report, November 1966.

5. D. L. McDanels, R. W. Jech and J. W. Welton, "Metals Reinforced with Fibers, Metal Prog., Vol. 78, No. 6, Dec. 1960, pp. 118-121.

6. J. Gatti, J. B. Higgins, R. Cree, E. Feingold, "Boron Carbide Continuous Filaments," TR AFML-TR-65-354, Nov. 1965.

ACKNOWLEDGEMENTS

Acknowledgement is given to Messers. R. Mehan and R. Grosso for their valuable assistance in this program and to Dr. W. H. Sutton who reviewed the manuscript.