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Seventh Quarterly Report

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

by

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ILLUSTRATIONS

SUMMARY

This progress report covers the period from 1 May 1968 to 1 August 1968. 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 selected for study were aluminum (or its alloys) and "ductile" epoxies reinforced with B_4C whiskers or with high modulus filaments, such as $B_4C/B/W$*, SiC/W, B/W, etc. Related tasks in the program include the development of a more economical process for growing B_4C whiskers, the investigation of deposition parameters, the production of continuous B_4C filaments, and the characterization of the individual constituents in the final composites. The latter task involves a study of the structural and chemical interactions of the combined elements (fibers, matrix, coatings, etc.).

The results obtained during this period are summarized as follows:

(1) Filament strength characteristics of liquid infiltrated aluminum-$B_4C/B/W$ composites were determined by etching away the aluminum matrix of a composite (#6 in the present series). A study of 96 tensile tests showed a maximum value of 590,000 psi, minimum values near 18,000 psi, and an average value of 357,000 psi.

(2) A series of 12 Al-$B_4C/B/W$ continuous filament composites containing 50 v/o filament were fabricated by liquid infiltration. Room temperature tensile strength values of these specimens varied considerabley ranging from 198,000 psi to 110,000 psi. It is hypothesized that the scatter in composite strength can be related to the position of the weak area of each filament in relation to its nearest neighbors. Thus completely randomized defects can lead to higher than average composite strengths, while aligned defects in a given cross section can lead to lower than average strength. Examination, both microscopically and by etching, of the failure area of the composites after fracture showed that only one break per filament occurs. Thus it appears likely that the composite failed at a stress which is determined by the failure stress of the first filament break. This lack of apparent cumulative damage behavior is rationalized by proposing a model based on an inhomogeneous strain criteria enabling weak fibers to be stressed above their individual test strength due to interaction with their stronger neighbors.

(3) Elevated temperature tensile tests at 500 C were attempted using identical specimens to those described above. However, no meaningful data could be obtained. Excessive shearing out of the gauge section always occurred and failures which were recorded resulted from grip damage during specimen mounting. Such tests were difficult to perform and meaningful results will not be

*This terminology denotes a multiphase filament in which B_4 is vapor deposited over a boron filament having a W-substrate core. Other filaments, such as SiC/W denote a SiC deposit on a W-substrate core, and such teminology is used throughout the test.

available until larger specimens can be fabricated.

(4) Aluminum specimens containing single, five-filament and ten-filament arrays of continuous $B_4C/B/W$ filaments were fabricated by a hot pressing technique developed earlier [4]. The series of specimens were tested at room temperature in tension both as a function of strain rate and filament-matrix bonding, The extent of matrix-fiber bonding was varied by sputtering iron on filament surfaces and using as-deposited filament surfaces and graphite-coated filament surfaces. Single filament specimens show a definite correlation between the apparent bonding efficiency and the number of pieces etched out of the specimens after tensile testing. A minimum number of pieces is counted for the graphite-coated filaments (2-3), an intermediate number is counted for the as-deposited filaments (4-6) and a larger number of pieces is counted for the iron coated filaments (10-11).

Multi-filament specimens, however, after tensile testing and matrix desolution do not show any variation with surface treatment. It appears that filament interaction dominates during fracture and only one break per filament is observed. These experiments are described.

(5) A qualitative discussion of the relationship between bond strength and composite strength is presented in an appendix attached to this progress report. First, a concept of critical bond strength is developed. Once the degree of bonding exceeds a critical level, matrix failure and subsequent adjacent filament fiber failure occurs. Also, by considering the change in effective cross-section as filament fracture occurs in a composite where filament interaction is possible, it is shown that the rule-of-mixtures can be modified to take the apparent deviation from this mixture rule into account.

It is thus shown that when a volume fraction of filaments becomes large enough to cause a critical spacing, that is, that spacing at which a single filament fracture begins to weaken those next to it, then the strength of the composite will begin to decrease even though the volume fraction of filaments is increased.

I. INTRODUCTION

From a reinforcing viewpoint, whiskers (single-crystal fibers) 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 strengths observed for these whiskers range from about 0.05 to 0.1 of their elastic moduli, the latter values 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 an 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 are a large number of imposing technical problems to be solved in order to achieve the high potential strengths. Many of these problems have not yet been solved.

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 percent of the predicted values.

A list of possible reasons for the low composite strength 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.

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 had been synthesized and characterized in previous studies.[1-4]

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 below:

(1) It is much easier to characterize the relevent 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 so that the effects of discontinuous reinforcements can be assessed.

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

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. Elastic Modulus

B. Matrix Variables

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

C. Composite Variables

11. Volume fractions of components--fiber and matrix
12. Homogeneity of whisker distribution
13. Whisker aspect ratio
14. Whisker orientation
15. Interfacial bond strength

This program was therefore initiated to investigate in detail the behavior of a ductile metal (aluminum) reinforced with various brittle fibers, such as $B_4C/B/W$, SiC/W, B/W, etc. (in both continuous and chopped lengths) to provide data which would be pertinent to whisker-reinforced metals. Included in this investigation was a parallel study using a "ductile" epoxy novolac, which in turn has led to the recognition and documentation of three failure modes possible in fiber-reinforced composite materials. This program is being conducted in two parts: (1) The development of a process to grow B_4C whiskers which would be amenable to eventual scale-up and (2) An investigation of the reinforcement of aluminum and "ductile" epoxies with brittle, high modulus filaments, such as B_4C which would simulate the B_4C whiskers.

A review of the results of the first year of effort [6] includes the following:

(1) Extensive studies were made of B_4C whisker growth systems which utilized chemical vapor deposition rather than direct B_4C bulk vaporization. These systems included boron tribromide + hydrogen + CCl_4 and the volatile substituted boranes, tributyl bo- ne and ethyl decaborane. This approach appeared to lend itself most easily to whisker growth scale-up.

(2) A ready supply of $B_4C/B/W$ filament materials was necessary to continue composite studies. A previously developed process [6] was modified so that purchased B/W filaments could be coated with a layer of B_4C while still maintaining the high strength capability of B/W filaments. B/W filaments coated in this manner were able to withstand molten aluminum for significant times without reaction, and thus allowed liquid infiltration techniques to be used in the preparation of composites.

(3) $B_4C/B/W$ filaments were the mainstay of the filament-composite work. However, many other filamentary materials including, B/W, SiC/W, B/SiO_2 and W were also examined for potential usage and to document the mechanical behavior characteristics of composite materials as a function of individual filament characteristics. A final phase of the characterization portion of this study considered the matrix-filament stability of aluminum-filament composites of the Al-$B_4C/B/W$ and Al-SiC/W systems.

(4) Composite studies encompassed a variety of filaments utilizing "ductile" epoxy novolac resins and aluminum matrices using both single and multiple filament arrays. This work has established experimentally the critical fracture modes of an epoxy matrix in the vicinity of a break in a high modulus, high strength filament. Three distinct failure modes were observed to occur and the nature of these three modes was explained through an analysis of the stress state in the matrix [6].

(5) Continuous filament Al-$B_4C/B/W$ composites were fabricated which showed full utilization of the strength potential of individually tested filaments in a number of instances. SiC/W-Al composites, however, suffered a large decrease in strength because of an unfavorable reaction between the filaments and a Ti/Ni coating which had been used to promote wetting.

(6) By being cognizant of the literature concerning composite mechanical behavior coupled with the present study, a tentative judgement of the expected mechanical behavior of all types of fiber composite materials was made through consideration of individual filament strength and modulus, bond strength between filament and matrix, matrix and filament ductility, and matrix and filament mechanical response to strain rate changes.

The report contained the work performed for NASA under Contract NASw-1543 and covered the period from 1 November 1966 to 31 October 1967. However, it was decided to continue the present program under the same charter and Contract No. (NASw-1543). Thus this progress report is No. 7 in the quarterly series.

Studies during this 7th quarter included a statistical study of B_4C/B/W filaments which had been infiltrated with aluminum and removed from the resulting aluminum matrix by etching with HCl.

Composite studies during this reporting period dealt exclusively with aluminum matrix B_4C/B/W composites of two types. A group of single layered composites were fabricated containing arrays of one, five and ten continuous filaments. These composites were then tested in tension at room temperature and their mechanical behavior examined. These results are described. A second group of aluminum-B_4C/B/W 50 volume percent composites were also fabricated by liquid infiltration and these specimens were tested in tension as a function of temperature and strain rate. These results are presented together with a proposed model to explain the results.

A qualitative discussion of the relationship between bond strength and composite strength is included in the appendix. This work is the rationale of the epoxy-filament studies presented in earlier progress reports.

II. EXPERIMENTAL PROCEDURES - RESULTS, DISCUSSION

A. Preparation of Continuous Boron Carbide Coated Filament

During the present reporting period, no new results were obtained nor was any additional filamentary material made. A careful evaluation of the processing parameters is now being made, however, and this summary will be an important part of the final year end report.

B. Characterization of Composite Materials, Filament Evaluation

Since this program is concerned with an investigation of the factors which control the mechanics and the physical and the chemical behavior of metal matrix composites reinforced with brittle discontinuous fibers, it is very important that parameters which affect this behavior be well identified and characterized. The approach used evolves simply from the 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.

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. During this reporting period, the materials extensively tested were $B_4C/B/W$ filaments which had been fabricated into a composite by liquid infiltration of aluminum at 720 C for 10 minutes and then the aluminum removed by etching in HCl solution (see Figure 1). Ninety-six filaments were tested on an Instron machine in tension at room temperature at a strain rate of .02 in./in./minute. The frequency/strength distribution curve derived from these data are shown in Figure 2. There is an obvious bi-modal distribution of strength peaking in the 100,000-200,000 psi range and the 400,000-500,000 psi range.

Figure 1. 300 $B_4C/B/W$ filaments which had been etched out of an aluminum infiltrated composite by a 50% solution of HCl (1/2 size)

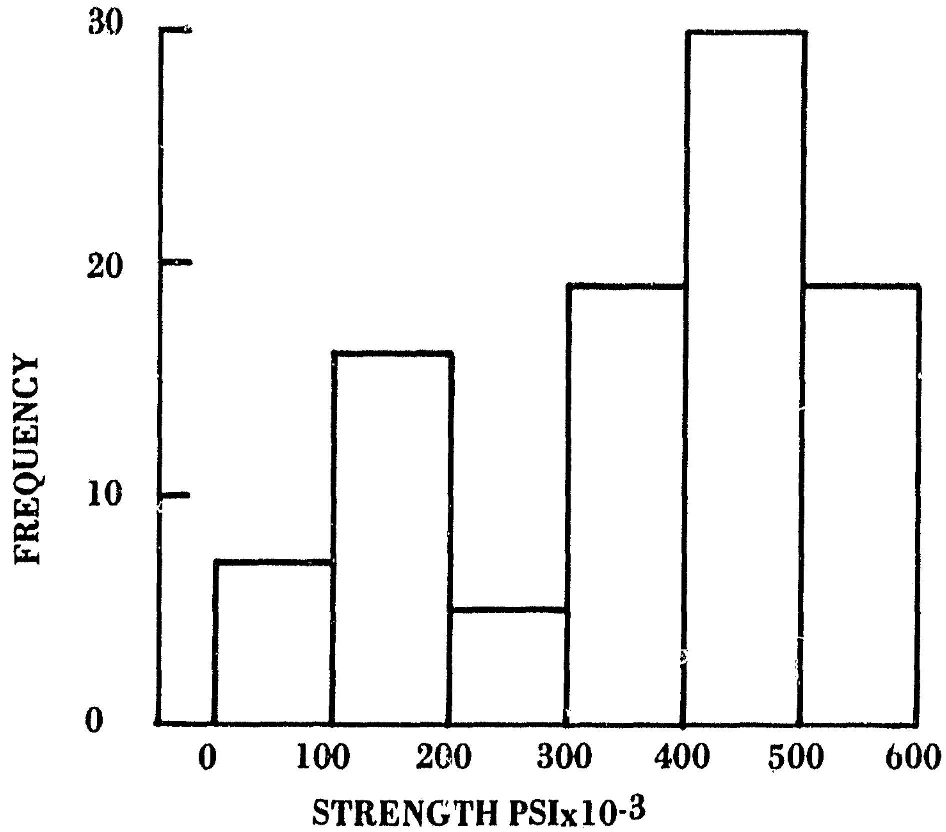


Figure 2. *Frequency/strength distribution curve for 96 etched out $B_4C/B/W$ filaments.*

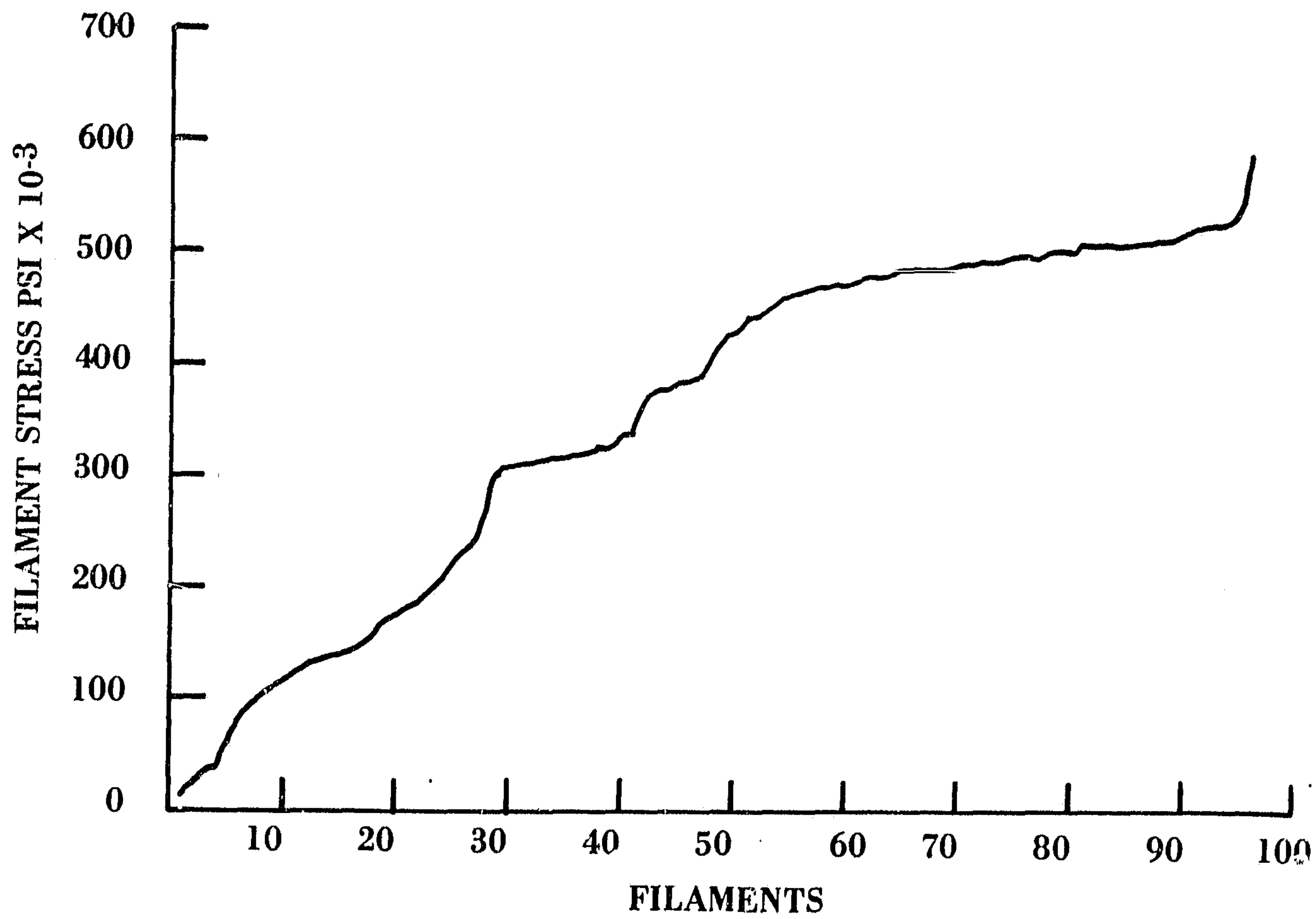


Figure 3. *Failure stress of 96 filaments plotted from lowest to highest values*

All the tensile data is shown in Figure 3, plotted from lowest to highest value as a convenience. This plot is utilized later to arrive at Figure 12 in Section II C. The lowest value recorded was 18,000 psi while the highest value reached by a single filament was 590,000 psi. The numerical average value of all the tests was 357,000 psi.

These filaments, which had received all the various processing manipulations necessary to form composites, did not show appreciable weakening when compared to virgin material tested in the as-deposited condition.

C. Aluminum Matrix Composite Systems

Prior work has shown that the strength predicted by the rule-of-mixtures was achieved in many continuous $B_4C/B/W$ composites. It was also shown that $B_4C/B/W$ filaments are ideal for aluminum-based composites, since they are capable of maintaining their chemical and mechanical stability at high temperatures. Also, studies with thin epoxy-filament samples had delineated the fracture modes to be expected in composite materials.

During this reporting period, thin Al-$B_4C/B/W$ specimens were fabricated to extend the epoxy-filament work to metal matrix systems, and also 50 v/o continuous filament $B_4C/B/W$-Al composites were fabricated to study the tensile behavior of large composites at both room and elevated temperatures.

1. Al-$B_4C/B/W$ SINGLE, 5-and 10-FILAMENT COMPOSITES CONTAINING CONTINUOUS FILAMENTS

a. Fabrication

A technique previously described[6] was used to produce single, five and 10-filament continuous composites. Briefly, 2 in. long by 1/8 in. wide by .025 in. thick aluminum strips were laminated together by hot pressing at 550 C for 10 minutes at 10,000 psi in a steel die. The filaments were incorporated into the resulting laminate before pressing by alternately laying .006 in. diameter aluminum wires and filaments so that the single filament array was centered in the laminate while the 5 and 10 filament arrays were equi-spaced in the cross section of the laminate. A series of three different filament surface treatments were used: including, as-deposited filaments (uncoated); sputtered with iron (coated); and graphite coated filaments (graphite).

Typical specimens produced by this method were approximately .030 in. thick, .135 in. wide and 2 in. long. All subsequent tensile tests were performed on an Instron tensile machine at strain rates of .02 in./in./minute and 2 in./in./minute with 1 in. gauge length.

b. RESULTS

Owing to very large scatter in the results obtained by tensile testing low volume $B_4C/B/W$-Al specimens, only a few very general remarks seem warranted. The tensile strength results are shown in Figure 4 and the total elongations to failure are shown in Figure 5. The extent of filament break-up as a function of surface treatment is shown, for single filament specimens, in Figure 6.

Although the data are limited, it would appear that a graphite coating reduces the bond strength while iron coating increases it (see Figure 6). It is also quite evident that filament break-up was confined to single filament specimens; that is, 5 and 10 filament specimens exhibited only 1 break per filament, regardless of the kind of surface treatment. Figure 7 is typical of failure in multi-filament specimens (matrix etched away to show the filament integrity). Note that the breaks appear not to be randomly distributed.

Figure 8 contains a pair of load elongation curves for single filament specimens and shows the abrupt load changes which are associated with filament break-up. Perhaps the least ambiguous of the scattered tensile test results (see Figures 4 and 5) is the effect of configuration on elongation to failure. Most of the single-filament specimens are slightly less ductile than plain aluminum specimens. The multi-filament specimens are markedly less ductile than aluminum and also less ductile than most of the single-filament specimens. Apparently, the concentration of filament failures (see Figure 7) induces, what, in a homogeneous specimen, would be designated as premature necking.

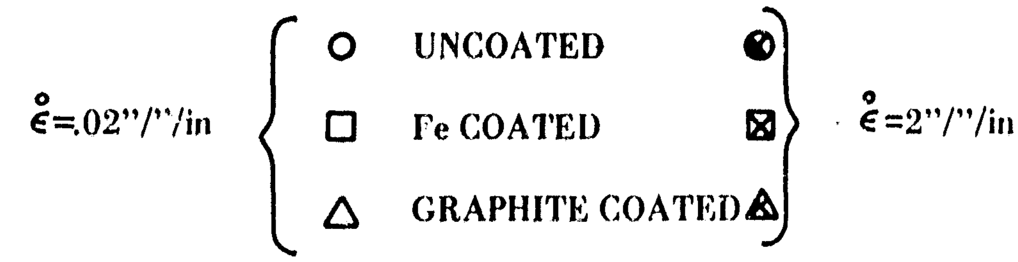
The cumulative damage rationale for composite strengthening involves the proposition that reasonably extensive filament break-up (at weak regions in brittle filaments) will not preclude reasonable composites strengths. These data, while not definitive, indicate the phenomenon of filament break-up will not occur in $B_4C/B/W$-Al composites having useful volume fractions. As will be discussed later, this has been confirmed for 50 volume percent composites which are quite strong without the cumulative damage phenomenon.

2. CONTINUOUS ALUMINUM-$B_4C/B/W$ COMPOSITES

a. Fabrication

A fabrication technique for producing round iron test bars containing a core of 50 volume percent $B_4C/B/W$-aluminum composites was described in the 6th Quarterly Progress Report. Several such specimens were made. However, attempts to use these specimens for high temperature tensile tests resulted in pull out due to insufficient shear area in the grips. These specimens are adequate for room temperature testing and will be used to determine the room temperature fatigue properties of these materials.

Others[7] had designed a flat tensile bar mold for liquid infiltration such that part of the filament containing volume could be physically held in serrated grips. The flat tensile bar is better illustrated



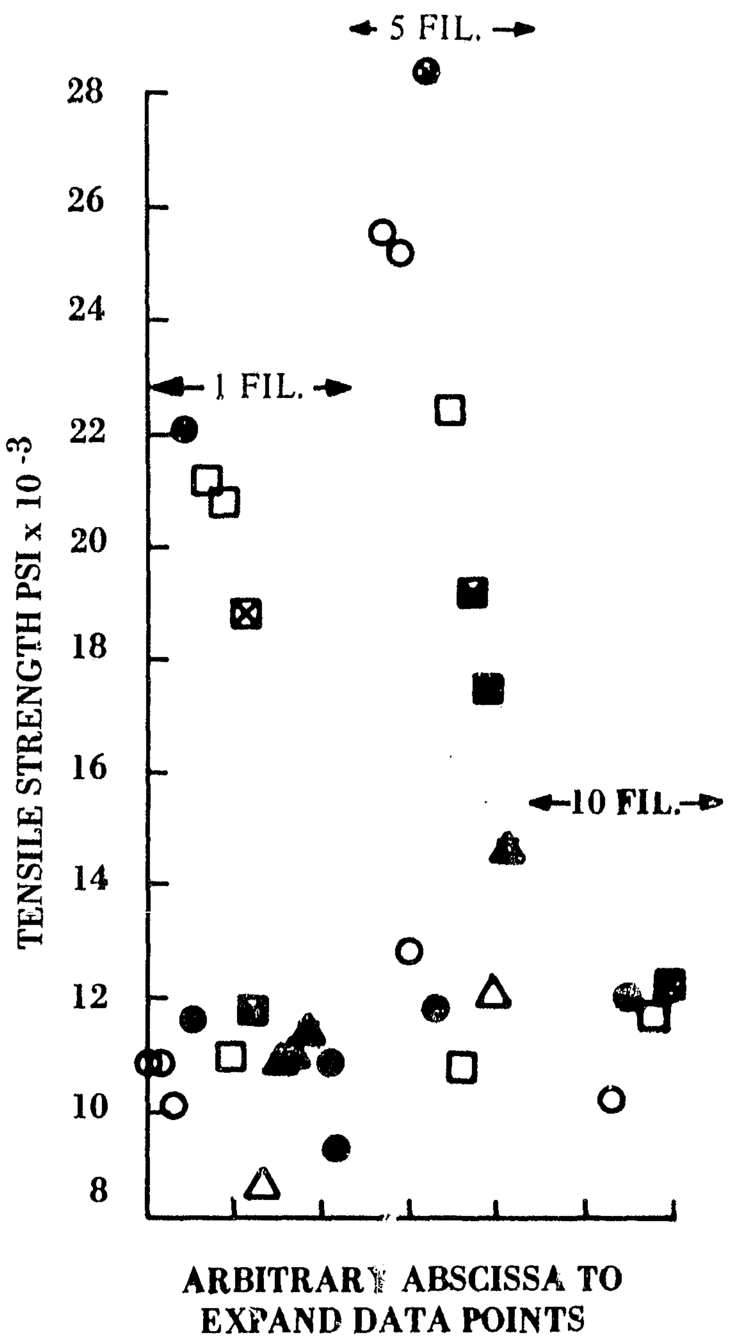


Figure 4. The effect of surface treatments on the tensile strength of low volume fraction $B_4C/B/W$-aluminum specimens

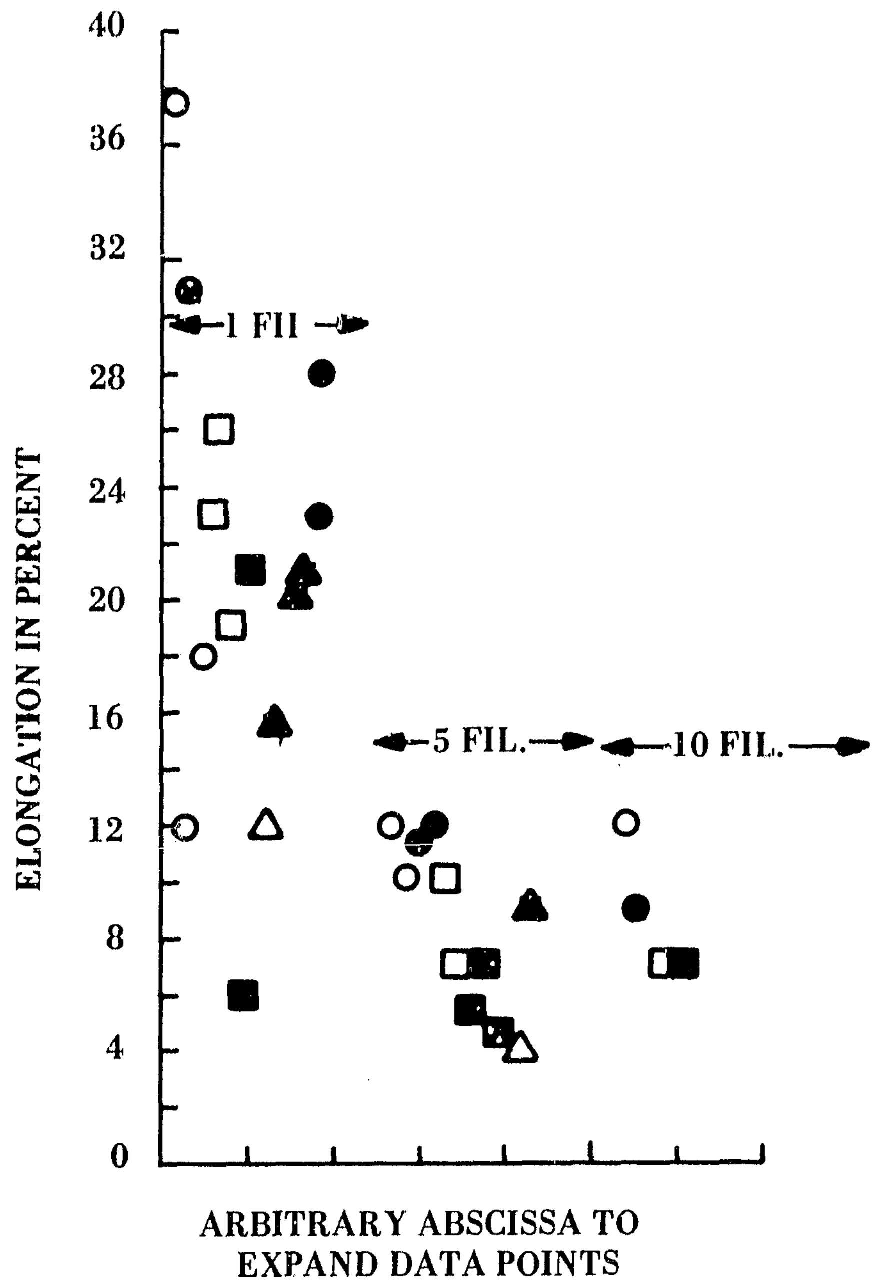


Figure 5. The effect of surface treatments on the tensile elongation to failure in low volume fraction $B_4C/B/W$–aluminum specimens

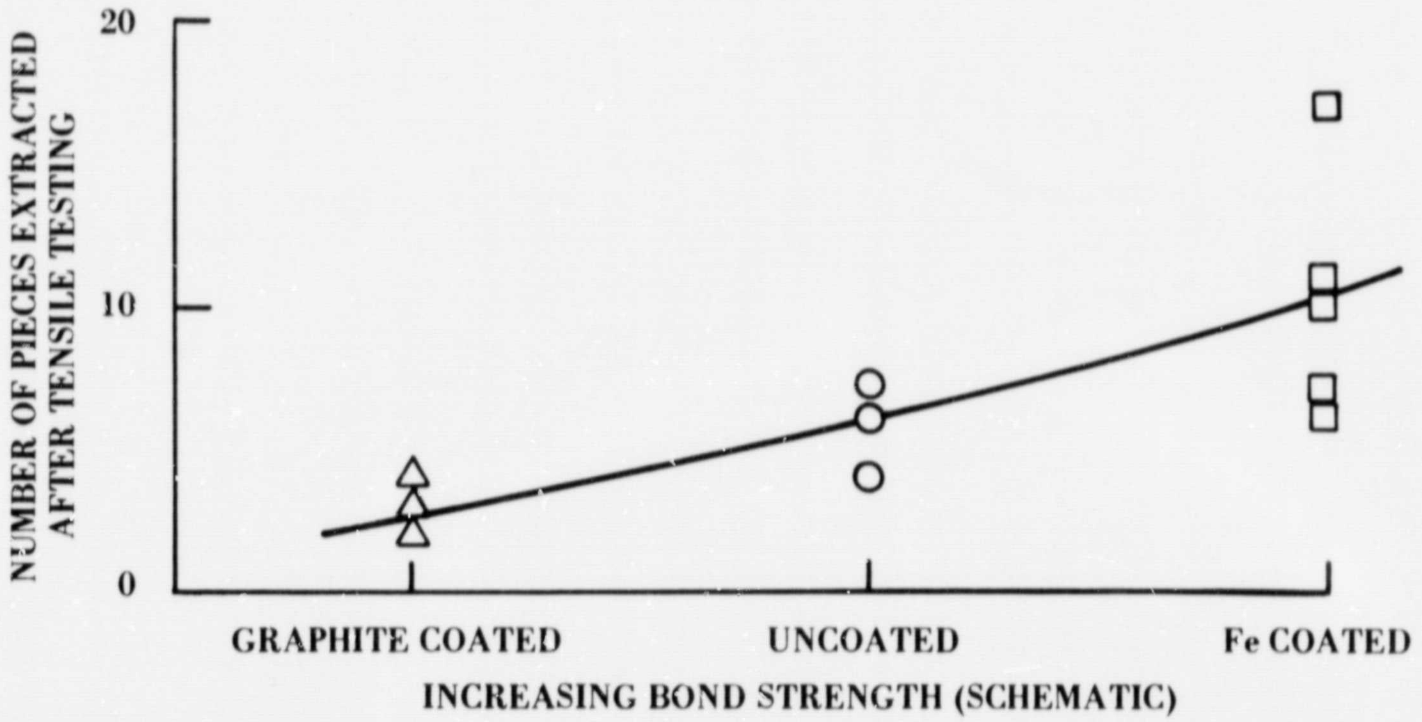


Figure 6. The effect of surface coatings on filament break-up in single filament $B_4C/B/W$-aluminum specimens

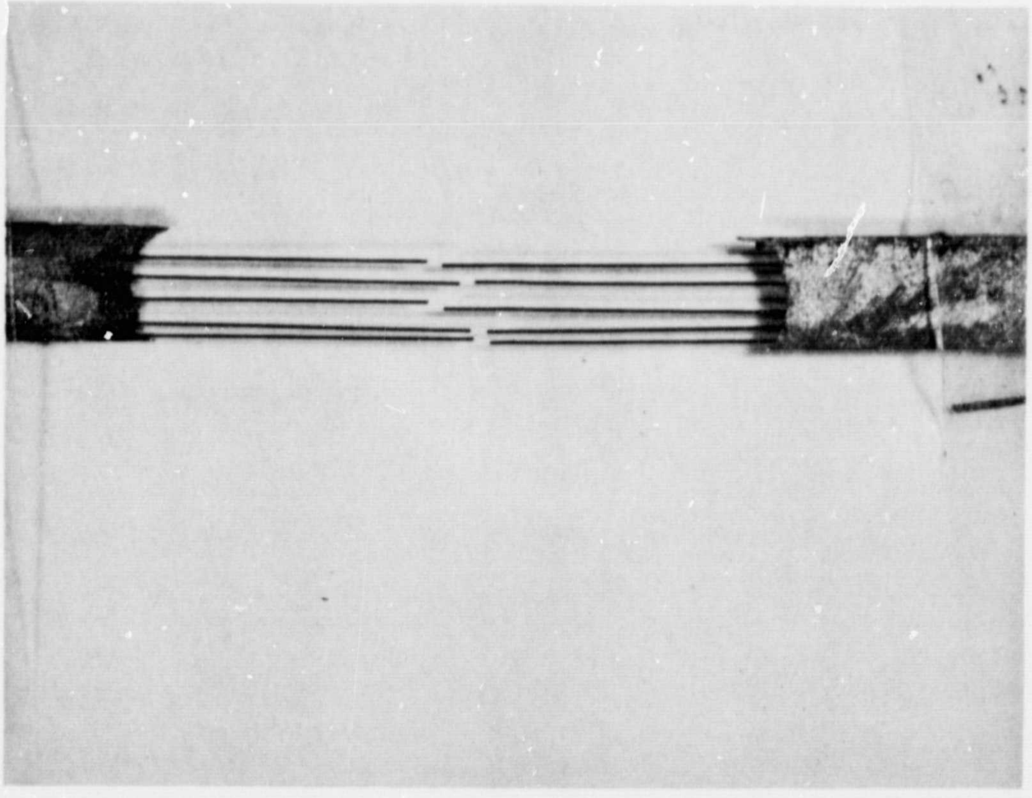


Figure 7. Typical 5-filament specimen after tensile failure (matrix dissolved to show filament integrity)

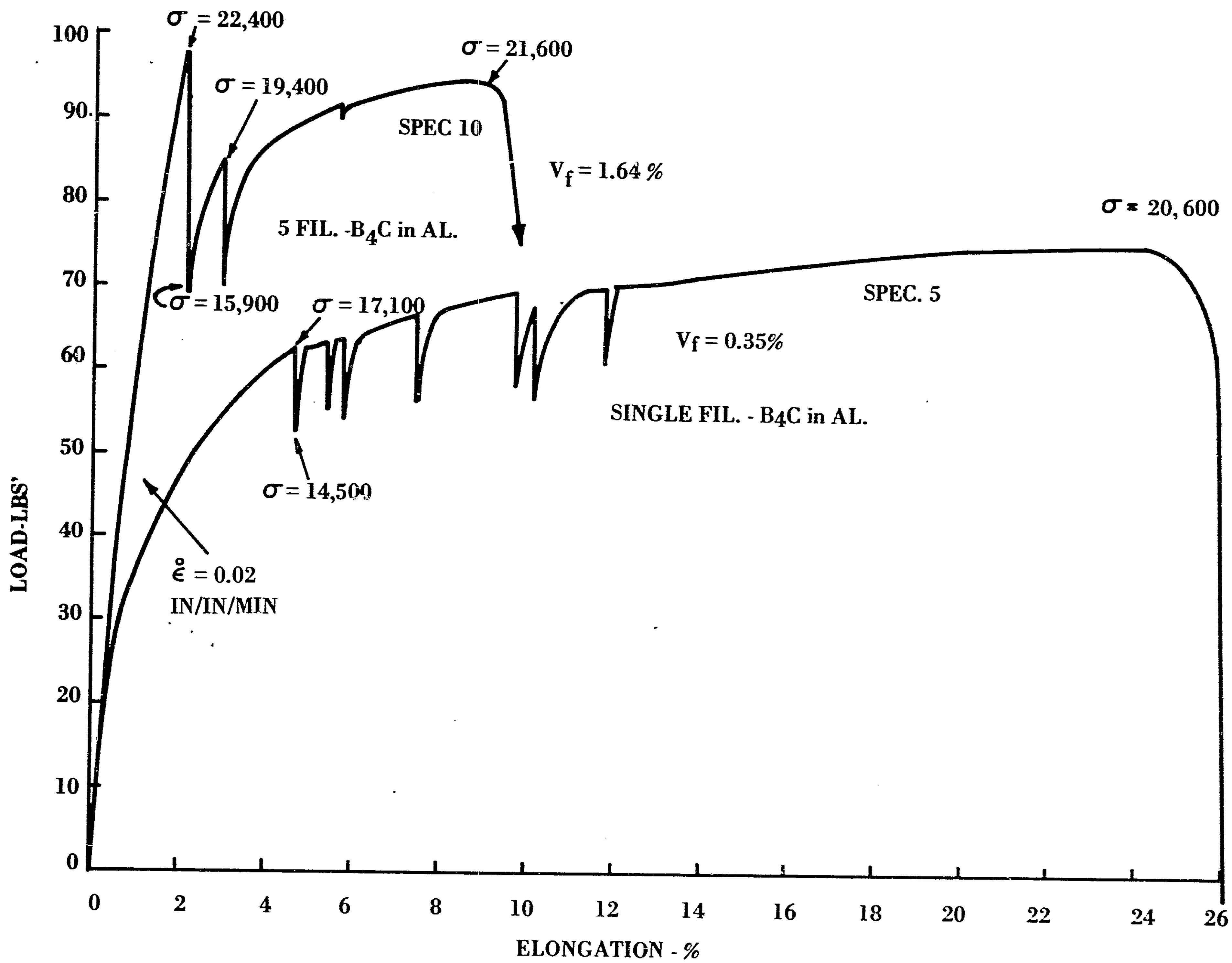


Figure 8. *Typical load-elongation curves for single and five-filament $B_4C/B/W$-aluminum specimens*

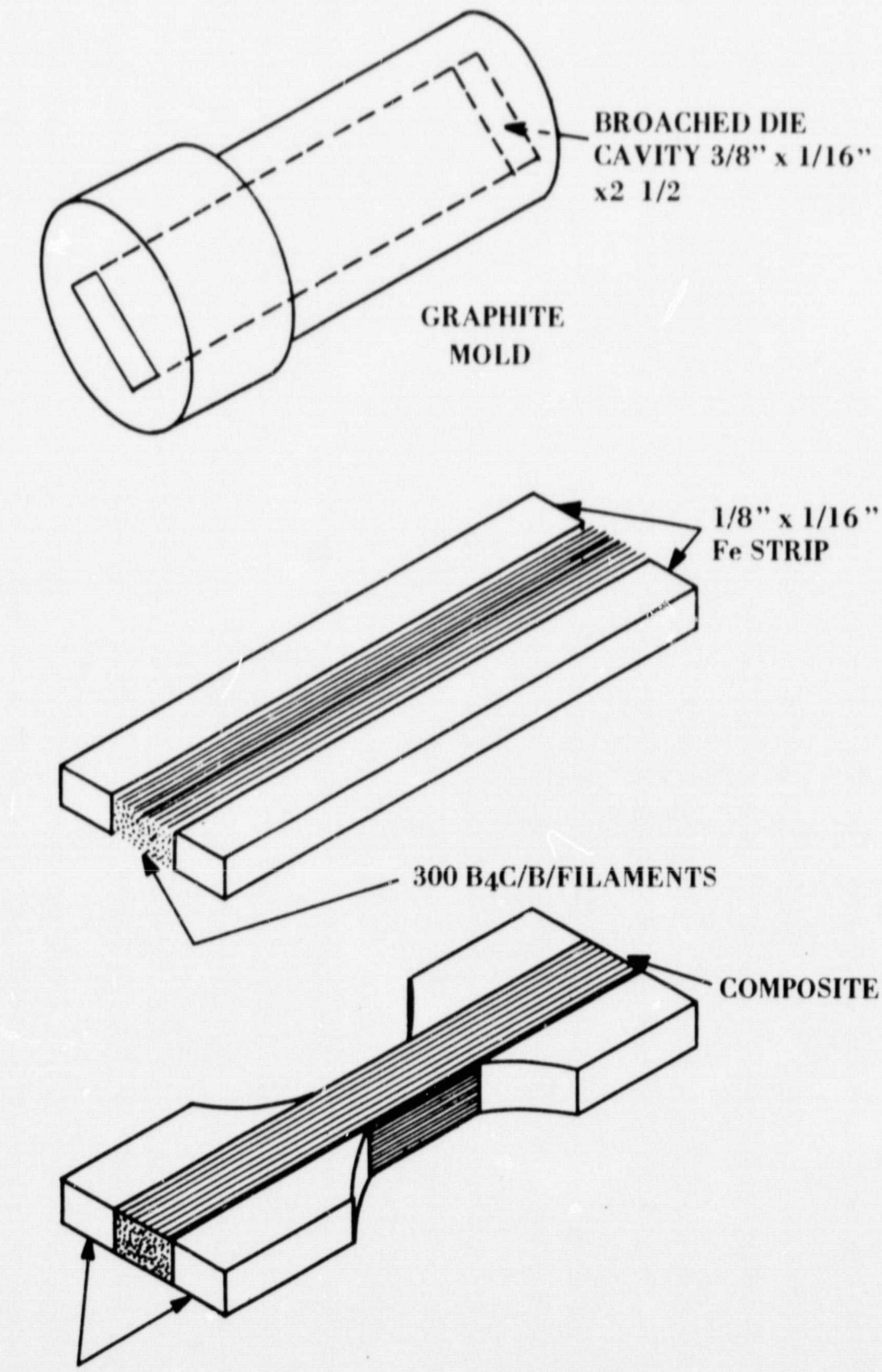


Figure 9. Schematic view of flat tensile bar molding and bar design

by Figure 9. The iron strips were incorporated in the mold cavity so that the number of filaments needed to completely fill the cavity was reduced by 2/3 as a filament conservation step. All tensile bars were designed to contain 50 volume percent $B_4C/B/W$ filament. This composite section of the specimen measured 1/16 in. x 1/8 in. x 2 in. and contained 300 filaments. After removing the iron from the test section, it is seen that a tensile bar containing a 1 in. gauge section of Al-$B_4C/B/W$ composite can be formed. Unfortunately, this design also proved incapble of testing at high temperatures and indeed also pulled out at room temperature. A reduced section in the composite area was also tried (Figure 10) so that only 1/3 of the original composite cross-section remained ($\sim$100 filaments) still without success. The twelve specimens fabricated were salvaged for room temperature measurements by stripping the iron slabs from the grip area and resorting to the technique [6] of inserting the composite bar into aluminum grip tabs with epoxy adhesive.

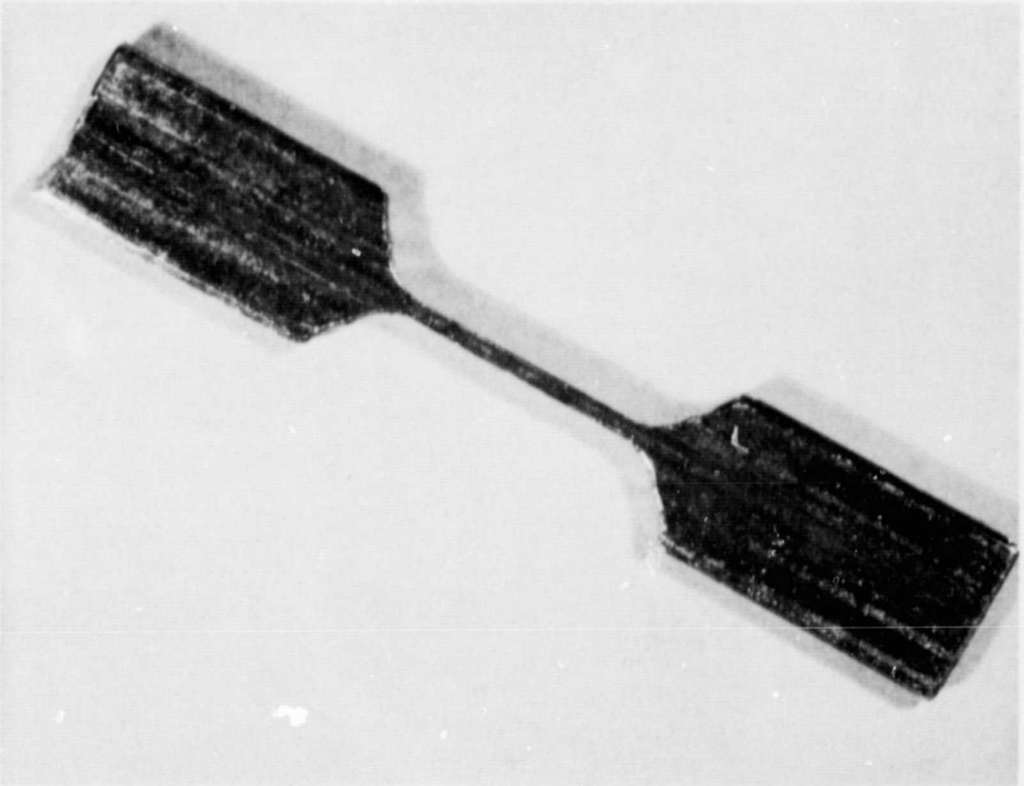


Figure 10. Flat tensile specimen containing reduced composite cross-section

b. Tensile Test Results

Table II contains a summary of the tensile test results obtained on the 50 volume percent continuous $B_4C/B/W$-aluminum composites fabricated during this quarter. Tensile tests were performed on an Instron machine. All specimens were tested at a strain rate of .02 in./in./Minute, unless otherwise designated in the table. (All specimens which pulled out of the grips, but were not otherwise damaged, were re-tested.)

Tensile data at 500 C, although recorded, is only a measure of the stress necessary to overcome the specimen shear area in the grips. All high temperature tests pulled out in this manner. Data obtained on specimens tested at 2 in./in./Minute strain rate also proved invalid because of inadequate pen response on the Instron. A simple exercise using 1/16 in. diameter drill rod specimens showed that the machine was only capable of reliably testing specimens at strain rates of .5in./in./Minute or less.

Figure 11 is a plot of all the room temperature tensile data for 50 v/o continuous B_4C/B/W-aluminum composites at .02 in./in./Minute strain rate, accumulated during this study period. Included in the firgure is the predicted (rule-of-mixtures) strength of composites using the average value of filament strength as reported in an earlier section. As can be seen, the value scatters both above and below the predicted value of 175,000 psi.

c. Discussion of Results

The tensile test data on continuous composites fell both above and below the rule-of-mixtures predictions. Considerations of "mechanical compatibility" might be invoked to explain sub-average results, but the higher strength observed requires additional considerations. As a beginning, it is useful to examine the matter of bundle vs. composite strength expectations.

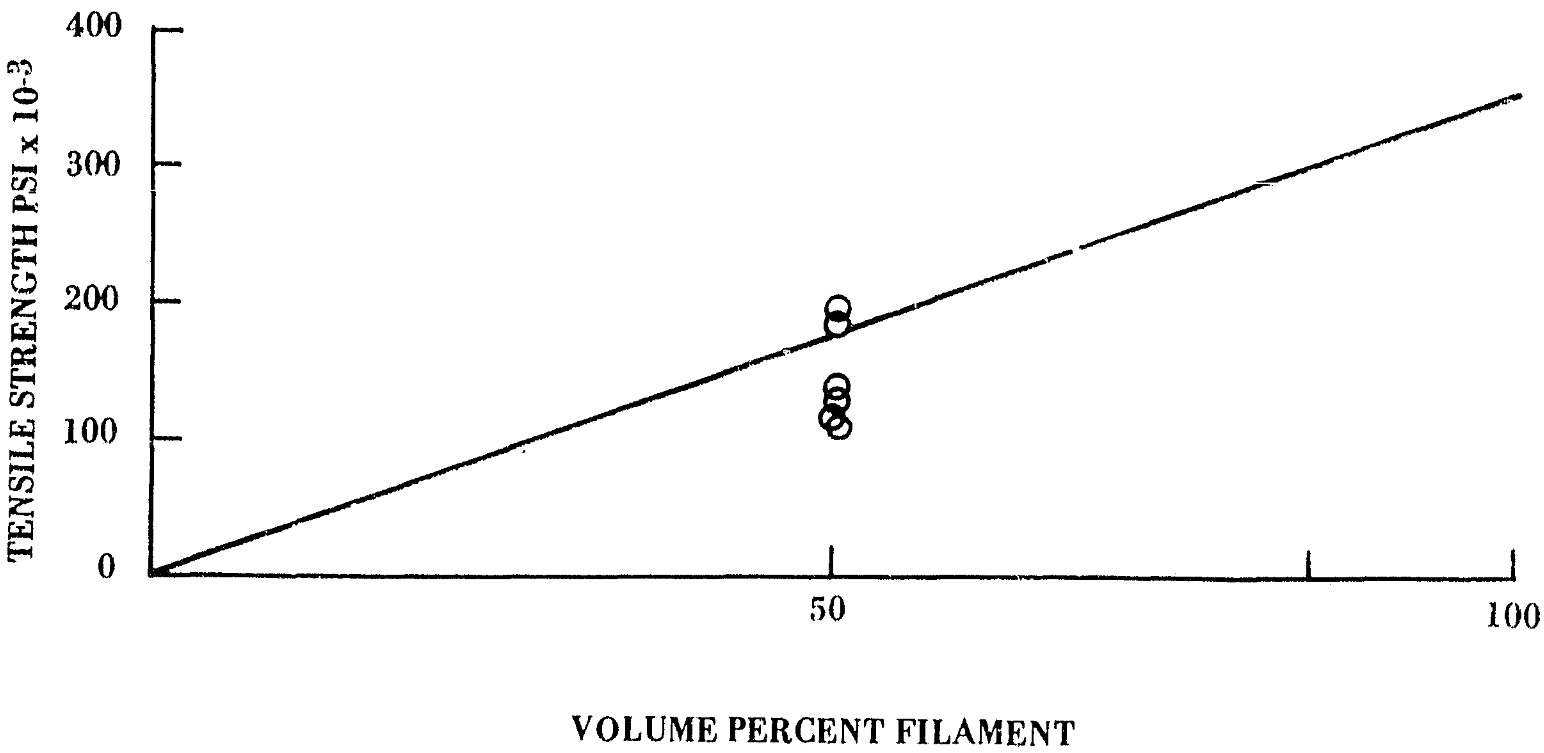


Figure 11. Room temperature tensile strength of 50 v/o continuous B_4C/B/W-aluminum composites compared to the predicted value using the rule-of-mixtures based on the average filament strength of 357,000 psi

The effect of the filament strength distribution on the expected bundle strength is shown in Figure 12. The strength distribution was obtained by extracting 96 filaments from a B_4C-Al composite and testing each individual filament in tension (Section II B). The strength values were arranged in the order of increasing strength, and the median of each group of eight specimens were plotted using data from Figure 3 (see also Figure 12) and the solid line, then, represents the estimated strength distribution for 100 specimens. The abscissa shows the number of filaments (in a bundle of 100) which would be broken as the stress *per unbroken* filament increased. The dashed-lines in Figure 12 show the stress per filament at constant load and they curve upward because the stress on the unbroken filaments increases as the number of filament failures increase. The intersection of these curves with the ordinate is the average stress on the 100 filaments if none had been broken.

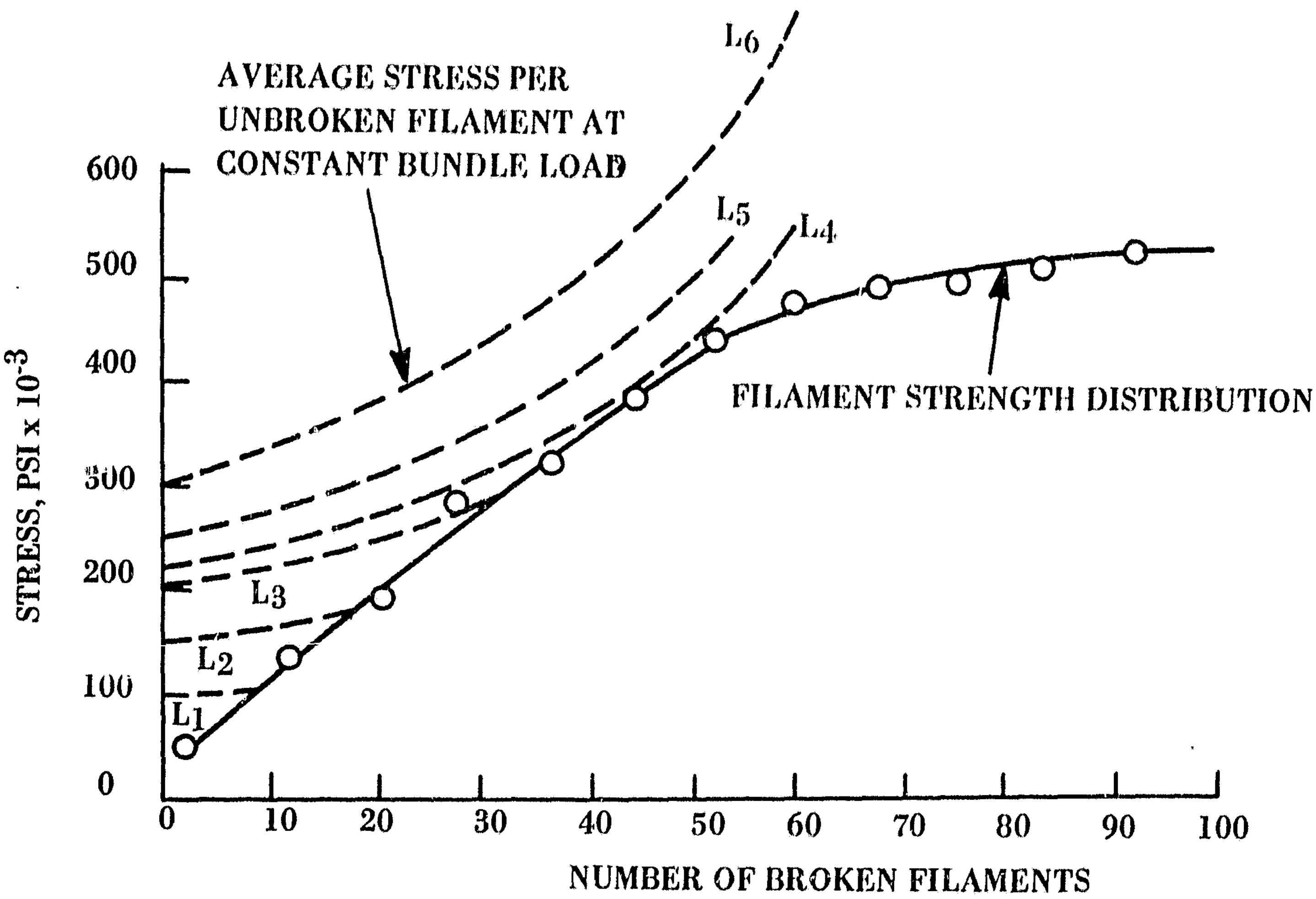


Figure 12. The effect of filament strength distribution on bundle strength

From an examination of Figure 12, it is possible to hypothesize a load deflection curve for the filament bundle. It is assumed that filaments will continue to fail at a constant load until the strength of the unbroken filaments becomes greater than the rising stress occasioned by failure of the weaker filaments. The bundle will fail at a load characterized by a dashed curve tangent to the strength distribution curve (L4 in Figure 12). This is, in this case, a load equivalent to an initial average stress of 220,000 psi/filament will cause the bundle to fail catastrophically, because the stress per unbroken filament is above and increasing more rapidly than the strength of the remaining unbroken filament.

Since the number of broken filaments will increase from approximately 2% at 50,000 psi to approximately 42% at the onset of catastrophic failure, the stiffness of the bundle will continue to decline as the load rises during the tensile test. The effect of filament failure (from Figure 12) on stiffness is also taken into account in synthesizing the bundle (non-linear) load-elongation curve in Figure 13. It is interesting to note that this non-linearity would not be easily distinguished from approximate 0.3% uniform plastic deformation in a ductile material. The flat transition region at the top of the curve would be expected to continue so long as the slope of the curve for rising stress on unbroken filaments was equal to the slope of the filament strength distribution curve (at the point of tangency in Figure 12). In this transition region, of accelerating filament breakage, the rapidly decreasing bundle stiffness precludes a bundle load increase (in a constant cross-head motion test).

There are several significant similarities and differences between the hypothetical bundle (see Figure 13) and the behavior of a B $_4$C/B/W-aluminum composite shown in Figure 14. Composite No. 9 (see Figure 14) contained 100 B_4C filaments (same batch as filament used in strength-distribution work, shown in Figure 12) in an (infiltrated) aluminum matrix (see Table II). The data for the plain aluminum was obtained on a specimen of approximately the same cross-section. The comparison is summarized in Table III.

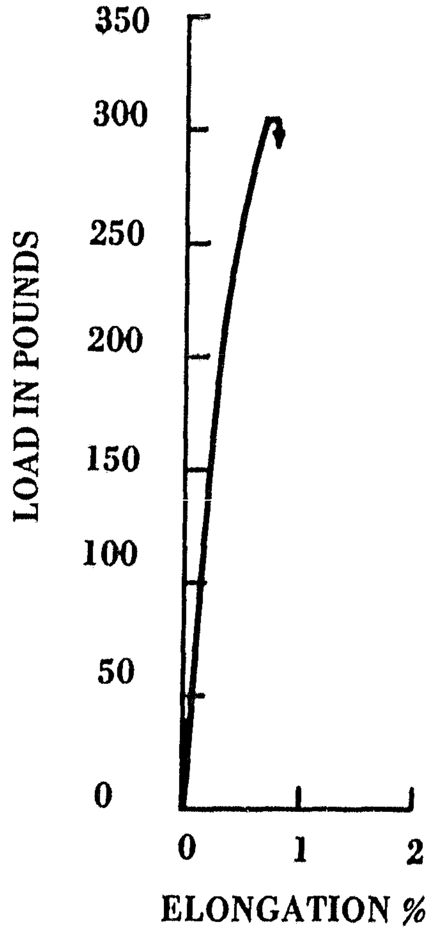


Figure 13. Synthesized load/deflection curve for a bundle of 96 filaments (from Fig. 3)

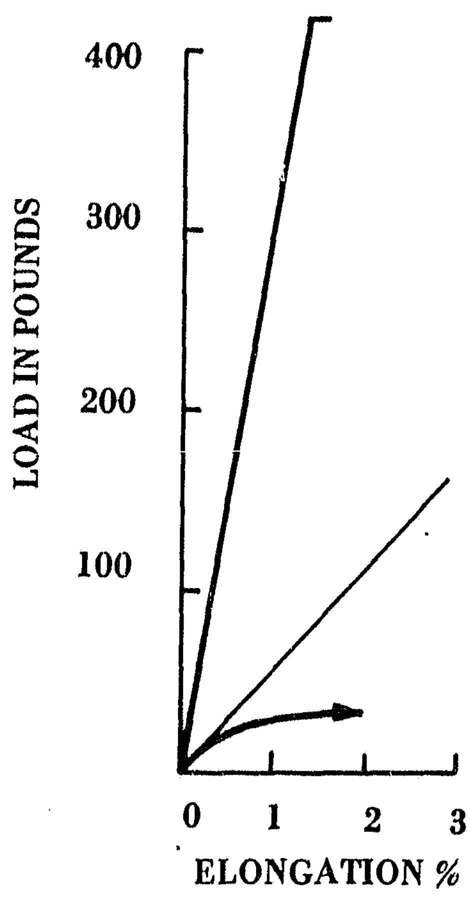


Figure 14. Load/elongation curves for specimen # 9 and pure aluminum

The stiffness difference need not be regarded as significant when comparing hypothetical with real tests owing to the "softness" of tension testing machines.

TABLE II. TENSILE TEST RESULTS OF 50 v/o CONTINUOUS $B_4C/B/W$-ALUMINUM COMPOSITES

Spec. #	Load (Lbs.)	Tensile Strength (P.S.I.)
1 (1)	---	---
2	330	141,000
3 (2)	---	---
3 (4)	135	55,500
4	230	110,000
5 (2)	187	67,800
6 (5)	---	---
7 (6)	480	198,000
8 (7)	307	116,000
9	420	186,500
10 (3)	310	131,000
10 (4)	140	56,700
11 (2)	---	---
12	Not tested	

(1) Specimen lost during machining
(2) Tested at 500 C – pulled out of grips
(3) Tested at room temperature – pulled out of grips
(4) Tested at 2 in./in./Min. strain rate
(5) Etched out filaments after infiltration cycle
(6) Cycled to 500 C once
(7) Tested at .5 in./in./Min. strain rate

TABLE III. COMPARISON OF HYPOTHETICAL BUNDLE OF 100 $B_4C/B/W$ FIBERS WITH COMPOSITE SPECIMEN

Load Deflection Characteristics				
Sample Decription	Stiffness	Shape	Transition Region	Max Load per Filament, lbs
Hypothetical Bundle	Based on Elastic Modulus	Non-linear	Flat	3.05
Composite 9	Less stiff than bundle	Linear	Flat	4.20 (3.92)[1] (3.45)[2]

[1] Load sharing by Al in plastic range

[2] Load sharing by Al elastic deformation extrapolation

It seems reasonable to assume that the shape differences in the load-elongation curves are significant. The results suggest one of two possibilities: (1) filaments break in increasing numbers as the load rises, but being well-bonded, their contribution to stiffness is not appreciably diminished or (2) few, if any, filaments break prior to catastrophy at stresses predicted by the bundle model. In either event, it is interesting to note that the introduction of a ductile matrix reduces the apparent ductility of a bundle of brittle filaments.

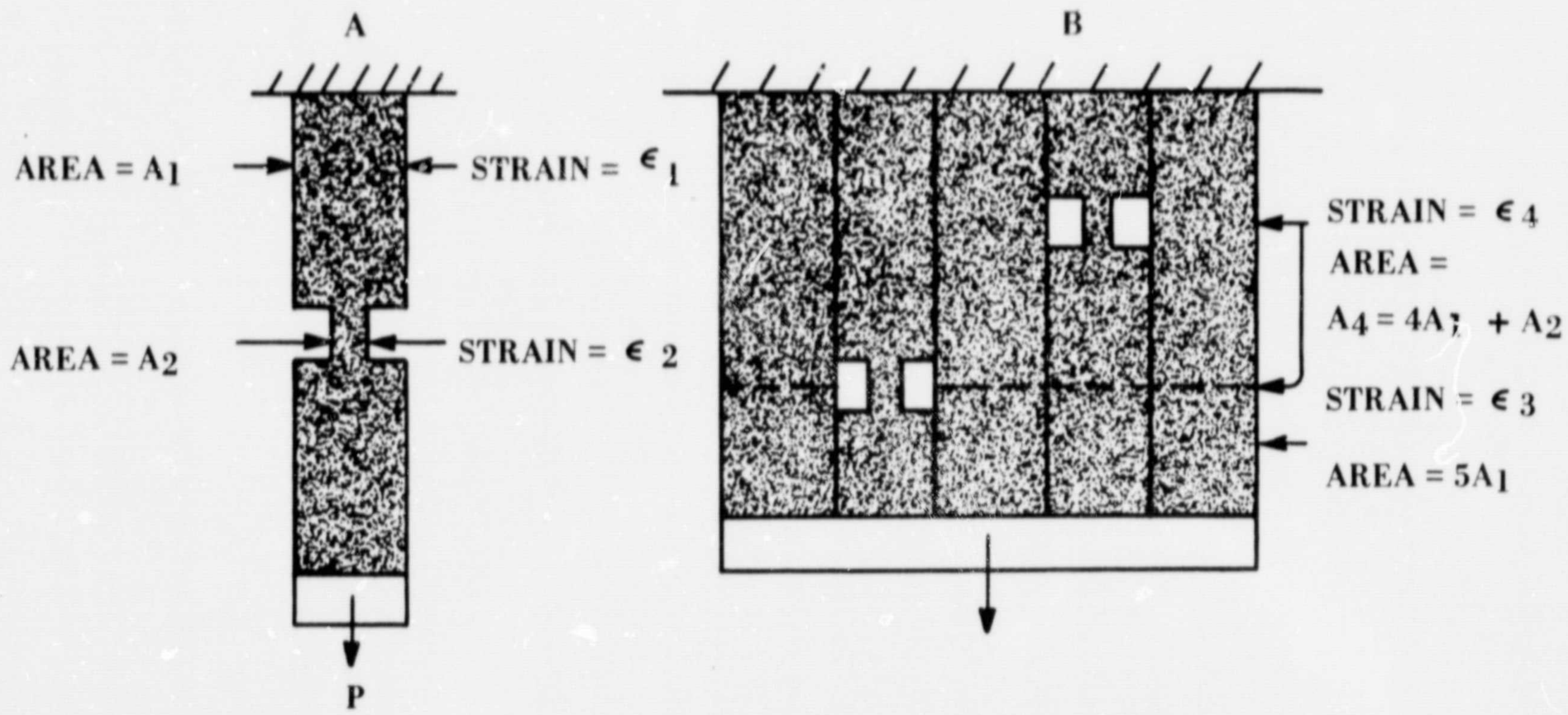
Both the hypothetical bundle and the real composites exhibit a flat load-deflection transition region at the onset of catastrophic failure. The remarkable shape similarity indicates that the catastrophic mechanism is similar for both.

The experimental composite is approximately 38% stronger than the hypothetical bundle and in another case (see Spec. 7, Table II) 57% stronger. These values may be reduced by taking into account the load bearing capacity of the aluminum matrix (see Figure 14) in which case the composites are only 28% or 48% stronger. Even more conservative is to consider that the aluminum does not deform plastically (as indicated) but shares the load in proportion to its elastic behavior; in this case, the composites are still 11% or 13% stronger than the bundle. Fabrication and experimental difficulties are often necessary to explain away less-than-bundle strengths; but, rational explanations seem necessary to explain composites which are stronger than bundles.

Superior results for composites can be rationalized by recalling a common characteristic of brittle filaments. Owing to a statistical distribution of flaw severity, short lengths are often stronger than longer gage lengths. In consequence, strength distributions obtained on 1-inch gage lengths are adequate only for bundle models, because a single failure precludes any further load sharing by the broken filament. However, if broken filaments continue to reinforce (as long as they exceed the critical length), and breaks are randomly located, composites can be stronger than bundles owing to the higher strength of the broken remnants. In such a situation, the specimen would remain stiff and be stronger than the bundle as is the case here. This "cumulative damage" model is, then, consistent with the observed mechanical behavior but it appears completely incompatible with another experimental observation: multiple filament $B_4C/B/W$-Al specimens almost never exhibit more than one break per filament and these breaks are not randomly located but situated at or very near to the region of catastrophic failure.

The fact that there is no direct evidence of cumulative damage shows that a number of randomly situated weak regions of filament do not bear their share of the load and that conventionally obtained strength-distribution data is inadequate for predicting composite behavior. The problem is to explain why weak regions of filament do not fail until after the average filament stress far exceeds the strength of the weak region. In what follows a model is suggested based on a different elastic strain distribution in the composite relative to that in a single filament test.

Filament defects may be small internal cracks having high (tip) stress concentrations. Since the net effect (for strength of single filaments) is equivalent to a local reduction in cross section, it is depicted as such for convenience in Figure 15A. Under this load. this filament will have an inhomogeneous elastic strain distribution along its length, the ratio of maximum to minimum being inversely proportional to the ratio of the respective cross-sectional areas. Failure in the tensile test will occur when the maximum elastic strain (located at the smallest cross-section) becomes equal to the fracture strain. However, when two such filaments are well-bonded to three defect-free filaments the strain inhomogeneity in the defect region will be markedly reduced by virtue of the strain constraint imposed by the neighboring filaments (as indicated in Figure 15B). Therefore, the 5-element specimen will sustain a greater nominal average stress before the fracture strain is reached at either defect. When one of the defects finally fails, the highest elastic strain in the specimen will be in the neighboring filaments in the region of the failed defect, not at some other 5-element defect region. This reasoning is numerically illustrated by assuming values of elastic modulus and a high and low strength shown in Figure 15. In summary, the elastic strain inhomogeneities characterizing loaded single filaments are reduced (by interaction constraints) when the filament is incorporated in a well-bonded composite. This reduces the strength-scatter and increases the average strength predicted on the basis of single-filament tests. This accounts for higher-than-average (predicted) composite strengths and is consistent with the one-break-per-filament observation. This model differs from conventional cumulative damage models in four respects: (1) useful load redistribution occurs prior to, not after, failure of weak regions; (2) it does not depend upon an inverse strength-gage length relationship; (3) it is consistent with few breaks per filament; and, (4) it predicts specimen failure nearly coincident with the first filament failure. The details of the example (Figure 15) were chosen to explain above- average strength. If the two defects were lined up in a single plane but still separated by defect-free filaments, the composite would exhibit average or near average strength. If, however, the two defects were adjacent, their common boundary would no longer have the same constraint against inhomogeneous deformations and the composite would exhibit less-than-average strength.



$$\epsilon_2 / \epsilon_1 = A_1/A_2$$

$$\epsilon_4 / \epsilon_3 = A_3/A_4 = \frac{5A_1}{4A_1 \quad A_2}$$

If max strength is 550,000 psi and elastic modulus is 55 x 10^6 psi, $\epsilon_f = \epsilon_2 = \epsilon_4 = 10^{-2}$.

If nominal minimum strength is 55,000, ($\epsilon_1 = 10^{-3}$) in single filament test,

then $\epsilon_2/\epsilon_1 = 10^{-2}/10^{-3} = 10 = A_1/A_2$ and $\epsilon_3/\epsilon_4 = 4/5 + 1/5\ A_1/A_2 = 41/50$

$\therefore$ $\epsilon_3 = 41/50 \times 10^{-2}$ (when $\epsilon_4 = \epsilon_f = 10^{-2}$)

and the nominal stress/filament in composite,

$$\sigma_c = \epsilon_3 E = 41/50 \times 55\text{x}104 \quad 451{,}000 \text{ psi}$$

The average of individual filament strength

$$\sigma_a = \frac{3 \times 550{,}000 + 2 \times 55{,}000}{5} = 352{,}000 \text{ psi}$$

If above 5 element configuration was unbonded (bundle) the nominal stress per filament,

$$\sigma_B = 3/5 \times 550{,}000 = 330{,}000$$

Figure 15. Schematic representation of model to account for composite strength greater than rule-of-mixtures predictions

Thus, the model chosen is consistent with the scatter of tensile data shown in Figure 11.

III. FUTURE WORK

The characterization of composite component materials is an important and necessary part of this program and will continue.

Further attemps will be made to design and fabricate composites suitable for testing at 500 C so that high temperature stability tests, including heat treatment, creep and tensile tests on the aluminum matrix/continuous filament composite systems can be made.

Extensive work still remains in the aluminum-discontinuous-filament composite system. Test specimens containing discontinuous-filament arrays are presently being made.

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APPENDIX

RELATION BETWEEN BOND STRENGTH AND COMPOSITE STRENGTH

This discussion by J. V. Mullen attempts to rationalize the fracture behavior observed in epoxy-boron composite materials which had been reported upon earlier. Underlying this treatment is the assumption that the cumulative damage mechanism of composite failure is valid. Present work with aluminum-B_4C/B/W composite materials has led to other models to explain their fracture behavior rather than the cumulative damage mechanism. It is anticipated that the next progress report will consider all the evidence for all systems studied and a unified, rational model will emerge.

Assume that a certain number of filaments of given modulus and strength distribution and a matrix of specific strength, modulus, crack sensitivity, etc, is available. Assume further that the capability of varying the bond strength as defined by the shear stress τ at the interface exists. If it were possible to degrade the bond so much that $\tau = 0$, the filaments would not be loaded by the matrix at all and the volume the filaments occupy could be considered as voids in the matrix. All stress would be carried by the matrix material and since the voids (filaments) would reduce the strength of the matrix due to stress concentrations, the composite strength would be less than than of an equal volume of matrix with no filaments. This may be construed as a zero volume fraction condition. This is point A in Figure 16 where none of the filament strength is developed. As the bond strength is increased very gradually the matrix begins to transfer load across the interface to the filaments without any fracture of filaments and the strength of the composite is increased by the contribution from the filaments. This process continues as the bond strength increased up to point B where the weakest filament fails. Because the total composite stress is still low the first weak filament fractures will be low energy breaks and will not throw out disk-shaped cracks [8] which could affect adjacent filaments. As these breaks occur at weak points in the filament, the bond strength will still be relatively low and considerable debonding will occur also. This debonding to some extent prevents the high energy breaks from occurring, until the bond strength is large enough to fracture filaments at the higher stress level corresponding to point C. At this point, high energy breaks begin to occur which throw out disk-shaped cracks affecting adjacent filaments. Each local fracture has a much more damaging effect than just the loss of one fiber and subsequent static redistribution of load, because this higher bond strength does not allow dissipation of energy through unbonding. For these reasons, the bond strength corresponding to point C is a critical bond strength τ_c for a given system, there is too much bond strength and fewer filament fractures can be sustained before the composite fails. Therefore, the overall composite strength is reduced as shown on CD. Now consider the implications of the bond strength variation in interpreting the rule-of-mixtures. In its ususal form, composite strength σ_c is expressed as:

$$\sigma_c = \sigma_m (1 - V_f) + V_f \sigma_f \ldots, \qquad (1)$$

where:

σ_m is matrix tensile stress

V_f is the volume fraction of filaments

σ_f is the average fiber strength

In the case where the bond strength is zero, the effective volume fraction, V_f, equals zero, since the fibers, although present, are not reinforcing the matrix. Equation (1) then reduces to:

$$\sigma_c = \sigma_m , \qquad (2)$$

the fibers reduce the strength of the matrix. For this reason, it is more realistic to use the form:

$$\sigma_f = \sigma_m (1 - V_f) + eV_f\sigma_f , \qquad (3)$$

where V_f is the volume occupied by filaments, and e is the mechanical compatibility of the fibers and matrix. The mechanical compatibility of fibers and matrix is a measure of how they complement one another, the filaments in reinforcing the matrix and the matrix in transferring load to and isolating the failures of filaments. There are several parameters which can influence the value of e, the most obvious being variation in bond strength. To illustrate, reconsider the variation in bond strength in the light of equation (3). First, consider what happens when the bond strength is zero. The matrix and filaments are acting separately with no coupling and the mechanical compatibility is therefore zero. Then

$$\sigma_f = \sigma_m (1 - V_f) + 0 ,$$

which is consistent with Figure 16 where composite strength is less than matrix strength because of the discontinuitites introduced by the filaments. As the bond strength increases, the mechanical compatibility increases, because there is sufficient bond strength to load the filaments to fracture, but some unbonding occurs at the newly formed ends keeping the fracture isolated. The increase in e results in an increase in the "effective volume fraction" (eV_f). This concept of an "effective volume fraction" takes account of the fact that having a lot of filaments in the matrix does not guarantee high strength. Rather it is the volume of the filaments able to reinforce the matrix, thereby adding to the strength of the composite, which determines the overall strength. For this reason, the highest value of e is unity, that is, every filament placed in the composite is providing reinforcement for the matrix.

As the bond continues to increase above point B, filaments begin to fail, but energy is absorbed in unbonding too so that single-filament fractures are not catastrophic. Therefore, e continues to increase, since more filaments are being used efficiently; therefore, mechanical compatibility increases. Finally, the bond strength becomes so great that the filament fractures are not damped by

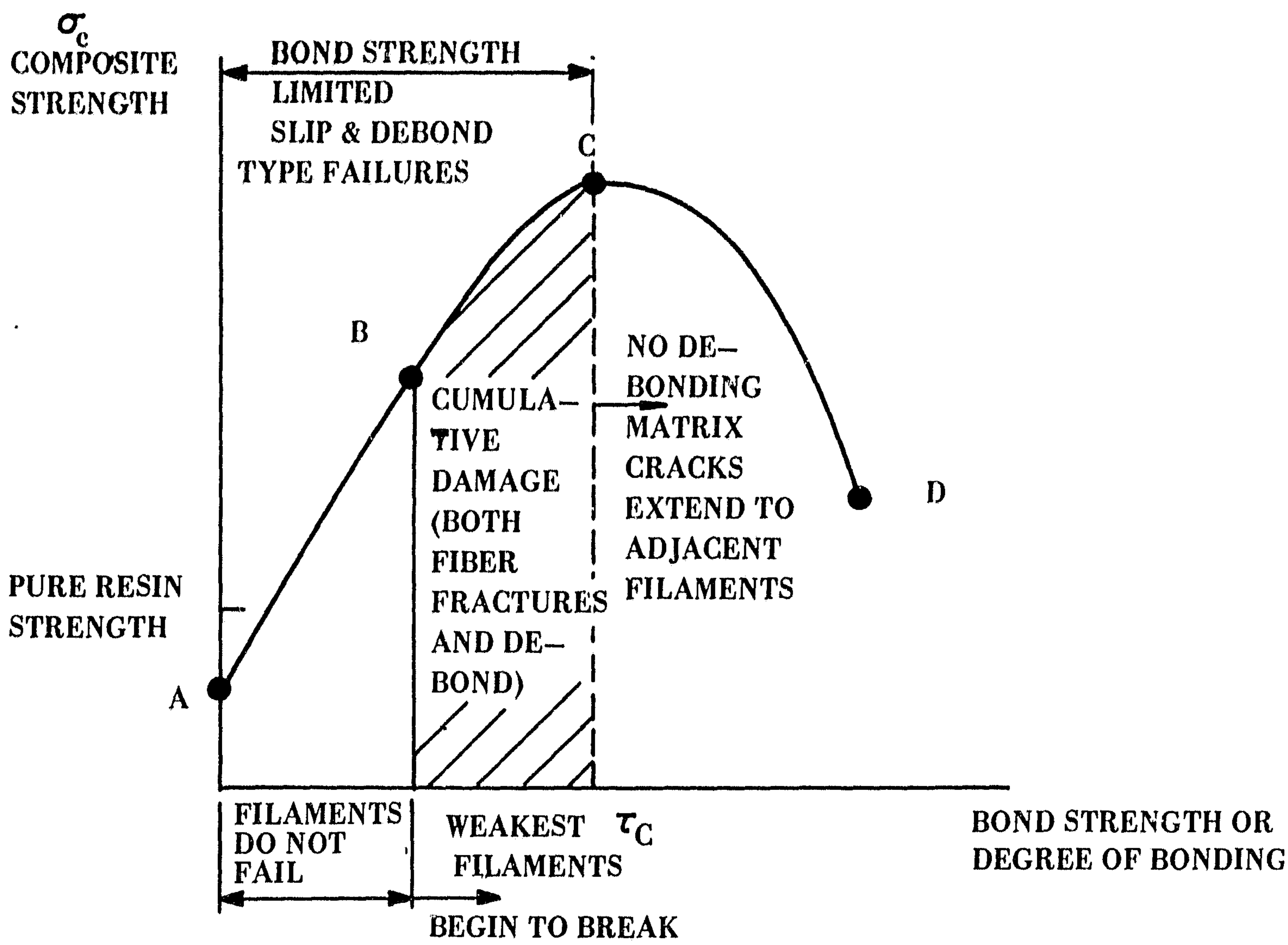

Figure 16. Proposed relationship between bond strength and composite strength for a resin–filament composite

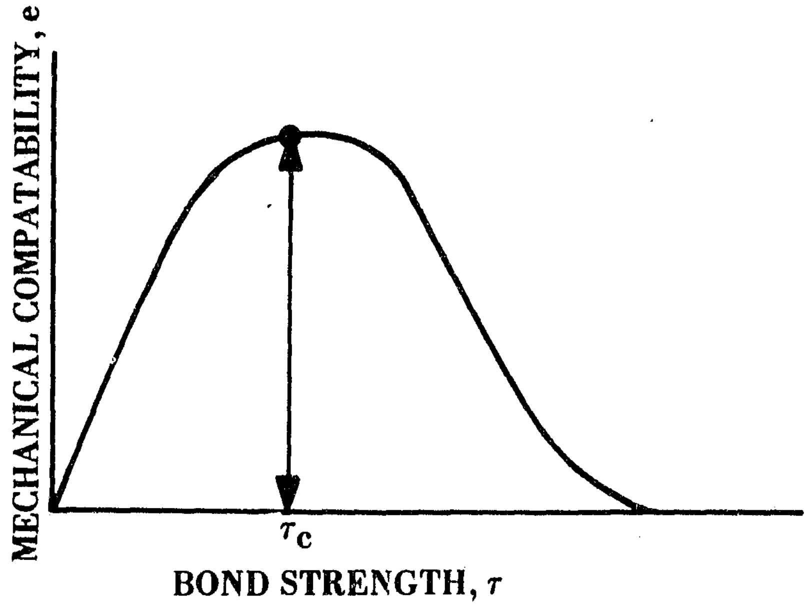

Figure 17. e vs. bond strength, τ

unbonding and each filament fracture influences adjacent filaments. Because each local failure results in considerable damage the effective volume fraction (eV_f) is greatly diminished, since premature failure will prevent the composite from developing its potential v lume fraction V_f. Therefore, for bond strengths in excess of that defining point C in Figure 16, the value of e is decreasing and this means the strength of the composite must be diminished. The plot of mechanical compatibility versus bond strength would appear as shown in Figure 17. There is an optimum value of bond strength and therefore an optimum value of e, which determines the strength of the composite, all other things being equal).

Now consider the case where the only variable is the actual volume fraction of filaments, such parameters as crack sensitivity, etc. remaining constant. Remembering that V_f is the actual volume fraction of fibers in the composite and (eV_f) is the "effective volume fraction", consider the case of zero volume fraction. When $V_f = 0$, there is no mechanical compatibility since there are no filaments; therefore, $\sigma_c = \sigma_m$, the strength of the matrix alone. When very small numbers of filaments are added ($V_f \sim 10\%$), the presence of these $\sigma_c = \sigma_m$, the strength of the matrix alone. This means that the value of e may be either zero, such that

$$\sigma_c = (1 - V_f)\,\sigma_m = .9\sigma_m$$

or very slightly negative (e.g., e = -.1) in which case

$$\sigma_c = (1 - V_f)\sigma_m + (-.1)\,V_f\sigma_f \; .$$

Either case results in a composite strength less than that of the unreinforced matrix. As the volume fraction V_f is increased, the strength increases because the distribution of fibers becomes more uniform, yet the fibers are not so close that their local fractures influence one another. The compatibility e may be unity during this phase where the strength increase is due entirely to the increase in V_f then

$$\sigma_c = (1 - V_f)\sigma_m + V_f\sigma_f \, ,$$

which is the standard expression for rule-of-mixtures behavior. Only when the value of V_f becomes large enough to cause critical spacing, that is, that spacing at which a single filament fracture begins to weaken those next to does the value of e begin to decrease. The greater the volume fraction V_f above the critical value (that causing critical spacing), the smaller the value of e. This is much more realistic than the usual form of the rule-of-mixture relation. A plot of the value of e against V_f, the actual volume of filaments, appears as shown in Figure 18. The corresponding composite strength is also given.

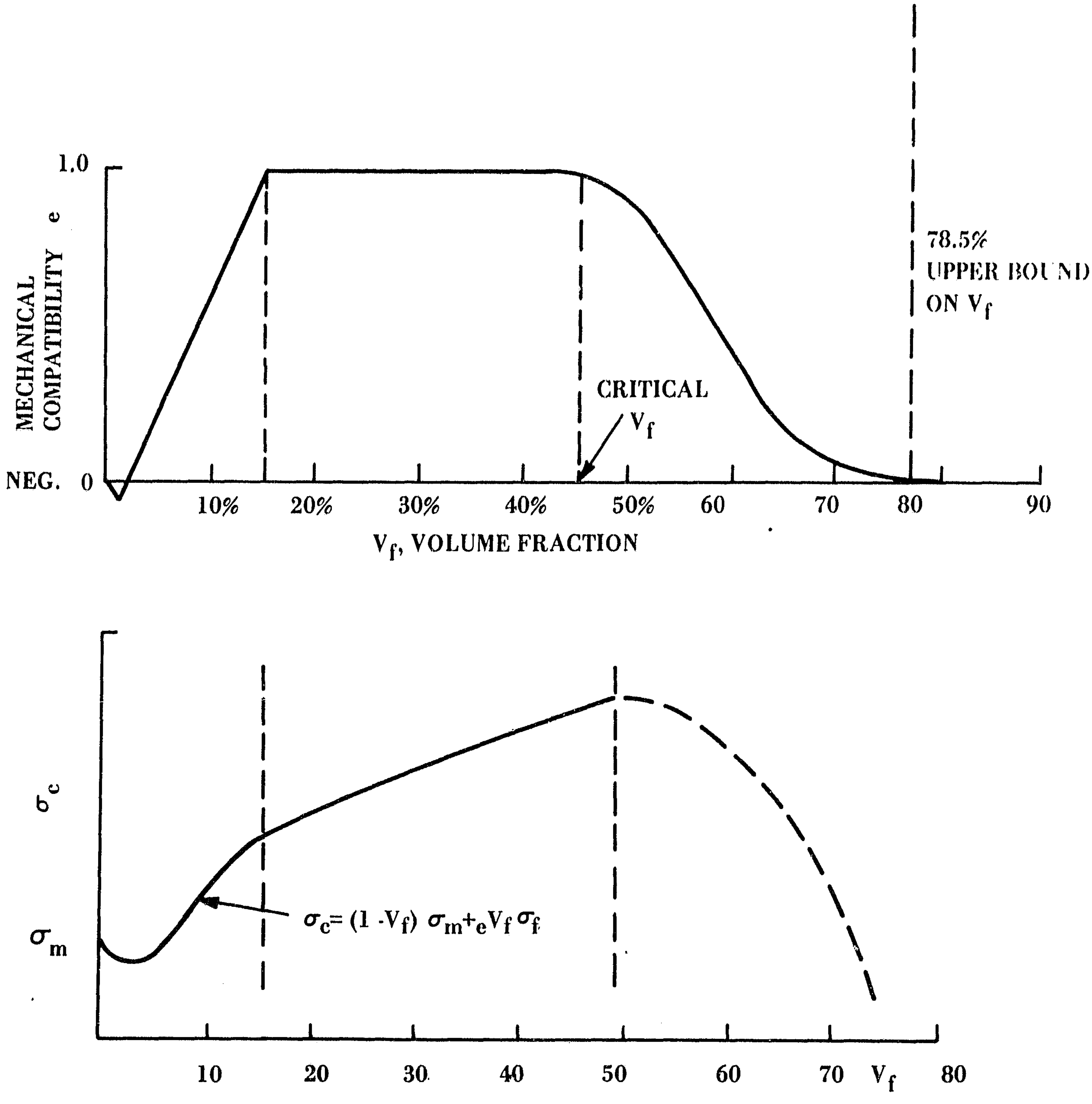

Figure 18. Variation in e and σ_c with V_f