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Annual Report

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#### INVESTIGATION OF MICROSTRUCTURAL FACTORS THAT CAUSE LOW FRACTURE TOUGHNESS IN SILICON CARBIDE WHISKER/AL ALLOY COMPOSITES

Submitted to:

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# TABLE OF CONTENTS

		Page
I.	INTRODUCTION	1
II.	BACKGROUND	3
III.	DESCRIPTION OF RESEARCH	5
	Results and Discussion	9 29 31

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#### SECTION I

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#### Introduction

The stigma of high cost which has inhibited the use of metal matrix composite materials for many years is slowly being removed. Improved manufacturing techniques for the reinforcements and composites, higher volume usage, and a realization that lifetime factors and efficiency improvements must be considered in an overall economic evaluation have led to increased incorporation of the materials into present day and future design concepts.

Additionally, many of the conventional metals and their alloying components (i.e., additives such as Ti, Cr, Co) are primarily imported from potentially unstable nations and have been placed on a critical materials list. For this reason, replacement of these materials may be a forced necessity in the very near future. Metal matrix composites are strongly being considered to fulfill this role.

One such materials system that has moved rapidly from its laboratory inception to near commercialization is a silicon carbide whisker reinforced aluminum composite material. This material is composed of typically 20 volume percent whiskers in various aluminum alloy matrices and has demonstrated strength values that equal or exceed most Al alloys and a Young's modulus that is 75% higher (18 x  $10^6$  psi [124 GPa]). These properties are quite similar to those obtained for many titanium alloys and at lighter weight hence specific properties for the whisker reinforced material are even more impressive. These properties along with additional advantages of being able to use conventional forming methods such as extrusion, forging, rolling, etc. make the material attractive from an economic as well as a convenience standpoint.

The development of SiC whisker reinforced composite material has followed a logical progression in that baseline mechanical property data has been established, as well as limited microstructural characterization. As with many materials, success comes quickly in the early stages of development only to succumb to more subtle problems later on. These subtleties are overcome only by completely understanding the system. Most frequently the limitations are microstructurally related and on an atomic level. Hence correlation of specimen history and properties with microstructural observations can lead to an understanding which could extend the range of properties for the material.

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#### SECTION II

#### Background

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This program deals with determining factors that influence ductility, fracture toughness, and elongation to failure in SiC whisker/Al alloy composites. Specifically, a microstructural study is being made in an attempt to define a model for the fracture process as it relates to the aforementioned properties. The model will then be tested by preparing composite specimens using formulas derived in the study (i.e. including alternate alloy systems) and evaluating their mechanical properties.

With the recognition that ultimate improvements in fracture toughness and elongation will only result from understanding and manipulation of microstructural features, the controlling factors much be defined from an atomistic standpoint. This emphasizes the roll of dislocations, their generation, movement, interaction, and overall contribution to the strengthening and/or fracture process of the composite system.

Transmission electron microscopic studies made on other composite systems have demonstrated the important roll that dislocations play in the fracture of this composite system. Stress buildup as a result of dislocation tangle and cell formation as well as interaction between interfacial and matrix dislocations play a strong part in the failure mechanism of the system.

A complicating factor in the present systems of interest (i.e. SiC/2124 and SiC/6061) is the roll of precipitates and eutectic particles. These intermetallic phases influence fracture toughness since they generally tend to have a brittle nature and form in strategic locations such as along critical crystallographic orientations where slip is active and between whiskers that are in contact or extremely close to each other. Most studies conducted to

date on the composites with 6061 or 2124 commercial alloys as matrices have yielded confusing results. The complexity of these systems (i.e. the large amount and number of solute elements) combined with the fabrication technique of exceeding the solidus temperature during ingot formation and subsequent thermal treatment creates many different phases that make microstructural correlation exceedingly difficult if not impossible.

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Because of these complicating factors, this program is attempting to understand matrix microstructural effects on toughness and elongation by utilizing simpler alloy systems (i.e. alloys with altered chemistry). This approach should avoid the formation of intermetallics thereby enhancing the understanding of microstructural interactions before proceeding to the more complicated precipitation hardening material and associated thermo-mechanical treatments. Typically increasing the number of precipitates causes the strength to go up but at the expense of toughness. Hence developing an understanding of the microstructural phenomena involved by using a systematic approach such as the one proposed could lead to alterations that will maximize both properties.

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#### Description of Research

Past microstructural studies have indicated that constituent particles in the 3-5 micron size range dominate failure in the present composite systems of interest (i.e. SiC/2124 and SiC/6061). These particles, indentified through X-ray analysis as: Al<sub>2</sub>CuMg, Al<sub>20</sub>Mn<sub>3</sub>Cu<sub>2</sub>, and FeCuMnAl<sub>6</sub>, definitely influence fracture toughness since they generally are brittle and form in critical locations, such as along whisker-matrix interfaces and along grain boundaries, where dislocations can move quite rapidly. These particles are also found to form in areas where whiskers are in contact. Figures 1 and 2 show scanning electron micrographs (SEM) of typical particles on the fracture surface of a 2124/20 v/o SiC composite, along with energy dispersive X-ray spectra, indicating the Cu, Fe and Mn content. Note the cleaved surfaces of the particles compared to the ductile appearance of the surrounding matrix. Also, in Figure 2 a crack through the particle is clearly visible, and pore formation can also be seen. Figure 3 shows transmission electron micrographs (TEM) of particle formation along the whisker-matrix interface, and where two whiskers are in contact.

Minimizing these detrimental particles is a first step toward improved fracture toughness. Improved extrusion methods developed at ARCO Silag have greatly improved whisker distribution throughout the matrix. In Figure 4, note the distribution and orientation of whiskers in "older" 2124 in (A), compared to more recently processed material in (B). With better whisker distribution, the probability of precipitate formation at whisker-whisker contact areas is greatly decreased. In addition to minimizing precipitate formation sites, matrix alloy chemistry can be altered to reduce the amount of elements present (i.e. Fe, Mn, Mg, Cu), which are the components of these precipitates.



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(A) Brittle intermetallic particle on 2124-20 v/0  $SiC_w$  fracture surface. (B) Corresponding X-ray spectrum.

FIG. 1 6



(A) Crack through particle, and void formation at particlematrix interface in 2124-20 v/o  $SiC_w$  composite. (B) Corresponding X-ray spectrum.



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(A) Example of past 2124 extrusion. (B) Improved extrusion methods, and altered alloy chemistry result in finer whisker distribution and alignment.

New alloys of altered Cu and Mg contents, and also a Lithium-containing alloy, were used to form composites with SiCw. The compositions and fiber volume fractions are listed in Table I. (These three composites, along with 1100/20 v/o SiCw and 2124/20 v/o SiCw are the materials being used in this investigation). The composites of altered chemistry were proposed by ARCO Silag. However, only a limited amount of material, less than 50 grams of each composition, was provided. So, only very limited thermo-mechanical testing of these new alloys has been done to this point. Vacuum-hot-pressing facilities are now operational at the University of Virginia; it is planned to manufacture more of these alloy composites for additional testing. Currently, most thermo-mechanical experiments are being conducted with 1100 and 2124 matrices.

Table II is a brief outline of the work plan currently in progress. Briefly stated, the plan is to compare the microstructure of as-received material to the products obtained after various thermo-mechanical processes, relate the processing to strength and elongation, and then optimize parameters for improved properties. Results obtained during the past year are discussed below.

#### Results and Discussion:

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#### 1. General Microstructure

The basis of metal matrix composite strengthening is the addition of high modulus reinforcement to lower modulus matrix material. There is a critical aspect ratio for the fibers which must be exceeded to fully utilize the high strength properties of the reinforcement. The aspect ratios of all materials listed in Table I were measured, in order to determine if, not only damage

#### TABLE I

#### Compositions of Composites Being Studied

### <u>Matrix</u>

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# Reinforcement

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A1 - 1.44 Mg A1 - 1.37 Mg - 2.95 Cu A1 - 0.85 Mg - 0.91 Cu - 1.66 Li 1100 A1 2124 A1 20 v/o SiCw (F-8) 19.3 v/o SiCw (F-8) 15.9 v/o SiCw (F-8) 20 v/o SiCw 20 v/o SiCw

#### TABLE II

#### Outline of Work Plan

- I. Microstructural Characterization of As-Received Material
  - A. SEM and TEM to examine:
    - 1. General apppearance
    - 2. Fiber size, distribution
    - 3. Inclusions and porosity
    - 4. Dislocations
  - B. Fracture

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- 1. SEM of tensile fracture surfaces, with stress axis along, and also transverse to the whisker axis
- 2. TEM of areas adjacent to fracture surface, examining for damaged fibers, interface
- 3. Correlation of microstructure with tensile properties (longitudinal and transverse)
- II. Characterize after Rolling
  - A. Investigate rolling temperatures coordinated with differential scanning calorimetry (DSC)
  - B. Fracture
    - 1. SEM and TEM on rolled specimens
    - 2. Compare microstructure and tensile properties before and after rolling

#### III. Heat Treating

- A. Treat samples to various tempers and aging conditions to determine baseline data; coordinated with (DSC)
- B. Mechanical testing of treated samples
- C. Compare microstructure and mechanical properties before and after heat treating
- IV. Thermal Cycling
  - A. Cycle material to high, and also to low temperatures
  - B. Mechanical testing; DSC
  - C. Compare mechanical properties before and after thermal cycling
- V. Define Failure Mechanism or Model
- VI. Propose Techniques and Alloy Compositions for Optimum Toughness and Elongation

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incurred during handling and processing of SiC whiskers was affecting final aspect ratio, but also if other factors such as formation of precipitates, volume fraction of the fibers themselves, and other component interactions, also have an influence.

Processing of all composites was held constant, while matrix composition and whisker volume fraction were varied. Figure 5 is a typical aspect ratio histogram obtained for all five systems tested. Aspect ratios are basically the same in all composites investigated. The average L/D range is 7-9, with 1100 having the slightly higher value. In each instance, the most probable ratio is 3 to 1. Slight volume fraction and matrix variations have no major effect on SiCw aspect ratio.

As seen in Figure 4, the new composites exhibit much improved whisker alignment and distribution. Surfaces for examining SiCw distribution with SEM were polished and etched. To verify that polishing was not influencing orientation, ion-milled TEM samples were examined, and found to exhibit the same, fine distribution of whiskers, as shown in Figure 6.

The amount of the large (3-5 micron size) particles common in 2124 and 6061 composites, have been drastically reduced by lowering the Cu and Mg content of the new matrix alloys, and also by using "cleaner" raw materials. Minimizing trace impurities such as Fe and Mn has also helped. Again, in Figures 4B and 6, the relatively clean matrix is evident. Very few precipitates were found in sizes larger than whisker diameters (0.5 micron). Attempts to X-ray map areas of Cu and Mg concentrations showed only even dispersion of these elements throughout the new matrix alloys. There are, of course, still some large intermetallics formed, as seen in Figure 7, but they are not very common.





Note also in Figure 7, the large pores. There is still a considerable amount (1-2% measured from SEM micrographs) of porosity in the compacted composites. Figure 8 shows higher magnification photos of pores between whiskers and at whisker ends. Still more improvement in powder metallurgy techniques is necessary to further reduce porosity.

Matrix-whisker interfacial areas are generally very clean in the Al-Cu-Mg and Al-Li-Cu-Mg alloy composites (see Figure 9). In the binary alloy of Al and Mg however, precipitates are seen to form along the interface; also needle-like Mg<sub>2</sub>Si precipitates are seen distributed throughout the matrix, as in Figure 10. Si in the Al raw material is the likely source for this precipitation phenomenon. The larger amount of brittle precipitates, as previously seen in 6061 and 2124 composites, has been greatly reduced in these new matrix alloys, resulting in cleaner interfaces.

Dislocations have been observed at fiber ends and running through grains adjacent to whiskers (Figure 11). Dislocation density measurements have not been made, but intense tangles or forests are generally not observed in the undeformed composite materials.

#### 2. Mechanical Properties and Fracture

The limited amount of material available for thermo-mechanical testing prohibits the use of specimens conforming to ASTM size standards. A summary of tensile tests completed is presented in Table III. Elongation values are preliminary and are presented for use on a comparative basis only; absolute magnitudes are questionable because of the sample size restrictions.

Tensile fracture tests are being used to compare several variables. First, comparisons of ultimate strength and elongation, with the stress axis applied in directions along the whisker axis (extrusion direction), and normal to the extrusion direction, were made. These directions are referred to as



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TEM of clean interfaces in (A) Al-Cu-Mg matrix and (B) Al-Li-Cu-Mg. One small precipitate is visible along whisker in (A).



 ${\rm Mg}_2{\rm Si}$  precipitates in Al-Mg matrix, and precipitates along matrix-whisker interface.



and at tip of SiC whisker.

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# TABLE III

# **Tensile Properties**

Matrix	Fiber Orientation	Ultimat (MP	e Strength Pa);(Ksi)	% Elongation
1100	Longitudinal	334	48.5	2.3 <sup>†</sup>
(extruded)	Transverse	331	48	24.0
Al-Cu-Mg	Longitudinal	396	57.5	8.5
(rolled)	Transverse	325	47.2	13.7
Al-Li-Cu-Mg	Longitudinal	461	66.9	7.5
(rolled)	Transverse	318	43.1	8.4
2124	Longitudinal	700	101	16.0
(extruded)	Transverse	469	72	9.8
2124	Longitudinal	520	75	16.4
(hot rolled) (55% reduction	Transverse )	430	62.4	17.0
2124 <sup>*</sup> (cold rolled) (18.7% reducti	Longitudinal	441	64.0	7.8

# <sup>†</sup>Longitudinal surfaces exhibited flaws

 $\star$  Cold rolled samples fracture on third pass through rollers.

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longitudinal and transverse, respectively. Longitudinal and transverse tests were conducted on extruded, rolled, heat-treated, and thermally cycled materials; comparisons of strength values and the fracture surface microstructure were then used to determine the effects of the thermal or mechanical processing. Generally transverse strength is approximately 75% of the longitudinal strength. Only the 1100 Al matrix shows similar values for both directions tested. However, 1100 longitudinal fracture surfaces exhibited large flows, areas of brittle fracture, at the edges of the specimens (see Figure 12) So, longitudinal values for 1100 may be artificially low. Also, there is a large discrepancy in 1100 longitudinal and transverse elongation values, with the longitudinal value very low in comparison. This also indicates premature failure. Further testing will have to be done. All the other composites, however, have transverse tensile strength values which are approximately 75% of the longitudinal values.

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Fracture surfaces in Figure 13 are from 1100 matrix composites, showing similarity between transverse and longitudinal failure modes. The ductility of the matrix is quite evident. The same comparison for 2124 matrix is shown in Figures 14A and B, longitudinal and transverse, respectively. Comparison of 1100 and 2124 shows much more porosity and brittle appearing failure in 2124, attributable to the greater amount of matrix precipitate present. In 2124, the dimples are smaller in size and are more numerous than in the cleaner 1100 matrix, which is attributable to the greater amount of precipitate particles. An interesting phenomenon observed only in transverse specimens , and in fact, one which is seen in transverse fracture surfaces of all the alloys studied, is the large pieces of matrix and whisker conglomerate which appear to have been torn out in chunks, leaving behind large pores and exposed whiskers. (see Figures 14C and D).



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(B)

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2124-20 v/o  $SiC_w$  fracture. (A) Longitudinal. (B),(C), and (D) transverse. In (C) and (D), note the large pieces of matrix-fiber conglomerate which decorate the surface, and also porosity.

These transverse vs. longitudinal observations are consistent with the following analogy, as in Figure 15. When stress is applied to the whiskers longitudinally, the effective aspect ratio is greater than when the stress axis is transverse to the whisker length. In the latter instance, the "length" of the whisker is essentially the same as the diameter, and L/D=1. Consequently, the smaller aspect ratio provides less strengthening in that direction. Another fracture occurrence observed in most of the composites is areas of whisker-free matrix which are pulled to very fine knife-edges, while the surrounding areas retain the usual dimpled appearance. Such areas in 1100, 2124, Al-Li-Cu-Mg, and Al-Cu-Mg are pictured in Figure 16.

#### 3. Rolling Effects

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Al-Mg, Al-Li-Cu-Mg, and Al-Cu-Mg alloy composites received from ARCO metals were shipped with the measured tensile values listed in Table IV. However, ultimate strengths measured here are considerably lower than ARCO's values. (Again elongation comparison is questionable because of sample size.) It is believed that the rolling process has considerably decreased strength properties by breaking down the fiber-matrix interface, although other studies have not demonstrated this.

Rolled Al-Cu-Mg and Al-Li-Cu-Mg transverse and longitudinal fracture surfaces are pictured in Figure 17. Note the large amount of whisker pullout (both the whiskers and pores can be seen) in the longitudinal surfaces. Compare these with the materials of comparable strength in Figures 13 and 14; there the material is not rolled and interfacial damage (pullout) is not evident. In the rolled transverse fracture surfaces of Figure 17B and especially in 17D, the tearing way of material at the whisker-matrix interface is quite evident. Fig. 17D also shows bundles of whiskers apparently not wetted by the matrix. The smaller effective whisker aspect



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Transverse

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Longitudinal

Comparison of aspect ratios in transverse and longitudinal directions. Arrows indicate stress axis. Higher L/D ratio provides more composite strengthening, as observed.

# FIG. 15

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Whisker-free matrix area ductility in (A) 1100,(b) 2124,

(C) Al-Cu-Mg, and (D) Al-Li-Cu-Mg.

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FIG. 16

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### TABLE IV

# Tensile Properties of Altered Alloy Matrices

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# UTS {Ksi,(MPa)}

### <u>% Elongation</u>

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	ARCO	UVa	ARCO	<u>UVa</u>
Al-Mg	not measured	38.9, (268)	* not measured	<1
Al-Cu-Mg	104, (716)	57.5, (396)	4.2	13.7
Al-Li-Cu-Mg	93, (641)	66.9, (461)	3.4	7.5

\* ARCO's reasons for not measuring Al-Mg tensile properties are inclusions and segregation.



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Interfacial damage, whisker pullout, induced by the rolling process. (A) Al-Cu-Mg longitudinal fracture surface. (B) Al-Cu-Mg transverse. (C) Al-Li-Cu-Mg longitudinal. (D) Al-Li-Cu-Mg transverse. ratio in the transverse direction generates greater interfacial damage at lower stresses.

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The decreased strength values and interfacial fracture damage observed in materials received in the rolled condition, prompted further investigation into the rolling process. Since exact rolling parameters-temperature, rolling speed, reheating times, etc. - were not known for the rolled materials received, rolling experiments were begun, and are in progress, using 2124-20 v/o SiCw and 1100-20 v/o SiCw extruded materials. Comparisons of tensile properties and the corresponding microstructures of the fracture surfaces, are being made for the extruded materials before and after rolling.

Different rolling temperatures are also being investigated. Differential scanning calorimetry (DSC) curves have been generated for the various composites under study. Endothermic or exothermic reactions taking place during heating (or cooling) are recorded as graphs of heat flow vs. temperature. One such curve for the Al-Cu-Mg altered chemistry material is shown in Figure 18. The reactions taking place can be related to microstructure with TEM studies. But, as yet, no correlations have been made. Still, the temperatures of interest found with DSC are being used as rolling temperatures to see if the corresponding reactions affect rolling properties.

Data from 2124/20 v/o SiCw transverse and longitudinal tensile strengths, before and after rolling, are listed in Table III. Hot-rolling (55% reduction) decreased longitudinal tensile strength 25%, and cold-rolling (18% reduction) reduced strength 36%. In the transverse direction, hot-rolled tensile strength is 13% lower. It was found that 2124 is not as suitable for cold-rolling, since it fractures after only a few passes through the rollers, whereas hot rolling at 493°C was found to introduce no visible damage even after 55% reduction. Although it has not been pursued as yet, values as high as 95% thickness reduction have been reported in the literature.



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Transverse and longitudinal fracture surfaces of hot-rolled 2124 are shown in Figure 19. These can be compared with the same material, not rolled, in Figure 13. Transverse fracture surfaces, before and after rolling appear The longitudinal surfaces also have similar appearances, but there similar. is an unusual observation in the hot-rolled surface (Figure 19A). The area pictured, which was a common occurrence across the surface, does not exhibit the usual dimpled structure in the matrix. It is not known what this phenomenon is, but further study will be undertaken. One possibility is that of localized incipient melting at a free surface (pores), which could have taken place during the hot rolling process. Note also, in Figure 19A, that there is a small area of ductility in the same region. The transverse surfaces again show the pulling away of fiber-matrix chunks, seen previously, and a crack through a brittle intermetallic particle. The fracture surfaces for 55% rolling reduction do not show any definite reasons for the observed strength decreases; more experiments are required to clarify this phenomenon.

In depth heat treating and thermal cycling studies have yet to be conducted. Again, DSC curves will be employed to identify reactions and temperatures where they occur. Interfacial effects of any such reactions are of particular interest. Once more, tensile testing and microstructural examination will be used to relate any thermal treatment to the mechanical properties.

#### Conclusions

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1. Minimization of raw material impurities decreases the amount of large brittle intermetallic precipitates. In addition, improved extrusion methods result in finer whisker alignment and distribution. Both of these factors result in improved fracture toughness.



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(C)



(B)

FIG. 19

Hot rolled 2124-20 v/o SiC composite fracture surfaces. (A),(B) - Longitudinal stress axis. (C), (D) - Transverse

2. Matrix composition and fiber volume fraction have minimal effect on aspect ratio. The most probable aspect ratio is: L/D=3 in all composites investigated.

3. Porosity (1-2%) is evident in extruded materials.

4. Fiber-matrix interfaces are generally clean and free of precipitates. 5. Ultimate tensile strength values in directions transverse to the whisker axis orientation are approximately 75% of the longitudinal direction values. The difference is attributable to the greater whisker aspect ratio in the longitudinal direction. Fracture surfaces show matrix ductility.

6. There are distinct differences between extruded and rolled material. Extruded composites have higher ultimate strength and lower elongation than the same material after rolling. Whisker pullout, interfacial damage is quite evident in rolled material; extruded composites have much less interfacial breakdown.

There have been reports of strength increases after rolling, but only decreases in strength have been observed here. However, investigation is continuing.

#### Plans for Next Year's Research

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During the next year the research program will continue as follows:

- Thermally treat the samples to various tempers and aging conditions to obtain baseline microstructural data.
- 2. Characterize basic as-fabricated microstructure of these samples utilizing transmission electron microscopy (to determine precipitate phases, location, etc.). Perform thermal cycling experiments to determine effect on matrix microstructures.
- Prepare composites containing 20 v/o SiC whiskers using established fabrication procedures.

- 4. Determine mechanical properties (particularly fracture toughness, strength, and elongation to failure) for these specimens.
- Perform fracture and microstructural analysis for correlation with mechanical properties (i.e. - determine the role of dislocations, precipitates, whiskers, etc.).
- 6. Define a mechanism and/or model for the failure process.

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7. Propose techniques and alloy compositions to produce composite specimens with optimum toughness and elongation.

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#### UNIVERSITY OF VIRGINIA School of Engineering and Applied Science

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The University of Virginia's School of Engineering and Applied Science has an undergraduate enrollment of approximately 1,500 students with a graduate enrollment of approximately 560. There are 150 faculty members, a majority of whom conduct research in addition to teaching.

Research is a vital part of the educational program and interests parallel academic specialties. These range from the classical engineering disciplines of Chemical, Civil, Electrical, and Mechanical and Aerospace to newer, more specialized fields of Biomedical Engineering, Systems Engineering, Materials Science, Nuclear Engineering and Engineering Physics, Applied Mathematics and Computer Science. Within these disciplines there are well equipped laboratories for conducting highly specialized research. All departments offer the doctorate; Biomedical and Materials Science grant only graduate degrees. In addition, courses in the humanities are offered within the School.

The University of Virginia (which includes approximately 2,000 faculty and a total of full-time student enrollment of about 16,400), also offers professional degrees under the schools of Architecture, Law, Medicine, Nursing, Commerce, Business Administration, and Education. In addition, the College of Arts and Sciences houses departments of Mathematics, Physics, Chemistry and others relevant to the engineering research program. The School of Engineering and Applied Science is an integral part of this University community which provides opportunities for interdisciplinary work in pursuit of the basic goals of education, research, and public service.

