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DEVELOPMENT OF A NEW GENERATION OF HIGH-TEMPERATURE  
COMPOSITE MATERIALS

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ABSTRACT

There are ever-increasing demands to develop low-density materials that maintain high strength and stiffness properties at elevated temperatures. Such materials are essential if the requirements for advanced aircraft, space power generation, and space station plans are to be realized. Metal matrix composites and intermetallic matrix composites are currently being investigated at NASA Lewis for such applications because they offer potential increases in strength, stiffness, and use temperature at a lower density than the most advanced single-crystal superalloys presently available. Today's discussion centers around the intermetallic matrix composites proposed by Lewis for meeting advanced aer propulsion requirements. The fabrication process currently being used at Lewis to produce intermetallic matrix composites will be reviewed, and the properties of one such composite, SiC/Ti<sub>3</sub>Al+Nb, will be presented. In addition, the direction of future research will be outlined, including plans for enhanced fabrication of aluminide composites by the arc spray technique and fiber development by the floating-zone process.

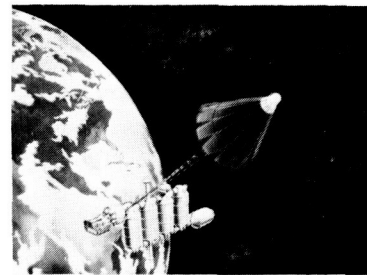
LEWIS INVOLVEMENT IN COMPOSITE MATERIALS DEVELOPMENT

Lewis has been working on composite materials for many years. Our efforts have focused on producing and characterizing metal matrix composite materials to obtain strength and density ratio improvements and higher operating temperatures than possible with currently available materials. Concurrently a technology base of high-temperature composite materials has been established. A model developed during the generation of this technology base is the well-known rule of mixtures (ROM), which is commonly used to predict composite properties from the behavior of the components. Some of the metal matrix composite systems that have been and are currently being examined at Lewis are listed in this figure alongside the areas for which these composites are targeted for possible use, including model systems studies and space power, space shuttle, and propulsion system components.

LEWIS INVOLVEMENT IN COMPOSITE MATERIALS DEVELOPMENT

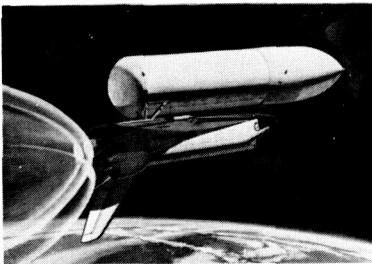
W/Cu (-Zr)  
W/FeCrAlY

W/Nb (-1Zr)  
Gr/Cu



MODEL SYSTEM STUDIES  
RULE OF MIXTURES

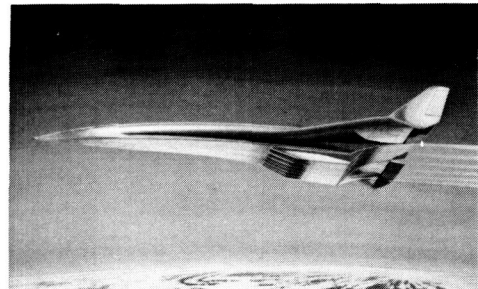
SPACE POWER COMPONENTS



W/FeCrAlY  
W/INCOLOY 903 AND 907  
W/WASPOLOY  
W/316 STAINLESS  
SiC/SUPERALLOY  
B<sub>4</sub>C-B/SUPERALLOY

Gr/Cu  
SiC/Fe-40Al  
SiC/NiAl  
SiC/Ti<sub>3</sub>Al + Nb

SPACE SHUTTLE MAIN  
ENGINE COMPONENTS



PROPULSION  
SYSTEM COMPONENTS

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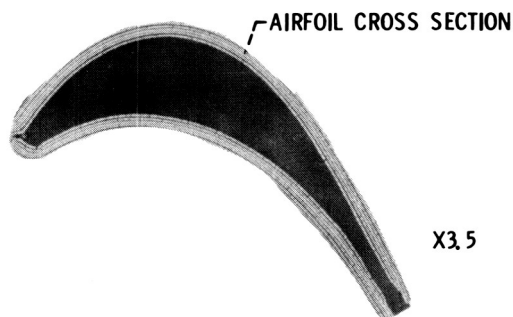
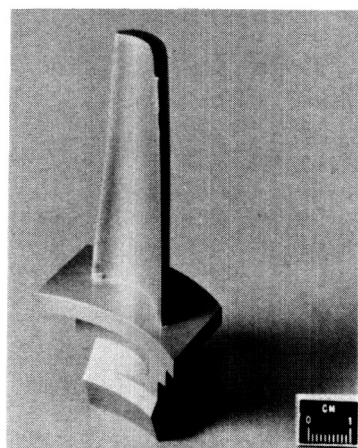
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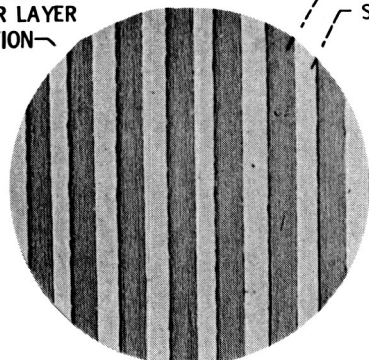
TUNGSTEN-FIBER-REINFORCED SUPERALLOY COMPOSITE BLADE

Another highlight of past work performed at Lewis is the fabrication of a tungsten-fiber-reinforced superalloy composite in the shape of a turbine blade. This proof of concept showed that production of intricately shaped composite components is indeed attainable. The composite blade was designed after a JT9D blade. It is hollow and contains cooling channels along the trailing edge. Note also that the uniformity of the fiber spacing in the longitudinal and transverse cross sections is uniform. The powder cloth method, which will be discussed shortly, was employed in fabricating this blade.

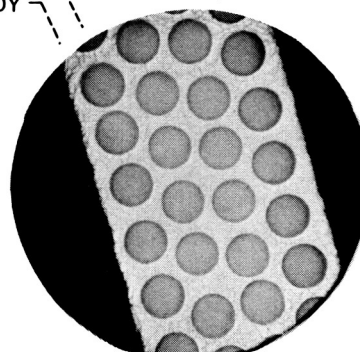
**TUNGSTEN-FIBER-REINFORCED SUPERALLOY COMPOSITE BLADE**



SPANWISE  
FIBER LAYER  
SECTION



TUNGSTEN FIBER  
SUPERALLOY



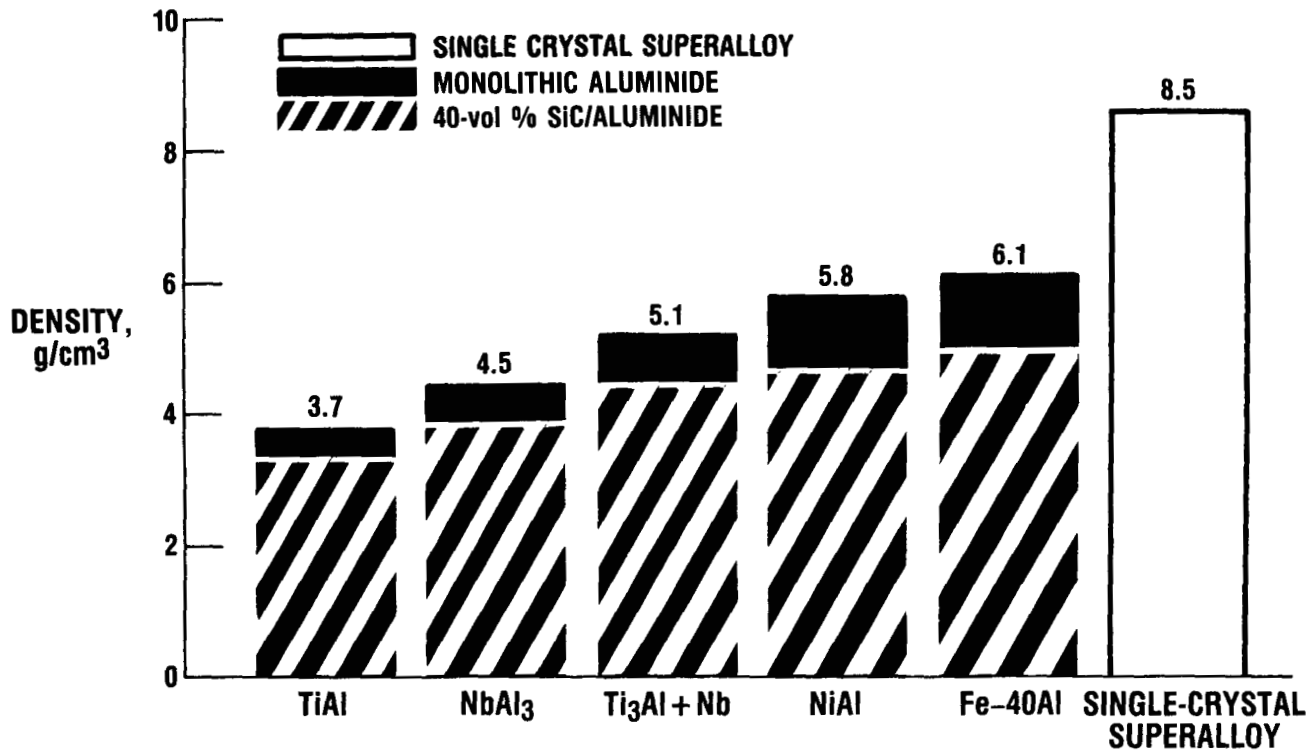
CROSS SECTION  
DETAIL

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## DENSITY COMPARISON OF ALUMINIDES AND SUPERALLOY

Our current efforts in advanced materials for aeropulsion applications center around the development of continuously reinforced aluminide matrix composites because of the potential these composites have to outperform existing superalloys. The primary properties requiring improvement if we are to realize hypersonic travel include lower density, strength at temperatures beyond 1800 °F, higher strength/density ratio and stiffness over the entire temperature range, and enhanced oxidation resistance or thermal barrier coating compatibility. Comparing the densities of several aluminides targeted for development and the nominal density of a superalloy clearly shows the advantage of pursuing aluminides. Furthermore, when these aluminides are reinforced with 40-vol % SiC, the densities are even more attractive. It is important to note here that even though monolithic aluminides offer potential over superalloys on a density basis, they are not as competitive on a strength basis unless reinforced. Thus fiber reinforcement is required to attain the best strength/density ratio improvement over superalloys.

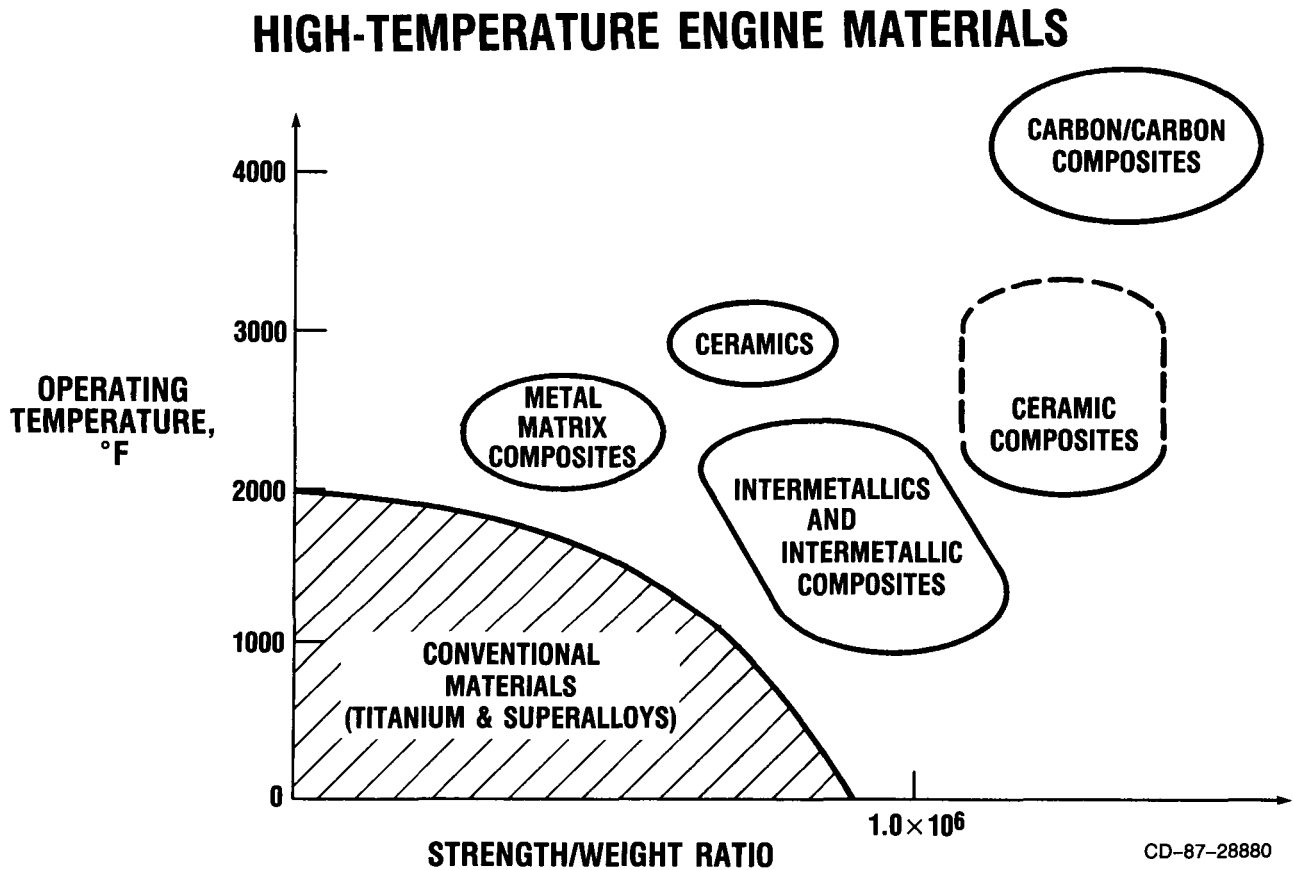
## DENSITY COMPARISON OF ALUMINIDES AND SUPERALLOY



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## HIGH-TEMPERATURE ENGINE MATERIALS

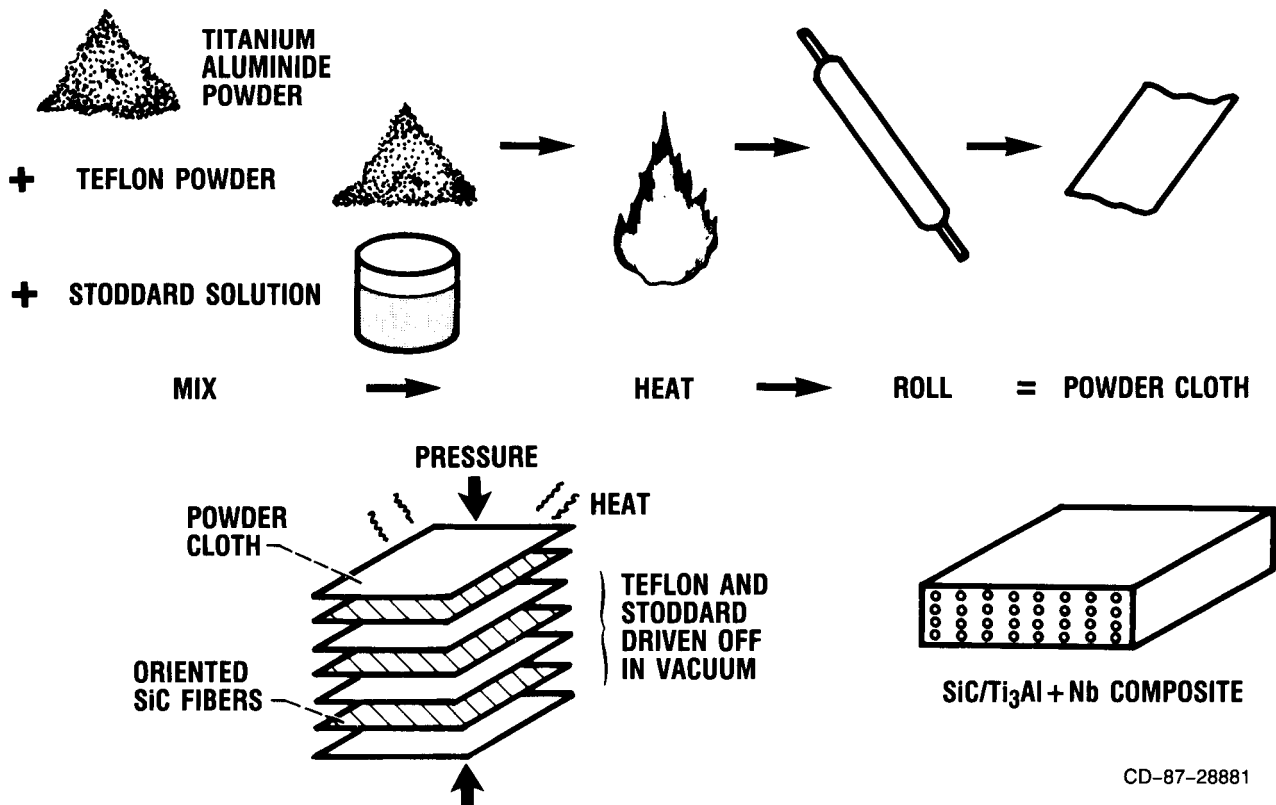
The enhanced strength/density ratio and increased operating temperature potential that metal matrix and intermetallic matrix composites offer over conventional materials are shown in this graph. Included also is the longer-term development of ceramics, ceramic composites, and carbon/carbon composites.



## PRESENT METHOD OF SiC/Ti<sub>3</sub>Al+Nb COMPOSITE FABRICATION

The process Lewis presently uses to produce aluminide matrix composites is illustrated here for SiC/Ti<sub>3</sub>Al+Nb. This fabrication process is called the powder cloth method. Prealloyed titanium aluminide powder is mixed with Teflon powder and a solvent in a blender. The mixture is heated to drive off the excess solvent and to provide the proper consistency for the rolling operation from which a powder cloth is obtained. These metallic powder cloths are the matrix of the composite. Mats of full-length SiC fibers are layered between the metallic powder cloths until the desired number of fiber layers is obtained. These layers of SiC fibers can be oriented as desired to obtain maximum properties in particular directions. The entire layup is placed in a hot press and diffusion bonded. The Teflon and remaining solvent are driven off in vacuum before the diffusion bonding occurs. The resultant SiC/Ti<sub>3</sub>Al+Nb composite is a 2- by 6-in. plate of a desired thickness, which is tested to characterize its properties.

## PRESENT METHOD OF SiC/Ti<sub>3</sub>Al+Nb COMPOSITE FABRICATION

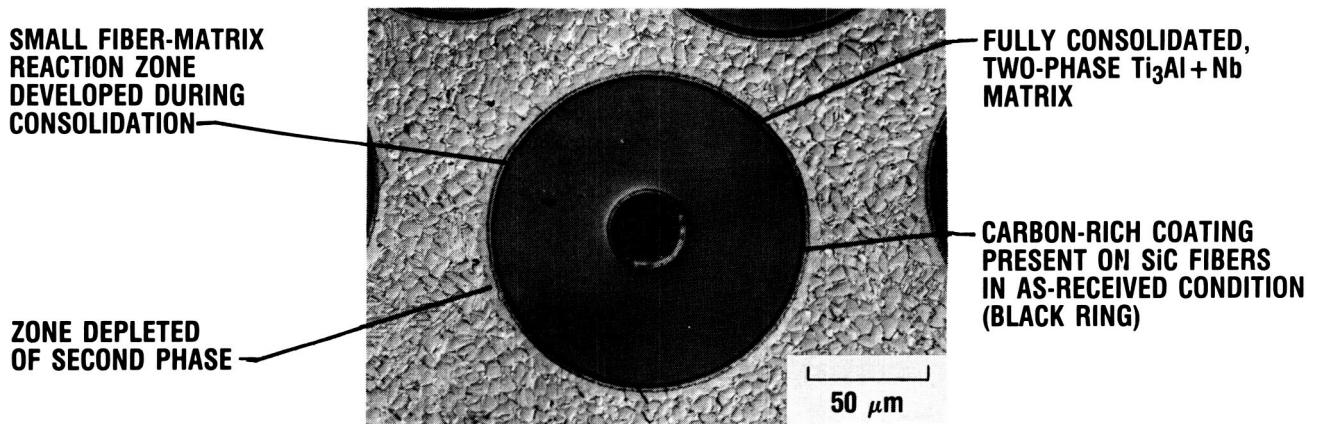
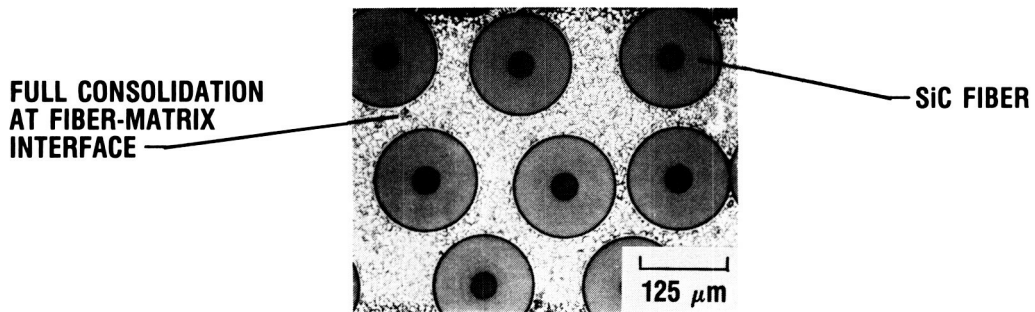


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AS-FABRICATED SiC/Ti<sub>3</sub>Al+Nb

Two views of an actual SiC/Ti<sub>3</sub>Al+Nb composite produced by the powder cloth technique are shown. The overall view shows a fully consolidated composite: no voids or cracks are evident in the matrix. The magnified view reveals the fiber and matrix to be fully consolidated. Other features include a two-phase Ti<sub>3</sub>Al+Nb matrix, a small reaction zone between the SiC fiber and the Ti<sub>3</sub>Al+Nb due to fabrication, and a zone surrounding the fiber and the reaction zone that appears to be depleted of the second phase. These features can be directly related to mechanical properties and will be elaborated on later.



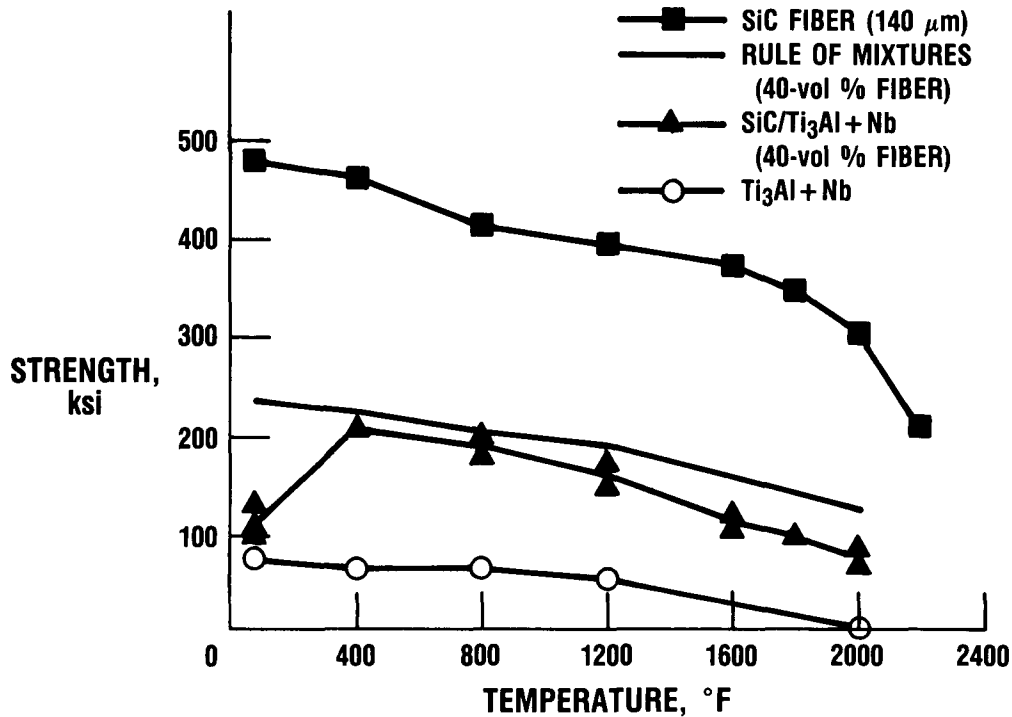
AS-FABRICATED SiC/Ti<sub>3</sub>Al + Nb

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## COMPARISON OF PREDICTED AND ACTUAL SiC/Ti<sub>3</sub>Al+Nb STRENGTH

The experimentally measured strengths of the SiC fiber, the Ti<sub>3</sub>Al+Nb matrix-only material, and the SiC/Ti<sub>3</sub>Al+Nb composite are plotted versus temperature. The fiber and the matrix-only data were used with the rule of mixtures (ROM) to compute predicted strengths. The ROM is basically a weighted average that predicts composite strength on the basis of the volume fractions of fiber and matrix present in the material. The composites tested contained 40-vol % SiC fiber. The actual composite values obtained were comparable to the ROM in the intermediate temperature regime, but lower than expected at room temperature and at 1200 °F and above. The presence of excess oxygen is known to limit matrix ductility in Ti<sub>3</sub>Al+Nb. The matrix material employed here contained 1000 to 1200 ppm oxygen. It was therefore thought that the oxygen was responsible for the lower strength values observed at room temperature by not allowing the fiber to attain its full strength potential. This was further substantiated by the lack of ductility observed in the room-temperature fracture surfaces. It should be possible to solve this room-temperature strength difficulty by using powder with lower oxygen content as well as through unique processing techniques. These ideas are being pursued. Debonding and fiber pullout are likely contributors to the strength falloff observed at elevated temperatures as shown in the next two figures.

## COMPARISON OF PREDICTED AND ACTUAL SiC/Ti<sub>3</sub>Al+Nb STRENGTH



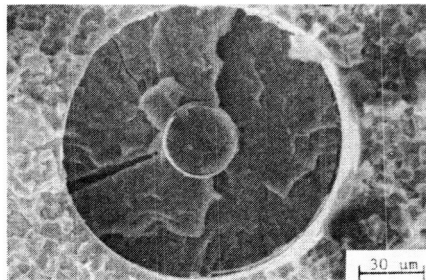
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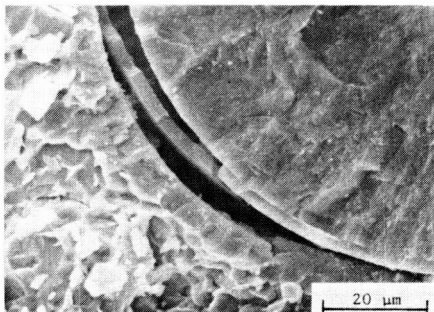
TENSILE FRACTURE SURFACES OF SiC/Ti<sub>3</sub>Al+Nb SHOWING DEBONDING

An understanding of the mode by which aluminide composites fracture is one of the goals of this work. This understanding will help us design these materials to obtain ROM strengths at all temperatures. This figure and the following one contain photographs of as-fractured SiC/Ti<sub>3</sub>Al+Nb surfaces after tensile testing. The test temperatures are indicated below each photograph. Note that as test temperature increases, so does the amount of debonding, or fiber-matrix separation. At room temperature the bond between fiber and matrix appears to remain intact, allowing for load transfer from the matrix to the fiber. However, at 1200 °F and above, some debonding is evident for these individual fibers. Since load transfer cannot occur in these debonded regions, it is plausible that debonding contributes to the falloff from ROM strengths observed at elevated temperatures.

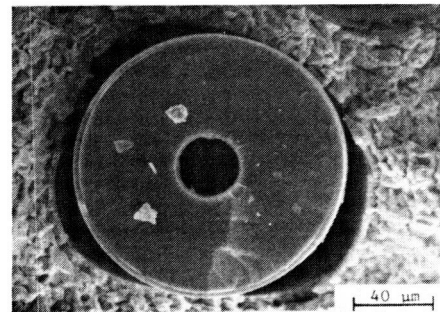
**TENSILE FRACTURE SURFACES OF SiC/Ti<sub>3</sub>Al + Nb  
SHOWING DEBONDING**



**23 °C (73 °F)**



**650 °C (1202 °F)**



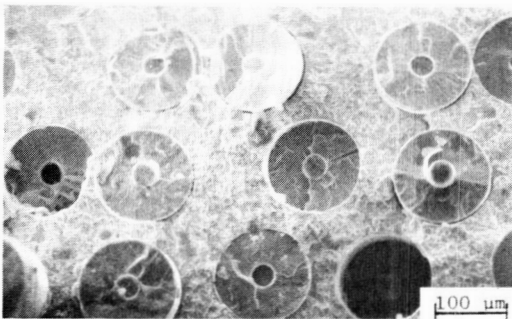
**1100 °C (2012 °F)**

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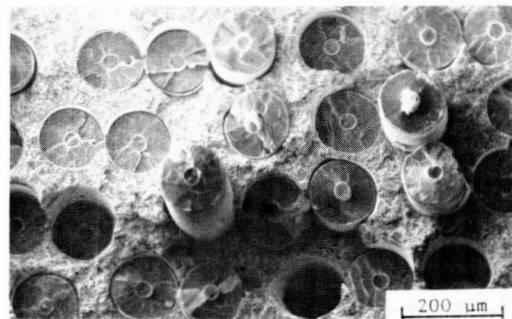
## TENSILE FRACTURE SURFACES OF SiC/Ti<sub>3</sub>Al+Nb SHOWING FIBER PULLOUT

Fiber pullout was also observed in the fracture surfaces of the SiC/Ti<sub>3</sub>Al+Nb composite tested over a range of temperatures. Note that very little fiber pullout is evident at room temperature but that increasing amounts of fiber pullout are obvious as the test temperature is increased. Such observations suggest that fiber pullout is another possible contributor to the falloff in ROM strengths observed at elevated temperatures.

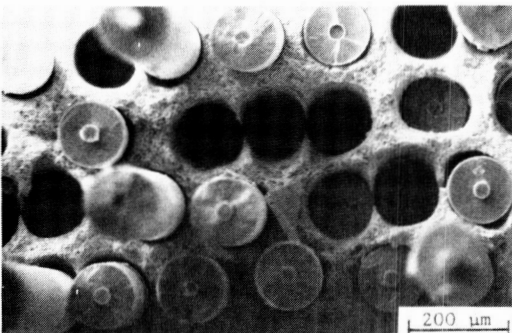
## TENSILE FRACTURE SURFACES OF SiC/Ti<sub>3</sub>Al + Nb SHOWING FIBER PULLOUT



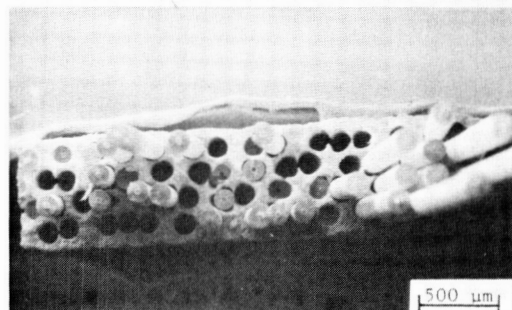
23 °C (73 °F)



425 °C (797 °F)



875 °C (1607 °F)



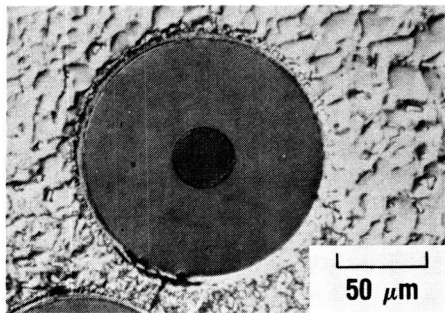
1100 °C (2012 °F)

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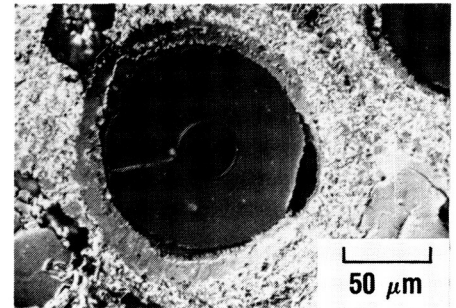
### UNDERSTANDING SiC/Ti<sub>3</sub>Al+Nb FIBER/MATRIX COMPATIBILITY

An understanding of fiber/matrix chemical compatibility is necessary to determine how long a particular composite will be able to function at a given operating temperature. In general, a large reaction zone between the fiber and the matrix is not acceptable because it is accompanied by a decrease in mechanical properties. However, a small reaction-zone is acceptable. The challenge is to determine the acceptable reaction-zone thickness for each composite system. The first step in determining acceptable limits is to anneal coupons of the composite at various times and temperatures. SiC/Ti<sub>3</sub>Al+Nb was annealed at 1800 and 2200 °F for 1 to 100 hr to determine the rate of chemical reaction between the fiber and the matrix at various temperatures. The results indicate that fiber/matrix reaction will probably limit the use temperature to 1800 °F for any application with extended life. The next step in determining reaction-zone effects on mechanical properties is to test SiC/Ti<sub>3</sub>Al+Nb containing various quantities of reaction zone over a range of temperatures.

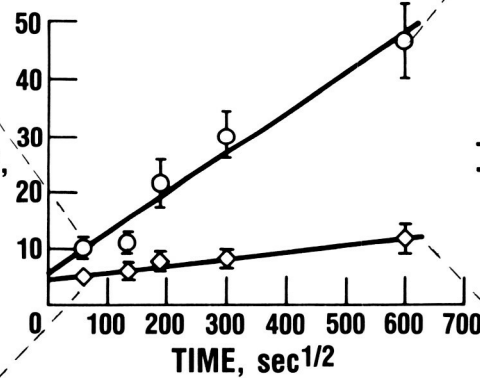
### UNDERSTANDING SiC/Ti<sub>3</sub>Al + Nb FIBER/MATRIX COMPATIBILITY



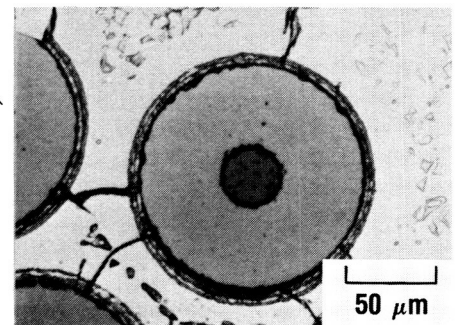
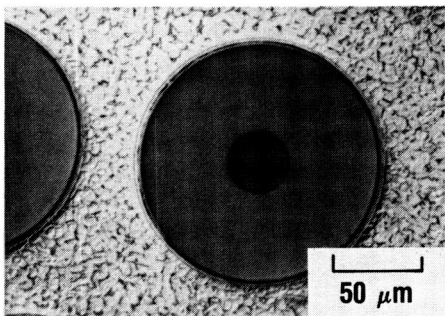
• RESULTS INDICATE FIBER/MATRIX INTERACTION WILL PROBABLY LIMIT USE TEMPERATURE TO 1800 °F



REACTION THICKNESS, μm



—◇— 1800 °F ANNEAL  
—○— 2200 °F ANNEAL

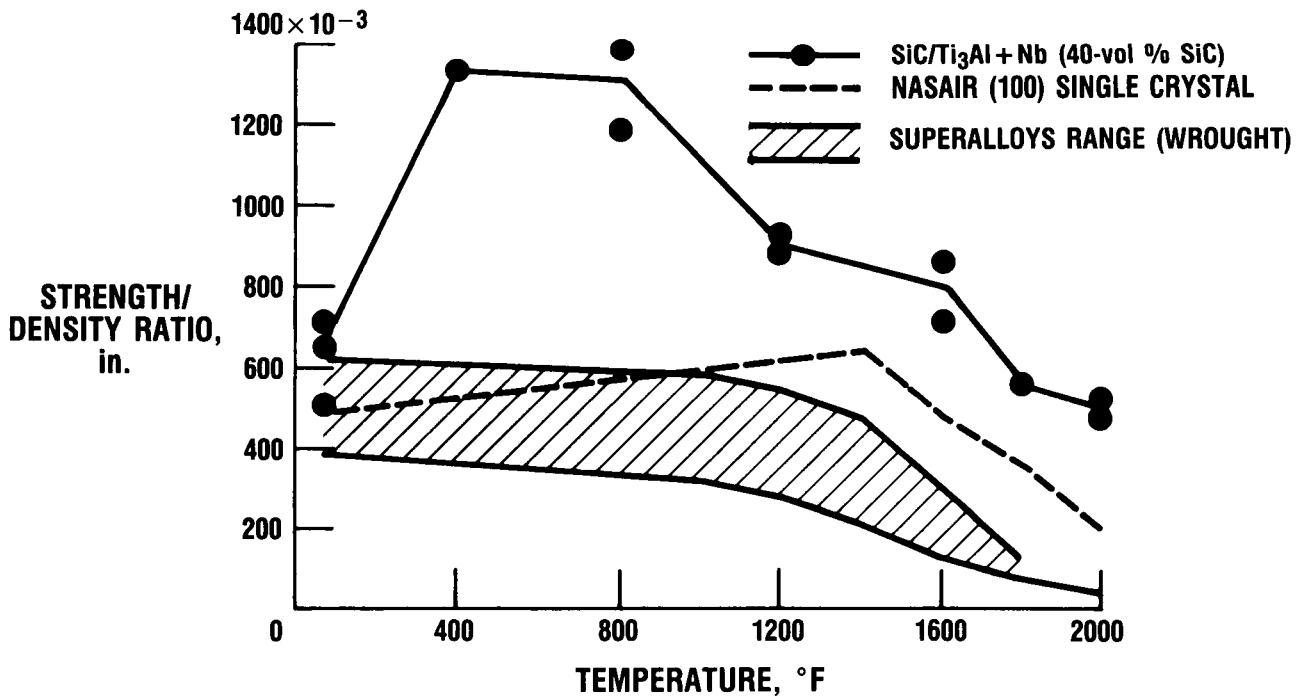


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SUPERIOR TENSILE PROPERTIES OF SiC/Ti<sub>3</sub>Al+Nb COMPOSITE COMPARED WITH EXISTING SUPERALLOYS

Comparing SiC/Ti<sub>3</sub>Al+Nb composite tensile properties on a strength/density ratio basis with those of a range of wrought superalloys and a single-crystal superalloy shows that the superior tensile properties predicted for aluminide matrix composites are attainable.

SUPERIOR TENSILE PROPERTIES OF SiC/Ti<sub>3</sub>Al + Nb COMPOSITE COMPARED WITH EXISTING SUPERALLOYS



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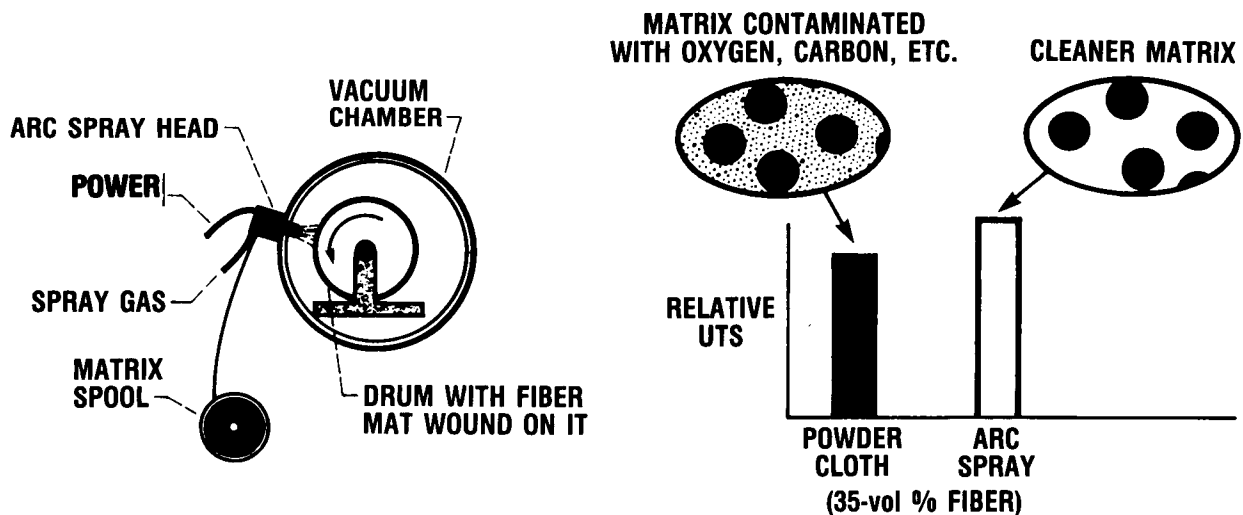
## FUTURE SiC/Ti<sub>3</sub>Al+Nb WORK EMPLOYS LEWIS-DEVELOPED ARC SPRAY FABRICATION PROCESS

Future work on aluminide matrix composites includes investigating alternative processing techniques in order to obtain higher production rates and, more importantly, cleaner matrix materials. Low-temperature ductility in the Ti<sub>3</sub>Al+Nb system, for instance, is greatly improved by decreasing the residual oxygen and carbon. For this reason we are presently pursuing the development of Ti<sub>3</sub>Al+Nb in wire form, since it is not readily available, to be used in our arc spray facility. It is anticipated that the arc spray process will maintain a Ti<sub>3</sub>Al+Nb matrix with a lower oxygen and carbon content than can the powder cloth technique for two reasons. First, powder inherently contains more oxygen than does wire because of its larger surface/volume ratio: there is more available surface for oxidation in the powder. Second, the capabilities of the arc spray process have been proven and documented in several composite systems, all of which show minimal oxygen and carbon pickup during processing.

## FUTURE SiC/Ti<sub>3</sub>Al + Nb WORK EMPLOYS LEWIS-DEVELOPED ARC SPRAY FABRICATION PROCESS

### BENEFITS

- HIGHER PRODUCTION RATES—AUTOMATED
- CLEANER MATRIX—ENHANCED DUCTILITY AND STRENGTH
- PROVEN PROCESS CAPABILITIES IN OTHER COMPOSITE SYSTEMS:  
W/SUPERALLOYS, W/Nb-1Zr, W/Cu, AND SiC/Nb-1Zr

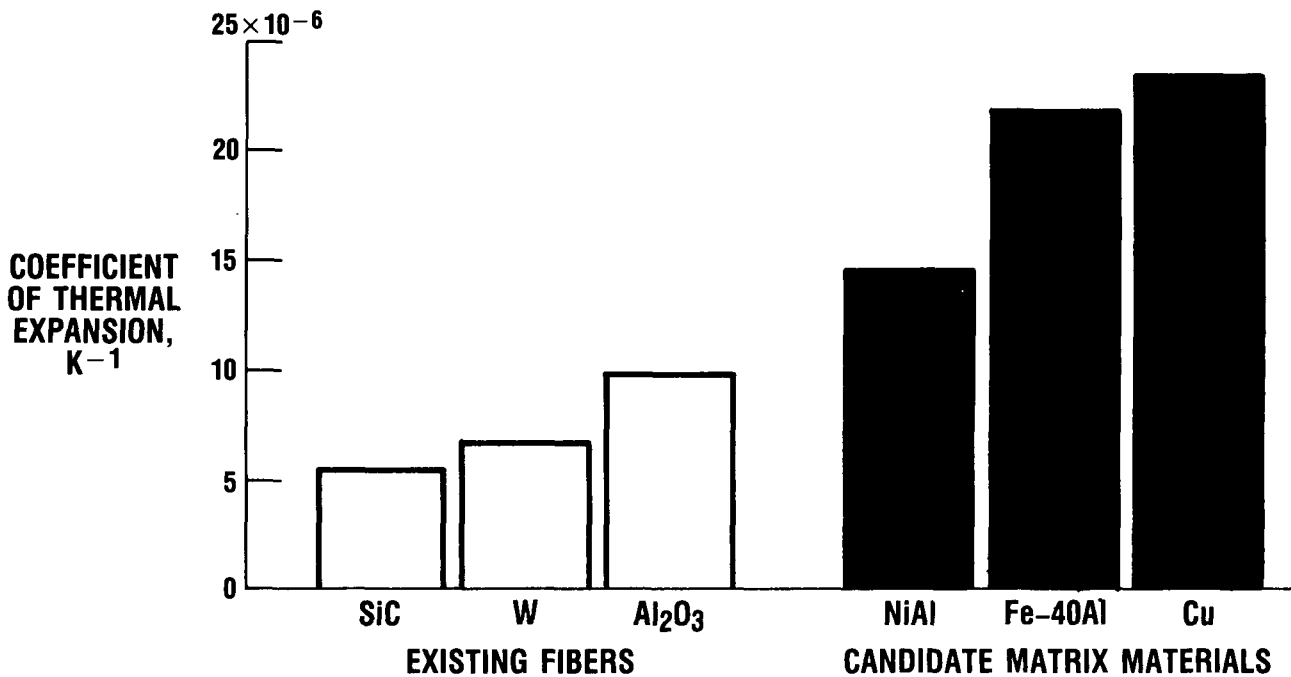


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## COMPARISON OF FIBER AND MATRIX EXPANSION SHOWING NEED FOR NEW FIBER DEVELOPMENT

Fiber development to match the fiber coefficient of thermal expansion closely to that of the matrix is planned to begin in the near future. A thermal expansion mismatch can result in a buildup of residual stresses within a composite subjected to thermal cycling during fabrication and service. The stress accumulation can cause premature failure of a composite component as a minimum, or catastrophic failure under severe thermal cycle conditions as an extreme. Thus the fiber and matrix expansion coefficients must be closely matched. An examination of thermal expansion coefficients of available fibers, such as SiC and Al<sub>2</sub>O<sub>3</sub>, and some aluminides quickly reveals the two- to fivefold difference in expansion between fibers and matrices. It is most reasonable to investigate the development of fibers from materials with thermal expansion coefficients nearer to that of the matrix, since the matrix materials have been chosen based on density, oxidation resistance, and higher operating temperature as previously discussed.

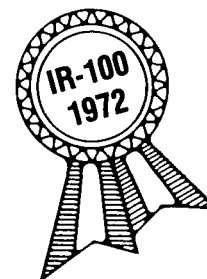
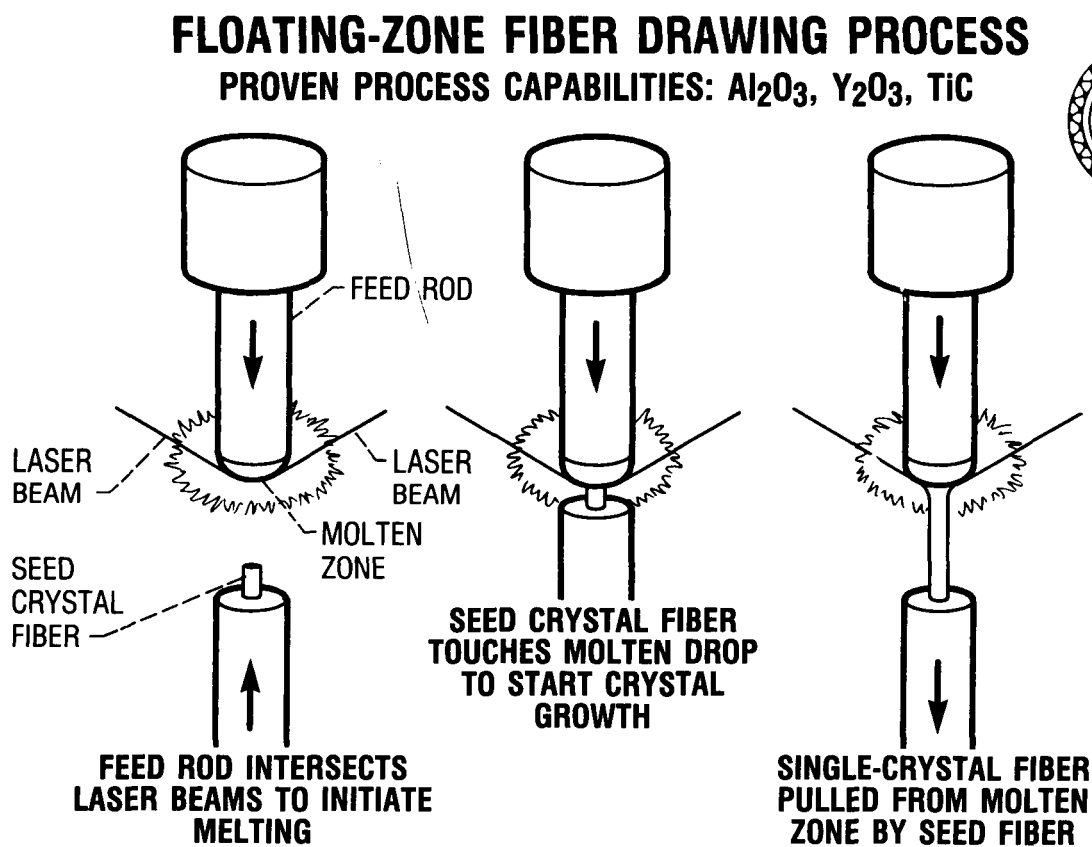
## COMPARISON OF FIBER AND MATRIX EXPANSION SHOWING NEED FOR NEW FIBER DEVELOPMENT



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## FLOATING-ZONE FIBER DRAWING PROCESS

A floating-zone fiber drawing process, developed in conjunction with A.D. Little, is being procured for laboratory-scale production of a range of fibers with high thermal expansion coefficients. In the floating-zone process a laser beam melts a polycrystalline feed rod. Once the feed rod becomes molten, a single-crystal seed rod of desired orientation is brought into contact with the feed rod to start crystal growth. A single-crystal fiber is then pulled from the molten zone as the feed rod is traversed through the laser beam. The capabilities of the floating-zone process have been proven in the production of  $\text{Al}_2\text{O}_3$ ,  $\text{Y}_2\text{O}_3$ , and  $\text{TiC}$  single-crystal fibers. This apparatus will be used to discern which materials of high thermal expansion coefficient are most promising for large-scale fiber processing, perhaps by chemical vapor deposition, on the basis of producibility, strength, and chemical compatibility with the matrix materials.



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