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DEVELOPMENT OF A NEW GENERATION OF HIGH-TEMPERATURE COMPOSITE MATERIALS

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SUMMARY

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 are to be realized. Continuous fiber-reinforced metal matrix composites and intermetallic matrix composites are currently being investigated at NASA Lewis Research Center for such applications because they offer potential increases in strength, stiffness, and use temperature at a lower density than that of superalloys presently available.

A brief review of key contributions from past metal matrix composite investigations precedes the primary topic of this paper: intermetallic matrix composites proposed to meet advanced aeropropulsion requirements. The powder metallurgy fabrication process currently being used to produce these intermetallic matrix composites will be presented, as will properties of one such composite, SiC/Ti_3Al+Nb (ref. 1). In addition, the direction of future research will be outlined, including plans for enhanced fabrication of intermetallic composites by the arc-spray technique and fiber development by the floating-zone process.

PAST RESEARCH

NASA Lewis' involvement in composite materials development is shown pictorially in figure 1 with the appropriate composites listed alongside each area of interest. Much of the past composite work has been composed of model system studies to determine tensile and stress rupture properties (refs. 2 to 6) and the effect of fiber-matrix reaction on mechanical properties (ref. 7). The model composite systems studied were primarily W-reinforced Cu (W/Cu) (refs. 2 and 3), W/Cu alloys (refs. 4 and 7), and W/superalloys (refs. 5 and 6). A model developed during the generation of this data base is the well-known rule of mixtures, which is commonly used to predict baseline composite properties from the behavior of the constituents (ref. 3).

A series of W-reinforced matrices (FeCrAl, Incoloy 907, Waspaloy, and 316 stainless steel) were examined by Rocketdyne for NASA Lewis (ref. 8) to determine their potential as alternate materials for turbine and compressor blades in advanced versions of the space shuttle main engine (SSME). These composites have been screened through a series of tests including severe thermal shock (cryogenic to 2000 °F in 0.3 sec), thermal fatigue, and high and low cycle fatigue. The most promising composite, W/Waspaloy, is continuing to be examined since it proved to have thermal fatigue properties equal to the currently used SSME blade material of directionally solidified Mar M 246 + Hf while being superior in thermal shock resistance and high and low cycle fatigue.

Another contribution in the area of metal matrix composites was a fabrication study performed by TRW for NASA Lewis (ref. 9) in which a W/superalloy composite turbine blade was made using a powder metallurgy approach. The composite blade, which is shown in figure 2, demonstrated both the feasibility of employing a continuous fiber composite to form a hollow blade and that the design requirements of a particular airfoil shape could be met. The airfoil of the W/superalloy blade has the external dimensions of a JT9D-7F first-stage, convection-cooled airfoil. The composite airfoil walls were designed to be thinner because of the increased strength and stiffness of the composite material. Designing thinner walls also reduced the composite blade weight to within 10 percent of the current Mar M 200 blade weight and allowed for more efficient cooling (ref. 10).

CURRENT RESEARCH

Our current efforts in advanced composites are also shown in figure 1 for aerospace applications. These endeavors center around the development of continuously reinforced aluminide matrix composites because of the potential these composites have to outperform existing superalloys. The primary properties required to realize advances toward hypersonic travel include lower density, improved strength at temperatures beyond 1800 °F, higher strength/density ratios, stiffness over the entire temperature range, enhanced oxidation resistance, and thermal barrier coating compatibility.

The densities of several aluminides targeted for development within NASA Lewis are compared in figure 3 with the nominal density of superalloys to show one advantage in pursuing aluminides. And, as expected, when aluminides are reinforced with SiC fiber, shown here at 40 vol % reinforcement, the density comparison is even more attractive. It is important to note that even though monolithic aluminides offer a weight savings, they typically are not competitive with superalloys on a strength basis unless they are reinforced. Thus, fiber-reinforced aluminide composites were chosen for examination. Three intermetallic matrix composite systems are currently under investigation with the following matrix materials: Fe-40 at. % Al (Fe-40Al), Ti-24 at. % Al-11 at. % Nb (Ti_3Al+Nb), and NiAl.

In any discussion of intermetallic materials it is important to note that limited ductility can be exhibited at low temperatures because their ordered atomic structures can limit the number of available, independent slip systems (ref. 11). This limited ductility in the matrix is one issue which needs to be overcome if intermetallics are to be successfully utilized in composites. In some cases ductility has been imparted to intermetallics by alloy addition to form a two-phase microstructure, as in the case of Ti_3Al+Nb (refs. 12 and 13), or by using an off-stoichiometric composition, as in the case of Fe-40Al (ref. 14). Thus, these aluminides were chosen as matrices for study because they exhibited ductility at room temperature and above, in addition to potential strength/density advantages over existing materials.

NiAl is also being pursued as a potential matrix material because of its high melting point of 2980 °F (ref. 15), and therefore high potential use temperature, and because it exhibits excellent oxidation resistance to 2012 °F (ref. 16). However, NiAl matrix composites are a longer term development than either Ti_3Al+Nb or Fe-40Al matrix composites because no means has yet been devised to achieve ductility in NiAl at low temperatures. Thus, NiAl is continuing to be researched to determine its potential as a suitable matrix material.

COMPOSITE DEVELOPMENT

A powder metallurgy approach is currently being used at NASA Lewis to fabricate intermetallic matrix composites, as illustrated in figure 4 for SiC/Ti_3Al+Nb. In this powder cloth technique, prealloyed aluminide powder is blended with Teflon powder and a solvent. 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 powder cloths until the desired number of reinforcement layers are achieved. These fiber layers can be oriented as desired to obtain maximum properties in particular directions. The entire layup is placed in a hot press and diffusion bonded, driving off the Teflon and remaining solvent in vacuum. The resultant SiC-reinforced intermetallic matrix composite is a 2- by 6-in. plate of desired thickness.

The microstructure of an actual SiC/Ti_3Al+Nb composite produced by the powder cloth technique is shown in figure 5. The lower magnification photomicrograph shows the three-fiber-layer composite, which is fully consolidated upon processing; no voids or cracks are evident in the matrix and the fiber-matrix interface appears to be well joined. The larger magnification view again shows the fiber-matrix interface to be fully consolidated. Other features include a two-phase Ti_3Al+Nb matrix, a small reaction zone between the fiber and matrix due to fabrication, and a zone surrounding the fiber and reaction zone that is depleted of second phase.

MECHANICAL PROPERTIES

Tensile properties at temperature for the AVCO SCS-6 SiC fiber, the Ti_3Al+Nb matrix material produced by the powder cloth technique, and the SiC/Ti_3Al+Nb composite tested in air are plotted in figure 6 (ref. 1). The fiber and the matrix-only data were used in the rule of mixtures (ROM) to calculate first-order composite properties. The ROM is a weighted average that predicts composite strength from the strength of the constituents (ref. 3). The data displayed here are for 40 vol % SiC fiber reinforcement. The actual composite strengths obtained were comparable to ROM in the intermediate temperature regime, but lower than expected at room temperature and at 1200 °F and above.

Consider first the room temperature results. It has previously been discussed that intermetallic compounds can be brittle at room temperature and that Nb has been successfully added to Ti_3Al to impart ductility at lower temperatures. It is also known that high levels of oxygen and carbon can negate the ductility benefits of Nb if present at levels of ~1000 ppm or greater. In this first generation of SiC/Ti_3Al+Nb composites, the oxygen level was ~1200 ppm.

It is therefore postulated that the oxygen was responsible for the low composite strengths obtained at room temperature resulting in cracking at low strains within the matrix and propagation through the brittle fibers such that the fibers were not able to attain their full strength potential. This proposed mode of failure was further supported by the lack of ductile features on the room temperature fracture surfaces as well as the overall flat fracture appearance of figure 8, in which the fibers and matrix failed in the same plane. It is hoped this room temperature strength difficulty can be solved by using matrix powder with lower oxygen contents. This is under investigation in the second generation of SiC/Ti_3Al+Nb composites in which powders with 480 to 620 ppm oxygen are being used.

With regard to the falloff in strength of SiC/Ti_3Al+Nb at 1200 °F and above, two features were observed on the fracture surfaces: debonding and fiber pullout. Figure 7 shows the presence and location of debonding which occurs at the fiber/fiber-coating interface and the fiber-coating/matrix interface after elevated-temperature tensile tests. At room temperature, the bond between the fiber and matrix appears to remain intact. However, at 1200 °F and above, the matrix separates from the C-rich coating of the fiber. Separation of the C-rich coating from the SiC fiber is sometimes observed in addition to the separation at the fiber coating/matrix interface, as shown here at 1200 °F.

Fiber pullout was also observed in the fracture surfaces of the SiC/Ti_3Al+Nb, as shown in figure 8. Note that very little fiber pullout was evident at room temperature but that increasing amounts of fiber pullout were obvious as test temperature was increased. These observations suggest that debonding and fiber pullout could have contributed to the deviation from ROM-predicted strengths at 1200 °F and above.

Fiber/matrix interface separations are not surprising when one considers the two- to five-fold differences in coefficient of thermal expansion (CTE) that exist between the currently available fibers for reinforcement and some of the candidate matrix materials, as shown in figure 9. Such large CTE mismatches are expected to be the most damaging under the thermal cycling and thermal mechanical fatigue conditions which these composites will surely encounter as components. Thus, it is imperative that thermal cycling and thermal mechanical fatigue characterization of these composites is performed. The need for new materials development to provide materials with higher CTE's is also clear, so as to minimize the damage induced in future generations of advanced composites during nonisothermal, cyclic conditions. The plans for new fiber development is discussed in the Future Composite Work section.

CHEMICAL COMPATIBILITY

Another area which requires characterization in any composite system is that of fiber-matrix chemical compatibility. In general, a large reaction zone between the fiber and matrix is unacceptable because it is accompanied by a decrease in mechanical properties (ref. 7). However, a small reaction zone may be acceptable. The first step in determining acceptable limits is to anneal coupons of the composite at various times and temperatures. Samples of SiC/Ti_3Al+Nb were annealed in vacuum at 1800 and 2200 °F for 1 to 100 hr to determine the rate of chemical reaction, as shown in figure 10. At short times, the reaction was not extensive at either 1800 or 2200 °F. However, for long periods of time, the reaction at 2200 °F is especially damaging. After 10 hr at 2200 °F, the C-rich fiber coating has been depleted, and extensive reaction has occurred. After 100 hr at 2200 °F, large voids within the reacted zone were also observed. In contrast, after 100 hr at 1800 °F, the C-rich fiber coating was still present, and the reaction thickness was on the order of 10 μm in thickness. These results indicated that fiber/matrix reaction will probably limit the use temperature of SiC/Ti_3Al+Nb to a maximum of 1800 °F. Direct measurements of strength with various quantities of reaction are required to determine the actual temperature and time limitations for applications of this composite.

Note also the matrix cracks which are present in the 1800 °F/100 hr photomicrograph of SiC/Ti_3Al+Nb in figure 10. These matrix cracks were induced during the several thermal cycles from room temperature to 1800 °F which were incurred during the accumulation of the 100 hr at 1800 °F. Again, these cracks most probably resulted because of the large CTE difference between the fiber and matrix combined with the low strain-to-failure of the composite components. This again points to the need for new fiber development of materials with larger CTE's, as discussed in the preceding section. One must also keep in mind that these data were from the high oxygen bearing material with low strain-to-failure. It is possible that the lower oxygen Ti_3Al+Nb being used in the second generation of composites may be able to accommodate more of the strain induced by the CTE mismatch. The amount of strain accommodation possible is under continued investigation.

To illustrate the potential of intermetallic matrix composites, the preliminary tensile properties of SiC/Ti_3Al+Nb were plotted on a strength/density basis versus temperature and compared with a range of wrought Ni-base superalloys and one single-crystal Ni-base superalloy in figure 11. These data show that the potential beyond superalloys anticipated for tensile properties of 0° fiber reinforced aluminide matrix composites is attainable.

FUTURE COMPOSITE WORK

Future work on intermetallic matrix composites includes investigating alternative processing techniques in order to obtain higher production rates and, more importantly, cleaner matrix materials. For this reason, we are presently pursuing the development of Ti_3Al+Nb in wire form to be used in our arc-spray facility (ref. 17), as shown schematically in figure 12. It is anticipated that the arc spray process will maintain a Ti_3Al+Nb matrix with a lower oxygen and carbon content than can be achieved by the powder cloth technique. Furthermore, the capabilities of the arc-spray process have been proven and documented in several composite systems including W/superalloys, W/Nb-1Zr, W/Cu, and SiC/Nb-1Zr, all of which displayed minimal oxygen and carbon pickup during processing.

New fiber development is also being pursued to investigate a wide range of materials which exhibit coefficients of thermal expansion similar to those of the intermetallics being considered as matrices. A floating-zone fiber drawing process (ref. 18), schematically shown in figure 13, is being procured and will be used to discern which materials are most promising on the basis of producibility, strength, and chemical compatibility. The most promising fibers will then be processed on a large scale, possibly by chemical vapor deposition, and subsequently employed in composites for evaluation and characterization.

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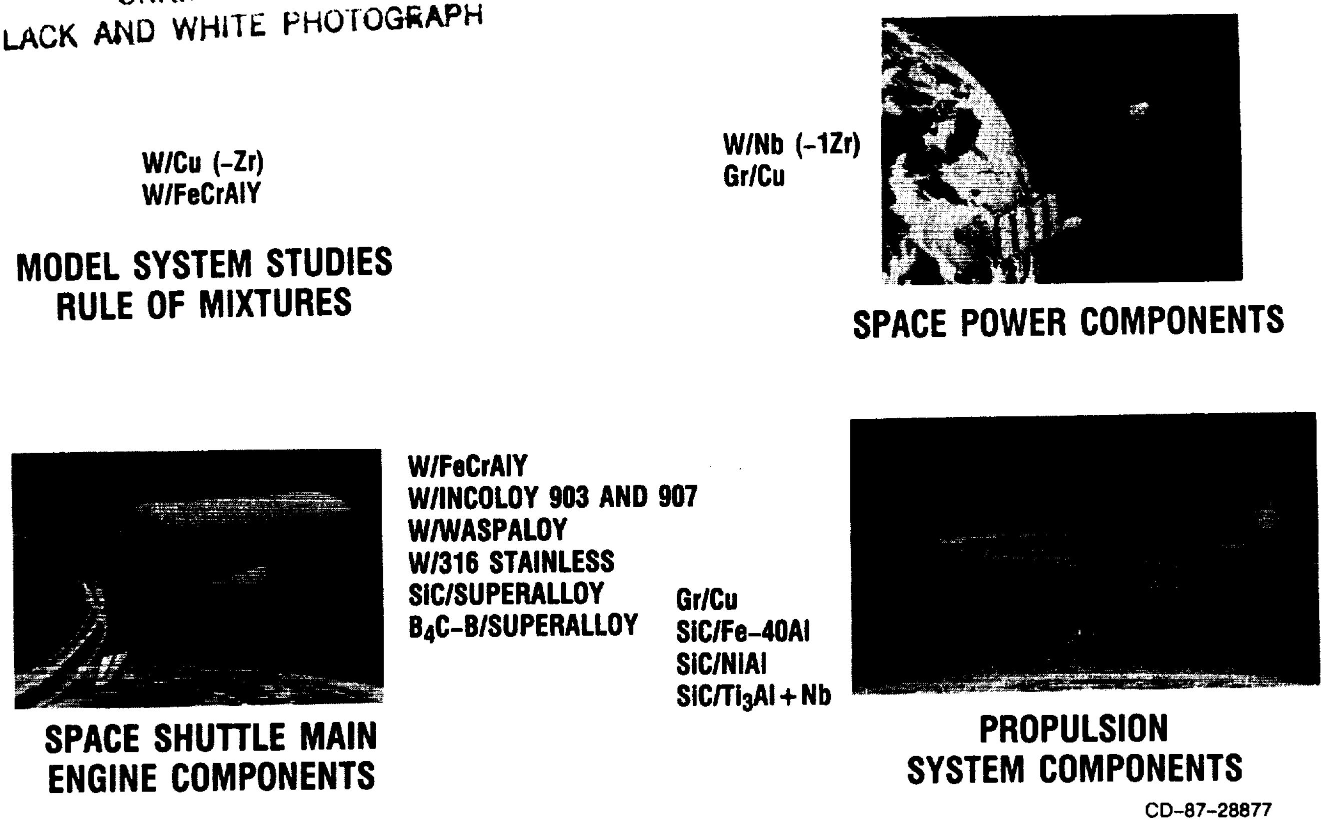

Figure 1. - NASA Lewis involvement in composite materials development.

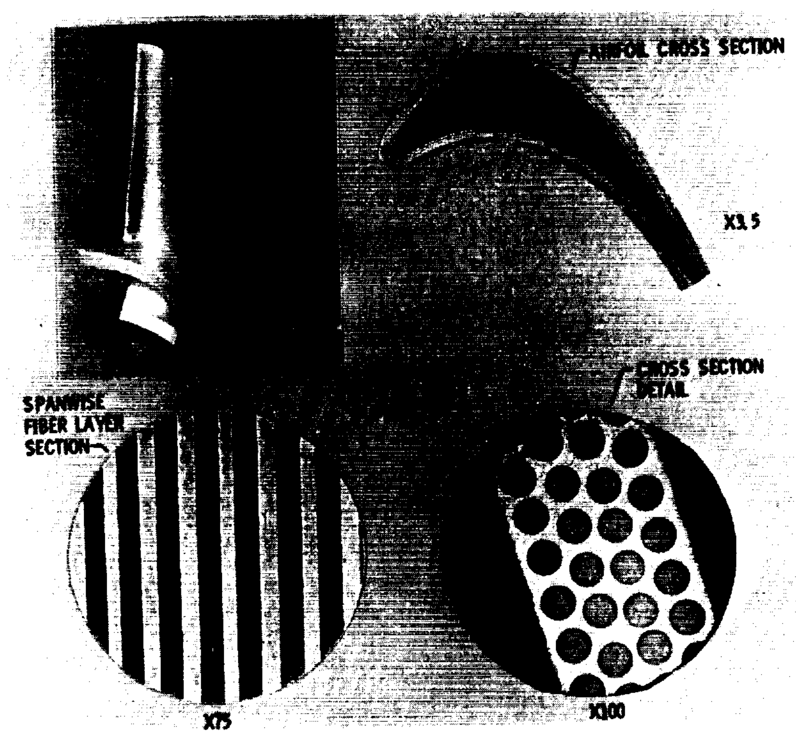

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Figure 2. - Tungsten-fiber-reinforced superalloy blade formed using powder metallurgy (ref. 8).

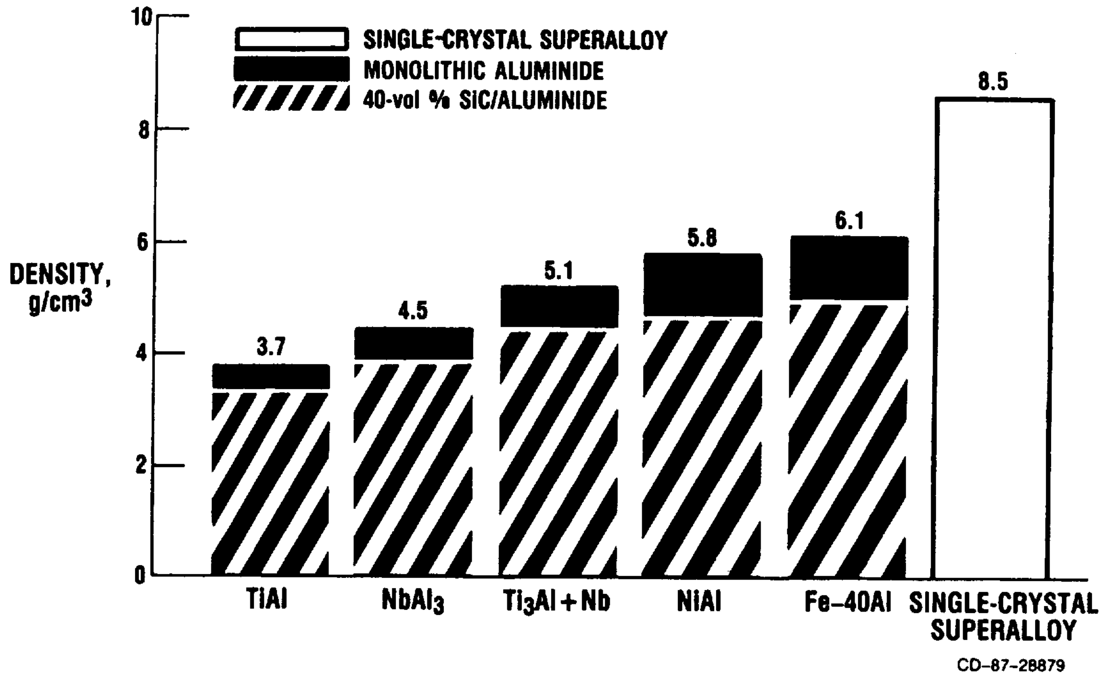


Figure 3. - Density comparison of superalloys, monolithic aluminides, and SiC-reinforced aluminides.

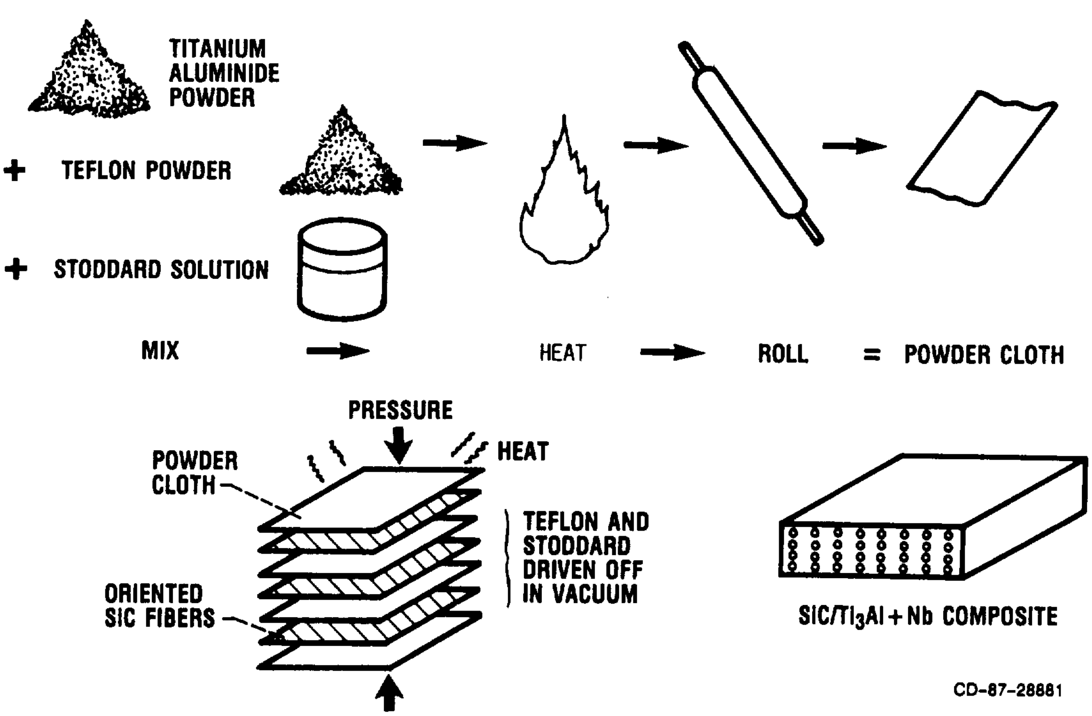


Figure 4. - Powder metallurgy approach currently being used at NASA Lewis to fabricate SiC/Ti_3Al+Nb intermetallic-matrix composite.

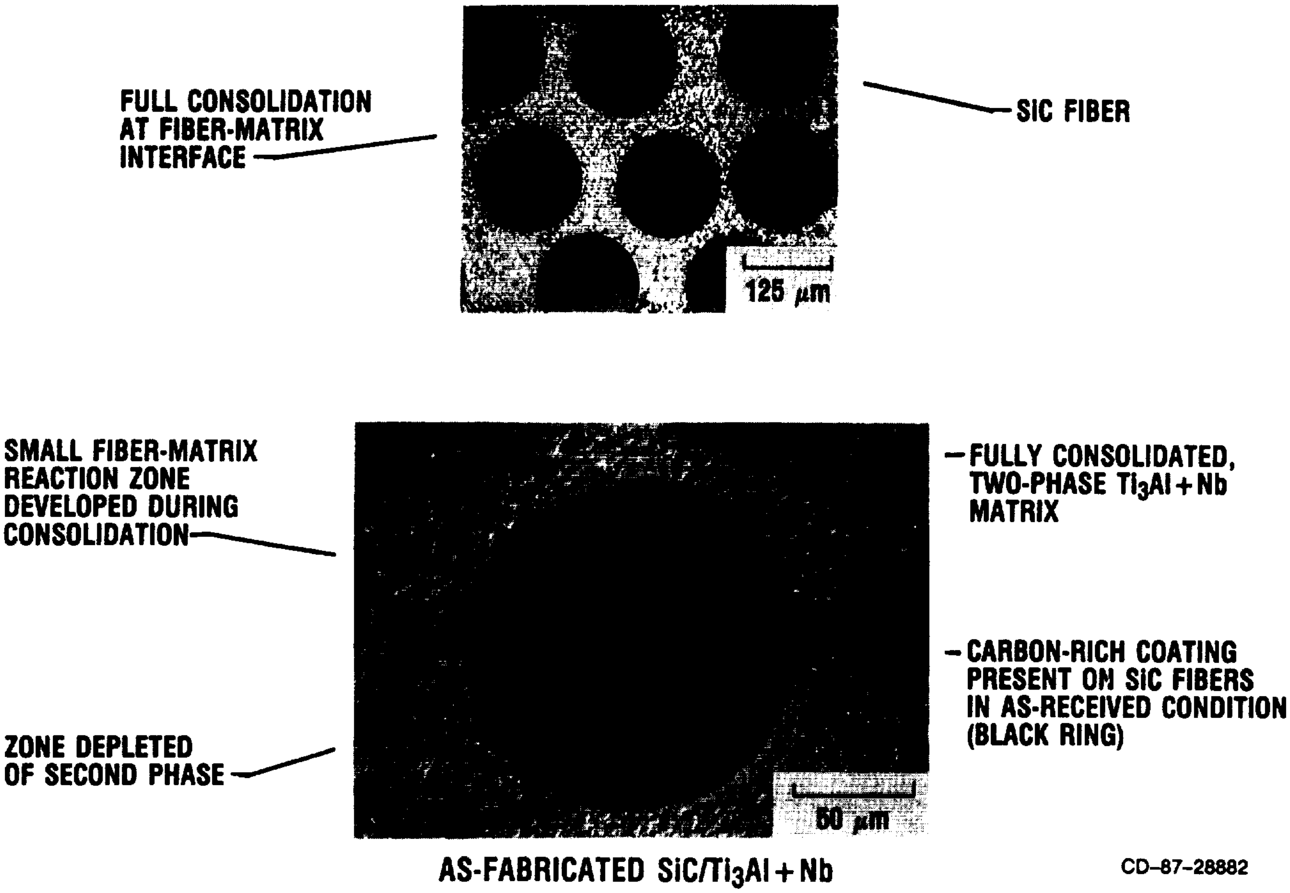


Figure 5. - Two views of SiC/Ti_3Al+Nb composite fabricated by powder cloth technique.

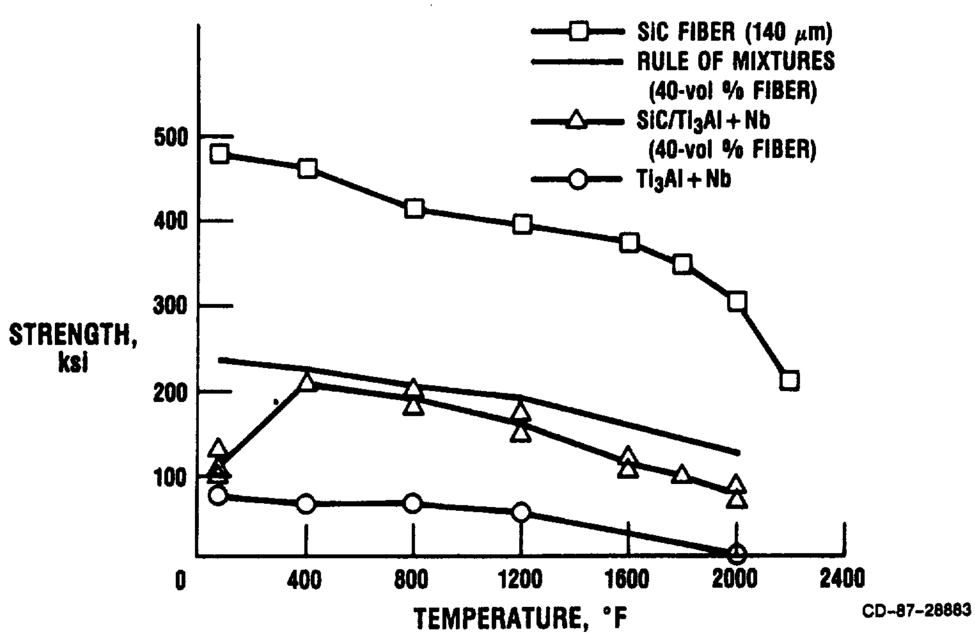
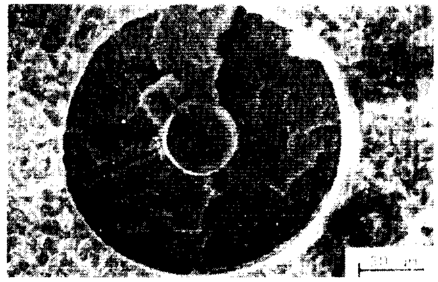


Figure 6. - Tensile properties at temperature for SCS-6 SiC fiber, Ti_3Al+Nb matrix material produced by powder cloth technique, and SiC/Ti_3Al+Nb composite tested in air. Fiber and matrix-only data were used in rule of mixtures (ROM) to determine first-order composite properties.



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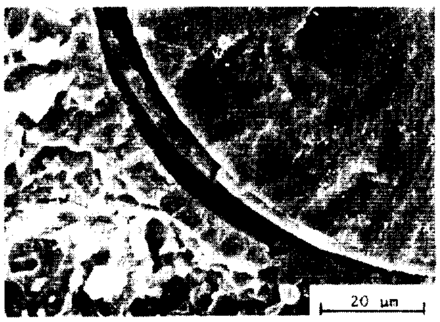
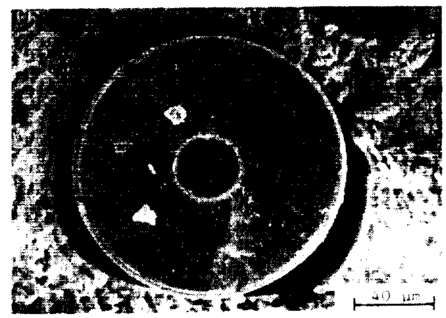


650 °C (1202 °F)



1100 °C (2012 °F)

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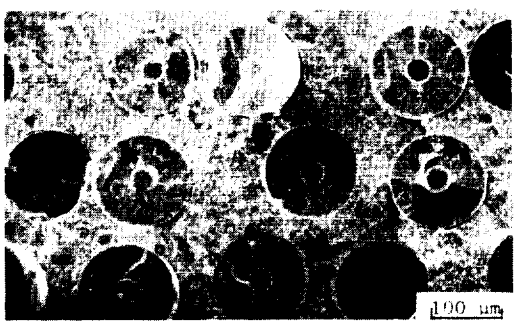
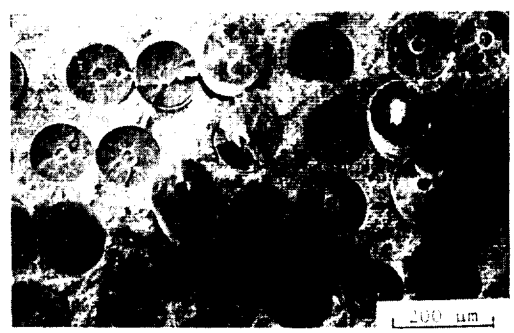
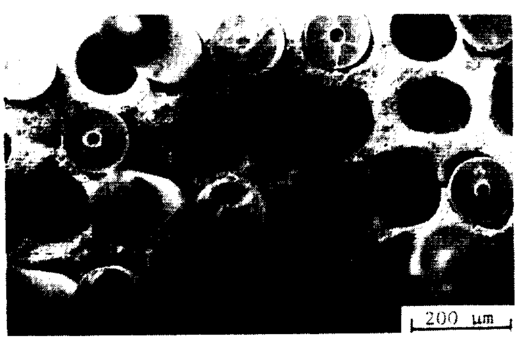
Figure 7. - Presence and location of debonding which occurred at fiber/fiber-coating interface and fiber-coating/matrix interface after elevated temperature tensile tests of SiC/Ti_3Al+Nb.



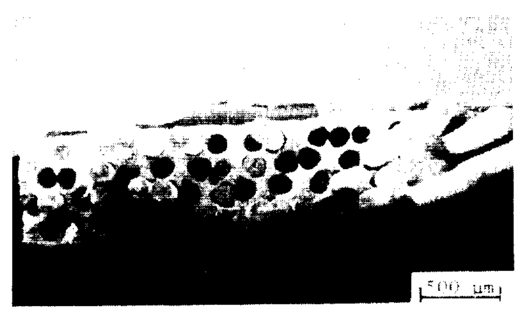
23 °C (73 °F)



425 °C (797 °F)



875 °C (1607 °F)



1100 °C (2012 °F)

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Figure 8. - Increased amount of fiber pullout observed as test temperature increased in tensile fracture surfaces of SiC/Ti_3Al+Nb.

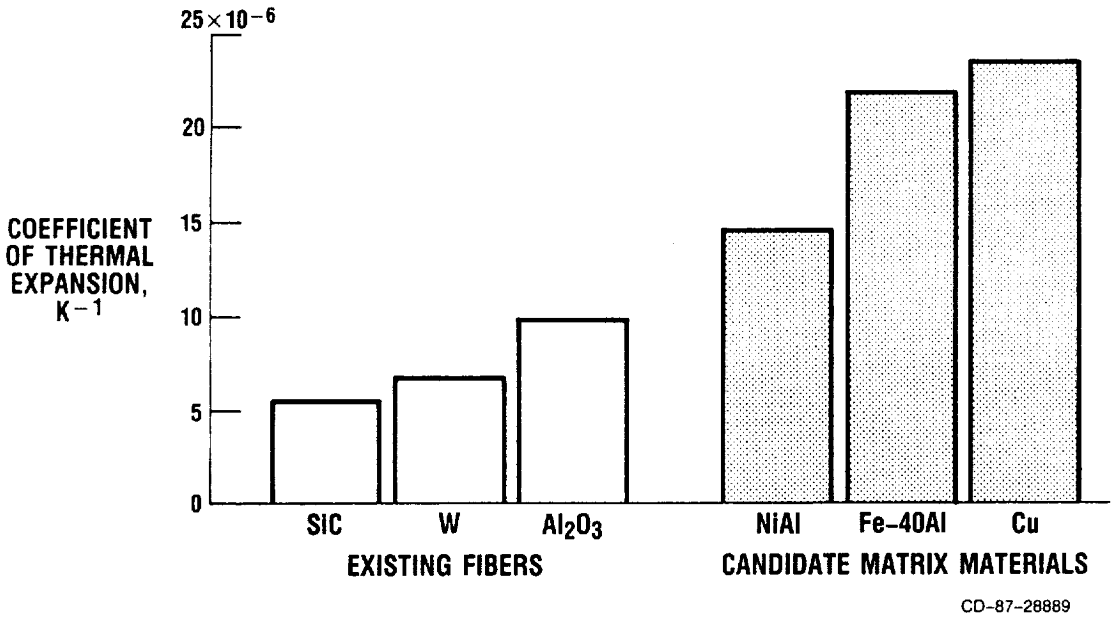

Figure 9. - The two- to five-fold difference in CTE that exists between currently available fibers for reinforcement and some candidate matrix materials point to need for new fiber development of materials with higher CTE's.

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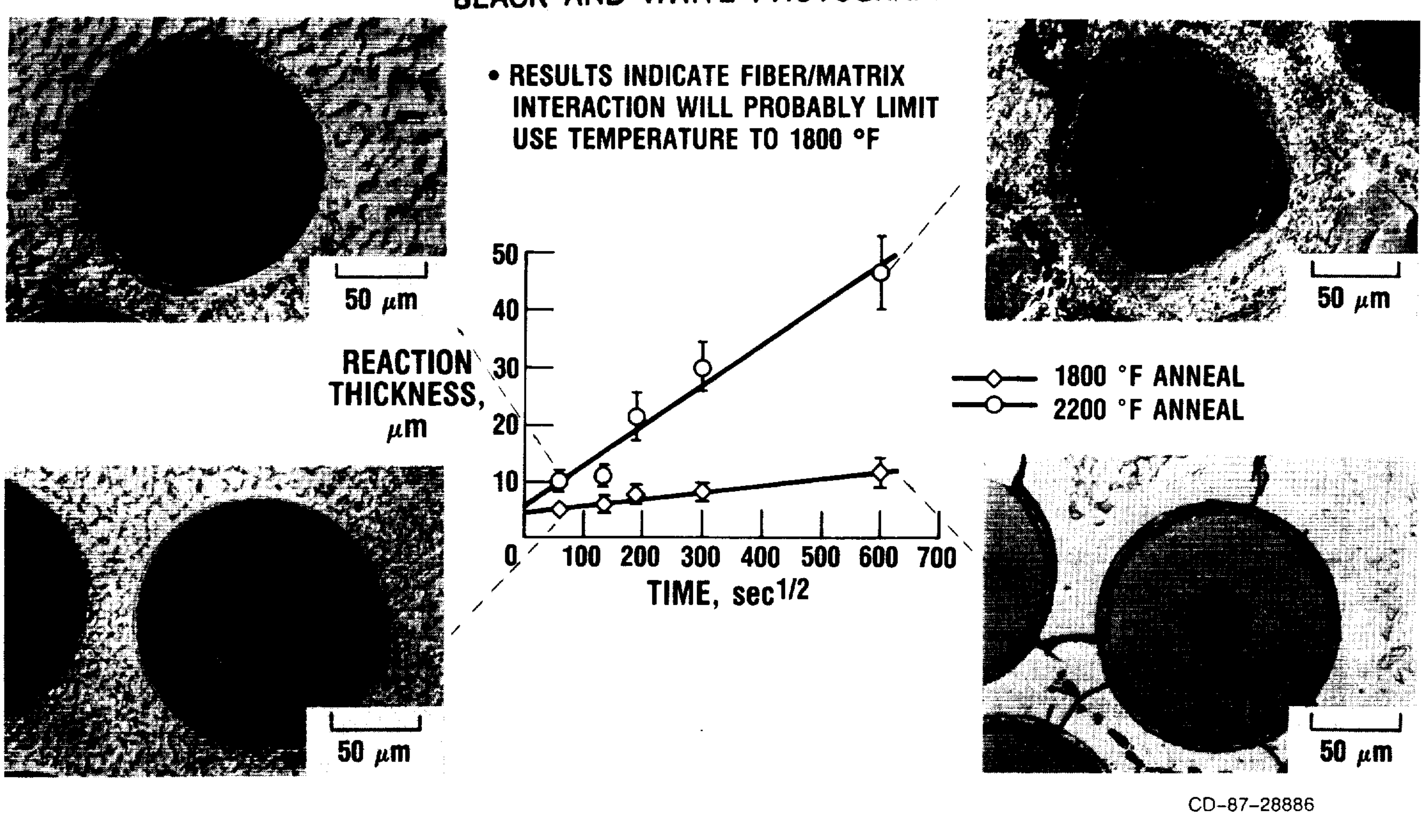

Figure 10. - Fiber-matrix chemical compatibility results of SiC/Ti_3Al+Nb annealed in vacuum. Fiber-matrix reaction will probably limit use temperature of this composite to 1800 °F.

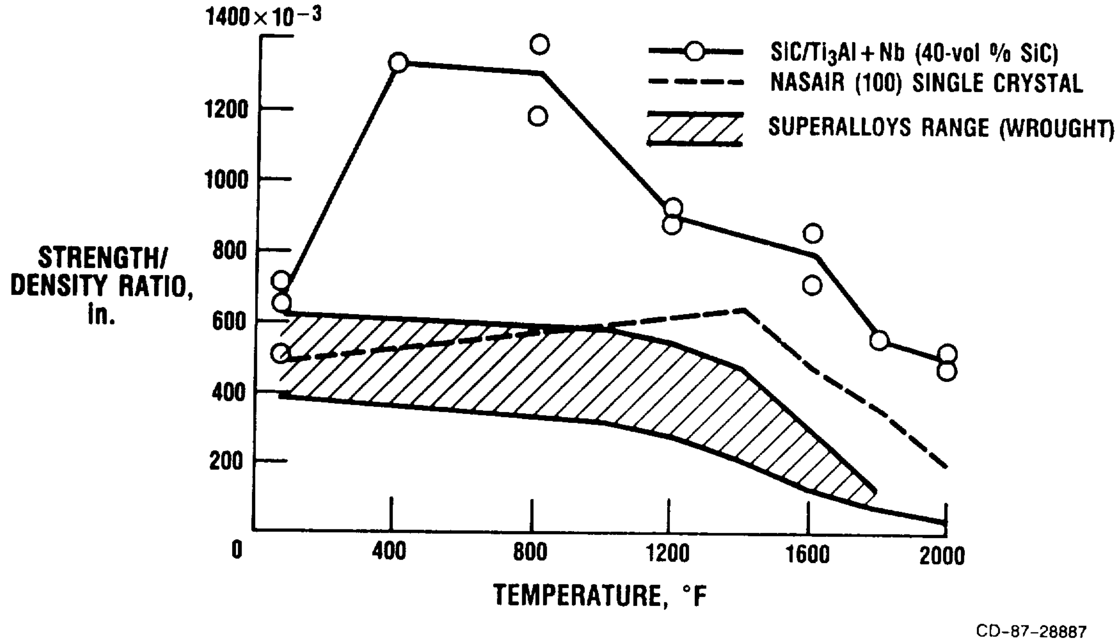

Figure 11. - Strength/density versus temperature comparison of SiC/Ti_3Al+Nb, range of wrought superalloys, and single-crystal superalloy.

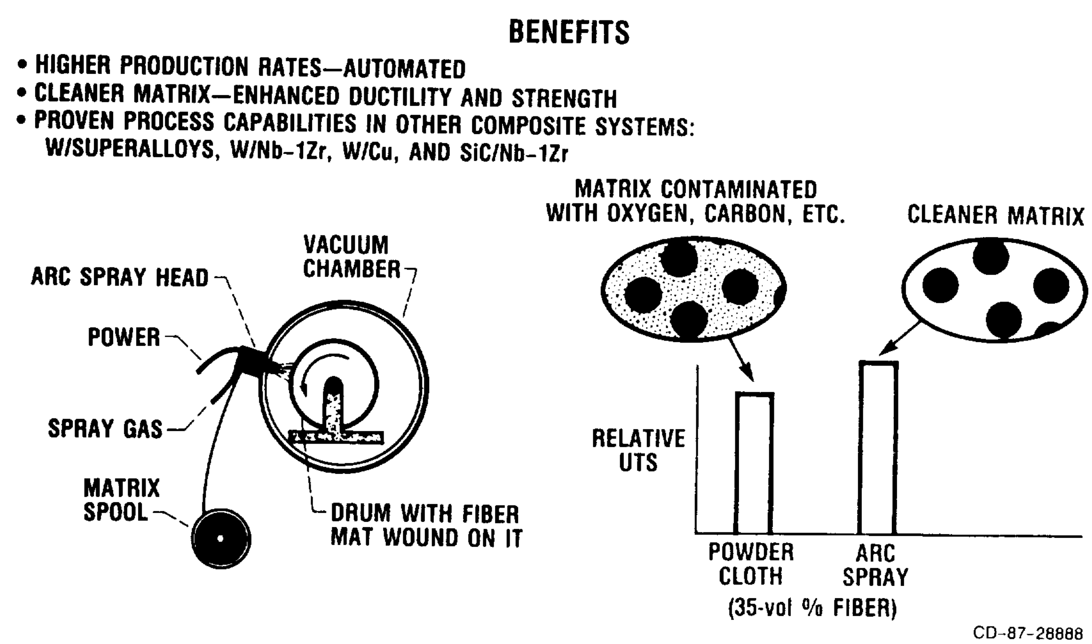

Figure 12. - Future SiC/Ti_3Al+Nb fabrication employing arc spray technique (ref. 17) in order to obtain cleaner matrix materials and higher production rates.

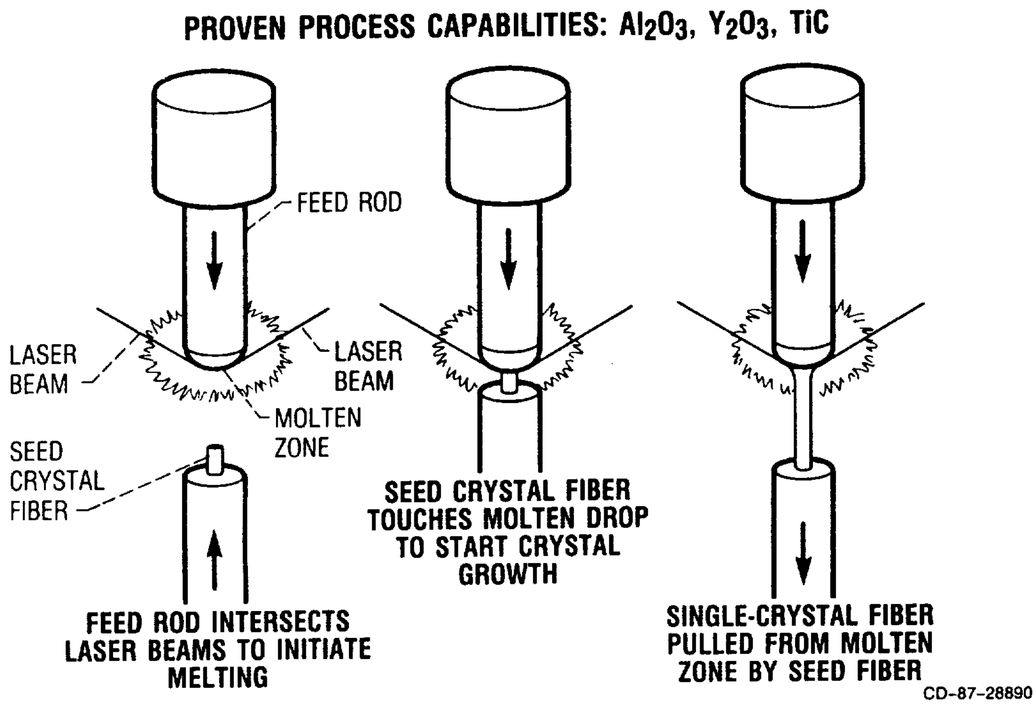

Figure 13. - Floating-zone fiber drawing process (ref. 18) being procured to develop lab-scale quantities of fiber which exhibit CTE's similar to intermetallics being considered as matrix materials.