Steel-SiC Metal Matrix Composite Development

DOE grant # DE-FG02-03ER83587

Phase 1 Final Report

7/21/2003-4/20/2004

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One of the key materials challenges for Generation IV reactor technology is to improve the strength and resistance to corrosion and radiation damage in the metal cladding of the fuel pins during high-temperature operation. Various candidate Gen IV designs call for increasing core temperature to improve efficiency and facilitate hydrogen production, operation with molten lead moderator to use fast neutrons. Fuel pin lifetime against swelling and fracture is a significant limit in both respects.

The goal of this project is to develop a method for fabricating SiC-reinforced high-strength steel. We are developing a metal-matrix composite (MMC) in which SiC fibers are embedded within a metal matrix of steel, with adequate interfacial bonding to deliver the full benefit of the tensile strength of the SiC fibers in the composite. In the context of the mission of the SBIR program, this Phase I grant has been successful.

The development of a means to attain interfacial bonding between metal and ceramic has been a pacing challenge in materials science and technology for a century. It entails matching or grading of thermal expansion across the interface and attaining a graded chemical composition so that impurities do not concentrate at the boundary to create a slip layer. To date these challenges have been solved in only a modest number of pairings of compatible materials, *e.g.* Kovar and glass, titanium and ceramic, and aluminum and ceramic. The latter two cases have given rise to the only presently available MMC materials, developed for aerospace applications. Those materials have been possible because the matrix metal is highly reactive at elevated temperature so that graded composition and intimate bonding happens naturally at the fiber-matrix interface. For metals that are not highly reactive at processing temperature, however, successful bonding is much more difficult. Recent success has been made with copper MMCs for cooling channels in first-wall designs for fusion¹.

The focus of the Phase 1 program has been the development of a new approach to achieving bonding between a SiC fiber and a metal matrix. We solve the interface problem by coating the fiber with a thin layer of the matrix metal *before* it is incorporated into the matrix. Achieving bonding at the SiC-metal interface is addressed by introducing adhesion layers and tempo transition layers as intermediate steps in the deposition process. When the coated fiber is then introduced into a metal matrix, the bonding is between identical metals and should proceed readily. This approach is proprietary and is the subject of a patent application that is in preparation. We expect that it will make it possible to form a metal matrix composite with virtually *any metal and*

any ceramic. The importance of this development is given context by the comments of another group who are developing ceramic-matrix composites (CMC) which pose similar problems:

“Adequate fiber coatings are requisite for providing damage tolerance (toughness) to CMC’s. Fiber coatings also protect fibers from environmental attack during composite fabrication and use. Fiber coatings must demonstrate chemical and mechanical stability in high-temperature corrosive environments in order to maintain the necessary fiber-coating-matrix de-bonding characteristics for damage tolerance in the composite. Unfortunately, the development of interfacial coatings has lagged behind the development of ceramic fibers. Therefore, the widespread use of CMC’s has been limited, to a large extent, by inadequate fiber coatings.”²

Given the difficulty of the project (and a series of unforeseen HR issues), we feel that enough progress has been made during the execution of this Phase I effort to warrant its continuation, even though we did not meet the original goal of fabricating a complete sample MMC for high-temperature creep testing.

We believe (and intend to demonstrate in this report and proposal) that the interfacial coating technology we are developing has the merit to continue into Phase II in its own right. Our Axial Thermal Evaporation (ATE) process, invented during the execution of this Phase I SBIR and currently in development, has the potential to improve any fiber-based composite utilizing metal as an interfacial coating component.

The reviewers’ comments received at the start of the Phase I were thoughtful, insightful, and pertinent. Based on their comments we strove to identify the most important “go/no-go” technical challenges of our approach, then solve as many of those challenges as possible.

It became obvious that the key such challenge is the development of a functionally graded system of ceramic/metal intermetallics at the fiber surface.

We believe that our Phase I proposal contained the correct technological approach, but that our strategy for implementing this approach was flawed on two accounts: The metallization method and the fiber incorporation method. During the execution of this Phase I, we have corrected both flaws in our strategy and turned them into strengths.

Revised Research Objectives:

1. Understand the reasons why the intended product has not been previously commercialized.
2. Develop a sound plan with countermeasures for all known previous failure modes.
3. Identify and procure the best candidate ceramic fibers for process development.
4. Identify and procure the best candidate metals for intermetallic formation.
5. Identify and procure the best candidate stainless steel for MMC development.
6. Commission a high-vacuum thermal evaporation unit for metal coating.
7. Develop a highly uniform metal coating process for the ceramic fibers.
8. Commission an RF induction furnace for MMC formation.
9. Develop an optimized thermal bonding process.
10. Measure high-temperature creep properties of the completed MMC sample.

Summary of Progress:

At the time of this writing we have completed the first six objectives and are in progress on objectives 7 and 8.

Objective 1: Why Doesn't the Product Already Exist?

The 3M™ Nextel™ family of fiber products has given rise to an entire product line of aluminum-based metal matrix composites (AMC's). These are perhaps the best example yet of the marketability and value of a well-designed MMC. 3M™ AMC's are marketed for use in automotive push rods, brake calipers, in rotating structure components, and for composite power conductors³.

Ti/SiC unidirectionally reinforced MMC's have been investigated for aerospace uses. It is not yet clear how well the composites will ultimately perform how widespread the applicability of the materials will be. These materials have attracted enough attention to have had quite a bit of serious characterization done by many groups. As such, the typical failure modes have already been researched and the results published in the literature. For instance, a SiC/Ti-15-3 MMC was studied by the TAMU Aerospace Engineering Department in 2000, and the failure modes found were typical of the material:

“Microstructural evaluation identified the primary damage modes for both the transverse and axial specimens. The axial specimens showed evidence of cracks *developing perpendicular to the loading direction starting from the fiber-matrix interface*. The transverse specimens showed cracks emanating from areas of poor consolidation resulting in cracks propagating in the loading direction along grain boundaries.”⁴

The failure mode is pictured in Figure 1. This is a direct consequence of the failure to produce bonding at the fiber/metal interface.

In discussion with the manufacturer of the selected ceramic fiber (Objective 3), we confirmed that the main issues for a steel/SiC MMC commercial product are the high work temperature and the high thermal expansion coefficient α of stainless steel. This, of course, was assumed at the outset of the original proposal. No one to date has succeeded in making steel/SiC composites.

Figure 1. Fiber pull-out failure in Ti/SiC unidirectionally reinforced MMC (from Ref. 5).

Objective 2: What will you do differently to be successful?

Strategy Changes $\Rightarrow$ New Ideas:

A comprehensive, integrated approach to interface engineering: Axial Thermal Evaporation (ATE)

We changed two aspects of our strategy in our effort to create a prototype steel/SiC MMC. The first aspect of the approach that required improvement was the fiber metallization, and we believe that we have come up with a compelling approach, which we have named Axial Thermal Evaporation (ATE). *ATE solves several of the problems associated with fiber coating and inter-metallic formation by performing the metallization and thermal annealing steps at the same time.*

We originally proposed develop interfacial bonding using RF sputtering coat to coat the fibers with metal. This method of physical vapor deposition (PVD) proved to be inappropriate for two reasons. First, RF sputtering produces a non-uniform coating on the surface of a round fiber, however. The center of the fiber gets a thicker coating than the edges (Figure 2a). Second, RF sputter coating is typically performed at relatively low temperature, and the deposited metal has too little thermal energy on the surface to enable it to chemically displace the largely stable bonds at the ceramic surface.

This is also the result of standard thermal evaporation, in which the crucible(s) or filament(s) are loaded to create a vapor cloud within the vacuum chamber, and the substrates to be coated are typically rotated some distance away from the evaporation source (Figure 2b). Since the main mechanism for heat transfer in vacuum is photonic radiative heating (Stephan-Boltzmann Law), the distance from the evaporation source keeps the substrate heating reasonably low.

In the present context, the goal is to advantageously create intermetallic species. If the metallization step is separated from the thermal anneal step, then any exposure to atmosphere immediately oxidizes the surface of the metal (barring, of course, noble metals: any process to use these becomes cost-prohibitive almost immediately) and complicates if not destroys the desired outcome.

In our approach, the object to be coated (the thin ceramic fiber) is threaded along the axis of a solenoidal evaporation filament. The fiber is arranged in a reel-to-reel fashion, and its linear speed through the filament can be controlled to very precise values by connecting a stepper motor to the vacuum rotary feedthrough. The placement of the fiber within the cross-section of the filament can also be positioned accurately with external fiber guides.

The ATE concept allows for control of all relevant process parameters:

- Ambient atmosphere
- Metal deposition rate
- Metal deposition thickness
- Fiber composition
- Fiber feed rate (linear velocity)
- Fiber thermal cycle (DT)
- Fiber thermal ramp rate
- Filament geometry

Figure 2. Three techniques for metal deposition on fibers: a) RF sputtering, b) unidirectional thermal evaporation, and (c) axial thermal evaporation (ATE).

ATE combines excellent radial uniformity with integrated thermal annealing. For the low thermal mass of thin ceramic fibers, this is a key intermetallic stabilization step that prevents atmospheric poisoning of the coated ceramic fiber.

In some cases it may be desirable to introduce specific quantities of some gas-phase species. This is a built-in feature of almost all thermal evaporators, and ATE benefits immediately from this feature, allowing for more complex interfacial phases to be created.

Another aspect of ATE that allows for precise interfacial control is the ability to create in-situ anneal-only zones. If additional anneal time is needed to allow for diffusion of the interface into the fiber surface, one only needs a blank filament (*e.g.* the W filament with no evaporation material loaded) set to the desired filament temperature and fiber speed.

Since the system operates in reel-to-reel mode, any number of independent depositions and/or anneal stations can be installed in a single vacuum system. The Denton DV-502 used during the Phase 1 effort has three high-current deposition/anneal stations in addition to the rotary-motion feedthrough. This is sufficient for the first metal system under development (Ta/Ti). It is expected that the DV-502's capabilities will be sufficient through scale-up.

A controllable method for unilaterally-reinforced MMC fabrication

COI Ceramics (U.S. distributor for Nicalon™ family SiC fiber products from Nippon) informed us that the fabrication process for their high-quality SiC fibers precludes the formation of single-strand fiber tows. The nature of their fabrication process creates literally hundreds to thousands of bindings per linear inch.

A review of the literature related to various methods of filling such tows, most notably chemical vapor infiltration (CVI) and other variants of MOCVD (metal-organic chemical vapor deposition) convinced us that this path was a dead-end. Our original approach intended to create a fully filled tow (the unrealized goal of many other researchers as well), but we instead sought to metalize the fibers, then weave the tow. This approach will not be achieved unless an independent vendor has been successful in creating very thin, flexible SiC fibers in bulk.

For the purpose of fuel cladding, the two load strain components are the axial weight of the fuel pin itself and the radial swelling from the core environment. It is not clear that a multi-layer composite composed of unidirectionally reinforced layers with alternating layer direction is inherently unsuitable. In fact, without a steel/SiC MMC to measure, any discussion of the relative merits of one layup over another is purely speculation.

Therefore we have chosen to design to a specification that results in a unidirectionally reinforced MMC product similar to existing Ti/SiC products. We believe that the correct approach to continuous processing is to create a single-layer tape-form MMC as the “unit cell” basis of any final-form MMC product that ATC would market.

To efficiently pack the matrix, we intend to wire and foil as illustrated in Figure 3. Using wires to fill the fiber layer ensures uniform fiber spacing and simplifies the task of filling voids with the foil. Varying the ratio of wire/fiber and the outer foil thickness provides very tight process control over the metal/composite ratio.

It also provides the potentially interesting idea of a multi-component matrix. The multi-component MMC concept may prove useful when trying to optimize bulk properties of the MMC like thermal or electrical conductivity. Multi-component MMC's may also prove to be the key to designing into a highly constrained problem (like nuclear materials).

Figure 3. Conceptual design of the prototype MMC, designed to be modular and allow for design flexibility with respect to fiber concentration and matrix composition.

Figure 4. SCS-6 SiC monofilament from Specialty Materials. We also have SCS-5A (~40AWG) that matches the 17-7PH wire. (Objective 3).

Figure 5. We decided to use 17-7PH stainless steel as our matrix due to its low thermal expansion coefficient. This is AWG40 wire custom-made for this project (Objective 5).

Figure 6. Custom tungsten evaporation filament and Denton Vacuum evaporator.

Objective 3: SCS SiC Fiber, Refractory Carbides

The starting point for the interface design is to determine the size of the thermal coefficient (delta-alpha) mismatch between the fiber and the matrix material. Specialty Materials specifies the coefficient of thermal expansion (alpha) at 20°C for their SCS SiC fibers as 4.1 ppm/°C. Whenever possible, we try to obtain alpha at the highest possible value in order to estimate worst-case values of the delta-alpha mismatch.

The entire diameter of an SCS SiC fiber is not approximately stoichiometric silicon carbide. Discussions with process engineers at Specialty Materials confirmed that the actual composition of SCS fiber is a center of C, followed by the bulk of the fiber being SiC, and a “significantly carbon-rich” outer layer. Specialty Materials did not have an assay of the Si/C ratio at the surface of their fibers.

What is important from this information is that the amount of carbide formation will be “significantly” higher than silicide formation. In the context of our approach, this is important, since in general refractory carbides have moderate to low alpha values and extremely high operating temperatures⁶. Additionally, good carbide layers can help prevent free carbon migration to the matrix interface, which could lead to failure nucleation sites due to surface C precipitation.⁷

Objective 4: Metal coatings for intermetallic layers

Selection of a suitable set of coatings is perhaps the most difficult of the planning steps associated with the fabrication of an MMC. Quite a number of programs exist to calculate binary diagrams, and these are one of the bases on which we selected the Ta/Ti system for our first attempt. Figures x through y show the relevant binary diagrams.

Thermal diffusion effects dominate intermetallic formation. As such, we desire to as completely as possible form a dual diffusion barrier against drift of both C and Si during post-coating thermal treatments of the metal matrix. Creation of refractory carbides appears to be the best path towards this goal.

Due to the extremely high melting point of tantalum (2996°C), a partial pressure of 10⁻⁸ Torr is obtained at a filament temperature of 2000°C.⁸ Therefore the deposition rate is expected to be quite low. The expected carbide and silicide phases can be read from the binary diagrams, if the specific ratio of C/Si at the surface of the fiber were known.

Table 1. Sampling of alpha values and melting points of several carbides.

Material	Symbol	MP (°C)	CTE @ 20°C (ppm/°C)
Tungsten Carbide	W ₂ C	2785	4
Tungsten Carbide	WC	2850	5.1
Tantalum Carbide	TaC	3880	6.3
Niobium Carbide	NbC	3500	6.7
Zirconium carbide	ZrC	3540	6.7
Titanium Carbide	TiC	3065	7.7
Molybdenum Carbide	Mo ₂ C	2520	7.8
Chromium Carbide	Cr ₃ C ₂	1800	10.3

The SCS composition data can be obtained, but is typically expensive for precise measurement. Evans, Texas (Round Rock, TX 77845) quoted us ~\$1000/scan for a high-precision diameter scan. We are investigating the capabilities and prices for high-end metrology at some of the TAMU centers that accept outside work. Irrespective of the exact ratios of carbides to silicides, it is clear from the binary diagrams that both types certainly exist at the expected deposition temperatures.

We have demonstrated the ability to quite readily melt titanium in our DV-502 (1660°C) using a tungsten evaporation filament. At this phase transition, the thin film deposition rate is at its theoretical maximum. As such, we can coat “thick” films (compared to the Ta layer, still expected to be <1000Å) of titanium directly onto the TaC/TaSi layer.

At this temperature it is evident that much more uncertainty exists for the final silicide phase than does for the carbide phase. It is not yet clear what quantity of Si would be actually present for TiSi formation. As our first ATE-processed samples are completed in the next three weeks, we should get better data to answer these questions.

It is not clear yet whether the formation of a higher- α TiSi ($\alpha = 13.2^\circ$) would be beneficial to effectively put the matrix metal under compressive shear. This effect can be modulated by the thickness of the Ta layer, and we have an external power supply more than sufficient to push the Ta filament temperature to well over 2500°C and greatly increase the deposition rate.

Objective 5: Matrix stainless steel selection

There are a very large number of steel products on the market, ranging widely in composition, microstructure, and formation method. The nuclear literature routinely discusses Sandvik HT9, which at best is a niche specialty product. This specific material is not widely available, and as such isn't the best candidate for the purposes of process development: We desire a proof-of-principle prototype with the ability to do many experimental runs over a wide range of process conditions. To this end we selected a more commercially available product than HT9. UNS-S42200 is the closest semi-commercial match that we could find to the literature's average values with regard to HT9 composition. Nevertheless, UNS-S42200 is difficult and expensive to obtain in either the wire or foil forms we desire for MMC fabrication, since it is typically cold in bar form for high-strength fastener applications (bolts).

Since some references cite Carpenter as a joint researcher for HT9, we compared their Carpenter 636 version of UNS-S42200 versus the literature average (see Table 2). Stainless steel families range in α (@20°C) from 11 to 16 ppm/°C, with UNS-S31600 (stainless 316) having the highest expansion, and both UNS-S42200 (stainless 422) and UNS-S17700 (stainless 17-7PH) having the lowest expansion.⁶ See Table 3.

Table 2. Comparison of HT9 and UNS-S42200 stainless steel composition.

Grade	Cr	Ni	Fe	C	Mo	W	Mn	Si	V	P	S
HT-9	11.83	0.53	bal	0.21	1.04	0.50	0.59	0.37	0.28		
UNS-S42200	12.50	0.75	bal	0.23	1.03	1.03	<1.0	<1.0	0.35	<0.04	<0.03

Figure 8. Phase diagrams for binary systems at Ti/Ta/C/Si interfaces: a) C-Ta, b) Si-Ta, c) C-Ti, d) Si-Ti, e) Ti-Ta.

Table 3. Comparison of coefficient of expansion α for several stainless steels and temperatures

Material	CTE @ 20°C (ppm/°C)	CTE @ 250°C (ppm/°C)	CTE @ 500°C (ppm/°C)
UNS-S17700	11	11.2	
UNS-S31600	16	16.2	17.5
UNS-S42200	11.2	11.3	11.9
UNS-S44000	10.2		

Objective 6: Fiber Temperature Control

An important aspect of the ATE process that must be controlled is the fiber temperature. This can readily be obtained by using a collimator. Collimators are routinely used in commercial PVD sputtering machines to achieve directional coating for high-aspect ratio contact fill.

In the typical semiconductor PVD application, the geometry of the collimator is a disc with a number of holes through it. Some metal is deposited in and on the collimator with every process run, decreasing its ability to allow metal to pass through its holes. Consequently, the metal deposition rate slowly falls over time as the collimator coats.

Normally the process time per wafer is periodically increased to maintain constant metal film thickness on the processed wafers. After some number of process runs (or fixed number of wafers), the collimator is replaced, and the process time adjusted back down to compensate for the deposition rate increase.

We use a cylindrical collimator in exactly the same way. Each hole in the collimator acts as a deposition point source, and the benefits discussed above of the axial thermal evaporation process are not affected in any way by the presence or absence of the collimator. The only aspects of the process that change are the fiber temperature and the metal deposition rate.

The motivation for this process element is straightforward. See Figure 9 below, in which the partial pressure for Ta and Ti are plotted as a function of their temperature. Since in vacuum the only heat transfer mechanism is radiative, we can use a poor heat conductor for the collimator material, we must shield the fiber as necessary by drilling the collimator holes accordingly. We will use a continuous reel-to-reel feed of fiber through a succession of solenoidal filaments, each depositing one of the layers in the graded desired graded composition of the metal coating. The effective substrate temperature of each deposition will be controlled using collimators on each filament.

Figure 9. Partial pressure (log scale) versus material temperature for Ta and Ti.

Figure 10. a) Experimental setup for axial thermal evaporation; b) detail showing Ti foil cylinder wrapped on W filament prior to heating to Coat Ti on the filament.

Objective 7: Development and characterization of Ti-coated SiC

The above development was completed by the end of the Phase 1 effort. The re-thinking of the deposition process took much of that period, and we succeeded in trial coatings only at the very end of that time. ATC continued its R&D thereafter to develop the ATE process, produce prototype coated SiC fibers, and characterize the interfacial bonding.

The experimental setup used to develop ATE coating is shown in Figure 10a. In a previous run of the evaporator, the W filament was coated with Ti by spiral-wrapping a strip of Ti foil on the W filament and then heating it to melt the Ti into the W surface, as shown in Figure 10b.

The SiC fiber is then supported on the axis of the Ti-coated W filament (Figure 11a). The evaporator system was evacuated to 10^{-7} Torr and the filament was heated to a temperature of ~ 1000 C (Figure 11b) and Ti is deposited on the fiber. The thickness of deposition is controlled by the current vs. time profile delivered to the filament.

The resulting Ti layer was imaged in the series of views of increasing magnification shown in Figure 12, Figure **13**, Figure 14. There are several things to notice in these micrographs. First, the layer is extremely well bonded to the SiC substrate. When the fiber is fractured, the layer does not pull away but adheres locally to the fractured segments, even to the point of tenaciously holding the broken pieces of the fiber together.

Second, Figure 15 shows an analysis of the stoichiometry of the interface, performed by scanning electron dispersion scanning across a transverse of the image shown in Figure 16. The composition is *graded* across the interface: C has diffused into the Ti layer, and Ti has diffused into the SiC fiber. This type of diffusion is pivotal in producing strong bonding at a disparate interface. It does *not* occur in sputtered interfaces; it only happens because of the 1000 C heating of the substrate fiber. That is the truly new innovation that is achieved with the ATE process.

Figure 11. a) Positioning the SiC fiber on the axis of the filament; b) ATE coating in progress.

Figure 12. 60x optical photo after Ti coating. Top fiber is uncoated, bottom fiber is Ti-coated.

Figure 13. 160x SEM image after Ti coating. The fiber was cracked in order to examine the interface.

Figure 14. SEM micrographs showing the Ti-SiC interface region: a) 1000X; b) 6500x.

Figure 16. 10,000x SEM image of Ti-SiC interface.

Figure 15. EDX analysis of composition profile through the Ti-SiC interface.

Conclusions

We have succeeded in producing an extremely well-bonded interface of a Ti surface layer on a SiC fiber. That is the key first step to a graded-interface bonding of SiC fibers within a steel matrix. The next steps will be fully develop and optimize this successful process, to coat steel onto the Ti-coated fibers, and finally to embed the steel-coated fibers within a steel matrix and demonstrate full bonding throughout.

We would like to emphasize that this development only attained the success described in the last section in December, 2004 – 8 months after the deadline for Phase 2 proposals. It was accomplished by the perseverance of the company and the PI, continuing the development using company funds because we believe this can make a revolution in composite materials.

It will be necessary to do a great deal more characterization to fully optimize the Ti-SiC interface. Ti, Si, and C form a host of intermetallics, as shown in the binary phase diagrams of c,d. These intermetallics can either serve beneficial purposes (stress pinning) or pose pathological problems (seeding growth of large-scale impurities that initial fracture). We will need to use micro-crystallography to characterize the nature and distribution of intermetallics in the interface.

We will have to undertake a similar development for a Ta layer that would be placed on top of the Ti layer (see its phase diagram in a,b,e). Then finally we must develop the steel bonding to the multi-layer coated fiber, optimize its bonding, and finally develop the MMC fabrication that utilizes the coated fibers.

ATC has a business model for development of this technology, in which we manufacture coated fibers that fully match from a ceramic fiber core to a desired metal outermost coating. The coated fibers would solve the most difficult challenges in MMC technology, and would be shelf-stable for shipment to customers who could incorporate them into MMC products with minimum complications and maximum performance benefit.

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