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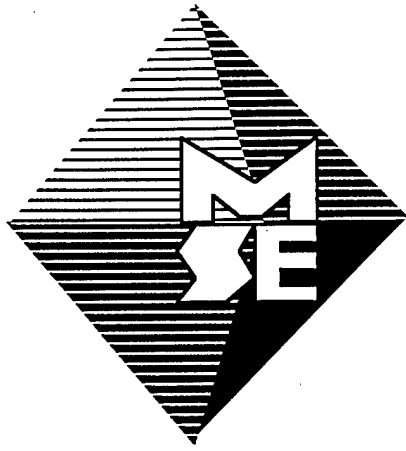
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Office of Naval Research/DARPA

Final Report
for
ONR Grant No. *N00014-91-J-4132*

*Intermetallic Matrix Composites
via in-situ Displacement Reactions*

Submitted to:

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Intermetallic Matrix Composites via *in-situ* Displacement Reactions

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This constitutes the final report on ONR Grant No. N00014-91-J-4132 which was performed at the University of Florida from 7/1/91-6/30/95. This report is divided into a summary section that indicates the program goals followed by highlights of the accomplishments with reference to the appropriate papers/dissertations that resulted from this work. The research students involved in this program are then described followed by a listing of papers, patents and presentations. Copies of the manuscripts and patents are included at the end.

A. Scientific Research Goals

Among the obstacles encountered in the development of intermetallic matrix composites are (1) the thermodynamic and mechanical incompatibility between the matrix and reinforcement phases and (2) the matrix-reinforcement interfacial properties which are not ideal from the standpoint of achieving optimum properties in the composite as a whole. In the past, the first of these problems has been addressed by coating the reinforcements with some sort of barrier or compliant layer before their introduction into the matrix. The various coating methods investigated (sol-gel, CVD, PVD) tend to be expensive, time consuming and unreliable with respect to producing sound coatings. This research program was initially concentrated on identifying more cost-effective and reliable schemes for developing the requisite coatings. The basic approach is to identify simple processing schemes whereby a beneficial reaction product (or products) is produced *in-situ* at the interfaces between the matrix and reinforcement during consolidation or subsequent heat treatment. As the research evolved, greater focus was placed on the need to achieve a better understanding of microstructural evolution during *in-situ* displacement reactions and on the correlation between the evolved microstructures and the mechanical properties. In the case of silicide-based IMC's, there was a clear need to determine the influence of discrete silica particles, incorporated during the consolidation of powder samples, on the mechanical properties and on both grain size and shape.

B. Highlights

Mo-Si-C

- Mechanical alloying of elemental molybdenum, silicon and carbon powders allows their consolidation at relatively low temperatures to produce silica-free, SiC-reinforced MoSi₂ composites. The reactions that occur during mechanical alloying and consolidation have been characterized in some detail (1,2).
- When mechanically alloyed Mo and Si powders are consolidated without carbon, the incorporated silica exists as discrete particles rather than as grain boundary films (1,2).
- The discrete silica particles present in the MoSi₂-20 vol. % SiC composites produced by more conventional blend and press methods appear to be detrimental to the high temperature strength of MoSi₂ (Jayashankar and Kaufman, in preparation).
- Silica-free composites with varying quantities of SiC (10-60 vol. %) can be synthesized from elemental powders using a combination of mechanical alloying followed by *in-situ*

displacement reactions. The variations in loading fraction allow the CTE to be tailored to match that of potential reinforcements. Likewise, it is also possible to match more closely the CTE of the SiC-MoSi₂ mixture with that of a substrate for those instances when the product material is to be used as a coating (3,4).

- Thermodynamic calculations revealed that the form of the Mo-Si-C ternary phase diagram proposed by Nowotny et al. is correct at all temperatures whereas that by van Loo is incorrect. Specifically, there is a MoSi₂+SiC+Mo₅Si₃C (Nowotny phase) three-phase field as opposed to the three-phase equilibria between MoSi₂, SiC and Mo₅Si₃ proposed by van Loo (5).
- The SiC size and volume fraction dictate the upper grain size based on Zener drag arguments such that the higher loading fractions correspond to finer grain sizes and vice-versa (Jayashankar and Kaufman, in preparation).
- The fine-grain material appears to be superplastic based on strain rate sensitivity measurements. This implies that grain boundary sliding is the dominant mode of deformation at elevated temperatures. It also suggests that it might be possible to superplastically form the fine-grain composite material (after consolidation of the sub-micron, mechanically alloyed powders) into a complex shape and then coarsen the grains to the "limiting grain size" dictated by the loading fraction of the SiC in order to increase the creep strength (6).
- The presence of silica appears to reduce the high temperature strength of MoSi₂ containing 20 vol. % SiC for similar grain sizes. The highest strengths are achieved in the coarsest-grain, silica-free materials containing only 5 vol.% SiC suggesting that it should be possible to tailor the strength somewhat (6).
- Previous reports of the brittle-to-ductile transition (BDTT) in MoSi₂ are questionable at best. It appears that the so-called BDTT is a misnomer in this and many other materials that exhibit high elongations at some intermediate temperature. Specifically, it is clear that MoSi₂ will be brittle at all temperatures (below the melting point) at sufficiently high strain rates due to the lack of a sufficient number of active deformation mechanisms (slip systems). The increased elongations at elevated temperatures and sufficiently low strain rates is again related to the onset of grain boundary sliding rather than a true change from brittle to ductile behavior (Jayashankar and Kaufman, in preparation).
- Investigations of the extent of the Mo₅Si₃C single-phase field have revealed that it is much narrower than that published in previous studies (Nowotny, et al., van Loo, et al.). Consequently, our attempts to produce single phase Mo₅Si₃C have met with limited success although we are able to produce structures containing only a small volume fraction of a second phase, the nature of which depends on the bulk composition (i.e., the direction of deviation from the single phase field) (7).

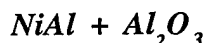
Mo-Si-Al

- Aluminum additions to Mo and Si can be used to produce silica-free MoSi₂ + Al₂O₃ composites. Depending on the amount of aluminum (substituted for silicon), the matrix phase is either the tetragonal C11_b structure or the hexagonal C40 Mo(Si,Al)₂ structure or a mixture of these phases (8-10).
- The volume fraction of alumina is lower than that of the silica due to the ~25% volume decrease associated with the silica to alumina structural transformation (8).
- The aluminum incorporated in the MoSi₂ can be used to reduce the oxide on pre-oxidized niobium to form a protective alumina coating. As expected, the kinetics of this reaction and the relative protection depend on the aluminum concentration/activity (11).

- Similar attempts to produce silica-free $\text{MoSi}_2 + \text{ZrO}_2$ composites using this approach were largely unsuccessful presumably because of the more sluggish transport/transformation kinetics of zirconium relative to aluminum as well as the very low solubility of zirconium in MoSi_2 , again in contrast to that for aluminum. This implies that high solubility solutes are more suitable for such reactions if there is a sufficient driving force for the reduction reaction. (Similar conclusions were drawn from the work on displacement reactions in NiAl using silica precursors as indicated below.)
- The Mo-Si-Al phase diagram appears to be only partially correct and attempts are being made to investigate the regions of the diagram surrounding the C40 phase field in order to determine the diagram more accurately and to elucidate the effects of other phases on the properties of the C40 structure (7).
- Unlike binary MoSi_2 which undergoes catastrophic oxidation "pest" at intermediate temperatures, the C40 $\text{Mo}(\text{SiAl})_2$ phase appears to be susceptible to pest-like degradation at room temperature in moist environments; further analysis to understand this phenomenon mechanistically is underway (Eason, unpublished results).
- In spite of their higher symmetry, the hexagonal C40 and Nowotny phases are extremely brittle much like the tetragonal C11_b MoSi_2 phase. Therefore, the transition to a higher symmetry structure (i.e., more slip systems) only enhances toughness and ductility when the dislocations are mobile at the temperature of interest (Ross and Eason, unpublished results).

Mo-Si-B

- *In-situ* solid-state displacement reactions have been used to synthesize MoSi_2 -molybdenum boride composites at temperatures lower than $0.7T_m$ (where T_m is the melting point of the boride). Finer structures are obtained when MoSi_2 and MoB are the product phases compared with the $\text{MoSi}_2 + \text{Mo}_2\text{B}_5$ composites. It appears that the finer scale structure is related to the fact that these phases grow in a coupled manner (12, 13).
- The discrete silica particles in these Mo-Si-B composites contain boron and, therefore, have a lower melting temperature than those without boron. Aluminum additions can be used to reduce the silica in these composites as in the silicides without boron; in this case, the structure contains alumina, the boride and MoSi_2 (or $\text{Mo}(\text{SiAl})_2$ depending on the aluminum concentration) (12-14).
- This concept was extended to the Mo-Si-B-Ti system in order to produce TiB_2 -reinforced MoSi_2 . The reactants were Mo_5Si_3 , Ti_5Si_3 and boron and the product phases were MoSi_2 and TiB_2 as expected (and silica since there was no deoxidant added). The structures tended to be submicron in scale consistent with the ternary alloys. Significantly, the temperatures required to form the TiB_2 and effect consolidation are considerably lower than those required using mixtures of these phases or those produced using Martin Marietta's XDTM process (12-14).
- Thermodynamic calculations of the Mo-Si-Ti-B quaternary system were made based on the available data and the results confirmed that there is a two-phase field between TiB_2 and MoSi_2 (14).
- Aluminum additions can be used to reduce the borosilicate phase in the TiB_2 -reinforced MoSi_2 as in the other materials described above (11-13).



- Molybdenum-reinforced NiAl composites were investigated using in-situ displacement reactions as the means of protecting the reinforcement. Carbon additions to the NiAl matrix resulted in the formation of an intermediate Mo_2C layer at the interface. However, pre-carburization of the molybdenum prior to reacting it with NiAl resulted in the carbon diffusing from the carbide into the NiAl matrix leaving behind molybdenum solid solution (15).
- Alumina reinforced NiAl can be produced by reacting aluminum with less stable oxides that are intentionally introduced into NiAl during powder processing. As with the MoSi_2 results, the more rapid diffusing species are better suited for the displacement reactions. For example, it is clear that silica transforms most rapidly whereas the other oxides studied (those based on iron, titanium, chromium, etc.) react initially before "shutting down" due to the sluggishness of the diffusion through the reaction layer (16).
- When the silica precursor was used, the resulting alumina phase appeared as a composite mixture consisting of about 75% alumina and 25% NiAl (with some silica in solution). This structure is related to the volume reduction accompanying the silica to aluminum reaction (16).

C. Students and Staff

S. Jayashankar, PhD: *Structure/Property/Processing Relationships in Molybdenum Disilicide/Silicon Carbide Composites*

S. Jayashankar's PhD dissertation work dealt with the carbothermal reduction and SiC formation in MoSi_2 matrix composites produced using in-situ reactions. The processing strategy involved using mechanically alloyed elemental powders of Mo, Si and C followed by hot pressing using a tailored approach designed to remove the gaseous reaction products prior to applying the uniaxial pressure. The resulting compacts were silica free and contained micron scale SiC particulate ranging in volume fractions from 5-40%. Jay's work consisted of (1) developing the processing methodology for producing high density compacts with controlled grain size, (2) microstructural characterization and verification of the absence of silica in the UF samples that were processed using the carbothermal reduction during consolidation, (3) determination of the influence of silica on the mechanical properties, (4) correlation of mechanical properties with grain size (related to the volume fraction of SiC) and (5) strain rate sensitivity tests that indicated that MoSi_2 is actually superplastic at the finest grain sizes. *Status: Completed dissertation and working at Seagate in Anaheim, CA.*

Andre Costa e Silva, PhD: *Synthesis of Molybdenum Disilicide Composites Using In-Situ Reactions*

Andre's PhD dissertation work involved the use of other in-situ displacement reactions that would reduce the silica and form solid state reaction products. Aluminum and zirconium were selected as the candidate elements for this purpose and Andre discovered that aluminum works well whereas the zirconium is too sluggish under the conditions used. Andre then extended his efforts to the in-situ production of borides in the MoSi_2 . Initially, the molybdenum borides MoB and Mo_2B were produced and he subsequently extended the efforts to TiB_2 where he was able to produce fine TiB_2 reinforced MoSi_2 at relatively low temperatures. Andre also carried out thermodynamic calculations using Thermocalc™ for the Mo-Si-C and Mo-Si-Ti-B systems and used the information to make predictions as to processing strategies and to help interpret the experimental results. *Status: Completed dissertation and teaching/working in Brazil.*

Scott Riddle - M.S.:

Microstructural Development and Characterization of Molybdenum Disilicide/Silicon Carbide Composites Formed by In-Situ Displacement Reactions

Scott worked with S. Jayashankar on the $\text{MoSi}_2 + \text{SiC}$ system and produced silica-free composites containing up to 60 v/o SiC. His materials were characterized as to their structure/property relationships and summarized in his MRS paper and his thesis. *Status: Completed thesis and working in industry.*

Prakash Krishnan - Non-thesis M.S.

Prakash worked on the NiAl composites and investigated the in-situ reactions leading to interface coatings and dispersoids of alumina. His work involved the reactions leading to protective interfaces in NiAl reinforced with refractory metals as well as the displacement reactions involving aluminum and the less stable oxides. The results led to two publications and a number of presentations. Unfortunately, he left with a non-thesis masters because his wife and he obtained jobs in Silicon Valley. *Status: Working in industry.*

Amelia Luisa Dempere - PhD

Amelia came to UF on a scholarship from her native country, Venezuela. After becoming familiar with the department, improving her English and her materials science background (chemistry undergraduate), she decided to work with myself and Andre on the silicide-boride system and perform more thorough investigations of the in-situ reactions leading to the TiB_2 -reinforced MoSi_2 . Her work was just beginning when this program ended and she is continuing to pursue this effort accordingly. *Status: Dissertation work continuing.*

Eli Ross - PhD

Eli has a B.S. from Johns Hopkins and is working on a PhD currently. His work began as an attempt to produce the Nowotny phase and study its properties but has evolved into a more general approach for investigating the structure and properties of intermetallics based on some of their characteristics. Specifically, he has developed a classification scheme in which the various intermetallics are categorized based on such properties as bulk modulus, ambient fracture toughness, etc., to be used by investigators who are attempting to develop the more exotic intermetallics for structural applications. *Status: Dissertation work continuing.*

Paul Eason - PhD

Paul continues to investigate the phase equilibria, physical and mechanical properties of the C40 $\text{Mo}(\text{SiAl})_2$ phase and their similarities and differences with respect to binary MoSi_2 . His results indicate that this phase is also very brittle at room temperature. However, there appear to be issues with respect to low incipient melting temperature as well as catastrophic oxidation (pest) at near ambient temperatures. This latter result is confusing and further investigation is underway. *Status: Dissertation work continuing*

D. Manuscripts, Patents and Presentations

Manuscripts

1. S. Jayashankar and M.J. Kaufman: "In-Situ MoSi₂ Composites by Mechanical Alloying," *Scripta Met.* **26** (1992) 1245-1250.
2. S. Jayashankar and M.J. Kaufman: "Tailored MoSi₂/SiC Composites By Mechanical Alloying," *J. Materials Research* **8** (1993) 1428-1441.
3. S. Jayashankar, S.E. Riddle, and M.J. Kaufman: "Synthesis and Properties of In-Situ MoSi₂/SiC Composites," *Mat. Res. Soc. Symp. Proc.*, **322** (1994) 33-40.
4. S.E. Riddle, S. Jayashankar, and M.J. Kaufman: "Microstructural Evolution in Compositionally Tailored MoSi₂/SiC Composites," *Mat. Res. Soc. Symp. Proc.* **322** (1994) 291-296.
5. A. Costa e Silva and M.J. Kaufman: "Phase Relations in the Mo-Si-C System Relevant to the Processing of MoSi₂-SiC Composites," *Met. Trans.* **25A** (1994) 5-15.
6. S. Jayashankar and M.J. Kaufman: "Effect of Oxygen Content on the Superplastic Behavior of MoSi₂/SiC Composites," *Advanced Synthesis and Processing of Composites and Advanced Ceramics II*, **79**, 163-173, 1996.
7. E.N. Ross, P.D. Eason, and M.J. Kaufman: "Processing of Low Silica MoSi₂-Based Compounds Using Carbon and Aluminum Additions," *Processing and Fabrication of Advanced Materials V*, Proceedings of 1996 Fall TMS Meeting, 347-360, 1996.
8. A. Costa e Silva and M.J. Kaufman: "Microstructural Modification of MoSi₂ Through Aluminum Additions," *Scripta Met.* **29** (1993) 1141-1145.
9. A. Costa e Silva, S. Jayashankar, P. Krishnan, and M.J. Kaufman: "Novel Processing of Intermetallic Matrix Composites Using In-Situ Displacement Reactions," in *Processing and Fabrication of Advanced Materials for High Temperatures Applications - II*, Proc. 1992 TMS Symposium, (eds.) V.A. Ravi and T.S. Srivatsan (1993), pp. 19-31.
10. A. Costa e Silva and M.J. Kaufman: "Applications of In-Situ Reactions to MoSi₂-Based Materials," *Mat. Sci. Engr.*, **A195** (1995), 75-88.
11. A. Costa e Silva and M.J. Kaufman: "In-Situ Formation of Alumina Coatings in Niobium Toughened Mo(Si,Al)₂," *Scripta Met.* **31**, (1994), 853-858.
12. A. Costa e Silva and M.J. Kaufman: "Synthesis of MoSi₂-Boride Composites Through In-Situ Displacement Reactions," *Intermetallics*, **5**(1), 1-15, 1997.
13. A. Costa e Silva and M.J. Kaufman: "Synthesis of MoSi₂ Matrix Composites Using In-Situ Reactions," in *Processing and Fabrication of Advanced Materials for High Temperatures Applications - III*, Proc. 1993 TMS Symposium, (eds.) V. A. Ravi, T. S. Srivatsan, and J. J. Moore (1994), pp. 585-598.
14. A. Costa e Silva and M.J. Kaufman: "The Use of Thermodynamics in the Design of In-Situ MoSi₂ Composites," in *Processing and Fabrication of Advanced Materials IV*, 1996 TMS Symposium, (eds.) T.S. Srivatsan and J.J. Moore, pp. 755-774.
15. P. Krishnan and M.J. Kaufman: "Development and Characterization of Interface Coatings in Molybdenum-Reinforced NiAl Matrix Composites," *Met. Trans.* **25A** (1994) 2111-2116.

16. P. Krishnan, A. Costa e Silva, and M.J. Kaufman: "Synthesis of NiAl/Al₂O₃ Composites via *In-Situ* reduction of Precursor Oxides," *Scripta Met.* **32** (1995) 839-844.

Patents

1. S. Jayashankar and M.J. Kaufman: "Refractory Metal Reinforced MoSi₂/SiC Composite with Matched Thermal Coefficients of Expansion," U.S. Patent #5,340,531, Issued Aug. 23, 1994, Assigned to the University of Florida.
2. S. Jayashankar and M.J. Kaufman: "Composite Silicide/Silicon Carbide Mechanical Alloy," U.S. Patent #5,454,999, Issued October 3, 1995, Assigned to the University of Florida.

Presentations

1. S. Jayashankar and M.J. Kaufman: "*In Situ* Reinforced MoSi₂ Composites by Mechanical Alloying," Presented at American Ceramic Society, Minneapolis, MN, April 1992.
2. A. Costa e Silva, S. Jayashankar, P. Krishnan, and M.J. Kaufman: "Novel Processing of Intermetallic Matrix Composites Using *In-Situ* Displacement Reactions," Presented at 1992 TMS Symposium, Chicago, IL, October 1992 - *Invited*.
3. M.J. Kaufman: "The Use of *In Situ* Reactions in the Development of IMC's Based on MoSi₂," Presented at High Temperature Materials Workshop, Wright Patterson AFB, OH, May 1993.
4. A. Costa e Silva and M.J. Kaufman: "Synthesis of MoSi₂ Matrix Composites Using *In-Situ* Reactions," Presented at 1993 TMS Symposium, Pittsburgh, PA, October 1993 - *Invited*.
5. A. Costa e Silva and M.J. Kaufman: "Solid-State Displacement Reactions in the Synthesis of MoSi₂ Matrix Composites," Presented at TMS Spring Meeting, San Francisco, CA, March 1994.
6. S. Jayashankar, S.E. Riddle, and M.J. Kaufman: "Synthesis and Properties of *In-Situ* MoSi₂/SiC Composites," Presented at Fall MRS Symposium, Boston, MA, November 1994.
7. S.E. Riddle, S. Jayashankar, and M.J. Kaufman: "Microstructural Evolution in Compositionally Tailored MoSi₂/SiC Composites," Presented at Fall MRS Symposium, Boston, MA, November 1994.
8. S. Jayashankar and M.J. Kaufman: "Development of MoSi₂/SiC Composites via *In Situ* Displacement Reactions," Presented at TMS Fall Meeting, Cleveland, OH, October 1995.
9. S. Jayashankar and M.J. Kaufman: "Effect of Oxygen Content on the Superplastic Behavior of MoSi₂/SiC Composites," Presented at American Ceramic Society Meeting, Cocoa Beach, FL, January 1996.
10. A. Costa e Silva, S. Jayashankar, and M.J. Kaufman: "Synthesis and Characterization of Low Silica MoSi₂ Composites Using *In Situ* Displacement Reactions," Presented at TMS Annual Meeting, Anaheim, CA, February 1996.
11. S. Jayashankar and M.J. Kaufman: "Effect of an Intergranular Silica Phase on the Elevated Temperature Behavior of MoSi₂/SiC Composites," Presented at TMS Materials Week '96, Cincinnati, OH, October 1996.
12. A. Costa e Silva and M.J. Kaufman: "The Use of Thermodynamics in the Design of *In-Situ* MoSi₂ Composites," Presented at 1996 TMS Symposium, Cincinnati, OH, October 1996 - *Invited*.

13. E.N. Ross and M.J. Kaufman: "Processing of Low Silica MoSi₂-Based Compounds I: Carbon Additions," Presented at 1996 Fall TMS Meeting, Cincinnati, OH, October 1996.
14. P.D. Eason and M.J. Kaufman: "Processing of Low Silica MoSi₂-Based Compounds II: Aluminum Additions," Presented at 1996 Fall TMS Meeting, Cincinnati, OH, October 1996.
15. S. Jayashankar and M.J. Kaufman: "Microstructural Effects on the Elevated Temperature Behavior of MoSi₂/SiC Composites," Presented at Fall MRS Meeting, Boston, MA, December 1996.
16. M.J. Kaufman, et al: "Overview: Processing MoSi₂-Based Intermetallics," Presented at 4th International Conference on High Temperature Intermetallics, San Diego, CA, April 1997 - *Invited.*