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1. REPORT DATE (DD-MM-YYYY) June 30, 2000		2. REPORT TYPE Final Technical Report		3. DATES COVERED (From - To) 5/01/93 - 4/30/00	
4. TITLE AND SUBTITLE  The Mechanics and Mechanical Behavior of High-Temperature Intermetallic Matrix Composites.				5a. CONTRACT NUMBER	
				5b. GRANT NUMBER AFOSR F49620-93-1-0289	
				5c. PROGRAM ELEMENT NUMBER	
6. AUTHOR(S)  R. Gibala, A.K. Ghosh, D.J. Srolovitz, J.W. Holmes, N. Kikuchi, R.O. Ritchie				5d. PROJECT NUMBER	
				5e. TASK NUMBER	
				5f. WORK UNIT NUMBER	
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) Ronald Gibala Department of Materials Science and Engineering The University of Michigan 2300 Hayward Street Ann Arbor, MI 48109-2136				8. PERFORMING ORGANIZATION REPORT NUMBER	
9. SPONSORING / MONITORING AGENCY NAME(S) AND ADDRESS(ES) Department of the Air Force Air Force Office of Scientific Research 801 North Randolph Street, Room 732 Arlington, VA 22203-1977 Attn: Dr. Craig S. Hartley				10. SPONSOR/MONITOR'S ACRONYM(S)	
				11. SPONSOR/MONITOR'S REPORT NUMBER(S)	
12. DISTRIBUTION / AVAILABILITY STATEMENT DOD - See DOD 5230.4 "Distribution Statements on Technical Documents." Approved for public release.					
13. SUPPLEMENTARY NOTES					
14. ABSTRACT This program has examined mechanics analyses and mechanical behavior of advanced intermetallic matrix composites based on the matrix materials MoSi <sub>2</sub> , Nb <sub>5</sub> Si <sub>3</sub> , NiAl, and TiAl, including Ti-based metal matrix composites. There has been a major focus on determination of strength of interfaces in composites under simple and complex loading configurations involving constrained deformation. Interface strength properties have been determined by use of novel experiments (e.g., asymmetric-notch shear testing of model laminates), finite element simulations, homogenization analysis, and first-principles calculations. Additional focus involved investigation of crack growth, process zone formation, and damage accumulation processes during creep, fatigue, shear, tension, and compression of monolithic matrix materials and ductile phase toughened composites. These experiments have been developed in conjunction with methods of theoretical modeling, e.g., by macroscopic mechanics of composite interfaces (finite elements, homogenization), analytical theory of eutectic microstructural evolution, and first-principles calculations of MoSi <sub>2</sub> -Mo and NiAl-Cr/Mo interfaces. Damage generation by thermal fatigue, thermal shock, and thermal misfit stresses has also been investigated experimentally and modeled analytically. The experimental program has included development of processing techniques to produce novel composites and microstructures for mechanical investigations: directional solidification of NiAl, Nb <sub>5</sub> Si <sub>3</sub> , and MoSi <sub>2</sub> -based alloy composites, powder processing of MoSi <sub>2</sub> toughened with particulates and uniquely shaped (e.g., wire-mesh) second phase dispersoids, hot pressing methods to form macrolaminates of MoSi <sub>2</sub> , Nb <sub>5</sub> Si <sub>3</sub> , and NiAl-based composites, and physical vapor deposition to produce finer scale micro-laminates of TiAl.					
15. SUBJECT TERMS Intermetallic matrix composites, silicides, aluminides, laminates, ductile phase toughening, creep, fatigue, thermal fatigue and misfit, fracture toughness, finite element analysis, homogenization analysis, interface properties, first-principles calculations					
16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT	18. NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON
a. REPORT	b. ABSTRACT	c. THIS PAGE			Ronald Gibala
Unclassified	Unclassified	Unclassified	Unlimited	70	19b. TELEPHONE NUMBER (include area code) 734-936-0178

Standard Form 298 (Rev. 8-98)  
Prescribed by ANSI Std. Z39.18

16 AUG 2000

**THE MECHANICS AND MECHANICAL BEHAVIOR OF HIGH-  
TEMPERATURE INTERMETALLIC  
MATRIX COMPOSITES**

AFOSR-MURI PROGRAM  
Grant No. AFOSR F49620-93-1-0289

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**FINAL TECHNICAL REPORT FOR THE PERIODS:**

Funding period: 5/01/93 – 4/30/97  
No-cost extension: 5/01/97 – 4/30/98  
Unfunded research period: 5/01/98 – 4/30/00

Air Force Office of Scientific Research  
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June 30, 2000



The University of Michigan

AFOSR-MURI Program

**The Mechanics and Mechanical Behavior of High-Temperature Intermetallic  
Matrix Composites**

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**I. EXECUTIVE SUMMARY**

The goal of this program has been to provide fundamental guidelines for alloy design of stable microstructures in intermetallic matrix composites (IMCs) and metal matrix composite (MMCs) for structural applications. We have developed an interactive experimental and theoretical framework that provides predictive understanding for achieving high strength at high temperatures and engineering toughness at lower temperatures. The framework includes analysis of monotonic and cyclic fatigue, creep, and creep/fatigue damage accumulation and development of atomic, microstructural, and macroscopic design principles for composite interfaces. The program includes synthesis and processing of IMCs, utilizing processing methods to produce microstructures that test guidelines and design principles developed.

The research program includes individual research projects which cover topics such as: interface adhesion and bonding by first-principles calculations; theory of microstructural stability of eutectic composites; homogenization analysis of composites; interface strength and toughness in model laminates; high rate physical vapor deposition (PVD) of micro-laminates; structure-properties-processing of in-situ eutectic composites; fracture, fatigue-crack growth and thermal fatigue in IMCs; and strength of carbon coatings on SiC in Ti-matrix MMCs. The materials emphasis was initially on MoSi<sub>2</sub>-based composites (including particulate, whisker and ductile-layer-toughened systems) and Nb-toughened Nb-Nb<sub>5</sub>Si<sub>3</sub> alloys. This was followed by emphasis primarily on NiAl-based materials, mainly in-situ eutectic NiAl-Cr and NiAl-Mo alloy composites and model laminates.

For convenience and reference throughout this report, the major results and accomplishments of the research program have been divided into three major categories: modeling, experimental mechanical behavior, and experimental infrastructure. Brief one- or two-sentence descriptions of each key result or accomplishment are given in the remainder of this executive summary. In the main body of the report, extended technical abstracts with accompanying figures which depict many of the key results are given for research projects. A list of publications, theses and presentations is also included.

## Major Results and Accomplishments 1993-2000

### Modeling: Structure and Bonding

- We have made first-principles predictions for interfacial adhesion in MoSi<sub>2</sub>-based composites and the effects of impurities on adhesion in these systems.
- We have successfully extended our own previous first-principles calculations, an ab-initio local density functional electronic structural code, of interface bonding and adhesion of MoSi<sub>2</sub>-Mo interfaces to NiAl-Cr and NiAl-Mo interfaces.
- We have developed an interactive computational procedure which greatly reduces the time required for making first-principles calculations.
- We have made first-principles predictions for interfacial adhesion in NiAl/Cr and NiAl/Mo composites.
- We have determined by first-principles calculations the effects of impurity solutes (interstitial and substitutional) on the strength and adhesion of NiAl-Cr and NiAl-Mo interfaces.

### Modeling: Microstructural

- We have developed a new analytical model for stability of lamellar and fiber eutectic microstructures at high temperatures and under stress.
- We have determined analytically (and experimentally) the role of elastic mismatch stresses in the deformation, fracture, thermal fatigue and thermal stability of NiAl-based laminates and eutectics.
- We have modeled the thermal misfit damage accumulation in IMC composites.
- We have successfully analyzed effects of reinforcement morphology on IMC matrix microcracking.
- We have developed an analytical model and boundary integral simulation method for examining crack nucleation.
- We have successfully analyzed twinning in coatings and thin films due to thermal mismatch and transformation stresses.
- We have made the first use of homogenization theory to predict the optimum volume fraction of particulate additions (SiC) in an IMC (MoSi<sub>2</sub>-SiC) and have extended the analysis to NiAl-base microstructures.
- We have modeled the deformation, fracture, and thermal fatigue behavior of IMCs by the finite element code ABAQUS and by advanced non-linear homogenization analysis.

### **Mechanical Behavior: MoSi<sub>2</sub>-base Materials**

- We have developed and mechanically tested novel particulate composites, including MoSi<sub>2</sub>-Nb wire-mesh "particles" with substantially improved toughness and fatigue crack resistance.
- We have successfully extrusion-formed MoSi<sub>2</sub> and melt processed MoSi<sub>2</sub>-TiSi<sub>2</sub> composites.
- We have successfully analyzed the competing roles of particulate strengthening and fine-grain-size weakening of MoSi<sub>2</sub>-based IMCs at elevated temperatures.
- We have achieved plastic deformation of polycrystalline MoSi<sub>2</sub> and its composites at temperatures as low as 700°C. In performing these experiments, we have determined the limiting deformability of a Von Mises slip-system-limited material.
- We have demonstrated that monolithic polycrystalline MoSi<sub>2</sub> has very poor thermal fatigue and thermal shock resistances, which are improved by SiC particulate additions.

### **Mechanical Behavior: NiAl-base Materials**

- We have determined plasticity and fracture initiation mechanisms in NiAl-based eutectics and laminates.
- We have determined the roles of residual interstitial impurities and substitutional-alloy-element partitioning on the strength and toughness of NiAl-based eutectics.
- We have determined the relative roles of intrinsic and extrinsic toughening mechanisms of NiAl-Cr and NiAl-Mo eutectics. We have compared and contrasted these results with our separate results on optimally ductile-phase toughened NiAl.
- We have demonstrated that NiAl-Mo eutectics exhibit stable (R-curve) crack growth behavior, whereas NiAl-Cr do not unless prepared with minimal residual interstitial impurities.
- We have determined that single-crystal NiAl can be dimensionally unstable under thermal fatigue conditions because of the large elastic anisotropy.
- We have demonstrated that NiAl-Mo and NiAl-Cr eutectics can exhibit large-scale plasticity-induced microstructural instability under thermal fatigue conditions.

### **Mechanical Behavior: Laminated and Model Interfacial Materials**

- We have characterized ductile phase toughening in several IMC-layered composites based on MoSi<sub>2</sub>, NiAl and TiAl.
- We have developed new or improved methods for measuring ambient and high temperature interface strength of laminated IMCs and model MMCs, with special

emphasis on interface shear strength measurements based on a novel technique involving asymmetric loading.

- We have made measurements of Mode I and mixed-mode fracture properties of IMC laminate composites of MoSi<sub>2</sub>- and NiAl-based materials.
- We have determined effects of processing variables on the microstructures and mechanical properties of carbon coatings on SiC fibers in Ti-base MMCs.

#### **Mechanical Behavior: Fatigue Related**

- We have characterized the fracture and fatigue-crack propagation in MoSi<sub>2</sub>-based particulate composites and NiAl-based in-situ eutectic composites. The composite additions to both materials improve toughness and fatigue resistance relative to the monolithic materials.
- We have determined the thermal fatigue and thermal shock behavior of MoSi<sub>2</sub> and its particulate composites and of NiAl and NiAl-Cr and NiAl-Mo in-situ eutectic composites. Compositing improves the thermal fatigue resistance of both materials.

#### **Experimental Infrastructure**

- We have synthesized and processed all materials investigated in this program by techniques afforded by AFOSR-MURI support or assistance: arc casting, directional solidification, crystal pulling, melt spinning, ball milling, hot pressing, HIPing, vapor deposition.
- We have developed capability for mechanical testing from room temperature to 1500°C. Testing in tension, compression, bending, compact tension and specialized loading environments has been used.
- We have developed hot hardness (microhardness) and nanoindentation techniques for use in this program.

The University of Michigan

AFOSR-MURI Program

**The Mechanics and Mechanical Behavior of High-Temperature  
Intermetallic Matrix Composites**

Grant No. F49620-93-1-0289

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**II. ABSTRACTS AND SUMMARIES OF MAJOR RESULTS  
AND ACCOMPLISHMENTS**

This section of the final report gives abstracts or short summaries of several of the research investigations undertaken during the period from May 1, 1993 through April 30, 2000. Abstracts or summaries are accompanied by one or more key figures which demonstrate the results obtained or the principles developed. This group of abstracts and summaries is not intended to be comprehensive, but to reflect the range of activities encompassed by the program and the fundamental depth to which the research was taken.

For easy reference, the abstracts and summaries have been divided into the same categories as the major results and accomplishments listed in the Executive Summary: modeling, experimental mechanical behavior, and experimental infrastructure. Various sub-categories used under each broad category are also maintained in this section. More detailed information about each project described in this section may be found in publications or theses listed in Section III or obtained by contacting any of the investigators listed as authors.

## Modeling: Structure and Bonding

### METHODS AND APPLICATIONS OF FIRST-PRINCIPLES CALCULATIONS

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The theoretical effort involving first-principles quantum-mechanical calculations focussed on the study of surface and interfacial energetics and adhesion in several intermetallic/ metallic systems. We have used first-principles computational techniques to calculate the total energy vs. separation curves for the systems NiAl/Cr, NiAl/Mo, MoSi<sub>2</sub>/Mo, and Al<sub>2</sub>O<sub>3</sub>/Cu. A typical result is given in Fig. 1. In addition to the adhesion energy, various electronic properties such as work functions, densities-of-states, and electronic charge-density distributions have been obtained. The first two (work functions and densities-of-states) allowed us to calibrate our calculations with experimental measurements and with previous calculations to ensure the accuracy of the results. Charge-density distributions allow us to examine the details of interfacial bonding. The only input for these calculations is the placement of the atoms in the slabs and atomic number for each atom.

For all of these systems, the total energy vs. separation of the interface was found to follow a universal form which decays as an exponential. This exponential decay indicates that wave function overlap plays a significant role in the bonding. Thus the interfacial bonding, including that for the ceramic/metal Al<sub>2</sub>O<sub>3</sub>/Cu system, exhibits covalent/metallic character. In all of these systems, the interfacial bonds are strong relative to the bond strengths of the constituent phases.  $W_{ad}$  for the interface is intermediate between the values for the constituents.

Since it is well known that fractional monolayers of impurities at interfaces can significantly affect fracture behavior, we chose to investigate how three common non-metallic impurities, namely, B, C, and S, and a refractory metal Nb affect interfacial adhesion in Mo/MoSi<sub>2</sub>. Our *ab initio*, electronic structure based results showed that, while substitution of Nb for Mo causes small changes explainable by the difference in elemental properties between Mo and Nb, inclusion of either one of the three non-metallic impurities results in significant decohesion when they act as interstitial impurities (i.e., increase the spacing between the Mo and MoSi<sub>2</sub> planes). It appears that the reduction of the ideal fracture energy or work of adhesion is proportional to the atomic size of the impurities. S, being the largest impurity of the non-metallic, shows the largest effect: the ideal fracture energy of this S-contaminated interface was less than half of the value for the impurity-free configuration. To clarify the ambiguity surrounding site preference of the impurities, we calculated a configuration in which S acts as a substitutional impurity. Our results suggested that S tends to take the substitutional site instead of the interstitial one. Results for the substitutional B-doped configuration indicate, however, that B strongly favors the interstitial site. The ideal fracture energy for the substitutional configuration is only about a half of that for interstitial case. As C is even smaller than B, the tendency toward favoring interstitial sites is even stronger for C at the Mo/MoSi<sub>2</sub> interface. The picture that emerges is that the main role of interfacial impurities is to change the spacing between the Mo and MoSi<sub>2</sub>. As the size of the impurity becomes larger, the interfacial spacing increases and the cohesive energy drops. The changes

in the electronic structure or bonding at the interface associated with adding the impurities, while they can be large, do not dominate the fracture energetics.

Impurity and magnetic effects can be responsible for the weakening of the interface in the NiAl/Cr. It is known that Cr is anti-ferromagnetic in the bulk and ferromagnetic on the surface. The inclusion of magnetic effects reduces the surface energy, thus lowering  $W_{ad}$  for the interface relative to the constituents. It is also known that impurities can have a significant effect on interfacial bonding. Calculations including magnetic and impurity effects have been performed. Our calculations show that magnetism does indeed show a 12% effect and that impurity effects are similar to those obtained for MoSi<sub>2</sub>.

The arrangement of atoms at the interface has a large effect on the bonding energy  $W_{ad}$ . The lower energy configuration of the NiAl/Cr interface is for Ni atoms to be in contact with Cr atoms while the free NiAl surface tends to be Al-terminated. This implies that there may be an effect on  $W_{ad}$  due to segregation processes, since Al atoms must segregate to the free NiAl surface. Estimates suggest a 20% reduction of  $W_{ad}$ . For Al<sub>2</sub>O<sub>3</sub>/Cu, the surface Al ions were found to relax by nearly 86%. By including this relaxation, good agreement with the measured  $W_{ad}$  is found, while disagreement by nearly a factor of three is obtained without it.

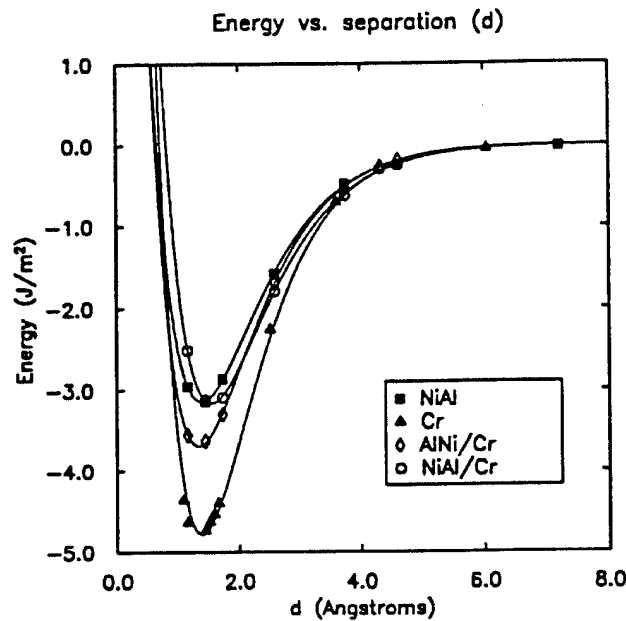


Fig. 1. Calculated adhesion curves for NiAl, Cr, and NiAl-Cr.

## Modeling: Microstructural

### MICROSTRUCTURE EFFECTS IN COMPOSITES

N. Sridhar, W. H. Yang and D.J. Srolovitz

Real engineering materials tend to have complex microstructures consisting a distribution of phases and defects. These second phase regions can be in the form of fibers, laminates or, more typically, as dispersed particles. These phases usually have different elastic, plastic, thermal expansion, and phase transformation properties than the matrix material. While the interaction of cracks with individual particles is routinely studied by continuum mechanics approaches, the behavior of cracks in these complex microstructures is not well understood. In the first part of our work, we focussed on the interaction of cracks with individual microstructural features and with complex microstructures. In the second part of this work, we examined the stability of directionally-solidified eutectic microstructures exposed to mechanical and thermal loading.

The method that we have developed for studying cracks in complex microstructure is based upon the boundary integral equation method, which incorporates Eshelby's procedure to handle inclusions within the matrix. The main feature of this method is that it can accurately account for the presence of several hundreds of particles more accurately and efficiently than other existing methods. This model was implemented for the two dimensional, generalized plane strain problem. We have applied this model to study the enhancement in the stress intensity factors at crack tips due to interactions with particles with different modulus and misfit relative to the matrix as a function of particle volume fraction. Changes in the local stress intensity factor at crack tips due to misfitting particles is crucial due to the thermal shock susceptibility of brittle intermetallics. High modulus particles were found to increase the maximum stress intensity factor at crack tips by as much as 35% at volume fractions as low as 20%. Additional work was performed using a related, discrete model. This model showed the effects of grain boundary embrittlement on toughness in polycrystals and the effects of interfacial toughness, misfit and modulus on lamellar microstructures.

An important consideration which determines the ultimate utility of these directionally solidified composites at high temperatures is the stability of these microstructures under operating conditions. In this work, we theoretically examined the effect of internal and applied stresses on the morphological stability of eutectic composites in order to identify combinations of material properties and operating conditions required to maintain microstructural stability and thereby provide the basic guidelines for alloy design and the expected lifetimes of the composites.

The morphological stability of many microstructures can be modified by the presence of internal or applied stresses. We first employed a linear stability analysis to examine the effect of stresses on the interface diffusion controlled morphological stability of lamellar eutectic composites. These stresses can be either due to misfit strains and/or due to externally applied loads. We examined the stability of the flat two-phase interfaces by slightly perturbing the profiles of the interfaces. The interface evolution is dictated by the spatial gradient of the interfacial chemical potential. This chemical potential along the perturbed interface took into account both curvature and elastic effects. Explicit evaluation of the elastic terms was performed by analytically solving the elasticity problem associated with the perturbed interface. For misfitting plates, we found that for dilute volume fractions of the second phase,

the nominally flat plate-matrix interface is unstable with respect to the growth of perturbations with wavelengths greater than a critical wavelength, provided that the plates are elastically stiffer than the surrounding matrix. For finite volume fractions, we found that the lamellar composite with a compliant minority phase is always stable. If the minority phase is stiff, the stability of the system is dependent on its volume fraction. Figure 1 illustrates the results in the form of a stability diagram. In contrast, for stresses generated by externally applied loads, we found that the flat plate-matrix interface is always unstable as long as the plate modulus and the matrix modulus are different. Consequently, the lamellar eutectic microstructure is destabilized by applied loads and the instability growth rate increases as the magnitude of the applied loads increases. In addition, our analysis revealed that misfit strains can either counteract or enhance the destabilizing influence of applied loads depending on the elastic properties of the plate and the matrix. We developed several forms of stability diagrams that identify material properties and operating conditions required to maintain a stable interface in lamellar eutectic microstructures.

We also employed linear stability analysis to examine the effect of stresses on interface diffusion-controlled morphological stability of fibrous eutectic composites. As opposed to lamellar eutectics, the rod morphology exhibits an interface-energy induced Rayleigh instability, even in the absence of elastic stresses. However, on including the elastic effects, we found that for a single misfitting rod in an isotropic matrix, the nominally cylindrical rod-matrix interface is unstable and has a maximally unstable wavelength which is smaller (greater) than that predicted in the absence of stresses, provided that the rods are elastically stiffer (softer) than the surrounding matrix. In addition, for elastically stiff rods, the growth rate corresponding to the maximally unstable wavelength can be much larger than the growth rate in the absence of elastic effects, and this implies that stresses can accelerate the Rayleigh instability. For stresses generated by externally applied loads, the instability wavelength is always smaller and the growth rates larger than the corresponding Rayleigh instability values as long as the rod modulus and the matrix modulus are different. In addition, our analysis revealed that misfit strains can either counteract or enhance the destabilizing influence of applied loads depending on the elastic properties of the rod and the matrix. We also observed that for dilute rod volume fractions, the Rayleigh instability can be suppressed in the misfit case provided the characteristic surface to strain energy ratio is below a critical value. We have developed stability diagrams, similar to that given in Fig.1, that identify conditions required to maintain a stable interface in these fibrous eutectic microstructures.

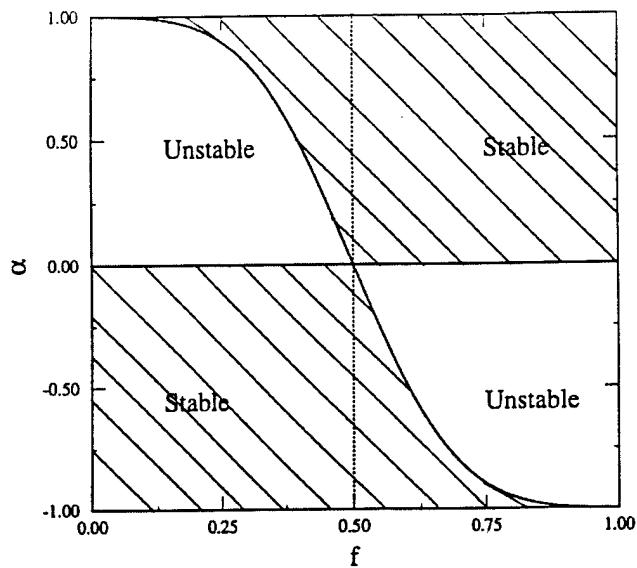


Fig. 1. Stability diagram indicating the stability of the flat interfaces in a lamellar microstructure consisting of a periodic array of misfitting plates as a function of the phase-A volume fraction ( $f$ ) and the elastic mismatch parameter ( $\alpha$ ). The mismatch parameter  $\alpha = (E_A - E_B) / (E_A + E_B)$ , where  $E_A$  and  $E_B$  are the moduli of the two phases, respectively. The shaded regions in the diagram indicate regions where the flat interfaces are stable.

## Modeling: Microstructural

### ANALYSIS AND DESIGN OF INTERMETALLIC MATRIX COMPOSITES BY THE HOMOGENIZATION METHOD, WITH APPLICATIONS OF DIGITAL IMAGE MODELING TECHNIQUES

K. Terada, D. Golanski, and N. Kikuchi

In order to design materials from a mechanics-based approach, it is generally necessary to analyze in detail the composite microstructure and to establish a relationship between the micromechanics of the composite microstructure and the macroscopic mechanical behavior of the composite. Most past work has concentrated on one approach or the other without effective coupling between the two extreme viewpoints. Most mechanics methods also use or construct an explicit, often phenomenological macroscopic constitutive equation, and apply it to non-homogeneous materials for all types of loading cases. However, many problems in composite mechanics involve nonlinear failure processes, e.g., accumulation/localization of micro-cracking and matrix/fiber debonding, and require effective coupling of micro- and macro-mechanics. In this program, we have applied the homogenization method (HM) to obtain such coupling. The HM is based on the rate formulation of nonlinear mechanics that assumes instantaneous linearity of the tangent modulus of the constitutive relation. The HM constructs an instantaneous sequentially linearized constitutive relation at every point in a material at each loading stage by solving through homogenization analysis the unit cell problem based on the properties and geometry of the material constituents. The macroscopic constitutive relation is formed implicitly at each point in the material with additional inputs into the unit cell problem such as interface characteristics, plastic properties of the constituents, crack initiation behavior in the microstructure, and localization and propagation of deformation fields. To incorporate geometrically complex, non-periodic domains that are characteristic of real microstructures of composite materials, a digital image-based technique has been developed for developing finite element models for any unit cell problem in which the non-linear HM is applied. This technique allows effective examination of the response of any three-dimensional structure or microstructure.

The initial progress in this project during the first two years of this program involved fundamental development of the nonlinear HM. The subsequent research included analysis of thermo-mechanical phenomena of importance to the materials under investigation (e.g., MoSi<sub>2</sub>-SiC alloys, NiAl-Cr and NiAl-Mo alloys), implementation of new computational schemes and introduction of the concept of digital image-based methods for analysis of microstructures. The formulation of the HM was also extended to include creep and other time-dependent viscous properties of materials, leading to a general methodology of the HM for application to a wide range of thermo-mechanical behaviors. The thesis of Terada describes the general methodology. We have applied the HM directly to problems involving IMCs under experimental investigation in this program: (1) thermal mismatch degradation of brittle particulate composites; (2) deformation damage of in-situ aligned eutectic fiber composites.

A major result involves application of the HM to brittle IMCs with particulate second phases. The MoSi<sub>2</sub>-SiC system has been examined with the goal of optimally designing the three-dimensional microstructure of particulate SiC in polycrystalline MoSi<sub>2</sub> matrices for thermal fatigue resistance. The experimental aspects of the project are given elsewhere in this report. The specific objective was to optimize the particle volume fraction for minimum thermal mismatch stresses while maximizing composite stiffness. Non-random distributions of SiC particles, a practical reality in real microstructures, were allowed by use of the digital image-based techniques described above. The optimization algorithm determined that 20 vol % SiC should be the optimum particulate content, in good agreement with the results obtained

experimentally in this program. A typical result from our computations is portrayed in Fig. 1. This method of optimization should be applicable to any other composite system.

The most recent applications of HM to materials under experimental examination in this program has concerned in-situ directionally-solidified NiAl-Cr and NiAl-Mo quasi-binary eutectics. These microstructures are ideal for investigation by HM because of the irregularities in the fiber distributions within individual cells and in the distribution of cells across cell boundaries involving much larger length scales. We have successfully analyzed the elastic modulus and thermal expansion distributions in these composites and applied them to conditions of thermal fatigue examined experimentally. A typical result for a NiAl-Cr composite is given in Fig. 2. As a result of these analyses, we have been able to understand qualitatively why highly aligned composites (with few cell boundaries) have poor thermal fatigue resistance compared to more cellular microstructures.

In summary, we have established the necessary computational tools for analysis and design of composite microstructures based on the nonlinear HM coupled with the digital image-based modeling technique. In future years, we will be able to develop applications to any specific composite systems under experimental study. Additional computations for Nb-Nb<sub>5</sub>Si<sub>3</sub>, TiAl-TiAl<sub>3</sub>Al and other MoSi<sub>2</sub>-based composites investigated as part of this program are being considered.

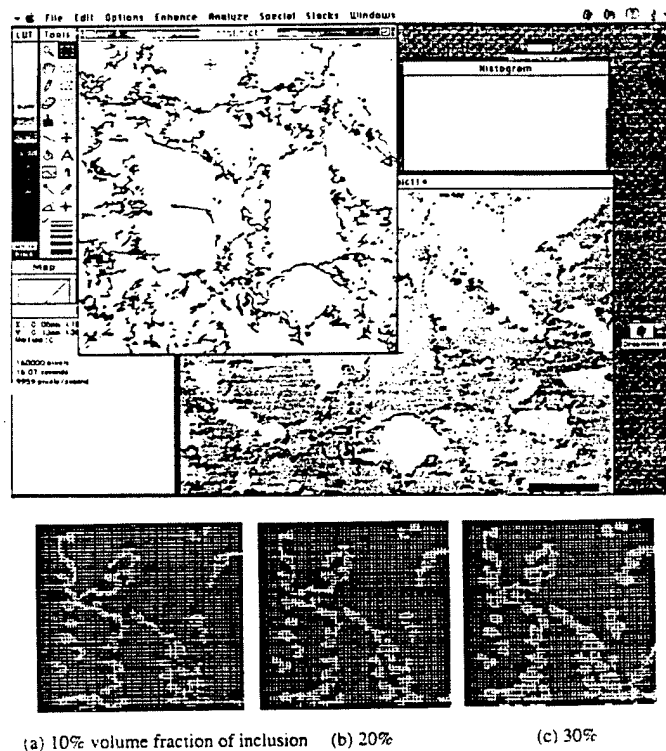


Fig. 1. Application of the Homogenization Method to thermal fatigue of MoSi<sub>2</sub>-SiC particulate composites, showing typical microstructure and digitized images on which analysis is done.

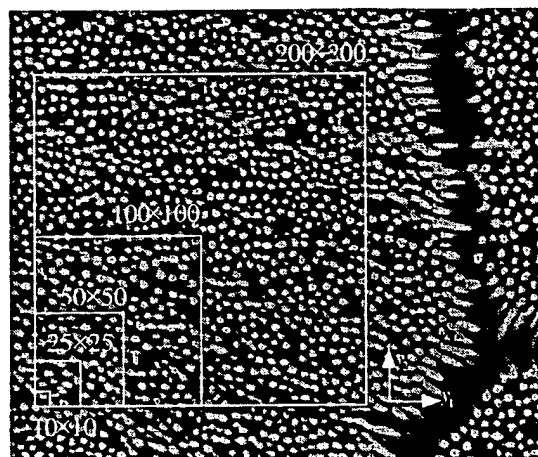


Fig. 2. Application of the Homogenization Method to the microstructure of a NiAl-Cr directionally-solidified eutectic alloy. The accompanying table below gives elastic properties and thermal expansion data derived from the HM analysis.

**Table 5.1 : Homogenized material properties of NiAl-Cr composite for different selected RVE region**

**	10*10	25*25	50*50	100*100	200*200	idealized	analytical***
E1*	197.85	197.58	197.54	197.48	197.42	197.43	195.91
E2	197.44	197.39	197.37	197.26	197.26	197.43	195.91
E3	200.51	200.46	200.40	200.39	200.38	200.41	200.40
G23	76.07	75.97	75.99	75.95	75.95	75.90	75.35
G31	75.85	76.07	76.02	76.06	76.06	75.90	75.35
G12	75.38	75.65	75.65	75.66	75.67	75.42	75.35
a11	1.3937E-5	1.3946E-5	1.3952E-5	1.3957E-5	1.3968E-5	1.4007E-5	1.4794E-5
a22	1.4037E-5	1.3980E-5	1.3983E-5	1.3988E-5	1.3993E-5	1.4007E-5	1.4794E-5
a33	1.3381E-5	1.3475E-5	1.3481E-5	1.3482E-5	1.3483E-5	1.3480E-5	1.1920E-5

\* GPa; for elasticity constants, /°C; for CTE.

\*\* The number of voxels in xy-plane.

\*\*\* Analytical Results implies: the results obtained by Halpin-Tsai equation (emperical parameter=0.3) for engineering elasticity constants and the relation of Schapery for CTE (Halpin, 1992).

## Mechanical Behavior: MoSi<sub>2</sub>-base Materials

### PLASTICITY ENHANCEMENT MECHANISMS IN MOSI<sub>2</sub>-BASE MATERIALS

H. Chang, C.M. Czarnik, J.P. Campbell, and R. Gibala

A major difficulty in the application of MoSi<sub>2</sub> as a structural material has been a lack of adequate ductility and fracture toughness at relatively low temperatures. "Low" temperatures for MoSi<sub>2</sub> are as high as 900°C-1400°C, where the final fracture is brittle with limited fracture toughness. At the lower end of this temperature range, MoSi<sub>2</sub> exhibits very limited plasticity, except in compression of high purity single crystals of selected orientations and under hardness indentations. Only toward the higher of these temperatures, with the onset of dislocation climb and diffusional creep processes, does MoSi<sub>2</sub> exhibit significant plasticity in compression, bending, and tension in both single crystals and polycrystalline materials. Within this temperature range, MoSi<sub>2</sub> undergoes a brittle-to-ductile fracture transition that appears to be dislocation-mobility or dislocation-density limited.

Many approaches have been taken to reduce the brittle-to-ductile transition temperature (BDTT) of MoSi<sub>2</sub> or to enhance the capability for plastic flow and obtain increased toughness at relatively low temperatures. Approaches include but are not limited to solid solution alloying, second-phase microstructural control, ductile phase toughening, and thermomechanical manipulation of grain size and texture. We examined three methods of plasticity enhancement that have proven effective previously in body-centered cubic refractory metals such as Nb, Ta, Mo and W, and B2 ordered intermetallic alloys such as NiAl and FeAl: high temperature prestrain, surface film coatings and second-phase particles. Such results have demonstrated that the ductility of bcc metals and B2 ordered alloys can be greatly enhanced by these methods because dislocation-mobility or dislocation-density limitations can be overcome. The dislocation-mobility limitation follows, for example, from the large difference in intrinsic mobility between edge and screw dislocations in the bcc structure and in B2 ordered alloys which deform by the motion of <111> dislocations. The dislocation-density limitation arises from a lack of sufficient numbers of surface or internal dislocation sources.

For MoSi<sub>2</sub>, insufficient knowledge exists concerning the relative mobilities of edge and screw dislocations, and information on the different dislocation types <100>, <110>, 1/2<111> and 1/2<331>, their glide planes, and the operative slip systems as a function of temperature, strain rate and crystallographic orientation is only partially developed. Because MoSi<sub>2</sub> exhibits qualitatively similar dependences of the yield strength on temperature, strain rate and crystallographic orientation as bcc metals and B2 ordered alloys, it is important to examine MoSi<sub>2</sub> for possible plasticity enhancement by processes that are operative in these materials. The purpose of this research has been to determine the extent to which the plastic deformation of MoSi<sub>2</sub> at and below the BDTT is bcc/B2-like in its sensitivity to high temperature prestrain, surface films, and second phases.

*Prestrain effects:* The effect of prestraining polycrystalline MoSi<sub>2</sub> at relatively high temperatures (e.g. 1300°C) on deformation at lower temperatures is illustrated in Fig. 1. In this particular experiment, the prestrained material can be deformed in compression to nearly twice the extent as the corresponding unprestrained material. We find that plastic deformation in compression can be achieved at temperatures as low as 700°C in

polycrystalline MoSi<sub>2</sub>. The results also suggest that materials which are limited in macroscopic plasticity because of the Von Mises criterion for operative slip systems can achieve approximately 1% plasticity prior to fracture.

*Surface film effects:* The effects of ZrO<sub>2</sub>-deposited films on the hardness of polycrystalline MoSi<sub>2</sub> are illustrated in Fig. 2. At all temperatures in the range 25°C to 1300°C, the Vickers hardness is reduced by application 100-500 nm thick films deposited by PVD techniques. It should be noted that the indentation depth is approximately 20-100 times the thickness of the films and that these experiments have been reproduced for several different polycrystalline and single crystalline materials. The surface film softening is also manifested in compression testing of single crystals.

*Effects of second phases:* We have found that 10-30 volume % TiC particulate additions to polycrystalline MoSi<sub>2</sub> not only increase the strength of the monolithic material, but also allow additional plasticity at stress levels not achieved in unalloyed MoSi<sub>2</sub>. A typical result is given in Fig. 3. The results may be understood in terms of enhanced dislocation activity in the MoSi<sub>2</sub> matrix afforded by dislocation nucleation at the matrix-particle interface. A critical element of the enhanced plasticity is associated with the ability of the TiC particles to deform prior to the MoSi<sub>2</sub> matrix. A typical micrograph which illustrates this is given in Fig. 4.

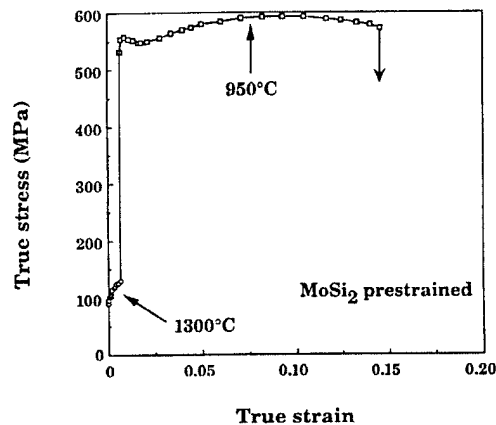


Fig. 1. Illustration of a typical prestrain experiment. MoSi<sub>2</sub> prestrained in compression to 1.0% true strain at 1300°C without strain-induced cracking can be subsequently deformed nearly 15% at 950°C.

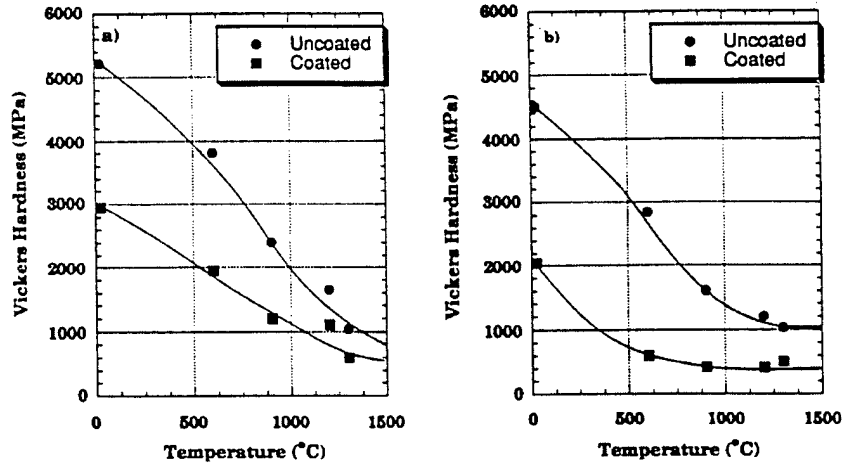


Fig. 2. Temperature dependence of the Vickers hardness of uncoated and ZrO<sub>2</sub>-coated polycrystalline MoSi<sub>2</sub>. (a) Commercially pure hot-pressed MoSi<sub>2</sub>. (b) High-purity mechanically alloyed MoSi<sub>2</sub>. Film thickness is 100 nm.

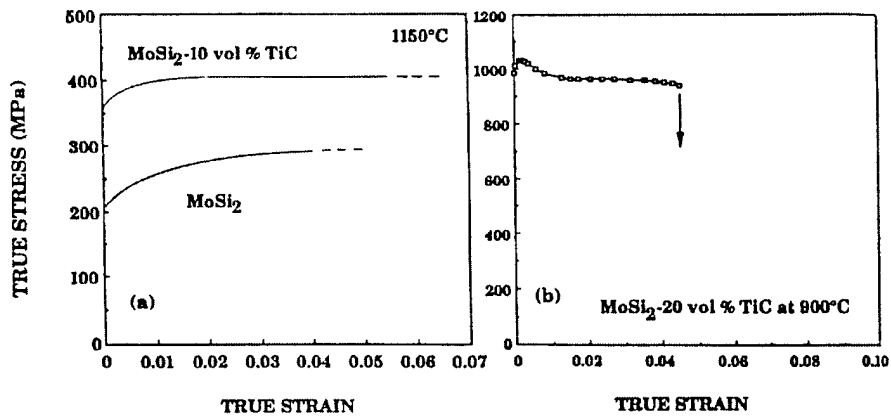


Fig. 3. Mechanical behavior of MoSi<sub>2</sub> and MoSi<sub>2</sub>-TiC particulate composites. (a) Comparison of stress-strain curves at 1150°C. (b) Deformation of a composite at 900°C.

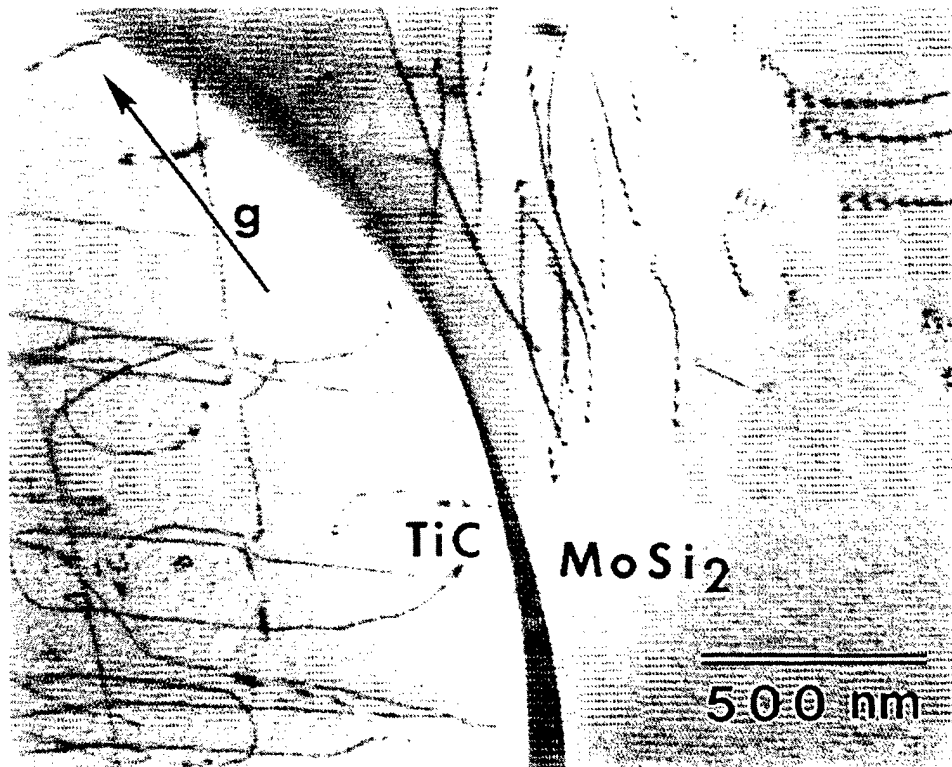


Fig. 4 TEM micrograph of dislocation substructures in a TiC particle and the adjacent MoSi<sub>2</sub> matrix in a MoSi<sub>2</sub>-10 vol % TiC composite deformed 0.5% at 1150°C.  $B=110$ ,  $g = 002$ .

## Mechanical Behavior: MoSi<sub>2</sub>-based Materials (also: Fatigue Related)

### FRACTURE AND FATIGUE-CRACK GROWTH RESISTANCE IN MOSI<sub>2</sub> COMPOSITES

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Molybdenum disilicide has been viewed as a candidate material for high-temperature structural use due to its high melting point (2020°C) and excellent oxidation resistance; in addition, it displays a brittle-to-ductile transition and better ductility than competing ceramics above 1200°C. However, as the toughness is very low (~ 2-4 MPa√m), improvements are essential if it is to be seriously considered for actual use. One approach is through the addition of a ductile phase, although previous attempts have often had limited success. Accordingly, the objective of this project was to develop a flaw-tolerant MoSi<sub>2</sub>-based material with significantly improved toughness, which would additionally show resistance to crack growth under cyclic loading.

To impede crack growth, the approach was to design the reinforcement phase/interface to develop intact ductile ligaments across the crack surfaces. Moreover, since the potency of such bridging is degraded under cyclic loads, the ligaments had to be resilient to fatigue. To meet these conditions, a ductile reinforcement with high aspect ratio and low interfacial strength was desired. Based on their earlier studies, Pickard and Ghosh developed a Nb-wire mesh reinforced MoSi<sub>2</sub> composite (Nb<sub>m</sub>/MoSi<sub>2</sub>), with a structure of ~20 vol % of nominally randomly distributed, 150-200 μm diameter Nb wires (aspect ratio ~7-13), in the form of a "criss-crossed" mesh. The behavior was compared with MoSi<sub>2</sub> reinforced with ~20 vol % of spherical Nb particulate (Nb<sub>p</sub>/MoSi<sub>2</sub>), with aspect ratio of order unity. The brittle reaction interface in both alloys had a composition consistent with Nb<sub>3</sub>Si<sub>3</sub> stoichiometry and provided a preferential path for cracking.

The addition of the Nb-wire mesh significantly toughened MoSi<sub>2</sub>. The R-curve behavior for this composite is shown in Fig. 1. Compared to unreinforced MoSi<sub>2</sub>, which failed catastrophically at  $K_c \sim 4$  MPa√m, the Nb<sub>m</sub>/MoSi<sub>2</sub> composite showed a steady-state toughness of 13 MPa√m. This is one of the highest toughnesses reported for a MoSi<sub>2</sub>-based material. Such a three-fold increase in toughness is to be compared with that of spherical Nb<sub>p</sub>-reinforced MoSi<sub>2</sub>, where unstable fracture occurred at  $K_c$  of ~5.2 MPa√m. In both composites, the behavior was associated with extensive debonding within the reaction layer and crack advance along the Nb/reaction-layer interface. With spherical reinforcements, this led to the crack simply circumventing the particles with only marginal toughening, whereas with the wire mesh reinforcements, significant crack bridging by the unbroken ductile Nb wires occurred over ~1.5 mm behind the crack tip. Such results were found to be consistent with existing models for ductile-phase toughening and small-scale particulate bridging.

Under cyclic loading, resistance to crack propagation was also far superior in the Nb wire-reinforced composite. The fatigue-crack growth rates as a function of the stress-intensity range are given in Fig. 2. Crack paths in fatigue again involved extensive crack deflection along the reaction-layer interface, although in contrast to R-curve behavior, little evidence of crack bridging by intact Nb-wire ligaments occurred. In fact, all the Nb-regions that intersected the crack path were observed to be broken, akin to behavior in many ductile-phase toughened intermetallics in fatigue. Some typical illustrative micrographs are given in

Fig. 3. However, with the present  $Nb_m/MoSi_2$  composite, despite this diminished role of bridging, the fatigue properties were still far superior to pure  $MoSi_2$  and the  $Nb_p/MoSi_2$  material, because other mechanisms of crack-tip shielding were developed. Specifically, the superior cyclic crack-growth resistance of the  $Nb_m/MoSi_2$  composite resulted from high levels of roughness-induced crack closure that were developed from the highly deflected crack paths as the crack followed the reaction-layer interfaces.

In conclusion, the addition of a high-aspect ratio ductile phase in the form of Nb wire mesh to molybdenum disilicide resulted in significant toughening (with stable crack growth up to  $\sim 13 MPa\sqrt{m}$ ) and vastly improved fatigue-crack propagation resistance, compared to the unreinforced matrix or Nb-particulate-reinforced  $MoSi_2$ . Such improved properties resulted from preferential cracking along a reaction-layer interface, which promoted ductile ligament bridging under monotonic loading and roughness-induced crack closure under cyclic loading.

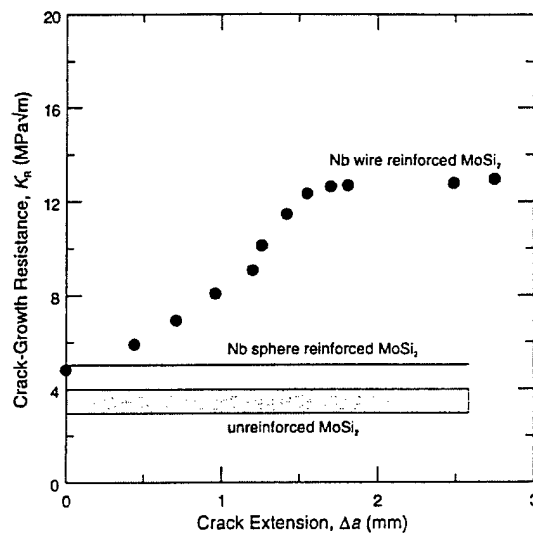
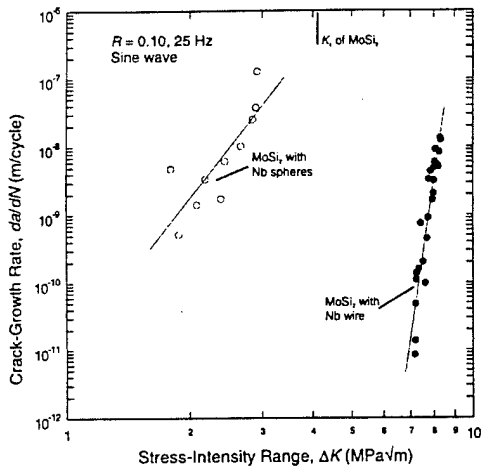
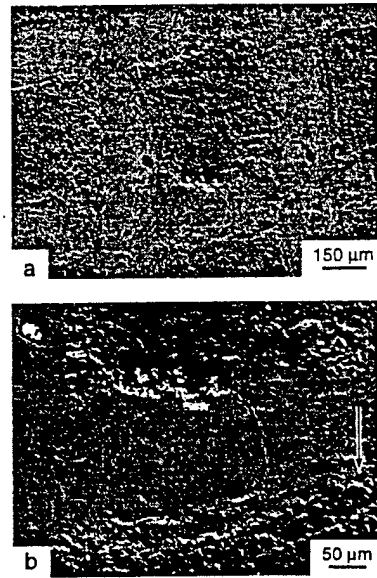


Fig. 1. Resistance curves for Nb-wire reinforced  $MoSi_2$ , compared to that for Nb-particulate  $MoSi_2$  and the unreinforced matrix material. Note that the R-curve for the  $Nb_m/MoSi_2$  composite shows a plateau after  $\Delta a \sim 1.5$  mm, which is on the order of the length of the bridging zone.



**Fig. 2.** Fatigue-crack growth rates as a function of the applied  $\Delta K$  for  $\text{MoSi}_2$  reinforced with 20 vol % Nb as wire mesh ( $\text{Nb}_m/\text{MoSi}_2$ ) and spherical particulates ( $\text{Nb}_p/\text{MoSi}_2$ ).



**Fig. 3.** Scanning electron micrographs of the crack/Nb wire interactions under cyclic fatigue loading in the  $\text{Nb}_m/\text{MoSi}_2$  composite showing (a) cracking along the weak reaction-layer interface, and (b) failure of the Nb phase (which limits any ductile-phase bridging.)

## Mechanical Behavior: NiAl-base Materials

### PLASTICITY AND TOUGHENING MECHANISMS OF DIRECTIONALLY SOLIDIFIED B2 NiAl-BASED EUTECTIC ALLOYS

A. Misra, Z.L. Wu and R. Gibala

We have investigated several directionally-solidified (DS) NiAl-based eutectic composites to gain insight on micromechanisms of plasticity and toughness in multiphase intermetallics, as well as to explore their viability as engineering structural materials. Two broad classes of materials have been examined: (1) composites for which the NiAl matrix and the reinforcing ductile second phase can be deformed compatibly due to easy slip transfer across interfaces, leading to high room-temperature tensile ductility and intrinsic fracture toughness; (2) composites for which interfaces are strong barriers to slip transfer and the metallic second phase has high strength and limited ductility, leading to incompatible deformation of the two phases and resulting in negligible tensile ductility and more limited, mainly extrinsic toughness enhancement. The former class of materials is typified by B2 Ni-Fe-Al alloys with a B2 Ni-Fe-Al matrix ( $\beta$ ) reinforced with a ductile fcc-based ( $\gamma$ ) Ni-Fe-Al second phase. For our work, an alloy of Ni-30 at.% Fe-20 at.% Al has served as a model material. The latter class of materials is typified by B2 NiAl-base  $\beta$  matrices with a bcc refractory metal second phase. NiAl-Cr and NiAl-Mo alloys with the respective compositions of Ni-33 at.% Al-34 at.% Cr and Ni-45.5 at.% Al-9.0 at.% Mo have served as model materials. The typical microstructure of the NiAl-9Mo alloy is shown in Fig. 1.

The major results can be summarized by two schematic figures. The geometry of the slip systems observed in the two classes of materials is illustrated schematically in Fig. 2. The nature of the operative toughening mechanisms is similarly illustrated schematically in Fig. 3. Tensile tests were performed on round specimens of 3 mm diameter and 10 mm gage length. Fracture toughness data were obtained from notched three-point bend tests on specimens 3 mm  $\times$  4 mm  $\times$  20 mm in size with a 1.5 mm  $\times$  150  $\mu$ m notch.

The highest tensile ductility (10-12%) and toughness (30-35 MPa $\sqrt{m}$ ) that we have observed in any NiAl-base material were observed in the Ni-30Fe-20Al alloys containing a ductile fcc-based second phase. Easy transfer of slip from the softer  $\gamma$  phase to the harder  $\beta$  phase, facilitated by the Kurdjumov-Sachs (KS) orientation relationship, allows compatible deformation of both phases and is manifested in appreciable plasticity and toughness, the latter dominated by intrinsic plasticity-based toughening mechanisms. Extrinsic mechanisms, such as crack blunting and crack bridging, also contribute to the toughness, but have lesser roles. These plasticities and toughnesses probably represent upper limits achievable in NiAl-base alloy materials.

In the NiAl-Cr and NiAl-Mo eutectic alloys, significant dislocation activity was observed in the crack tip plastic zone. However, the higher yield strength of the bcc metal phase as compared to the NiAl phase and the cube-on-cube orientation relationship between the  $\beta$  and bcc phase did not favor slip transfer geometrically. Incompatible deformation of the two phases due to difficulty of slip transfer across  $\beta$ -bcc interfaces thus leads to early crack initiation. As a result, little (<0.5%) or no tensile ductility is observed in these materials, and the fracture toughness of the  $\beta$ -bcc eutectics is controlled primarily, but not exclusively, by extrinsic toughening mechanisms, e.g. crack trapping, deflection and bridging, as illustrated

in Fig. 3. The fracture toughness of NiAl-Mo eutectics is approximately 15 MPa $\sqrt{m}$ ; that of NiAl-Cr eutectics is in the range 7-20 MPa $\sqrt{m}$ , with the higher values corresponding to materials with very low residual interstitial contents. Such impurity effects were not observed for the Mo-containing materials.

Although the  $\beta$ -bcc alloys have lower plasticities and toughnesses than the  $\beta$ - $\gamma$  alloys, they possess a better combination of room temperature toughness and high temperature creep strength. In an attempt to improve the toughness of the NiAl-Mo and NiAl-Cr eutectics, we investigated the role of Re alloying to possibly toughen the bcc metal phase. Re is known to solid-solution soften and lower the ductile-to-brittle transition temperature of various bcc refractory metals; it also preferentially partitions to the bcc phase in these NiAl-base eutectics. Re alloying was found to be effective in reducing the hardness of the refractory metal fibers or lamellae, but also reduced the fiber/lamellae alignment relative to the solidification direction. As a result, the Re-containing alloys had lower longitudinal-orientation toughness than the corresponding unalloyed eutectic. Only by use of very slow, impractical solidification growth rates could better longitudinal alignment be obtained. However, the combination of high thermal gradient and low growth rate needed to fully align the fibers in Re-containing alloys could not be achieved with available facilities. In additional alloying investigations, Hf and Si were also examined as possible alloying elements. Appreciable strengthening was observed, but at a significant cost in loss of toughness which partition preferentially to the NiAl-base matrix phase.

An additional outcome of investigating both the  $\beta$ - $\gamma$  and  $\beta$ -bcc alloys was the ability to determine crystallographic factors which control the occurrence of global plasticity versus initiation of cleavage cracks. Using simple models by Stroh and by Smith and Barnby, we were able to use calculations of dislocation pile-up stresses to predict whether slip is activated prior to cleavage or vice-versa for a given set of respective yield strengths and crystallographic orientation relationships between the two phases. The results have been summarized efficiently in terms of slip versus cleavage maps using stress and/or orientation factors as the pertinent variables.

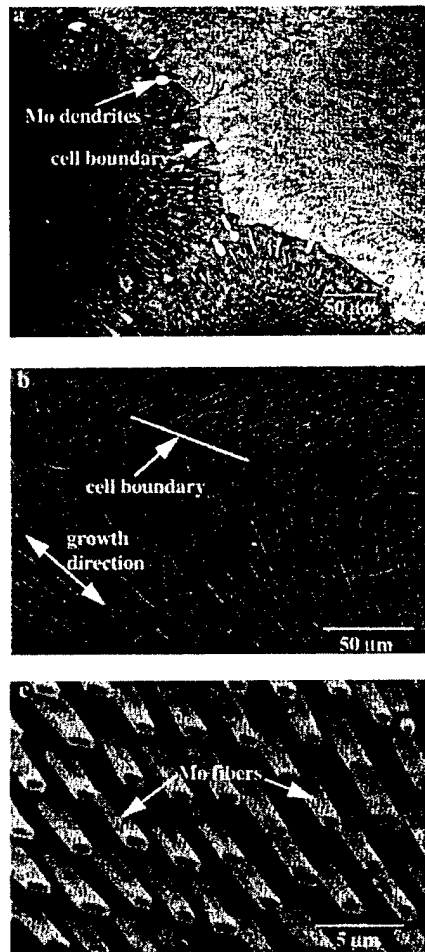


Fig. 1. The microstructure of the NiAl-9Mo alloys DS at  $\sim 10 \text{ mm h}^{-1}$ : (a) optical micrograph showing the transverse section; (b) and (c) SEM micrographs showing the longitudinal sections with the matrix selectively etched. Notice the cellular eutectic microstructure with some Mo dendrites at cell boundaries in (a), the curling of the fibers towards the cell boundary in (b), and the faceted fibers in (c).

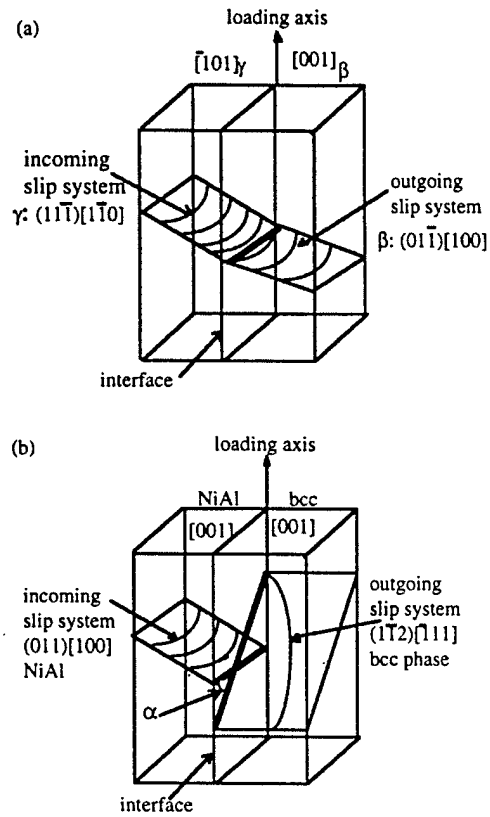
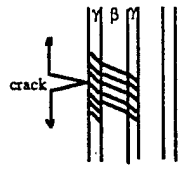
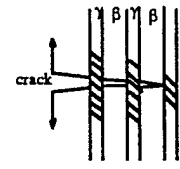
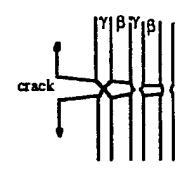
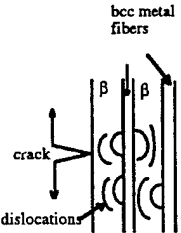
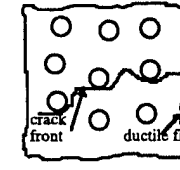
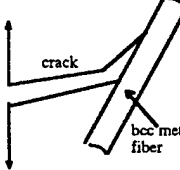
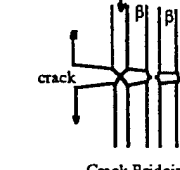
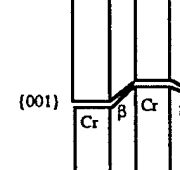


Fig. 2. Schematic illustration of the geometry of slip systems in the two types of alloys studied: (a) type I alloys in which slip planes are nearly parallel and have a common line of intersection at the interface due to the KS orientation relationship  $((110)\beta // (111)\gamma, [1\bar{1}1]\beta // [0\bar{1}1]\gamma)$ ; (b) type II alloys, which exhibited a cube-on-cube orientation relationship in which slip planes are not parallel and the angle  $\alpha$  between the lines of intersection of slip planes with the interface is large. Geometry favors slip transfer in (a) but not in (b).

Alloy	Toughness (MPa√m)	Intrinsic Mechanisms	Extrinsic Mechanisms
Ni-30Fe -20Al	30	 <p>Slip Transfer</p>	 <p>Crack Renucleation, and Crack Blunting by Slip in <math>\gamma</math></p>  <p>Crack Bridging</p>
9Mo 34Cr*	15 20	 <p>Plasticity in NiAl</p>	 <p>Crack Trapping</p>  <p>Crack Deflection Along Interfaces</p>  <p>Crack Bridging</p>  <p>Mismatch of Cleavage Planes **</p>

\* These toughening mechanisms were observed only in the high purity CELZ processed NiAl-34Cr material; In the low purity NiAl-34Cr alloy, catastrophic failure by cleavage in both phases and extensive fiber pull-out was observed ,

\*\* This mechanism was observed only in NiAl-34Cr alloy. In the NiAl-9Mo alloys, the Mo fibers failed by necking.

Fig. 3. Summary of toughening mechanisms observed in NiAl-based eutectics.

## Mechanical Behavior: NiAl-based Materials (also: Fatigue Related)

### FATIGUE-CRACK GROWTH OF SMALL CRACKS IN DS-NIAL WITH MO ADDITIONS

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NiAl has been contemplated for structural use in aero-engines for many years because of its low density and high oxidation resistance. However, its use has been compromised limited by low ductility/toughness. One approach to improving the toughness is through the use of *in situ* composite reinforcement, specifically by directional solidification of two-phase NiAl eutectic microstructures, by ternary alloying additions of refractory metals. Aligned-eutectic composites of NiAl-Cr, NiAl-V and NiAl-Mo have been reported to be much tougher than NiAl. Such ductile-phase toughening has been attributed to crack bridging and subsequent plastic deformation of the eutectic phase. However, since such mechanisms can degrade under cyclic loading, the aim of this work was specifically to examine the fatigue properties of the toughened alloys. Since for engine applications, it is the near-threshold properties of small (<500  $\mu\text{m}$ ) flaws that are lifetime limiting, studies were primarily focused on small-crack behavior in a directionally solidified NiAl-9 at.% Mo alloy to determine the resiliency of the toughening mechanisms under cyclic loads.

The DS NiAl-9 at.% Mo *in situ* composite was arc-melted and directional solidified by Kush, Misra and Gibala to give a cellular eutectic structure consisting of a NiAl phase (~100-200  $\mu\text{m}$  diameter "islands") within the NiAl-Mo eutectic (~5-15  $\mu\text{m}$  long, 1-2  $\mu\text{m}$  diameter Mo rods, aligned nominally parallel to the solidification direction).

Fatigue-crack propagation rates for small (<400  $\mu\text{m}$ ) surface cracks were measured in cantilever-bend specimens cycled at 25 Hz at load ratios of  $R = -0.3, 0$  and  $0.35$ . Results in the form of crack-growth rates in the near-threshold regime ( $\sim 10^{-10}$ - $10^{-6}$  m/cycle) as a function of stress intensity range were obtained. The crack-growth resistance increased with increasing load ratio and showed a strong dependency on stress intensity. See Fig. 1. The crack growth rates are several orders of magnitude slower than those for monolithic NiAl single crystals. Scanning electron microscopy of the small-crack growth revealed differing crack paths depending upon the local orientation of the Mo rods with respect to the loading direction. In regions where the Mo rod axes were nominally aligned parallel to the crack front, the particles remained unbroken and the crack tended to circumvent them. Conversely, where the rod axes were perpendicular to the crack plane, the crack bisected the rods causing their failure. No evidence of crack bridging by intact Mo-particle ligaments was detected, in contrast to behavior under monotonic loading where extensive crack bridging by the eutectic phase was reported to be responsible for the significant R-curve toughening. Cyclic loads clearly result in the premature failure of the metallic particles, because cyclic plastic strain and fatigue damage are more readily accumulated in the ductile phase.

Regression fits to the crack-growth rate data confirmed the marked dependency on stress intensity. In terms of the power-law relationship ( $da/dN \propto \Delta K^m$ ), growth rates were an approximate function of  $\Delta K^7$ , where  $\Delta K$  is the stress-intensity range. However, this relationship masks the true dependency on the maximum stress intensity,  $K_{\text{max}}$ , as shown in Fig.1. By including the effects of both  $\Delta K$  and  $K_{\text{max}}$  in the power-law, the growth-rate relationship for NiAl-9Mo was found to be  $da/dN \propto (K_{\text{max}})^6 (\Delta K)^1$ , which confirmed the dominant role of  $K_{\text{max}}$ . It was concluded that the  $K_{\text{max}}$ -dependence resulted from crack advance primarily by static fracture of the brittle NiAl and NiAl-Mo eutectic phases, whereas the weak  $\Delta K$ -dependence resulted from intrinsic fatigue failure of the Mo phase and the consequent cyclic-loading-induced suppression of crack bridging behind the crack tip.

In summary, fatigue-crack growth in the DS NiAl-Mo alloy was found to be strongly dependent on  $K_{\text{max}}$  and weakly dependent on  $\Delta K$ , with an  $R$ -independent fatigue threshold  $\sim 37\%$  of  $K_{\text{Ic}}$ . Unlike reported behavior under monotonic loading where crack bridging by

unbroken Mo rods acts to promote R-curve toughness, no evidence of such crack bridging is detected under cyclic loading.

Similar experiments to the ones described for NiAl-Mo eutectic alloys were also performed for NiAl-Cr eutectics. These latter materials, while known to have better oxidation resistance, exhibited poor behavior, both in terms of crack-growth resistance and fatigue-crack growth.

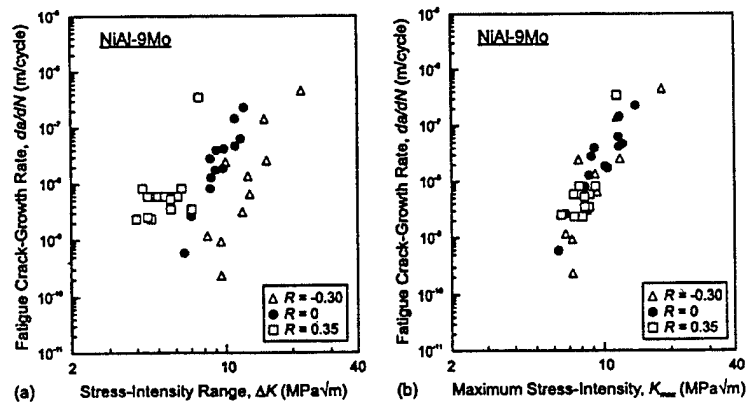


Fig. 1. Variation in ambient-temperature fatigue crack-growth rates,  $da/dN$ , with applied load ratio  $R$  for small surface cracks ( $< 400\mu\text{m}$ ) in directionally-solidified NiAl-9Mo *in situ* composite, with data plotted as a function of the applied (a) stress-intensity range,  $\Delta K$ , and (b) maximum stress intensity ( $K_{max}$ ).

## Mechanical Behavior: Laminated and Model Interfacial Materials

### INTERFACE SHEAR STRENGTH AND FRACTURE BEHAVIOR OF NiAl/METAL LAMINATES

M.R.Fox and A.K.Ghosh

Intermetallic matrix composites with ductile phase reinforcement are being considered for high-temperature structural applications. Increases in room temperature toughness over that of monolithic NiAl have been achieved in directionally-solidified NiAl/Mo and NiAl/Cr eutectic composites, as discussed elsewhere in this report. Since the nature of interface fracture is critical to understanding toughening effects in these composites, NiAl/Mo and NiAl/Cr model laminates were prepared to create interfaces for structural examination and mechanical testing. Three-layer laminate composites were fabricated in vacuum by forging and diffusion bonding at 1100°C. Forging at a strain rate of  $10^{-4} \text{ s}^{-1}$  resulted in a 40% thickness reduction. Load and temperature were then maintained on the samples during the 4 h long diffusion bonding step, producing a well-bonded interface.

Structural examination of the interfaces has revealed the presence of reaction layers, which vary in thickness with bonding conditions. The reaction layer was  $\text{Mo}_3\text{Al}$  in NiAl/Mo laminates and  $\text{NiCr}_2\text{O}_4$  in NiAl/Cr laminates. Fracture occurred between the reaction layer and Mo in NiAl/Mo and between the reaction layer and NiAl in NiAl/Cr. Fracture surfaces of NiAl/Mo reveal a replicated structure with some evidence of plastic deformation.

Three different sample geometries, shown in the enclosed Fig. 1, have been utilized to study interface mechanical response under various loading conditions. The double cantilever drilled compression (DCDC) specimen was used to produce primarily Mode I loading at the interface. This geometry has been used successfully by other investigators to produce stable Mode I cracks. To produce primarily Mode II loading, the offset notch shear/compression specimen was developed. To produce a combination of Mode I and Mode II loading, the asymmetrically loaded shear (ALS) specimen was used. In much of our most recent research, the ALS test was modified to a three-layer laminate in which Mo is the interior component and one of the outer NiAl components is compressed. The thickness of the Mo layer can be altered to produce a stress on the interface which is predominately shear with a minor component of superimposed compression. Under such conditions, upon crack initiation a stable crack is generated from either the top or bottom interface.

Finite element analysis (FEA) was used to determine the interface stresses present in ALS specimens prior to cracking. These stresses were used to develop fracture initiation diagrams for the NiAl/Mo and NiAl/Cr interfaces. After cracking, FEA was used to calculate the J-integral around the crack tip to determine the interface steady-state fracture resistance.

Some NiAl/Mo laminate interfaces were fractured in four-point bending. These specimens were two-layer laminates notched in the NiAl phase. The fracture toughness measured under these conditions is mixed mode, with nearly equal components of shear and tensile stress. FEA was used to model the experimental tests to determine interface fracture resistances in the same manner performed for other tests.

The maximum interface strength which can be measured using the offset notch geometry is limited by the strength and ductility of the NiAl matrix. The bond at the NiAl/metal interface is strong, and fracture did not often initiate at the interface in these tests. The stress state at the interface is mainly shear with some superimposed compression normal to the interface. As the relative notch spacing decreases, the amount of superimposed compression increases, resulting in a higher average shear stress required to initiate a crack. Due to these limitations of the shear/compression test, the ALS test was developed to determine shear strength and toughness of the interface more reliably.

The major results of this project have been obtained on both NiAl/Mo and NiAl/Cr model laminates and involving Mode II interface fracture by use of ALS specimen geometries. These results demonstrate that small amounts of plasticity play a critical role in the crack initiation process, just as intrinsic toughening mechanisms play an important role in directionally-solidified eutectics of these materials. Some typical results are shown in the enclosed micrographs in Fig. 2.

In-situ observation of crack growth by optical microscopy showed development of large-scale slip near the crack tip in the NiAl adjacent to the interface during steady-state crack propagation. After testing, the local slope and curvature of the interface were characterized at intervals along the interface and at locations where slip was observed. Slope and curvature data indicate the types of local stresses that are present at and just ahead of the crack tip. Results showed that the greatest percentage of slip occurred where closing forces on the crack tip would be near the maximum, but decreasing as the crack tip moves forward. Based on these results and previous work studying crack initiation in this system, a mechanism for crack propagation was developed, supported by optical microscopy of the crack tip in an interrupted test. The basic elements of the mechanism are described in the enclosed schematic model (Fig. 3) and the accompanying micrograph (Fig. 4).

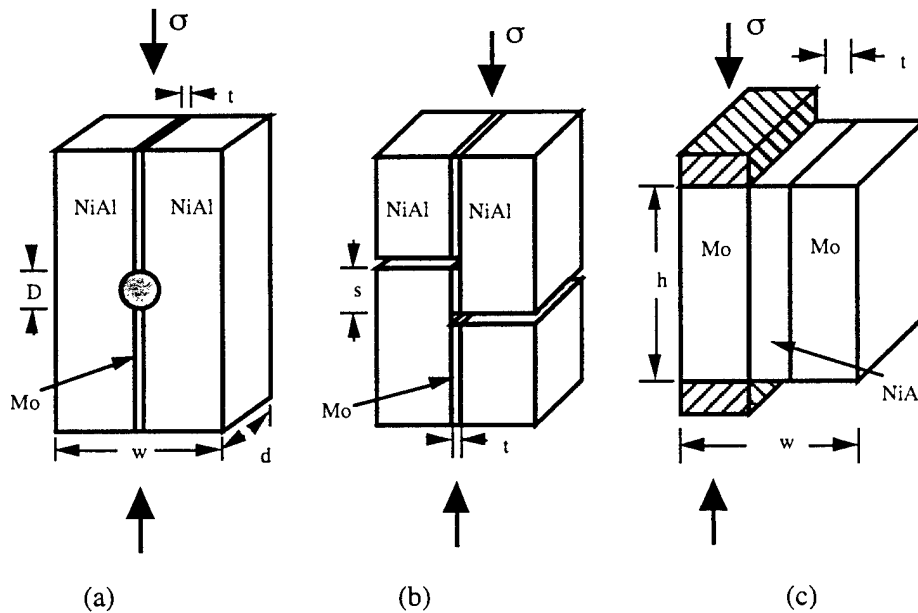
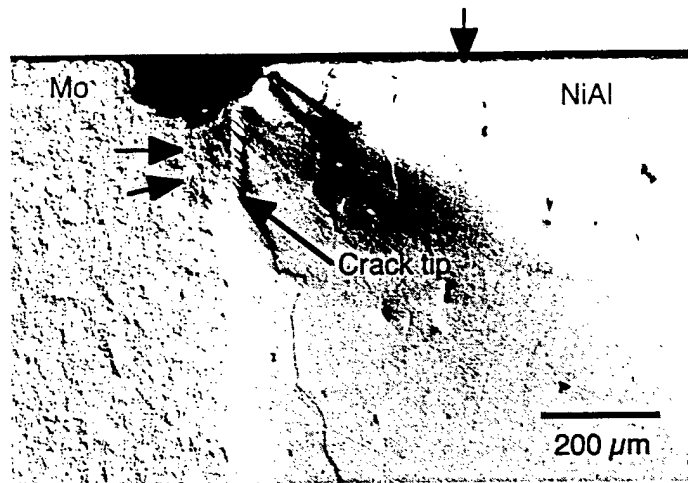
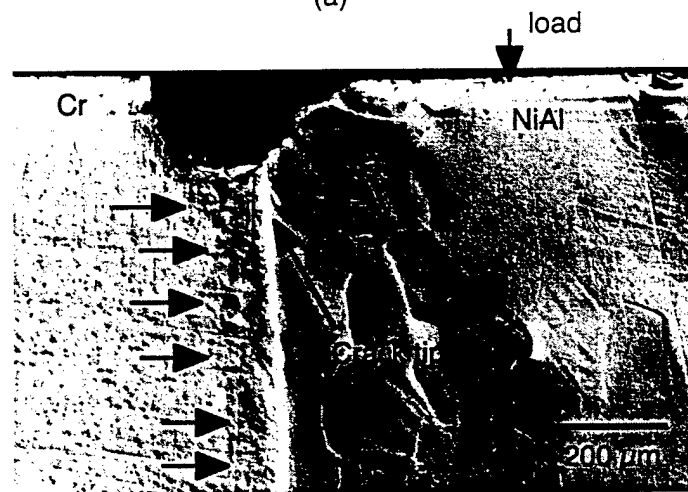


Fig. 1. Three of the sample geometries used in this study: (a) DCDC, (b) shear/compression, and (c) ALS.

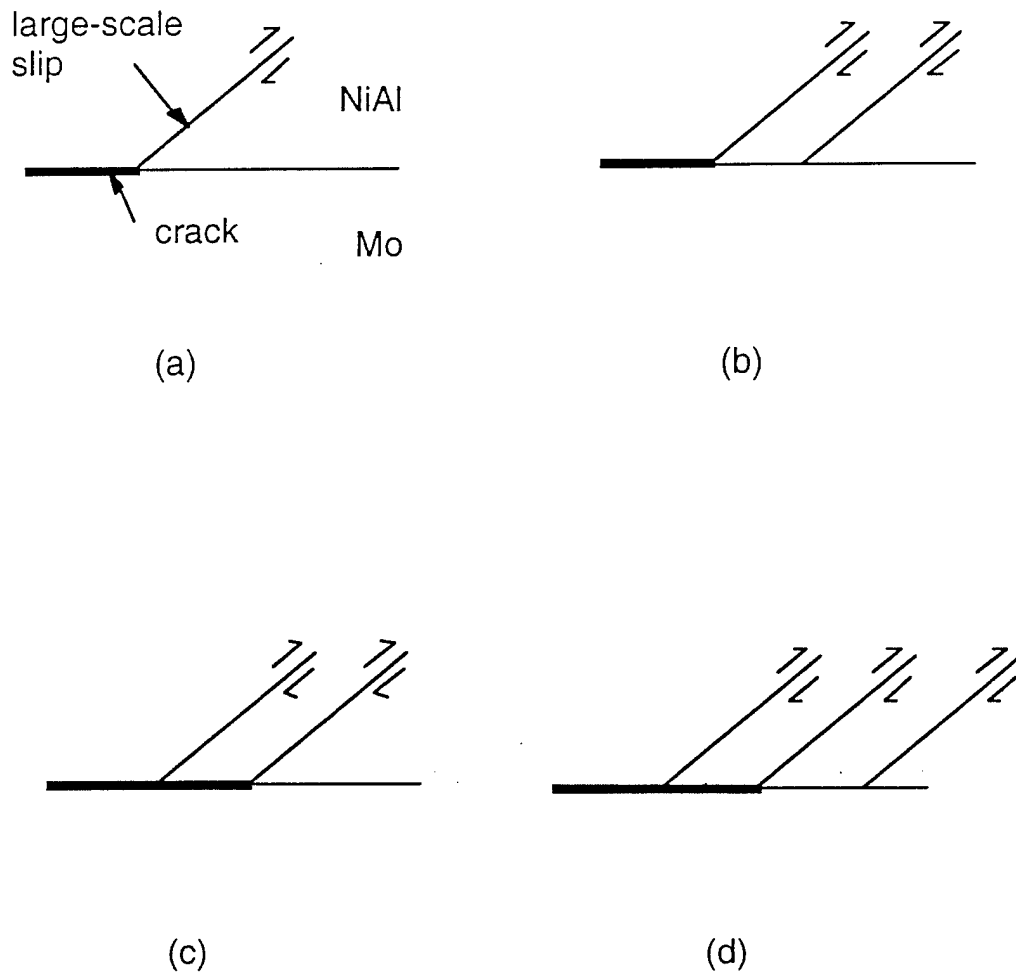


(a)



(b)

**Fig 2.** Plastic deformation in (a) NiAl/Mo and (b) NiAl/Cr model laminate asymmetrically-loaded shear (ALS) specimens after testing under similar loading conditions. (a) Extensive plastic deformation (indicated by arrows) in the Mo is visible near the crack tip. (b) Plastic deformation in the Cr extends well beyond the crack tip.



**Fig. 3.** Crack propagation along the NiAl/Mo interface, when associated with large-scale slip, is an incremental process. (a) The crack first initiates along the interface at the location where a large-scale slip band intersects the interface. (b) Next, new slip bands develop ahead of the crack tip. (c) Then a crack forms at the new slip band and extends back to the main crack, thus producing crack extension. (d) The crack continues to propagate in this incremental process.

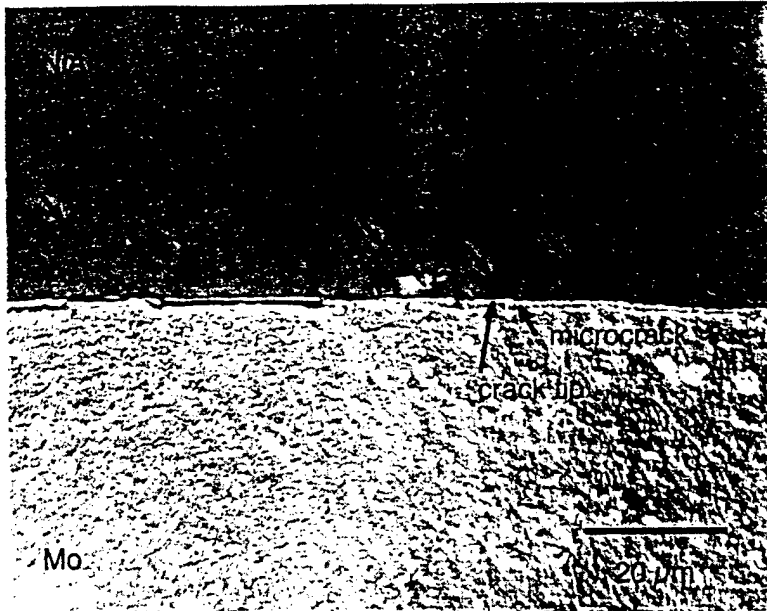


Fig. 4. An optical photograph of the crack tip highlights the large-scale slip lines. Large-scale slip and microcracking ahead of the main crack tip were observed.

## Mechanical Behavior: Laminated and Model Interfacial Materials

### HIGH RATE DIRECT DEPOSITION OF STRUCTURAL INTERMETALLICS USING PVD

S.M. Pickard and A.K. Ghosh

To make use of the improved mechanical properties of nanostructural materials, rapid and economical methods of synthesis must be developed. In this study, high rate evaporation of elemental sources is used to directly deposit vapor clusters to form bulk intermetallics and alloys. TiAl has been the model material selected for study, although Mo-Si and Nb-Si alloys have also been examined. Potential benefits of the nanoscale processing route are fine grained homogenous microstructures with compositional variations confined to the nanometer size of the microstructure. The potential property combinations available through the nanophase processing are higher strength, improved toughness and superplastic forming capability at a much lower temperature than for conventional grain size material. Ni and Ti based aluminides offer the potential for good oxidation resistance and high temperature strength, combined with lower density than nickel base superalloys. Our aim has been to explore the mechanical property advantages offered by PVD nanoscale processing of materials which otherwise show compositional inhomogeneities in the cast microstructure and low room temperature ductility.

Independent Ti and Al elemental sources are directly heated using 5kW electron beams and the evaporant is deposited as a nanolaminated structure at a rate of 1-2  $\mu\text{m}/\text{minute}$  on a rotating substrate (1000-2000 rpm) within a TEMESCAL evaporator unit with a substrate temperature of  $\sim 400^\circ\text{C}$ . In-situ reaction occurs on the heated substrate to produce a homogeneous nanograined bulk intermetallic deposit. Both relatively thin (30-40  $\mu\text{m}$ ) and thick (140-180  $\mu\text{m}$ ) film deposits have been produced. To examine bulk mechanical properties of the TiAl alloy, a stack consolidation scheme has been devised in which the thin deposit layers are stacked and diffusion bonded for 3.5 h under high vacuum ( $10^{-6}$  torr) at  $940^\circ\text{C}$  to produce a thick laminated material (1-1.5 mm thick). Compositional variations (in the range Ti 30-40 at% Al) are introduced into the thin stack layers to provide a two-phase  $\text{Ti}_3\text{Al}/\text{TiAl}$  microstructure after consolidation of the TiAl alloy. Process variables of time and temperature and applied pressure during consolidation have been optimized to retain the mechanical property advantages of high hardness and fine grain structure of the original deposit, while providing good interlayer bonding with minimal porosity. Compositional analysis of the as-deposited alloys using RBS indicates a compositionally banded appearance of the as-deposited material with compositional variations of submicron scale which cannot be fully resolved in the SEM.

TEM analysis of plane sections parallel to the growth direction illustrates a fine single phase structure with grain size of about 20-50 nm throughout the thickness of the foil (50-100 nm) with extensive strain field contrast in the bright field images, indicative of a highly defective material. The crystal structure is expected to be a disordered non-equilibrium supersaturated solid solution phase within the compositional range of the  $\text{Ti}_3\text{Al}$ -TiAl region of the equilibrium phase diagram. Annealing of the deposit for a minimum of 2 h at  $500^\circ\text{C}$  allows phase separation to occur with the formation of submicron size  $\gamma$ -TiAl grains in an  $\alpha_2$  matrix of the deposit. The defective regions which are believed to result from substrate temperature variations during deposition, and turbulence created during the deposition process, are reduced at higher substrate temperature. An important observation is void healing on annealing the porous regions for 2 h at  $700^\circ\text{C}$  with disruption of the nano-layered  $\text{Ti}_3\text{Al}/\text{TiAl}$  appearance of the deposit. It appears that diffusion of Al is the primary mechanism for this phenomenon. It is interesting to note that twin lamellar microstructure characteristic of these materials can be completely avoided by this processing route. The grain growth of the constituent phases appears to be somewhat restricted by the two-phase grain structure, which provides mutual grain pinning, as reported for conventionally processed two-phase TiAl alloys. Examination of the thick as-deposited material discloses processing-induced defect structures with porous seams of material parallel to the growth direction up to 1-5  $\mu\text{m}$  wide. Additional column-like defective regions appear parallel to the growth direction and appear to contain voided material.

Hardness characterization of the as-deposited film shows a Vickers hardness value of about 5.5 GPa, which is much higher than the expected value for an as-cast alloy of similar composition (2-3 GPa). Microstructural studies of annealing behavior of the deposit have been accompanied by progression of hardness measurements using small loads (20-100 grams). These studies show a transition from a nanolayered initial deposit to a fine equiaxed microstructure which begins after annealing for 2 h at 500°C, with an approximately 40% reduction in hardness. Vickers hardness measurements on the stack-consolidated materials give hardness variations which correspond to the compositional variations of the layered structure from Ti<sub>3</sub>Al to TiAl. Elastic modulus was determined using an instrumented nanoindenter which gave an unloading modulus of 180-200 GPa for the annealed TiAl alloy (2 h at 700°C). This value is slightly higher than that for conventionally processed TiAl (180 GPa).

The stack-consolidated TiAl alloy, loaded in compression along the stack layers, exhibited an initial flow stress of 700-800 MPa for the Ti<sub>3</sub>Al/TiAl dual-phase microstructure, higher than that of conventional cast material (which is less than 500 MPa). Failure of the specimen is by catastrophic delamination of the diffusion bonded layered structure which ensues shortly after the onset of non linear deformation response at a plastic strain of less than 0.75% (see Fig. 1). This observation indicates the presence of strength anisotropy in the nanoscale deposit. Final fracture path appears to follow closely the boundary between the more brittle and stiffer TiAl phase and the Ti<sub>3</sub>Al phase. The rupture strength of the material measured using three-point bending of laminated specimens cut parallel to the lamination direction is 450-500 MPa for Ti-30-40 at% Al, with linear-elastic loading response to failure, indicative of extrinsic defect-controlled strength. Some limited crack deflection into the more brittle TiAl occurs on failure and is indicative of a potential for crack deflection toughening in the dual-phase intermetallic microstructure.

In summary, PVD processing of TiAl shows property advantages of increased hardness and strength. The two-phase  $\alpha_2+\gamma$  equiaxed microstructures in the nanophase material appear to offer the possibility of microstructural stability at elevated temperature. However, restricted plasticity in the intermetallic at lower temperature remains a problem. The deposit can be thermomechanically processed to produce optimum mechanical properties with microstructural and compositional variations in a laminated material. An improved understanding of process control is necessary to minimize grown-in defects in the layer including layers of porosity.

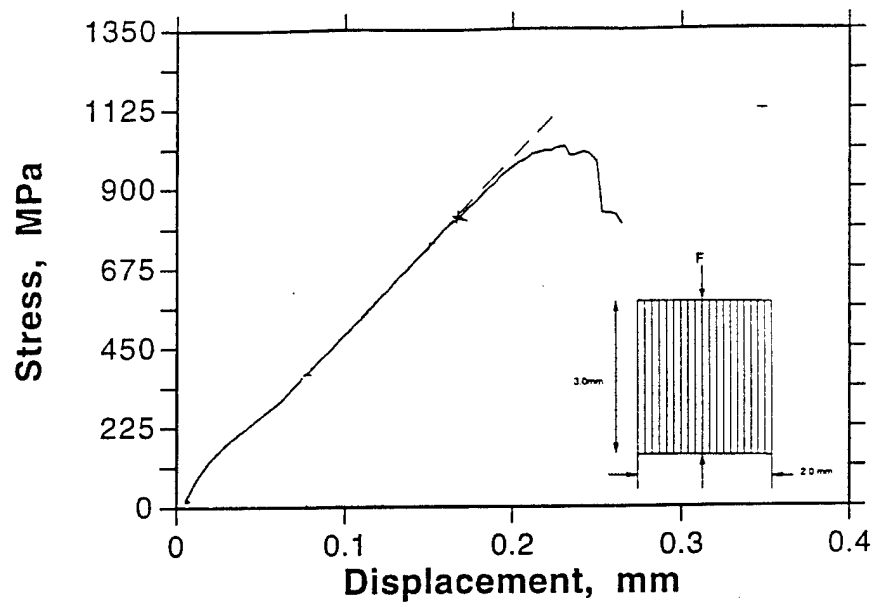


Fig. 1. Engineering stress-displacement curve for the stack consolidated Ti-Al alloy. The dashed line on the curve marks first deviation from linear loading.

Mechanical Behavior: Laminated and Model Interfacial Materials

**MICROSTRUCTURAL AND MECHANICAL CHARACTERIZATION OF  
CARBON COATINGS ON SiC FIBERS**

K.L. Kendig, R. Gibala, and D.B. Miracle\*

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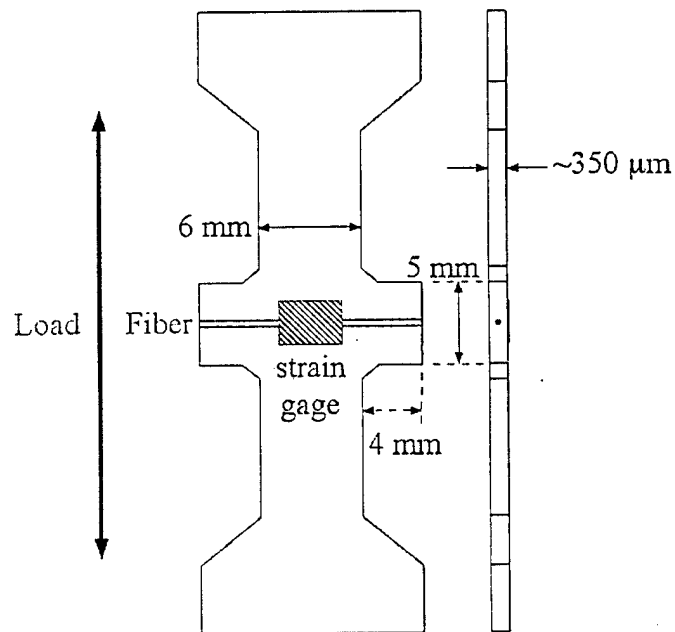
Thin carbon coatings, less than 5  $\mu\text{m}$ , are often used as coatings on the SiC fibers in a continuous-fiber metal matrix composite (MMC). These coatings serve several important roles in the composite, which include increasing the tensile strength of the SiC fiber to as much as twice the uncoated monofilament strength and acting as a weak interfacial region which decreases the risk of the catastrophic failure of the composite. When a metal matrix composite is used in a rotating component application, the fibers are typically wound with the fiber direction parallel to the direction of maximum applied tensile stress. Due to the rotation of the part, there also exists a lesser component of tensile stress transverse to the fiber direction. Experiments have shown that, in transverse tension, composite failure most often begins in the carbon coating, and commercially available SiC fiber coatings have exhibited transverse tensile strengths ranging from essentially 0 to 300 MPa. Thus, the carbon coating region is generally regarded as the weak link in transverse composite performance and is therefore an important area of research.

In order to optimize the transverse strength of carbon coating, the microstructural factors affecting the transverse strength were investigated. A series of three carbon coatings were deposited on the SiC monofilaments using chemical vapor deposition. One processing variable, electrical power through the substrate, which is approximately equivalent to a change in the substrate temperature during deposition in the range 920°C–1080°C, was examined. Three different substrate temperatures (920°C, 1000°C, 1080°C) were employed, corresponding to respective power settings of 180, 200, and 220 watts, as indicated in the enclosed figures. Transverse tension tests (cruciform tests, see Fig. 1), nanoindentation, and a semi-quantitative fiber-direction tensile test were used to examine the mechanical response of each coating. The microstructure of the carbon was observed using various types of optical and electron microscopy.

Transmission electron microscopy (TEM) was used to examine the structure of the three deposited carbon coatings and the substrate fiber. Selected area diffraction (SAD) of the carbon coatings verified the presence of a turbostratic-type pyrolytic carbon structure and revealed differences in the degree of texturing of this carbon. The coating deposited at 920°C exhibited the least amount of texturing of turbostratic "crystallites." The coatings deposited at 1000°C and 1080°C were similar to each other and exhibited a higher degree of texturing. Similar observations were made using high resolution TEM (HRTEM). SAD and HRTEM also revealed changes in the grain size of the SiC fiber near the SiC-C interface.

Some typical HRTEM images of the carbon coatings and the SiC-C interfaces are given in Figs. 2 and 3. The change in deposition temperature correlates with observed changes in the grain size of the SiC substrate fiber within 3  $\mu\text{m}$  of the SiC-C interface and in the texture of the basic structural units of carbon within the coatings. Changes in the ordering at the

interface and the texture of the carbon coatings correlate with observed changes in fracture location in transverse tension. A rule-of-mixtures model describes the change in transverse elastic modulus, as measured by nanoindentation (see Fig. 4), in terms of the change in width of the basic structural units of carbon. The change in deposition temperature did not significantly affect the transverse failure strength of the tested composites, despite the observed changes in microstructure, coating adhesion strength, elastic modulus of the coating, and transverse tension failure location. A model has been developed for which the interface strength and the coating strength are affected differently by deposition temperature and the corresponding microstructural changes. Fig. 5 summarizes the qualitative elements of the model.



**Fig. 1.** Schematic of a cruciform sample. The intersection of the fiber-matrix interface with the free surface for the single fiber is isolated from the stressed area of the specimen.

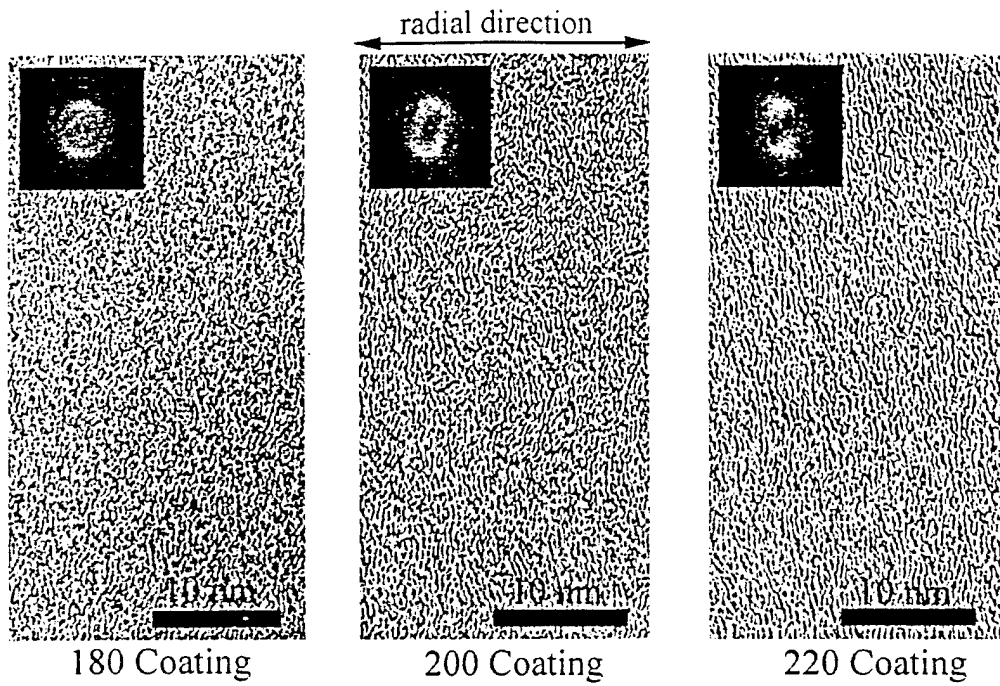


Fig. 2. HRTEM micrographs and corresponding fast-Fourier-transforms (FFTs) of carbon coatings near the SiC-C interface. The FFTs are aligned with the corresponding micrographs. The texturing of the carbon increases with increasing deposition temperature, i.e. from 180 (920°C) to 200 (1000°C) to 220 (1080°C).

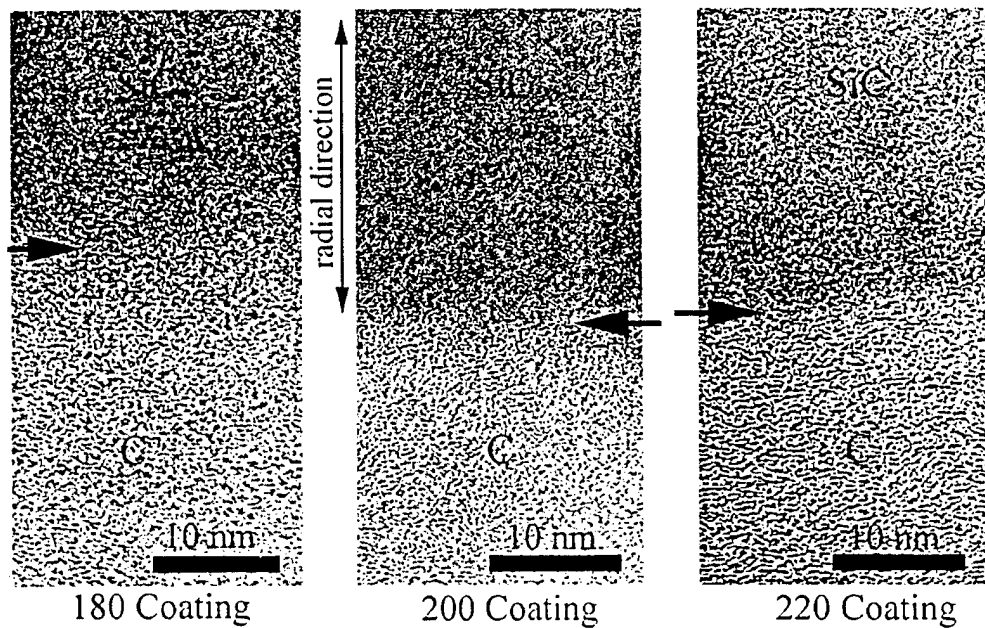


Fig. 3. HRTEM images of SiC-C interface for each of the three coated fibers. The interfaces between the SiC and C are horizontal and are indicated by arrows.

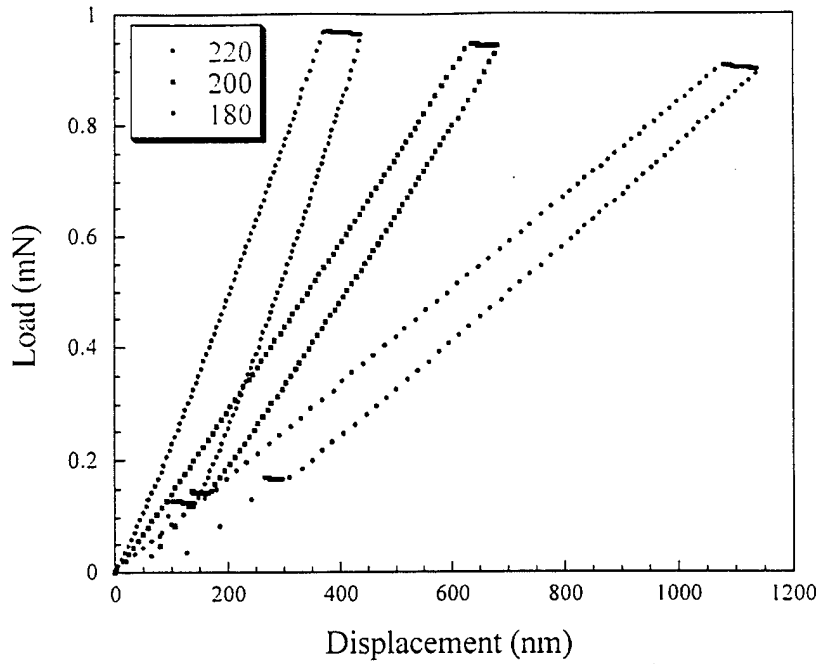


Fig. 4. Representative load-displacement plots for the 180, 200, and 220 coatings as obtained by nanoindentation. The modulus increases with increasing deposition temperature.

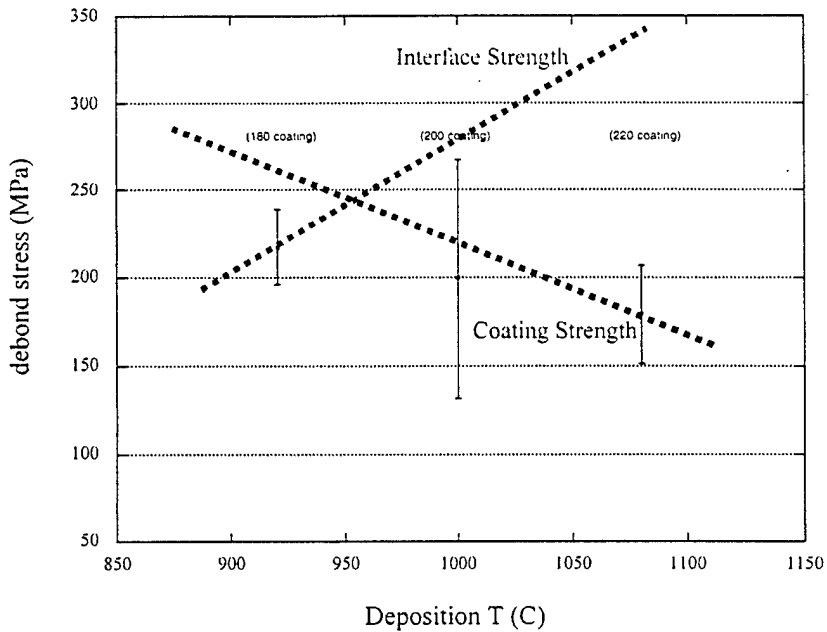


Fig. 5. Schematic of the effect of deposition temperature on the SiC-C interface and carbon coating strengths implied by the results of adhesion experiments, cruciform tests and microstructural determinations.

## Mechanical Behavior: Fatigue Related

### THERMAL FATIGUE DAMAGE ACCUMULATION IN MoSi<sub>2</sub>- AND NiAl-BASED COMPOSITES

M.T. Kush, R. Gibala and J.W. Holmes

Thermal fatigue resistance is a concern for many high temperature applications, but very little is known about the thermal fatigue resistance of MoSi<sub>2</sub>-based composites or NiAl-based eutectic composites, which have been the principal materials under investigation in this program. The goal of this project has been to determine the thermal fatigue behavior of MoSi<sub>2</sub>, NiAl, and MoSi<sub>2</sub>-based and NiAl-based composites and more generally to determine the effect of second phases on the thermal fatigue life. The use of thermal cycling experiments also allows fundamental study of the role of misfit strains in the mechanical and thermal stability of these and other IMCs. For example, the results of these experiments have allowed comparison with microstructural stability predictions from analytical work described elsewhere in this report.

The techniques developed for measuring thermal fatigue resistance of IMCs have been described in several of our MURI publications. The experimental method uses the skin effect during high frequency inductive heating of metallic materials to develop thermal gradients across the radius of 17 mm diameter, 2 mm thick disk-shaped specimens. Many different thermal cycles have been utilized. Most materials have been cycled between 700°C and 1200°C with individual cycles that include: (a) heating from 700°C to 1200°C in 5 s; (b) a 15 s hold at 1200°C; (c) convective cooling in 5 s to 700°C; and (d) a 15 s hold at 700°C. In some experiments longer heating and cooling times, e.g., 30 s in steps (a) and (c), were used. In the results described below, we use the terms 5 s profile, 30 s profile, etc. to describe some of the specific temperature-change histories employed. These cycles closely simulate engine conditions encountered by the leading edge of advanced gas turbine airfoils during idle and takeoff. The resulting thermoelastic (circumferential) stress-strain curve for the material is a very asymmetrical hysteresis loop. In one cycle, the material is initially driven substantially into compression, e.g., to approximately 120 MPa for [001]-oriented NiAl, then modestly into tension, about 10 MPa for NiAl. A typical result for single-crystalline NiAl in the [001] and [011] orientations is given in Fig. 1.

*Monolithic MoSi<sub>2</sub> and MoSi<sub>2</sub>-SiC Composites:* The thermal fatigue resistance of monolithic polycrystalline MoSi<sub>2</sub> is very poor. When MoSi<sub>2</sub> is subjected to a 5 s profile, the material usually cracks transgranularly on the initial cycle during the ramp from 700°C to 1200°C. The monolithic material can be successfully cycled when subjected to a less severe 30 s profile, but when subjected to 5000 cycles exhibits thermal fatigue cracking. By contrast, MoSi<sub>2</sub>-SiC particulate composites, with a typical particle size of ~1 μm, exhibit very good thermal shock and thermal fatigue resistance. For example, 10 vol % SiC particulate composites can be subjected to 5000 cycles of a 5 s profile without evidence of failure. This material, as well as 20% and 30% vol % SiC composites, form an adherent SiO<sub>2</sub> oxide layer, about 200 nm thick, during the thermal fatigue tests. There is no evidence of microcracking in the matrix due to the thermal stress cycling. Moreover, the higher SiC loadings produce better thermal fatigue resistance, consistent with predictions of homogenization analysis discussed elsewhere in this report.

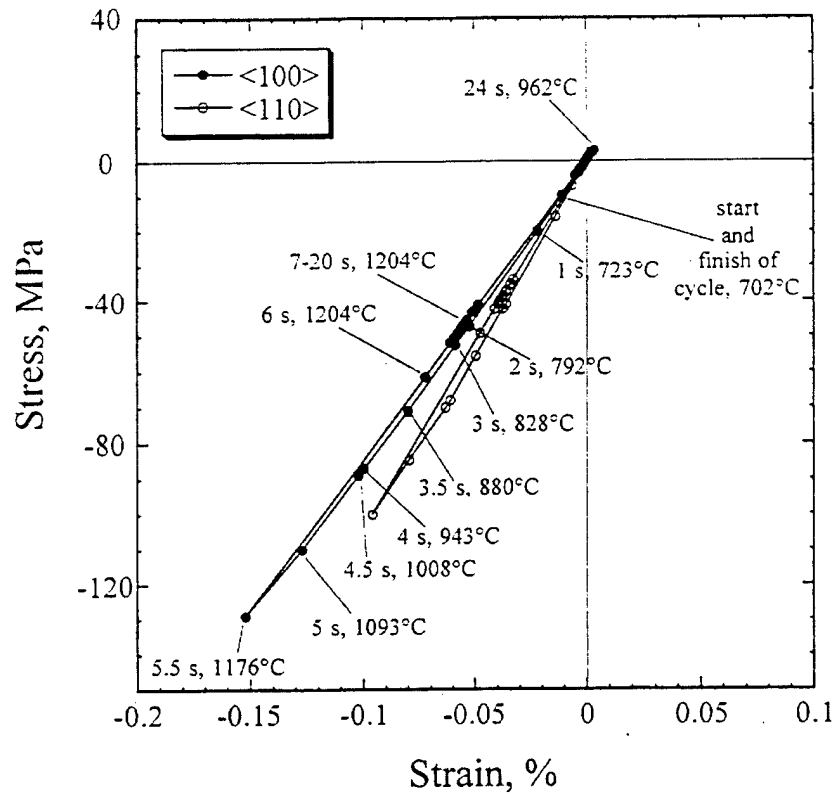
*NiAl Single Crystals:* Single crystals of [001]-oriented NiAl single crystals were subjected to thermal fatigue in an argon atmosphere using several time-temperature heating and cooling profiles to produce different thermal strain histories in specimens cycled between 700°C and 1200°C. After thermal cycling, pronounced shape changes in the form of diametrical elongations along <100> directions with accompanying increases in thickness at and near the <001> specimen axes were observed. An example is shown in Fig. 2. The deformations were

analyzed in terms of operative slip systems in tension and compression, ratchetting (cyclic strain accumulation), and the elastic properties of NiAl. The shape changes could not be accounted for in terms of known operative slip systems of NiAl or yield or flow anisotropies associated with specific slip systems. The results do correlate well with thermal stresses associated with the large elastic anisotropy of NiAl, which can result in approximately 50% higher stresses in the elastically soft  $\langle 100 \rangle$  directions. However, to obtain such shape changes requires additionally a significant applied stress asymmetry, which is afforded by the largely compression-driven asymmetric, thermoelastic stress-strain curves of Fig. 1.

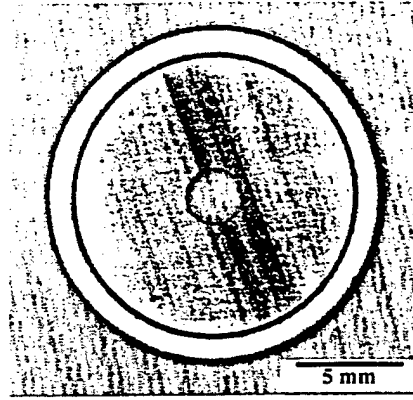
*NiAl-base In-Situ Eutectic Composites:* We have done extensive thermal fatigue experiments on NiAl-9 vol % Mo and NiAl-34 vol % Cr in-situ eutectic composites. These materials exhibit cube-on-cube orientation relations between the B2 matrix and the bcc metal fiber or lamellar second phase. Directionally-solidified NiAl-Mo composites can be subjected to the 5 s profile for 5000 cycles without failure. However, the material develops an extensive oxide layer on the surface that penetrates into the interior. The degradation is associated with both NiAl and Mo oxidation, especially that of Mo.

The microstructural stability of the NiAl-9 at.% Mo alloy was investigated, mainly under various conditions of thermal cycling between the temperatures 700°C and 1200°C. Two different microstructures were examined: a cellular microstructure in which the faceted second-phase Mo rods in the NiAl matrix formed misaligned cell boundaries which separated aligned cells approximately 0.4 mm in width and 5-25 mm in length, and a nearly fault-free fully columnar microstructure especially well aligned along the [001] direction. Both microstructures resisted coarsening, especially under strictly elastic loading, under thermal cycling, but plastic deformation induced by thermal stresses introduced significant specimen shape changes. Surprisingly, the cellular microstructure, for which the cell boundary region apparently acts as a deformation buffer, exhibited better resistance to thermal fatigue than the more fault-free and better aligned columnar microstructure. In the cellular material, yielding occurred locally at the cell boundaries leading to substantial shape changes of the thermal fatigue specimens after 120 h (10,800 cycles of the 5s profile) and coarsening of the microstructure at the cell boundary. The macroscopic length scale features are illustrated in Fig.3. In the aligned columnar grained material, the thermal fatigue specimens changed shape after 20 h (2,160 cycles). The lack of a soft cell boundary region for localized deformation to occur caused the individual columnar grains to yield over large length scales in the latter material.

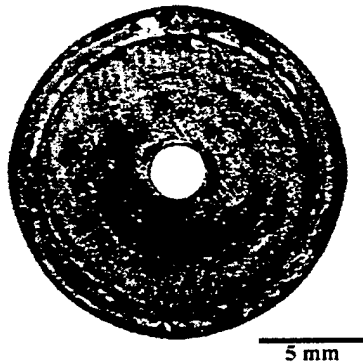
Similar experiments have been completed for the NiAl-34Cr alloys. In spite of better oxidation resistance, these materials had poorer thermal fatigue resistance, and exhibited greater sensitivity to growth conditions and details of the solidification microstructures. In a separate section of this report, similarly poor static crack-growth resistance and fatigue-crack growth behavior are discussed for these alloys.



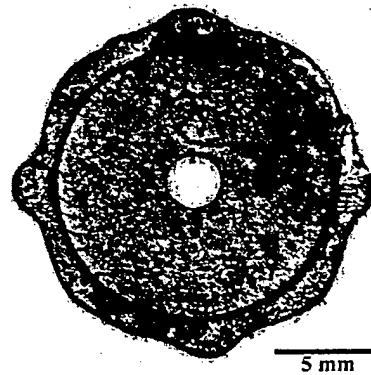
**Fig. 1.** Circumferential thermoelastic stress-strain curves along the periphery of NiAl and NiAl-9Mo alloys cycled between 700°C and 1200°C for <100> and <110> crystallographic directions. These curves give the lower and upper bounds of stresses in the transverse plane.



(a)

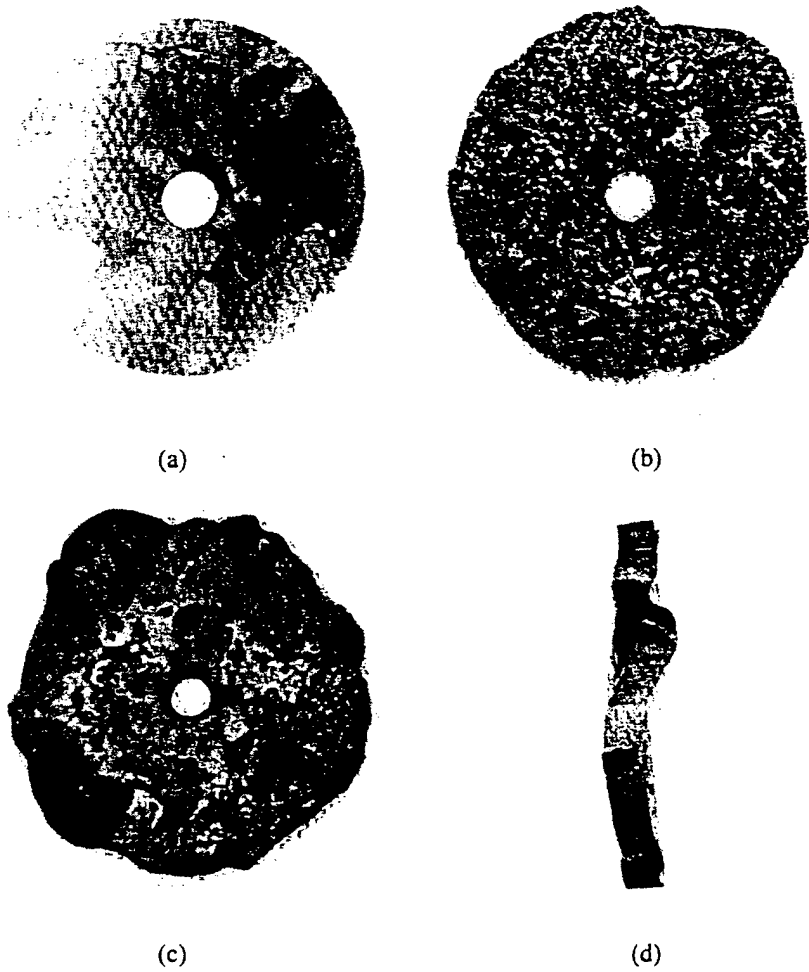


(b)



(c)

Fig. 2. Stepped-disk specimens of  $\langle 001 \rangle$  NiAl single crystals. (a) Original specimen. (b) After 45 cycles between 700°C and 1200°C. (c) After 2,160 cycles. The large in-plane extensional deformation occurs along the elastically soft  $\langle 100 \rangle$  directions.



**Fig. 3.** Thermal fatigue specimens of a NiAl-9Mo alloy cycled between 700°C and 1200°C. (a) Original specimen. (b) After 20 h. (c) After 120 h. (d) After 120 h, a side view showing substantial out-of-plane deformation.

## OTHER PROJECTS

Additional projects have been underway and completed as part of this program and are listed below with the key contributors. More detailed information on each of these projects is given in previous progress reports and in publications listed elsewhere in this report.

- Low Temperature Consolidation Techniques for Processing NiAl-Mo Laminates. (K. Mukai, S.M. Pickard, M. Fox, and A.K. Ghosh).
- Analysis of Effects of Reinforced Morphology on Matrix Microcracking and Thermal Misfit Damage in Particulate Composites (N. Sridhar and D.J. Srolovitz).
- High Temperature Mechanical Behavior of Nb<sub>5</sub>Si<sub>3</sub>/Nb and MoSi<sub>2</sub>/Nb Laminates. (W.R. Provancher, S.M. Pickard, and A.K. Ghosh).
- Mechanical Behavior and Creep Modeling of Monolithic MoSi<sub>2</sub> and MoSi<sub>2</sub>-SiC and MoSi<sub>2</sub>-TiC Composites. (A.K. Ghosh, A. Basu, H. Chang, and R. Gibala).
- Modeling and Measurement of Ductile Phase Toughening of Laminated Composites. (X. Wu, J.W. Holmes, H. Zhang, S.M. Pickard, K. Mukai, and A.K. Ghosh).

### III. LISTS OF PUBLICATIONS, THESES, PRESENTATIONS

The publications and presentations associated with this program will continue to grow over the next few years beyond the following lists of approximately 100 papers and 100 presentations on the next several pages. These lists, while not comprehensive, are intended to give a good indication of the breadth of topics covered and the intensity of the effort. These communications are based largely on the theses of 16 students (6 MS and 10 Ph.D) and the research of eight post-doctoral associates. We anticipate, finally, approximately 120 total research and review papers and 120 total presentations on the work accomplishments of this program because the basic results will continue to engender new ideas, interpretations and models. Two students (Czarnik and Mukai) have not defended their Ph.D. theses as of the writing of this report. All of their research is complete; the written theses have not been submitted.

#### A. PUBLICATIONS

##### Modeling: Structure and Bonding

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### **Modeling: Microstructure**

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#### **Mechanical Behavior: Laminated and Model Interfacial Materials**

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- W.R. Provancher and A.K. Ghosh, "High Temperature Mechanical Behavior of Nb<sub>5</sub>Si<sub>3</sub>/Nb Laminates," Cer. Engin. Sci. Proc. 17 (1996).
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- S.M. Pickard and A.K. Ghosh, "High Rate Direct Deposition of Intermetallics Using PVD," Proc. Third Int'l. Conf. on Composites Engin., ICCE/3, 675 (1996).
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#### **Mechanical Behavior: Fatigue Related**

- M.T. Kush, J.W. Holmes, and R. Gibala, "Investigation of the Thermal Fatigue of MoSi<sub>2</sub> Particulate and Short Fiber Composites," Intermetallic Matrix Composites III, J.G. Graves et al., eds., MRS Symp. Ser. 350, 189 (1994).
- M.T. Kush, J.W. Holmes, and R. Gibala, "Thermal Fatigue of MoSi<sub>2</sub> and MoSi<sub>2</sub> - 10 vol % TiC Composite," High Temperature Silicides and Refractory Metals, C.L. Briant et al., eds., Mat. Res. Soc. Symp. Proc. 332, 261 (1994).

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K. Badrinarayanan, A.L. McKelvey, K.T. Venkateswara Rao, and R.O. Ritchie. "Fracture and Fatigue-Crack Growth in Ductile-Phase Toughened Molybdenum Disilicide: Effects of Niobium Wire vs. Particulate Reinforcements," Mater. Trans. 27A, 3781 (1996).

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### **Experimental Infrastructure**

All papers based on enabling experimental techniques and methods, including equipment purchases, are included in other parts of this section. Nearly all of the equipment purchases or fabricated equipment were made with funds provided as University cost sharing. A listing is provided at the end of this report.

## B. THESES AND POST-DOCTORAL RESEARCH PROJECTS\*

### Ph.D. Theses

Ajoy K. Basu, "Processing and High Temperature Mechanical Behavior of MoSi<sub>2</sub> and MoSi<sub>2</sub>-based Composites," July, 1995.

Hugo Chang, "Mechanical Behavior of MoSi<sub>2</sub> and MoSi<sub>2</sub>-TiC Composites," January, 1995.

Wuhua Yang, "Computer Simulation and Microstructural Modelling of Deformation and Fracture of MoSi<sub>2</sub>-based Alloys and Composites," December, 1995.

Sridhar Narayanaswamy, "Stress Effects on the Stability of Microstructures," September, 1996.

Kenjiro Terada, "Global-Local Modeling for Composites by the Homogenization Method," June, 1996.

Matthew Kush, "Stability of Directionally Solidified NiAl-9Mo," May, 1998.

Kevin Kendig, "Microstructural and Mechanical Characterization of Carbon Coatings on SiC Fibers," September, 1999.

Matthew Fox, "Interface Shear Strengths of NiAl-Metal Laminates," December, 1999.

Kevin Mukai, "Interfacial Mechanical Properties of Model Intermetallic Matrix-Based Laminates," in preparation.

Cory Czarnik, "Effects of Surface Films on Mechanical Behavior of MoSi<sub>2</sub>, in preparation.

### M.S. Theses

Keith Patrick, "Mechanical Behavior of MoSi<sub>2</sub>-based Composites," August, 1994.

Matthew Fox, "Mechanical Behavior of IMC Laminate Materials," April 1995.

William Provancher, "High Temperature Mechanical Behavior of Nb<sub>5</sub>Si<sub>3</sub>/Nb Laminates," August, 1995.

Matthew Kush, "Thermal Fatigue of MoSi<sub>2</sub> and MoSi<sub>2</sub> Particulate Composites," August, 1995.

Kevin Mukai, "Interface Strength Measurements in Model Laminates," May, 1996.

Kevin Kendig, "Nanoindentation Response of Ti Alloy/SCS-6 Fiber MMCs," June, 1997.

### Post-Doctoral Projects

Dr. Tao Hong (Research Associate, 1993), "Electronic Structure Calculation of Strength and Adhesive Properties of Mo/MoSi<sub>2</sub> Interfaces."

Dr. Xin Wu (Research Associate, 1993), "Creep, Fracture, and Ductile Phase Toughening of Model Metal-Ceramic Composites."

Dr. Zili Wu (Research Associate, 1993-1995), "Synthesis of High Temperature Intermetallics by Melt Processing and Extrusion."

Dr. Hongyan Zhang (Research Associate, 1993-1995), "Mechanics Analysis of Mechanical Behavior of Intermetallic Matrix Composites."

Dr. Sion Pickard (Research Associate, 1993-1996), "Structure-Properties-Processing Relationships and Model Systems Analysis of Laminated Microstructures Based on Intermetallic Matrices."

Dr. James Reynolds (Research Associate, 1994-1996), "First-Principles Calculations of Strength and Adhesion Properties of Intermetallic Matrix Composites."

Dr. Hugo Chang (Research Associate, 1995-1996), "NiAl-Base Eutectic Alloys."

Dr. Amit Misra (Research Associate, 1996-1997), "Structure-Mechanical Properties-Processing Relationships of NiAl-Base Eutectic Alloys."

\* Also included are post-doctoral researchers and students with Prof. R.O. Ritchie at the University of California, Berkeley: K.T. Venkateswara Rao (1993-1995), J. M McNaney (1995-1997), M.-H. Hong (1995-1996), K. Badrinarayanan (1993-1995) H. Endo (1993-1995) and A. McKelvey (1993-1994). These researchers worked on fracture toughness and fatigue crack growth of MoSi<sub>2</sub> and NiAl, their alloys and composites.

## C. PRESENTATIONS (\*invited talks)

### Modeling: Structure and Bonding

T. Hong, J.R. Smith, and D.J. Srolovitz, "Impurity Effects on Adhesion: Nb, C, O, B, and S at a Mo/MoSi<sub>2</sub> Interface," American Physical Society, 1993 March Meeting, Seattle, 1993.

T. Hong, D.J. Srolovitz, and J.R. Smith, "First Principles Study of Interfacial Adhesion: the MoMoSi<sub>2</sub> Interface With and Without Impurities," Materials Research Society 1993 Spring Meeting, San Francisco, April 1993.

T. Hong, J.R. Smith, and D.J. Srolovitz, "Impurity Effects on Interfacial Adhesion," American Society for Metals, 1993 Fall Meeting, Pittsburgh, October, 1993.

J.E. Reynolds, J.R. Smith, G.L. Zhao, and D.J. Srolovitz, "NiAl/Cr Interfacial Adhesion: First-Principles Results," TMS Fall Meeting, Cleveland, October, 1995.

### Modeling: Microstructure

\*N. Kikuchi, "Homogenization Method and its Application to Material Design for Composites," in COMMP'93, Int'l. Conf. on Computer-Aided Material Design and Process Simulation, Tokyo, Japan, September, 1993.

\*N. Kikuchi, "An Optimal Design Method of Microstructure Using the Homogenization Method," 3<sup>rd</sup> IUMRS Int'l. Conf. on Advanced Materials, Sunshine City, Tokyo, Japan, August 31, 1993.

\*N. Sridhar, "Thermal Misfit Induced Damage in Brittle Composites," Materials Laboratory Colloquium, January, 1994, NIST, Gaithersburg, MD.

N. Sridhar, "Thermal Misfit Induced Damage in Brittle Composites," Department of Materials Science and Engineering Colloquium, April, 1994, University of Michigan, Ann Arbor.

N. Sridhar, J.M. Rickman, and D.J. Srolovitz, "Thermal Misfit and Thermal Fatigue-Induced Damage in Brittle Composites," MRS Spring Meeting, San Francisco, CA, April, 1994.

N. Sridhar, J.M. Rickman, and D.J. Srolovitz, "Modeling Thermal Misfit and Thermal Fatigue-Induced Damage in Brittle Composites," American Ceramic Society Meeting, Indianapolis, IN, April, 1994.

H. Zhang, A.K. Ghosh, and N. Kikuchi, "A Study of Crack Initiation, Propagation, and Interface Debonding in Intermetallic Matrix Composites," Symposium on High Temperature Fracture Mechanisms in Advanced Materials, TMS Fall Meeting, Rosemont IL, October 1994.

\*N. Kikuchi and K. Terada, "Nonlinear Homogenization Methods for Large Deformation Plasticity," Plasticity '95, International Conference on Plasticity, Sakai, Japan, July, 1995.

\*N. Kikuchi, "Microstructural Design Optimization for Thermal Mismatch," Workshop on Material and Structural Design Optimization, NIST, University of Utah, August, 1995.

\*N. Kikuchi, "Homogenization Methods for material Design," Annual Meeting of Japan Society of Applied and Industrial Mathematics, Okayama, Japan, September, 1995.

N. Sridhar and D.J. Srolovitz, "Temporal Evolution of Particle Shape in a Stressed Solid," MRS Fall Meeting, Boston, MA, November 1995.

\*N. Kikuchi and K. Terada, "Applications of the Homogenization Method for Computational Material Design," International Symposium on Computational Material Design, Kin-Zai Ken (National Institute for Metals), Tsukuba-shi, Japan, January, 1996.

\*N. Sridhar, "Stress-Induced Microstructural Instabilities," Department of Metallurgy, Indian Institute of Science, Bangalore, India, February, 1996.

\*N. Kikuchi, K. Terada, and T. Itoh, "Application of the Homogenization Method for Coupled Structure and Flow—Viscoelastic Solids," 3<sup>rd</sup> US-Japan Symposium on Large Scale Scientific Computation in Flows, Army High Performance Computing Research Center, University of Minnesota, March, 1996.

\*N. Sridhar, "Computational Material and Structural Design Optimization," HP-Convex High-Performance Computing Symposium for Automotive Engineering, Detroit, MI, April, 1996.

N. Sridhar and D.J. Srolovitz, "Morphological Instability of Thin Film Multilayers," MRS Spring Meeting, San Francisco, CA, April, 1996.

N. Sridhar, J.M. Rickman, and D.J. Srolovitz, "Stress-Induced Morphological Instabilities in Eutectic Composites," MRS Spring Meeting, San Francisco, April, 1996.

\*K. Terada, T. Minura, and N. Kikuchi, "Digital Imaged-Based Modeling to the Homogenization Analysis of Intermetallic Composites," Localized Damage '96, Kyushu, Japan, June, 1996.

\*N. Kikuchi, "Application of the Homogenization Method for Material Design and Analysis," Army WES Center for High Performance Computing, Mississippi, 1996.

#### **Mechanical Behavior: MoSi<sub>2</sub>-base Materials**

H. Chang and R. Gibala, "Plasticity of MoSi<sub>2</sub> Below 900°C," Symposium on High Temperature Silicides and Refractory Alloys, MRS Fall Meeting, Boston, MA, November, 1993.

\*R. Gibala, H. Chang, and C.M. Czarnik, "Plasticity Enhancement Processes in MoSi<sub>2</sub>-Base Materials," MRS Fall Meeting, Boston, MA, November, 1993.

A.K. Ghosh and A. Basu, "The Effect of Grain Size and SiC Particulates on the Strength and Ductility of MoSi<sub>2</sub>," MRS Fall Meeting, Boston, MA December, 1993.

A. Basu and A.K. Ghosh, "The Effect of Deformation and Reinforcement Particles on the Grain Growth Behavior of  $\text{MoSi}_2$ ," MRS Fall Meeting, Boston, MA, December, 1993.

A.K. Ghosh and A. Basu, "Microstructural Effects of Creep Strengthening of  $\text{MoSi}_2$  Matrix Composites," Intermetallics Conference, Wright-Patterson Air Force Base, 1993.

A.K. Ghosh and A. Basu, "Microstructural Effects of Creep Strengthening of  $\text{MoSi}_2$  Matrix Composites," Critical Issues in High Temperature Materials Conference, Hawaii, 1993.

\*A.K. Ghosh, "Grain Growth in  $\text{MoSi}_2$  With and Without Deformation," Lehigh University, Bethlehem, PA, 1993.

A. Basu and A.K. Ghosh, "Grain Growth in  $\text{MoSi}_2$  With and Without Deformation," Intermetallics Conference, San Diego, CA, May, 1994.

J. Campbell, H. Chang, and R. Gibala, "Characterization of High-Temperature Deformation Substructures of  $\text{MoSi}_2$ ," MRS Fall Meeting, November, 1994.

C.M. Czarnik and R. Gibala, "Effects of Thin Films on the Plasticity of Single Crystalline  $\text{MoSi}_2$ ," TMS Fall Meeting, Cleveland, OH, October, 1995.

H. Chang, and R. Gibala, "Plasticity Enhancement of  $\text{MoSi}_2$  and  $\text{MoSi}_2$ -TiC Composites Through Elevated Temperature Prestrain," TMS Fall Meeting, Cleveland, OH, October, 1995.

S.M. Pickard and A.K. Ghosh, "Processing and Mechanical Properties of  $\text{MoSi}_2$  Reinforced Alumina Grown by Directed Melt Oxidation," 20<sup>th</sup> Annual Cocoa Beach Conference and Exposition on Composites, Advanced Ceramics, Materials and Structures, January, 1996.

\*R. Gibala, H. Chang, and C. Czarnik, "Plasticity Enhancement Mechanisms in  $\text{MoSi}_2$ ," International Conference on High Temperature Structural Silicides, Hyannis, MA, May, 1998.

\*R. Gibala, "Surface Film Effects in and Nano-indentation of  $\text{MoSi}_2$ ," Sandia National Laboratories, Livermore, CA, February, 1999.

\*R. Gibala, "Structural Silicides—An Overview," International School of Solid State Physics, Silicides: Fundamentals and Applications, Erice, Sicily, Italy, June, 1999.

\* R. Gibala, "Plasticity Enhancement Mechanisms in Structural Silicides," International School of Solid State Physics, Silicides: Fundamentals and Applications, Erice, Sicily, Italy, June, 1999.

### **Mechanical Behavior: NiAl-base Materials**

R. Gibala, H. Chang, C.M. Czarnik, K.M. Edwards, and A. Misra, "Plasticity Enhancement Mechanisms in Refractory Metals and Intermetallics," Conference on Critical Issues in the Development of High Temperature Structural Materials, Kona, HI, March, 1993.

\*R. Gibala, H. Chang, C.M. Czarnik, K.M. Edwards, and A. Misra, "Plasticity Enhancement Processes in NiAl and MoSi<sub>2</sub>," International Workshop on Ordered Intermetallics, Hangzhou, China, June, 1993.

R. Gibala, H. Chang, C.M. Czarnik, K.M. Edwards, and A. Misra, "Plasticity Enhancement Processes in NiAl and MoSi<sub>2</sub>," First International Symposium on Structural Intermetallics, Seven Springs, PA, September, 1993.

R. Gibala and A.K. Ghosh, "An Overview of AFOSR-MURI Research on Structural Metallic Materials at the University of Michigan," Congressional Staff Presentation, Washington, DC, May, 1995.

Z.L. Wu and R. Gibala, "Effects of Rhenium in NiAl and NiAl-Mo In situ Eutectic Composites," TMS Fall Meeting, Cleveland, OH, October, 1995.

\*R. Gibala, "Strong Interface Toughening of Intermetallics," TMS Fall Meeting, Cleveland, OH, October, 1995.

\*R. Gibala, "Strong Interface Toughening of Intermetallics," JIM/TMS Meeting, Honolulu, HA, December, 1995.

\*R. Gibala, "Deformation and Fracture of NiAl-base Eutectics," TMS Fall Meeting, Cincinnati, OH, October, 1996.

\*R. Gibala, "Design of Interfaces for Enhanced Plasticity," MRS Fall Meeting, Boston, MA, December, 1996.

Z. Wu and R. Gibala, "Effect of Rhenium Alloying on the Microstructures and Mechanical Properties of Directionally Solidified NiAl-Mo and NiAl-Cr Eutectics," MRS Fall Meeting, Boston, MA, December, 1996.

\*A. Misra, Z. Wu, and R. Gibala, "Deformation and Fracture Properties of NiAl-Base Eutectics," Fourth International Conference on High-Temperature Intermetallics, San Diego, CA, April, 1997.

\*R. Gibala, "Some Recent Experiments on Directionally Solidified Eutectics: From BCC Metals and Back Again," Los Alamos National Laboratory, T.E. Mitchell Symposium on Defects in Crystals, May, 1997.

\*R. Gibala, "Surface Film Softening as a Probe for Cleavage Fracture," TMS Fall Meeting, Indianapolis, IN, September, 1997.

A. Misra, Z. Wu, R. Noebe, B. Oliver, and R. Gibala, "Toughening Mechanisms in Directionally Solidified B2-NiAl-Based Eutectic Alloys," Second International Symposium on Structural Intermetallics, Seven Springs, Champion, PA, September, 1997.

\*R. Gibala, "In-situ Processed Intermetallic Matrix Composites." University of Michigan, MSE Colloquium Series, Ann Arbor, MI, April, 1998.

\*R. Gibala, "Mechanical Behavior of In-situ Processed Intermetallic Matrix Composites." University of Illinois, Department of Materials Science and Engineering, September, 1998.

\*R. Gibala, "Interface-Enhanced Plasticity," Sandia National Laboratories, Livermore, CA, October, 1998.

\*R. Gibala, "Interface-Enhanced Plasticity in Metallic Materials," University of California, Lawrence Berkeley Laboratories, Berkeley, CA, April, 1999.

\*A. Misra and R. Gibala, "Plasticity in Multiphase Intermetallics," ASM International Materials Congress, Cincinnati, OH, October, 1999.

R. Gibala, "Surface Film Softening Experiments as Input for Modeling of Deformation and Fracture," MRS Fall Meeting, Boston, MA, November, 1999.

\*R. Gibala, "Plasticity Enhancement Mechanisms in Quasi-Brittle Materials," University of Minnesota, Minneapolis, MN, February, 2000.

A. Misra and R. Gibala, "Towards Microstructural Design of Multiphase NiAl-base Intermetallics for Optimization of Toughness and Strength," TMS Annual Meeting, New Orleans, LA, February, 2001.

#### **Mechanical Behavior: Laminated and Model Interfacial Materials**

S.M. Pickard and A.K. Ghosh, "Toughened Microstructures for Ductile Phase Reinforced Intermetallics," MRS Fall Meeting, November, 1994.

S.M. Pickard and A.K. Ghosh, "Crack Growth Behavior and Process Zone Formation in Intermetallic Matrix Composites," ASM-TMS Fall Meeting, Rosemont, IL, October, 1994.

W. Provancher and A.K. Ghosh, "High Temperature Mechanical Behavior of Nb-Nb<sub>5</sub>Si<sub>3</sub> Laminates," MRS Fall Meeting, MA, November, 1994.

\*A.K. Ghosh, "Ductile Phase Toughening in Intermetallic Matrix Composite Laminates," Conference on the Mechanics and Physics of Layered and Graded Materials, Engineering Foundation, Davos, Switzerland, August, 1995.

S.M. Pickard and A.K. Ghosh, "Interface Shear Properties and Toughness in NiAl/Mo Laminates," 20<sup>th</sup> Annual Cocoa Beach Conference and Exposition on Composites, Advanced Ceramics, Materials and Structures, January, 1996.

W. Provancher, K. Tanada, and N. Kikuchi, "Measurement and Modeling of Mechanical Behavior of Intermetallic Matrix Composites from Nb-Si System," 20<sup>th</sup> Annual Cocoa Beach Conference and Exposition on Composites, Advanced Ceramics, Materials and Structures, January, 1996.

K. Kendig, D. Miracle, and R. Gibala, "Micromechanical Response of Ti-15-3/SCS-6 MMCs by Nanoindentations," Seventh Annual AeroMat Conference and Exposition (AeroMat '96), Dayton, OH, June, 1996.

S.M. Pickard and A.K. Ghosh, "High Rate Direct Deposition of Intermetallics Using PVD," Symposium on High Temperature Materials, ICCE/3, July, 1996.

S.M. Pickard and A.K. Ghosh, "Shear Strength and Toughness of NiAl/Mo Laminates," Symposium on High Temperature Materials, ICCE/3, July, 1996.

K. Kendig, D. Miracle, and R. Gibala, "Analysis of Residual Stressed Ti-Alloy, SCS-X MMCs by Nanoindentation," TMS Winter Meeting, Orlando, FL, February, 1997.

K. Kendig, R. Gibala, R. Shatwell, and D. Miracle, "Processing-Structure-Property Relationships for Carbon Coatings on Silicon Carbide Fibers in Metal Matrix Composites," TMS Annual Meeting, San Antonio, Tx., February, 1998.

K. Kendig, D. Miracle, R. Shatwell, and R. Gibala, "Processing-Structure-Property Relationships for Coatings on SiC Fibers," Gordon Research Conference on Physical Metallurgy, June, 1998.

K. Kendig, R. Gibala, R. Shatwell, and D. Miracle, "Microstructure and Mechanical Characterization of Carbon Coatings on SiC Fibers, TMS Annual Meeting, Nashville, TN, March, 2000.

K. Kendig, R. Gibala, R. Shatwell, and D. Miracle, "Microstructure and Mechanical Characterization of Carbon Coatings on SiC Fibers, Aeromat 2000 Conference and Exposition, Seattle, WA, June, 2000.

#### **Mechanical Behavior: Fatigue Related**

\*R.O. Ritchie, "Fatigue of Intermetallic and Ceramic Matrix Composites," FATIGUE '93, 5<sup>th</sup> International Conference on Fatigue, Montreal, Canada, May, 1993.

\*R.O. Ritchie, "Cyclic Fatigue-Crack Growth Behavior in Advanced Structural Materials: Properties of Monolithic and Composite Ceramics and Intermetallics," Department of Mechanical and Aerospace Engineering, University of California, Irvine, CA, May, 1993.

\*R.O. Ritchie, R.H. Dauskardt, and K.T. Vankateswara Rao, "Cyclic Fatigue-Crack Propagation in Advanced Ceramics and Intermetallics," Eighth International Conference on Fracture (ICF-8), Kiev, Ukraine, June, 1993.

\*R.O. Ritchie, "Fatigue Behavior in High-Temperature Ceramics and Intermetallics," Third International Symposium on Ultra-High Temperature Materials," Tajimi, Japan, December, 1993.

\*K.T. Venkateswara Rao, and R.O. Ritchie, "Fatigue-Crack Propagation and Fracture Behavior of Intermetallics and Their Composites," TMS Fall Meeting, Pittsburgh, PA, October, 1993.

M.T. Kush, J.W. Holmes, and R. Gibala, "Thermal Fatigue of MoSi<sub>2</sub> and a MoSi<sub>2</sub>-10 vol% TiC Composite," MRS Fall Meeting, Boston, MA, November, 1993.

M.T. Kush, J.W. Holmes, and R. Gibala, "Thermal Fatigue of MoSi<sub>2</sub> Particulate and Short Fiber Composites," MRS Spring Meeting, San Francisco, CA, April, 1994.

\*K.T. Venkateswara Rao and R.O. Ritchie, "Fatigue and Fracture of Intermetallics and Intermetallic-Matrix Composites at Elevated Temperatures," Symposium on The Relationship Between Microstructure and Damage Mechanisms, Society of Engineering, College Station, TX, October, 1994.

K.T. Venkateswara Rao and R.O. Ritchie, "Fatigue Crack Growth and Fracture Resistance of Intermetallic Alloys and Their Composites," ASM/TMS Materials Week, Rosemont, IL, October, 1994.

L.C. Shen, W.O. Soboyejo, K.T. Venkateswara Rao, R.O. Ritchie, and R.J. Lederich, "Fatigue and Fracture Behavior in Transformation Toughened MoSi<sub>2</sub>," ASM/TMS Materials Week, Rosemont, IL, October, 1994.

S.M. Pickard, A.K. Ghosh, and K.T. Venkateswara and R.O. Ritchie, "Tough Microstructures for Ductile Phase Reinforced Intermetallics," MRS Fall Meeting, Boston, MA, November, 1994.

\*K.T. Venkateswara Rao and R.O. Ritchie, "On the Fracture Toughness and Fatigue-Crack Propagation Behavior of Aluminide and Silicide Intermetallic-Matrix Composites at Elevated Temperatures," TMS Annual Meeting, Las Vegas, NV, February, 1995

\*R.O. Ritchie, "Damage Tolerance in Low Ductility Materials," Spring Meeting of the Japanese Institute of Metals, Tokyo, Japan, April, 1995.

K.T. Venkateswara Rao, and R.O. Ritchie, "Fatigue and Damage Tolerance in Structural Intermetallics and Ceramics at Ambient and Elevated Temperatures," 27<sup>th</sup> National Symposium on Fatigue and Fracture Mechanics, Williamsburg, VA, June, 1995.

\*R.O. Ritchie, "Damage Tolerance in Advanced Ceramics and Intermetallics," International Symposium on Advanced Ceramics for Structural and Tribological Applications, 34<sup>th</sup> Annual Conference of Metallurgists, Vancouver, BC, Canada, August, 1995.

\*R.O. Ritchie, "Crack Propagation in Metal-Matrix Composites: Mechanisms of Fatigue-Crack Growth," NATO Advanced Study Institute on Mechanical Behavior of Materials at High Temperatures, Sesimbra, Portugal, September, 1995.

\*K.T. Venkateswara Rao and R.O. Ritchie, "Microstructural Control for Fracture and Fatigue-Crack Growth Resistance in Intermetallics Alloys," 117<sup>th</sup> Meeting of the Japan Institute of Materials, Honolulu, HI, December, 1995.

K.T. Venkateswara Rao and R.O. Ritchie. "Fatigue and Fracture Properties of Aluminide and Silicide Intermetallics and their Alloys at Ambient and Elevated Temperatures." 117<sup>th</sup> Meeting of the Japan Institute of Materials, Honolulu, HI, December, 1995.

M.T. Kush, J.W. Holmes, and R. Gibala, "Thermal Fatigue Damage Accumulation in MoSi<sub>2</sub>- and NiAl-Based Composites," Conference on Composites, Advanced Ceramics, Materials and Structures, American Ceramic Society Meeting 1996, Cocoa Beach, FL, January, 1996.

K. Badrinarayanan, K.T. Venkateswara Rao, and R.O. Ritchie, "Fracture and Fatigue-Crack Growth Resistance of MoSi<sub>2</sub> Composites," TMS Annual Meeting, Anaheim, CA, February, 1996.

M.T. Kush, J.W. Holmes, and R. Gibala, "Microstructural Stability of Directionally Solidified Eutectics Under Static and Thermal Cycling Conditions," MRS Fall Meeting, Boston, MA, December, 1996.

M.T. Kush, J.W. Holmes, and R. Gibala, "The Effect of Stress on the Microstructural Stability of NiAl-Mo Eutectics During Thermal Fatigue." TMS Annual Meeting, San Antonio, TX, February, 1998.

\*R. Gibala, "Thermal Fatigue of NiAl Single Crystals," Sandia National Laboratories, Livermore, CA, November, 1998.

M.T. Kush, J.W. Holmes, and R. Gibala, "Thermal Fatigue of NiAl Single Crystals," MRS Fall Meeting, Boston, MA, December, 1998.

M.T. Kush, J.W. Holmes, and R. Gibala, "Microstructural Stability of a NiAl-Mo Eutectic Alloy," MRS Fall Meeting, Boston, MA, December, 1998.

R. Urbance, K. Kendig, M. Kush, and R. Gibala, "Thermal Fatigue of a NiAl-Cr Eutectic Alloy," MRS Fall Meeting, Boston, MA, December, 1998.

### **Experimental Infrastructure**

Although there have not been papers, theses, or presentations based solely on experimental equipment developed for this program, the facilities obtained as part of this program have been instrumental in effecting nearly all of the research output. Moreover, this experimental infrastructure will be enabling for much of our future research on intermetallics and other materials. A brief listing of the major program facilities obtained by a combination of AFOSR and University funds follows:

- A large-throughput PVD vacuum co-deposition unit for synthesis of laminates and surface coatings.
- A plasma-arc melt spinning unit for alloy synthesis.
- A hot isostatic press (HIP) unit for powder processing of intermetallics.
- Several Instron- and MTS-based high temperature vacuum mechanical testing and hot pressing units.

- A crystal puller, arc melting and induction melting facilities for preparation of single crystals and starting materials for other processing methods.
- A directional solidification furnace with temperature capability to above 2000°C for processing of high temperature eutectics, single crystals, and textured materials.
- Mechanical testing equipment - tension compression, fatigue, creep, thermal fatigue - and accessories, including specialized in-house fabricated techniques.
- A hot hardness tester for micro-hardness testing to 1300°C.
- A high temperature (to 1600°C) dilatometer.
- An ion-beam assisted deposition (IBAD) system for synthesis of microlaminates and surface coatings (partial purchase).
- An ion mill for preparation of specimens for transmission electron microscopy (TEM) observations (partial purchase).
- Computers and workstations for modeling investigations.
- Major accessories for TEM and SEM units used in the program research.
- Optical microscopes and metallographic polishers for conventional optical metallography conducted.
- Nanoindentation and microindentation systems for high resolution hardness measurements of fine microstructures (includes AFM accessories).

**IV. OTHER PERTINENT DATA (INTERACTIVE ACTIVITIES)**  
(For space consideration, some of the following are partial listings only.)

**A. Personnel**

The research personnel directly involved and/or supported over the entire research period of 5/01/93 – 4/30/00 include the following:

1. Faculty: Ronald Gibala  
Amit K. Ghosh  
John W. Holmes  
Noboru Kikuchi  
Robert O. Ritchie\*  
David J. Srolovitz
  
2. Research Associates/Engineers: Hugo Chang  
M.-H. Hong\*  
Tao Hong  
J.M. McNaney\*  
Sion Pickard  
James Raynolds  
K.T. Venkateswara Rao\*  
Xin Wu  
Zili Wu  
Hongyan Zhang
  
3. Collaborators: Daniel Miracle<sup>x</sup>  
Ronald Noebe<sup>\*\*</sup>  
Jeffrey M. Rickman<sup>\*\*\*</sup>  
Eric Roddick<sup>\*\*\*\*</sup>  
John R. Smith<sup>\*\*\*\*</sup>
  
4. Graduate Student Research Assistants: K. Badrinarayanan\*  
Ajoy Basu  
Hugo Chang  
Cory Czarnik  
Hiroshi Endo\*  
Matthew Fox  
Daerk Golanski  
Kevin Kendig  
Matthew T. Kush  
Andrea McKelvey\*  
Kevin Mukai  
Keith Patrick

William Provancher  
Sridhar Narayanaswamy  
Kenjiro Terada  
Wuhua Yang

5. Undergraduate Student Assistants: Josh Campbell  
Brian Dunaway  
Laura Ely  
Robert Hutchinson  
Marcus Sprow  
Randall Urbance  
Brian Wishnow<sup>xx</sup>

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- \* University of California, Berkeley  
\*\* NASA Lewis Research Center, Cleveland, OH  
\*\*\* Lehigh University  
\*\*\*\* General Motors Technical Center, Warren, MI  
x Wright Laboratories, WPAFB  
xx Johns Hopkins University

## B. Participation/Presentations

See Part III, Section C, which contains an extensive list of meetings and conferences at which talks were given. Talks given at AFOSR grantee/contractor meetings are not included.

## C. Consulting/Advisory/Collaborations

- R. Gibala: Los Alamos National Laboratory, consultant, 1992-1995  
Wright Laboratories, WPAFB, research collaborator, 1995-2000  
University of Virginia, departmental reviewer, 1998  
Sandia National Laboratories, visiting researcher, 1998-1999  
Kansas State University, research reviewer, 1999  
Colorado State University, research reviewer, 1999  
Lawrence Berkeley National Laboratory, program reviewer, 1999-2000
- A.K. Ghosh: Oak Ridge National Laboratories, consultant, 1994-1995  
Wright Laboratories, WPAFB, visiting scientist, 1997-1998
- D.J. Srolovitz: ARPA Defense Research Science Council, consultant, 1988-1996
- N. Kikuchi: SES Executive Committee on Computational Mechanics, 1981-present  
IACM Int. Advisory committee, 1992-present
- R. O. Ritchie: President, International Congress on Fracture, 1997-2001  
Associate Editor, Journal of Mechanics of Materials, 1987-present  
Associate Editor, Journal of Engineering Failure Analysis, 1994-present  
Consulting: Applied Materials, General Motors, Northrop-Grumman,  
General Electric Aircraft Engines and several others, 1993-present.

#### **D. Transitions:**

R. Gibala and J.W. Holmes: thermal fatigue of Mo/Si<sub>2</sub>-SiC particulate composites have been explored as a possible new material for high-temperature furnace elements.

R. Gibala: research on plasticity enhancement by surface films and second-phase microstructures has been examined for possible use in the design of advanced NiAl-based high temperature engines.

A.K. Ghosh and R. Gibala: on-going collaboration with Wright Laboratories, WPAFB, on, respectively, deformation processing (L. Semiatin), and interface strength measurements (D. Miracle).

N. Kikuchi: application of homogenization methods and digital image-based modeling techniques by laboratories over the entire world as one of the most advanced FEA techniques available.

D.J. Srolovitz: collaboration with Biosym/MSI to implement iterative computational method for first-principles calculations into commercial codes.

D.J. Srolovitz: computer simulation techniques for fracture paths and microstructural stability have been utilized by many industrial, government, and university laboratories (Exxon, General Electric, Los Alamos National Laboratory, Lawrence Livermore National Laboratory, Carnegie Mellon University).

R.O. Ritchie's analyses of fatigue crack growth behavior of materials have been used and cited extensively throughout the world.

#### **E. New Discoveries/Inventions/Patents**

These have been listed in other sections, including the Executive Summary which contains an extensive listing of new scientific results and discoveries. Examples include first-principles interactive computational techniques, new methods for interface strength measurements, new high toughness IMCs (Mo/Si<sub>2</sub>-Nb mesh), Mo/Si<sub>2</sub>-SiC<sub>p</sub> composites for furnace windings, simulation techniques for deformation, fracture, and microstructural stability.

#### **F. Honors/Awards (partial listing since 1992)**

R. Gibala: Peter Winchell Memorial Lecturer, Purdue University, 1992  
B.T. Matthias Fellow, Los Alamos National Laboratory, 1992  
NASA Outstanding Paper Award, Materials Science Division, 1992  
Distinguished Merit Award, University of Illinois, 1998  
Frances E. Van Vlack Professorship, University of Michigan, 1998  
Life Member, ASM International, 1998  
President, Materials Research Society, 1999

A.K. Ghosh: ASM International, Fellow, 1993

- J.W. Holmes: ASM International. Henry Marion Howe Medal. 1992
- N. Kikuchi: Best Paper Award, ASME, Design and Automation, 1993.
- R.O. Ritchie: Rosenhain Medal, Institute of Materials, 1992  
Top 25 Most Cited Researchers in Materials Science, 1995  
Distinguished Structural Materials Scientists/Engineer Award,  
TMS-AIME, 1996  
Van Horn Distinguished Lecturer, Case Western Reserve University,  
1997  
President, International Congress on Fracture (ICF), 1997  
Southwest Mechanics Series Lecturer, 1997-98  
J. Testing & Evaluation, Most Outstanding Article Award, ASTM, 1998  
C.J. Beevers Memorial Lecturer, International Fatigue Congress  
(IFS), 1999
- D.J. Srolovitz: ALCOA Foundation Fellow, 1992  
Michael Visiting Fellow, Weizmann Institute of Science, 1993-1994  
ASM International, Research Silver Medal, 1994  
NASA Technical Transfer Award, 1995  
Edward deMille Campbell Professorship, University of Michigan, 1997  
American Institute of Chemical Engr., Outstanding Paper Award, 1997  
Fellow, ASM International, 1998  
Fellow, Institute of Physics (Great Britain), 1999.
- N. Sridhar: Materials Research Society, Graduate Student Research Award, 1995