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# COMPATIBILITY OF POTENTIAL REINFORCING CERAMICS WITH Ni and Fe ALUMINIDES

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In fulfillment of grant contract #NAG 3942

This report summarizes the accomplishments achieved during grant #NAG 3942. The original objective of this research was to examine the structure and chemistry of the interfacial region created between a variety of ceramic reinforcing phases and NiAl and FeAl matrices.

(NASA-CR-192232) COMPATIBILITY OF  
POTENTIAL REINFORCING CERAMICS WITH  
Ni AND Fe ALUMINIDES Final  
Technical Report (Ohio State  
Univ.) 6 p

N93-18784

Unclas

G3/27 0146045

## INTRODUCTION

There is a great deal of interest in the possible utilization of intermetallic compounds in advanced high temperature gas turbine engines [1,2]. These compounds exhibit a variety of promising properties, including reasonable strength, high melting points, relatively low densities, and good corrosion resistance [3]. However, in general, they also show limited ductilities and toughness, and less than optimum creep strengths at elevated temperatures. In addition, in applications involving advanced gas turbine engines, it is often necessary for candidate materials to have large elastic moduli.

To overcome these limitations, attention has been focussed on the possible use of composite materials. Aluminide phases are particularly attractive for high temperature applications because of their combination of high strength and excellent oxidation and corrosion resistance. It should be possible to further enhance the mechanical properties of these alloys at elevated temperatures by the incorporation of high strength ceramic fiber(s), to form a composite microstructure. However, in order to achieve this, it is first necessary to identify those metal/ceramic combinations which will bond strongly to one another without the formation of any gross reaction product(s). Misra [4-6] has carried out thermodynamic calculations in order to predict which ceramic reinforcements would be stable in an aluminide environment. Combinations of a binary aluminide matrix with binary carbides, borides, silicides, nitrides and oxides were considered, and the activities of the four constituent elements used to predict which binary products would be stable at a given temperature. While this represents an attempt to predict which composite systems might be viable, two limitations should be noted. Firstly, only binary products were considered; in such systems many ternary compounds may be possible. Secondly, the effect of reaction kinetics was neglected; these may lead to the formation of metastable phases.

The present study is part of a program whose objective is to identify a high temperature fiber reinforced composite. The approach adopted in this work was to fabricate laboratory samples of the combinations of materials considered by Misra, in order to determine the extent to which the thermodynamic calculations can predict phase stability. As many of the ceramic phases considered are not currently available in fiber form, they were added as particulates to the alloy matrices. This report describes the ways in which the materials were produced and evaluated.

## BACKGROUND

When ceramic reinforcements are added to intermetallic compounds, there are two principal areas of concern. Firstly, it is generally considered that the interfacial region between the matrix and reinforcing phase exerts a dominating influence on the mechanical properties of the composite. It is, therefore, important that the two phases in question are thermodynamically compatible, so that during prolonged thermal exposure embrittling reaction zones do not form [6]. Also, in order for the ceramic reinforcement to increase the modulus, there must be a reasonable bond established between this phase and the matrix. Finally, in order to retain reasonable toughness, this bond should not be too strong, so that an advancing crack can be deflected along the interface rather than through the ceramic

phase.

The primary objective of this project was to examine the compatibility of candidate ceramic reinforcement materials with intermetallic matrices suitable for high temperature composite systems. Powders of FeAl and NiAl were mixed with ceramic powders, consolidated, followed by hot isostatic pressing and subsequent heat treatment. The microstructures of these composites, and the nature of the ceramic/matrix interfaces, were assessed using a variety of electron-beam techniques, so as to determine chemical and phase stability of the intermetallic matrix with the various ceramic particles. The microstructural and chemical analyses of these systems were compared to the thermodynamic calculation.

## PROCESSING

Commercially pure ceramic powders with a particle size of approximately 44  $\mu\text{m}$  were obtained from Cerac Inc. In all, eighteen different powders were used and these had the following stoichiometry compositions;

Oxides;	HfO <sub>2</sub> , CeO <sub>2</sub> , Y <sub>2</sub> O <sub>3</sub> , Al <sub>2</sub> O <sub>3</sub> , La <sub>2</sub> O <sub>3</sub> , Sc <sub>2</sub> O <sub>3</sub> , CaZrO <sub>3</sub> .
Carbides;	TiC, ZrC, B <sub>4</sub> C.
Nitrides;	AlN, TiN, ZrN, HfN, BN.
Borides;	TiB <sub>2</sub> , HfB <sub>2</sub> .

Each of these powders was blended with powders of FeAl (60 at.% Fe) and NiAl (50 at.% Ni) in a 9:1 volume ratio of aluminide and ceramic. Heptane was added to the mixture to produce a slurry and promote homogeneity throughout processing. Approximately 10 grams of each slurry mixture was packed into a stainless steel fabricating canister, 6 mm in diameter and 15 cm in length, which was sealed at one end. The canisters were evacuated, then heated to promote evaporation of the heptane and any residual moisture, and finally sealed by crimping and welding the open ends.

All of the sample canisters were then hot isostatically pressed (HIP-ed) at a pressure of 140 MPa. The HIP-ing of the FeAl and NiAl based samples was performed at 980 °C for 3 hours and 1250°C for 4 hours, respectively. Optical examination of the hot pressed composites was used to determine the optimal processing parameters necessary to obtain a completely dense microstructure. Those systems that showed no gross reaction products were then heat treated (HT) in an argon atmosphere. The FeAl and NiAl based samples were held for 25 hours at 1225°C and 1425°C, respectively.

## CHARACTERIZATION

Sections of the HIP-ed samples were cut from various portions of the canisters and mounted for optical and scanning electron microscope (SEM) examination. The specimens were mounted in epoxy resin, ground, polished, and examined unetched using optical microscopy. They were also examined in a JEOL 820A scanning electron microscope at an accelerating voltage of 25kV. Energy dispersive X-ray spectroscopy (EDS) was used in the SEM for compositional analysis. For the lighter elements, compositional analysis was

performed by wavelength dispersive X-ray spectroscopy (WDS) on a Cameca SX-50 electron microprobe microanalysis (EPMA) system.

The samples were categorized according to extent of any reaction which had occurred between the particle and a given matrix as being;

- a) highly reactive,
- b) partially reactive, and
- c) non-reactive.

The "highly reactive" systems are those in which some distinct reaction phase(s) is formed either during HIP-ing or during the subsequent heat treatment. Several combinations exhibited this behavior: for example, the system FeAl/B<sub>4</sub>C produced a number of reaction products during HIP-ing. The complex nature of these products and their interactions with the surrounding phases are expected to reduce the effectiveness of the ceramic reinforcement and so these highly reactive systems are not considered desirable in the present context. In the "non-reactive" systems, no reaction products or interdiffusion of constituent elements resulting from processing or heat treatment could be detected in the present study. For example, this behavior was exhibited by the Al<sub>2</sub>O<sub>3</sub> particles in both the FeAl and NiAl matrices. In all such cases the ceramic particles did not adhere well to the matrix, and tended to fall out of the sample. Clearly, with no interfacial bond between the particles and matrix, there will be no reinforcing effect and hence these systems are also unsatisfactory for the contemplated applications. In the "partially reactive" systems there is some compositional or structural change but no large scale, third phase, reaction product. These systems appear to be the most promising composites as they probably bond to one another while retaining the inherent properties for which they were chosen.

The partially reactive systems were then characterized in more detail using transmission electron microscopy (TEM). Specimens were prepared by cutting 3 mm diameter discs from the heat treated samples, mechanically polishing and ion milling to perforation using 6kV Ar ions at room temperature. All TEM experiments were performed on a JEOL 200CX at an accelerating voltage of 200 kV and a Philips CM-12 at 120 kV.

CANDIDATE PARTICULATE	PREDICTED FORMATION	OBSERVED REACTION
CeO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub> /Al <sub>2</sub> Ce	Yes
Al <sub>2</sub> O <sub>3</sub>	None	None
Y <sub>2</sub> O <sub>3</sub>	None	None
Sc <sub>2</sub> O <sub>3</sub>	None	None
HfO <sub>2</sub>	None	None
La <sub>2</sub> O <sub>3</sub>	None	Yes
CaZrO <sub>3</sub>	Al <sub>2</sub> O <sub>3</sub> /Al <sub>2</sub> Zr	None
TiC	None	None
ZrC	None	None
B <sub>4</sub> C	FeB/C	Yes
TiB <sub>2</sub>	AlN/Al <sub>2</sub> Zr	None
ZrB <sub>2</sub>	None	None
HfB <sub>2</sub>	None	None
HfN	None	None
AlN	None	None
TiN	None	None
BN	None	Yes
ZrN	AlN/Al <sub>2</sub> Zr	None

### SUMMARY

A large range (18) of ceramic compounds have been assessed as candidate reinforcement materials for the intermetallic aluminides FeAl and NiAl; more complete details have been published elsewhere [7-9]. A summary of the predicted and experimentally examined microstructures are displayed in the Table above. In general, this Table indicates good agreement between the predicted microstructures and the observations made in this study. The samples were prepared by powder metallurgy techniques and provided representative microstructures with which to evaluate the compatibility of the reinforcing material with the intermetallic matrix. Microscopic observations made of the matrix/reinforcement interface regions were compared to theoretical predictions of the

thermodynamic compatibility of the various phases with these two matrices; the interfaces were classified as either being partially or not reacted, depending on whether the presence of a reaction zone was detected. In general, highly or partially reacted samples were rejected from further consideration because the reaction zones are usually considered to be brittle, and therefore limit the potential application of that particular candidate sample.

### ACKNOWLEDGEMENTS

The authors would like to thank Drs. H. Gray and S. Draper for helpful discussions and H. Colijn for expert technical assistance. This work was funded by NASA-Lewis Research Center under contract #NAG 3942.

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