PROCESSING AND PROPERTIES OF FeAl-BONDED COMPOSITES

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Abstract

Iron aluminides, which exhibit excellent oxidation and sulfidation resistance, are thermodynamically compatible with a wide range of ceramics such as carbides, borides, oxides, and nitrides, which makes them suitable as the matrix in composites containing fine ceramic particulates. Potential applications for these new materials will be outlined. For ceramic contents varying from 30 to 60 vol.%, composites of Fe-40 at. % Al with WC, TiC, TiB₂, and ZrB₂ were fabricated by conventional liquid phase sintering of powder mixtures. For ceramic contents from 70 to 85 vol.%, pressureless melt infiltration was found to be a more suitable processing technique. In FeAl-60 vol.% WC, flexure strengths of up to 1.8 GPa were obtained, even though processing defects consisting of small oxide clusters were present. Elimination of such defects, and improvement in the interfacial bonding strength are needed to further improve the strength of these materials. Room temperature fracture toughnesses were determined by flexure testing of chevron-notched specimens. FeAl/WC and FeAl/TiC composites containing 60 vol.% carbide particles exhibited K_{Ic} values around 20 MPa m^{1/2}. These values are similar to those found in WC/Co hard metals with similar binder volume fractions. In view of the relatively brittle behavior of monolithic iron aluminides this result is surprising. It is rationalized by the ductile necking and debonding of the thin (typically 1 μ m) FeAl ligaments observed on the fracture surfaces. Slow crack growth measurements carried out in water and in dry oxygen suggest a relatively small influence of water-vapor embrittlement. It appears therefore that the mechanical properties of iron aluminides in the form of fine ligaments are quite different from their bulk properties. Further study will be needed to fully understand the relevant mechanisms. Measurements of the oxidation resistance, dry wear resistance, and thermal expansion of iron aluminide composites suggest many potential applications for these new materials.

Introduction

Iron aluminide intermetallics such as Fe₃Al and FeAl (1,2) offer several useful features making them of interest in composite or cermet applications, in particular when compared to more common matrix alloys based on Co or Ni. To start with, iron aluminides use readily available and inexpensive elements. The density of iron aluminides is relatively low. For example, the density of Fe-40 at. % Al is only 6 Mg/m³. Iron aluminides are also very resistant to oxidation, sulfidation, and molten nitrate salts (3). For sufficiently high Al concentrations (≥ 30 at. %) they are not ferromagnetic, which makes them useful in die applications for magnetic materials. Unlike Co or Ni, they are not perceived to be carcinogenic. Therefore, they are likely to be more environmentally benign than those materials. In addition, Co is undesirable in radioactive environments, because it decays into reaction products with long half lives. Iron aluminides are therefore well suited as an alternative binder for cermets used in hostile environments.

However, the successful processing of iron aluminide bonded composites requires several conditions to be met. The iron aluminides and respective ceramics have to be thermodynamically compatible not only in the solid state, but also when the iron aluminide is liquid during processing. Successful liquid phase sintering requires also that the reinforcing particulates are wetted well by liquid iron aluminides. In particular, for high ceramic volume fractions, the solubility of the ceramics in the liquid iron aluminide must be high, or excessive porosity may be encountered. While the first two requirements are met for several FeAl/ceramic systems, the solubilities will be seen to be relatively low. This problem may be overcome by a newly developed pressureless melt infiltration technique.

In order to provide a better understanding of this new class of iron aluminide composites and cermets, we will address the following points in this paper:

- Thermodynamic compatibility and wetting behavior

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- Conventional liquid phase sintering of mixtures of FeAl and ceramic powders, and the resulting mechanical properties
- Processing via a one-step pressureless melt infiltration technique and the resulting mechanical properties
- Resistance against crack growth, oxidation, and wear
- Thermal expansion.

We would also like to note that a similar, and complimentary, class of composites containing an Ni₃Al binder rather than an FeAl binder has been patented in 1990 by Tiegs and McDonald (4) and has formed the subject of several investigations (5-7).

Experimental Procedure

The iron aluminide used in this research was prealloyed, argon atomized -325 mesh powder (particle size $\leq 45 \mu\text{m}$) with the composition Fe-40 at. % Al. Unless otherwise stated, the TiC and WC powders ranged from 2 to $10 \mu\text{m}$ in size. The TiB₂ and ZrB₂ powders were -325 mesh. Metallographic sections of the boride composites showed the boride particles to be in the 5 - $10 \mu\text{m}$ size range.

In one processing approach, ceramic composites were produced by mixing prealloyed Fe-40 at. % Al powder (Fe40Al or FeAl for brevity) with the desired ceramic powder, placing the mixture into an Al₂O₃ crucible, heating the uncovered crucible within approximately 5 ks to a temperature of 1723 K in a vacuum of 10^{-4} Pa, holding for 900 s at this temperature, and finally cooling in the furnace. In the infiltration approach to processing, FeAl powder was placed onto cold-pressed ceramic compacts. The infiltrations were carried out in vacuum in covered alumina crucibles with a titanium getter placed in the crucible. Typical processing conditions were 1723 K for 0.5 h. Some composites were also hot isostatically pressed (0.5h/1373 K/200 MPa) after liquid phase sintering or melt infiltration. Metallographic cross sections were prepared by standard techniques and examined in optical and scanning electron microscopes (SEM). Transmission electron microscope (TEM) specimens were prepared by ion beam milling and examined by energy dispersive analysis using a thin-window detector. Bend bars for flexure strength measurements and chevron-notched specimens for fracture toughness measurements with dimensions $3 \times 4 \times 25 \text{ mm}$ were fabricated by electro-discharge machining and grinding. The triangular sections in the center of the chevron-notched specimens had a width of 3 mm and a height of 2.5 mm. Flexure strength and fracture toughness were measured in three-point bending with a span L of 20 mm and a crosshead speed of $10 \mu\text{m/s}$. The flexure strength, σ_f , was determined from the load P_f at fracture, the specimen height h , and the specimen width w as:

$$\sigma_f = 1.5 \times (L P_f) / (w h^2). \quad (1)$$

The fracture toughness, K_{Ic} , was determined from the energy, W , absorbed during the tests, the area, A , swept out by the crack, and the plane strain Young's modulus, $E' = E / (1 - \nu^2)$, as:

$$K_{Ic} = [(W/A) E']^{1/2}. \quad (2)$$

The E' values for the composites were calculated from the Young's modulus of the iron aluminide matrix, E_m , and the Young's moduli of the respective ceramic particulate, E_p , using Ravichandran's (8) eqn. (8) with a value of $\nu = 0.2$ for Poisson's ratio:

$$E' = \frac{(cE_p E_m + E_m^2)(1 + c)^2 - E_m^2 + E_p E_m}{(cE_p + E_m)(1 + c)^2}, \quad (3)$$

where

$$c = (1/V_p)^{1/3} - 1 \text{ with } V_p + V_m = 1. \quad (4)$$

and V_p and V_m are the volume fractions of the ceramic and the iron aluminide, respectively.

Thermodynamic Compatibility and Wetting

Recent thermodynamic calculations by Misra (9) show iron aluminides to be thermodynamically compatible with a wide range of ceramics at 1273 K. These ceramics are listed in Table I. WC was not included in Misra's calculations. Also, his calculations did not address the question whether these ceramics would be stable in liquid FeAl (the melting point of Fe-40 at. % Al is 1690 K). To date, the compatibility of the following ceramics with liquid iron aluminide has been examined: TiC, WC, TiB₂, and ZrB₂. In none of these systems did SEM examinations of the microstructures obtained after liquid phase sintering suggest any reactions (10). This finding was substantiated by X-ray diffraction (with the exception of ZrB₂, where a small volume fraction of FeBO₃ may have formed) (11). Consistent with the absence of significant reactions, TEM examination of a liquid phase sintered FeAl/TiB₂ composite (Fig. 1) did not show any evidence for reaction products. In the TiB₂, no Fe or Al was detected, whereas the FeAl contained some B and Ti. This suggests some solubility of TiB₂ in FeAl.

Thermodynamic calculations using the code ThermoCalc[®] were also carried out. As an example, the calculated solubility of TiC in liquid FeAl at 1723 K is shown in Fig. 2. The solubility is low ($< 1 \text{ at. \%}$), and the Ti and C concentrations in the liquid FeAl depend on the stoichiometry of the TiC, which can vary within wide limits. Similar calculations for WC (12) and TiB₂ indicate also small solubilities ($< 2 \text{ at. \%}$). In summary, the calculations and experiments carried out to date for liquid FeAl containing

Table I. Compilation of ceramics which, according to thermodynamic calculations, are stable in contact with FeAl at 1273 K (9).

Carbides	Borides	Oxides	Nitrides
HfC	HfB ₂	Al ₂ O ₃	HfN
TiC	ScB ₂	BeO	
ZrC	TiB ₂	La ₂ O ₃	
	ZrB ₂	Sc ₂ O ₃	
		Y ₂ O ₃

Fig. 1 - TEM micrograph of Fe-40 at. % Al/TiB₂ composite

Fig. 2 - Calculated section of Fe-40 at. % Al/TiC phase diagram at 1723 K.

TiC, WC, TiB₂, or ZrB₂ suggest little or no reaction, and low solubilities of the ceramics in the liquid FeAl. These systems are thus quite different from the Co/WC system, in which liquid Co dissolves substantial quantities (10-20 at. %) of WC. This difference has to be taken into account in the processing of iron aluminide composites and cermets.

In addition to thermodynamic stability, the liquid phase sintering approach to the fabrication of iron aluminide-ceramic composites requires good wetting, i.e., low wetting angles. In the case of the carbides and borides, this requirement seems to be fulfilled. For example, a small amount of FeAl was placed on a 97% dense polycrystalline TiC substrate and heated to 1723 K in vacuum. The wetting angle appeared to be small, typically 20°. The FeAl penetrated the grain boundaries of the TiC to a depth of approximately 100 μm, which is also an indication of good wetting. However, because of the grain boundary penetration, a reliable evaluation of the liquid-solid wetting angle was not possible. TiC single crystals would be required in order to measure wetting angles reliably.

Processing by Liquid Phase Sintering of Mixed Powders

The liquid phase sintering (LPS) of mixtures of prealloyed iron aluminide powders and ceramic powders is very successful as long as the ceramic volume fractions are not too high. Near-net fabrication is readily accomplished and is illustrated in Fig. 3 (11). Figure 4 shows the microstructure of an FeAl/WC composite. While almost complete densification was achieved in this example, it is generally not reached for higher WC volume fractions. For example, with 60 vol. % WC, 98 to 99% of the theoretical density (TD) is reached, whereas only 92% TD is reached for 70 vol.% WC (11). The hardness measurements in Fig. 5 illustrate this limitation. As soon as the ceramic volume fraction exceeds some critical value (which seems also to depend on the type and/or particle size of the ceramic), the hardness drops significantly due to excessive porosity. The porosity is due to two reasons. First, the prealloyed iron aluminide powder is relatively coarse. For example, a large FeAl particle surrounded by fine carbide particles, once liquid, will infiltrate these particles and may leave a void behind. Second, the solubility of the ceramic particles in the liquid FeAl is relatively low. This makes it difficult to enhance the particle packing by dissolving material away from touching carbides. This limitation might in principle be overcome by increasing the processing temperature or the sintering time. However, Al tends to evaporate from liquid FeAl held in vacuum, resulting in a gradual shift of the iron aluminide composition to more iron-rich compositions (11).

Fig. 3 - Near-net shaped Fe-40 at. % Al/50 vol. % ZrB₂ iron aluminide composite.

Fig. 4 - SEM micrograph of a metallographic section through Fe-40 at. % Al/60 vol. % WC fabricated by liquid phase sintering of mixed powders.

Fig. 5 - Rockwell A hardnesses of composites consisting of Fe-40 at. % Al and TiC, WC, TiB₂, or ZrB₂. A scatterband depicts the hardnesses of WC/Co materials. Unless indicated otherwise, the materials were processed via liquid phase sintering of mixed powders.

Other processing issues in the LPS of mixed powders concern the reproducibility of microstructure and properties, the presence of residual flaws, and the effect of heat treatment. Figure 6 illustrates the three-point flexure strengths of FeAl/WC composites. Nominally identical processing by LPS does not guarantee identical strengths: the two bar clusters annotated LPS on the left-hand side of the graph show quite different flexure strengths. In an attempt to remove any residual porosity, additional consolidation by hot isostatic pressing (LPS+HIP) was carried out. This resulted in degradation of the average flexure strength. Although one bend bar exhibited a high strength close to 1800 MPa; the other specimens were much weaker. The large scatter in the data indicates a wide spread in the flaw size distributions of the specimens. Interestingly, when the LPS and HIP processing was followed by an anneal for 1 day at 773 K in vacuum, the scatter was reduced, 2 out of 5 bars exhibited a flexure strength close to 1800 MPa, and the average strength increased to (1747 ± 56) MPa. This can be understood as follows: the HIP schedule involved a fairly fast cool from the 1373 K HIPing temperature (about 1000 s). Since iron aluminides are known to contain large concentrations of thermal vacancies (e.g., 1 at. % at 1273 K), it is likely that a substantial concentration of vacancies was frozen in during the fast cool-down. These vacancies increase the yield strength, and reduce the fracture toughness (13). This could very well lead to a reduction in the flexure strength due to increased flaw sensitivity. The subsequent anneal at 773 K most likely reduced the yield strength of the FeAl matrix, while increasing its ductility and fracture

toughness. The expected improvement in the flaw tolerance of the composite may be responsible for the improved flexure strengths and decrease in the scatter.

Fig.6 - Flexure strength of Fe-40 at. % Al/60 vol.% WC composites. LPS = liquid phase sintering; HIP = hot isostatic pressing; 773 K refers to a 1 day anneal at this temperature.

As has been noted before (12), the FeAl/WC composites processed by LPS contain clusters of oxide inclusions which may limit their strength. These clusters are also found after HIPing. For example, Fig. 7 shows a cluster of aluminum oxide particles found on a fracture surface of HIPed FeAl/WC. This illustrates the difficulty in using alumina-forming matrix materials such as FeAl as binders in cermets: whereas alumina formation on the surface of finished parts is desirable in service, it is a disadvantage during processing.

Fig. 7 - Cluster of aluminum oxide inclusions on fracture surface of Fe-40 at. % Al/60 vol.% WC material. Some Al_2O_3 inclusions are indicated by arrows.

In recent work by Thomas (14), FeAl/WC cermets were fabricated using much more elaborate powder-metallurgical techniques than those employed in the present work. Prealloyed Fe-40 at. % Al powder was blended with 61 vol.% (80 wt%) WC powder in isopropanol, compacted in uniaxial compression, cold isostatic pressed, liquid phase sintered in vacuum, and, in the last step, gas-pressure sintered (1.9 MPa/1768 K). In spite of the involved processing, these materials contained residual pores. The nature of these pores was not further identified. In spite of the possibility of internal pores, the 4-point flexure strength of this material reached a high value of 1800 MPa, which is similar to the values obtained in our three-point tests.

Processing by Pressureless Melt Infiltration

As pointed out before, the relatively low solubility of ceramics in FeAl is a limitation in the processing of iron aluminide composites. Subramanian et al. (15) recently devised a one-step pressureless infiltration technique to overcome this limitation. The process is shown schematically in Fig. 8. A TiC preform is prepared by cold-pressing and FeAl powder is placed on top of it. Once the iron aluminide becomes liquid during heat-up in a vacuum furnace, it infiltrates the TiC preform completely. Because this process avoids the incorporation of large FeAl particles in the preform (which may give rise to porosity), and because considerable particle rearrangement may occur as the liquid penetrates the preform, composites with very high ceramic volume fractions may be fabricated in this manner. Figure 9 illustrates the microstructure of a FeAl/TiC composite processed in this way, with a nominal TiC volume fraction of 80%. Such composites exhibit typically 99% of the theoretical density. The flexure strengths for such high ceramic volume fractions are somewhat lower than those for 60 vol. %. For example, Subramanian et al. (15-17) obtained flexure strengths of 1030 and 1200 MPa in FeAl composites containing 70 vol.% TiC or WC, respectively. However, the hardnesses of these materials are very high and fall in the range of Co-bonded WC (see Fig. 5). Almost fully dense FeAl/TiB₂ composites containing 70 vol.% TiB₂ have also been fabricated and exhibit Rockwell A hardnesses in the vicinity of 90.

The particular melt-infiltration technique employed here has several attractive features. First, a separate sintering step to make a preform prior to infiltration is not required: the composite is fabricated in a single heating cycle. Second, the infiltration is presumably accompanied by less oxide formation than the sintering of mixed powders, since the iron aluminide advances through the preform as a liquid front.

Fig. 8 - Schematic representation of one-step pressureless melt infiltration technique.

Fig. 9 - SEM micrograph of melt-infiltrated Fe-40 at. % Al/80 vol. % TiC.

Crack Growth Resistance

Figure 10 shows the fracture toughnesses of FeAl/WC composites containing different WC volume fractions (10). As expected, the fracture toughness decreases as the WC volume fraction increases. The literature on cemented carbides (18,19) suggests that the toughening is to a large extent determined by the ductile deformation and energy absorption of ligaments of the binder phase. For a given

volume fraction, the toughening depends on the plastic parameters of the matrix such as yield strength, work hardening, ultimate tensile strength, and strain to fracture. Iron aluminides with the B2 structure are usually considered to suffer from low ductility. Their ductility in air rarely exceeds 10%. For a given volume fraction of ceramic particulates, one would therefore expect the toughening due to iron aluminides to be significantly less than that due to cobalt. Approximate values for the fracture toughnesses of WC/Co with different Co volume fractions were taken from the data of Sigl et al. (18) and the compilation of Ravichandran et al. (19), which lists the results of many different authors using different testing techniques. These values were plotted in Fig. 10. Surprisingly, Fig. 10 shows that the fracture toughnesses of FeAl composites and WC/Co are of comparable magnitude. Also, fracture surface observations show that the FeAl matrix undergoes debonding accompanied by extensive plastic deformation as illustrated in Fig. 11. While the high ductility is in qualitative agreement with the high fracture toughnesses, the interfacial debonding suggests limited interfacial strength. The interfacial strength may thus limit the flexure strength of iron aluminide-bonded cermets.

Fig. 10. Fracture toughness of FeAl/WC composites as a function of the WC volume fraction. Representative WC/Co values are indicated.

Another surprising result is shown in Fig. 12. Slow crack growth measurements were carried out with an Fe40Al-60 vol. % TiC composite in either water or in dry oxygen. Iron aluminides are very sensitive to water vapor as first discovered by Liu et al. (20). Lynch and Heldt (21) report a tensile ductility in dry oxygen or in vacuum of 9%, which decreases to 1% for a relative humidity of 100%. Although Fig. 12 shows a decrease in the energy absorbed during the slow fracture of a chevron-notched specimen in water as compared to dry oxygen, this decrease (i.e., the

Fig. 11. Ductile deformation and interfacial debonding of iron aluminide ligament on Fe-40 at. % Al/60 vol.% WC fracture surface. This particular composite was fabricated from WC with a particle size of 29 μm .

Fig. 12. Load-displacement curves for the 3-point bending of chevron-notched Fe-40 at. % Al/60 vol.% TiC composites in either dry oxygen or water at room temperature. The crack propagation rate was approximately 0.5 $\mu\text{m/s}$.

difference in the areas included under the respective curves) is approximately 50%, which is relatively small. For comparison, the energy absorbed in (humid) air during slow crack growth of monolithic iron aluminides may be lower by more than a factor of four than that absorbed during fast crack growth (where water vapor embrittlement is suppressed due to kinetic reasons) (22). This suggests the possibility that FeAl in the form of thin ligaments may not be as sensitive to humidity embrittlement as macroscopic, monolithic FeAl. More research will be needed to substantiate the possibility that the environmental embrittlement of iron aluminides is alleviated in iron aluminide composites.

Oxidation Resistance

Since high oxidation resistance is one of the merits of iron aluminides, it is obviously of interest to find out to what extent this oxidation resistance translates into the composites. WC/Co, the most commonly used hard metal, was chosen as a basis for comparison. Figure 13 shows indeed that the oxidation resistance of the iron aluminide matrix leads to a high oxidation resistance of the composites as compared to WC/Co.

Fig. 13. Comparison of the oxidation behavior of WC/Co, FeAl/WC, FeAl/TiC, and FeAl/TiB₂ (oxidation in air for 1 day at 1173 K).

Wear Behavior

A preliminary study of the dry wear resistance of FeAl/TiC was carried out. In Table II, the wear losses are compared against those of WC/Co with a similar volume fraction of Co. The mass loss of the FeAl/TiC composite is much lower than that of WC/Co. However, because of the high density of WC, the situation is reversed for the volume losses. Nevertheless it can be stated that iron aluminide bonded composites exhibit a high resistance to dry wear.

Table II. Dry wear test results according to ASTM G65A (dry sand/rubber wheel)

Composition	Fe-40 at. %Al/ 80 vol.% TiC	Co/80 vol.% WC (WC-13wt%Co)
Density, Mg/m^3	5.16	14.2
Mass Loss, g	0.0501	0.0931
Volume loss, mm^3	9.7	6.6

Thermal Expansion

The thermal expansion behavior of iron aluminides has been determined by Porter and Maziasz (23). Figure 14 shows the thermal expansion of a monolithic iron aluminide to be higher than that of iron. Also shown in this plot is the thermal expansion of TiC. Clearly, the thermal expansion of an FeAl/60 vol.% TiC composite lies between two extremes, namely, that of FeAl on one hand, and that of TiC on the other. A cursory inspection shows that the CTE of the composite follows roughly a rule of mixtures. By adjusting the TiC volume fraction, it is in principle possible to adjust the coefficient of thermal expansion (CTE) of an FeAl/TiC or FeAl/WC composite to that of a steel. This might be a useful property when considering, for example, wear-resistant sleeves in cast iron.

Fig. 14. Comparison between the thermal expansions of Fe and FeAl (FA-385 = Fe-36.5 at. % Al with minor alloying additions), TiC, and FeAl/60 vol.% TiC.

Potential Applications

The new class of iron aluminide bonded composites or cermets reviewed in this work may be useful in a variety of wear applications. Of particular relevance are wear applications in erosive and corrosive environments, where the resistance of iron aluminides against oxidation, sulfidation, and molten salts can be exploited. Applications of interest include various types of dies, bearing sleeves (where CTE matching might be advantageous), mechanical face seals, and impellers for slurry pumps. Since TiB_2 is of interest in molten aluminum applications, the FeAl/ TiB_2 system should be considered for the handling of molten or semi-solid aluminum. Other features relevant for future applications are of course the potentially low price of iron aluminides, their low mass density, and, in comparison to Co, Ni, or Cr, their greater environmental friendliness.

Conclusion

We have shown that ceramics, such as TiB_2 , ZrB_2 , TiC, and WC, may be liquid phase sintered or melt-infiltrated with iron aluminide intermetallics, and are sufficiently stable thermodynamically in contact with iron aluminides. Near-theoretical densities have been found for ceramic volume fractions up to 85%. In the FeAl/WC system, flexure strengths and fracture toughness values on the order of 1800 MPa and $20 \text{ MPa m}^{1/2}$ were achieved for a WC volume fraction of 60%. The flexure strengths may be limited by clusters of small oxide inclusions and insufficient interfacial bonding between the FeAl matrix and the ceramic particulates. Whereas HIPed specimens showed significant scatter in their flexure properties, a subsequent low temperature anneal (773 K) improved the strength, while at the same time reducing the scatter. This is attributed to the softening of the iron aluminide matrix and the associated lower flaw sensitivity. In view of the relative brittleness of monolithic iron aluminides, the fracture toughnesses of iron aluminide composites are surprisingly high. Also, the water vapor embrittlement is not as severe as might be expected. This may be due to the fact that the iron aluminide occurs in the composites in the form of very fine ligaments. Other properties such as oxidation resistance, wear resistance, and thermal expansion have been examined and make this new class of iron aluminide composites of interest in a variety of applications.

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