

## Liquid-Phase Sintering of Iron Aluminide-Bonded Ceramics

J. H. Schneibel and C. A. Carmichael

Metals and Ceramics Division  
Oak Ridge National Laboratory,  
P. O. Box 2008, Oak Ridge, TN 37831-6115, U.S.A.

### Abstract

Iron aluminide intermetallics exhibit excellent oxidation and sulfidation resistance and are therefore considered as the matrix in metal matrix composites, or the binder in hard metals or cermets. In this paper we discuss the processing and properties of liquid-phase sintered iron aluminide-bonded ceramics. It is found that ceramics such as  $\text{TiB}_2$ ,  $\text{ZrB}_2$ ,  $\text{TiC}$ , and  $\text{WC}$  may all be liquid phase-sintered. Nearly complete densification is achieved for ceramic volume fractions ranging up to 60%. Depending on the composition, room temperature fracture point-bend strengths and fracture toughnesses reaching 1500 MPa and  $30 \text{ MPa m}^{1/2}$ , respectively, have been found. Since the processing was carried out in a very simple manner, optimized processing is likely to result in further improvements.

### I. Introduction

Iron aluminide intermetallics such as  $\text{Fe}_3\text{Al}$  ( $\text{D0}_3$  structure) and  $\text{FeAl}$  ( $\text{B2}$  structure) offer a number of potentially useful properties, which are discussed in recent conference proceedings on intermetallics (Horton et al., 1995), or, more specifically, on iron aluminides (Schneibel and Crimp, 1994). Iron aluminides are very resistant against oxidizing or sulfidizing environments, they exhibit comparatively low densities ( $5.6 \text{ Mg/m}^3$  for Fe-50 at. % Al), they have high work hardening rates suggesting high wear resistance, and consist of inexpensive raw materials. Most conventional hard metals contain significant amounts of Co or Ni, both of which are not only expensive, but also toxic when occurring as fine particulates. Since iron aluminides are presumably less toxic than Co or Ni, they may have an advantage over these elements as environmentally benign matrix materials or binders. The major drawback of iron aluminides is their low ductility in air, although appropriate processing may result in ductilities in excess of 15% (see for example Schneibel and Crimp, 1994). The low ductilities are, at least in part, due to hydrogen generated via the reduction of water vapor by aluminum. Consequently, much higher ductilities may be achieved in vacuum or dry oxygen environments. Another limitation is the poor creep strength of iron aluminides, which has, however, recently been improved (McKamey and Maziasz, 1994).

Recent thermodynamic calculations by Misra (1990) showed iron aluminides to be thermodynamically compatible with a wide range of ceramics at 1273 K. Also, since ceramics such as  $\text{TiB}_2$  form from  $\text{Fe}_3\text{Al}$  melts containing small amounts of Ti and B (McKamey et al., 1991), it was thought possible that  $\text{TiB}_2$

The submitted manuscript has been authored by a contractor of the U.S. Government under contract No. DE-AC05-84OR21400. Accordingly, the U.S. Government retains a nonexclusive, royalty-free license to publish or reproduce the published form of this contribution, or allow others to do so, for U.S. Government purposes.

and some other ceramics might be stable in liquid iron aluminides. It was decided to investigate composites of iron aluminides with  $\text{TiB}_2$ ,  $\text{ZrB}_2$  (which has the same density as Fe-40 at. % Al),  $\text{TiC}$  [which may be successfully bonded with  $\text{Ni}_3\text{Al}$  intermetallics (Tiegs et al., 1995; Plucknett et al., 1995), and WC (which is the most common ceramic in cobalt bonded hard metals).

## II. Experimental Details

Iron aluminide/ceramic composites were produced from mixtures of prealloyed Fe-40 at. % Al powder and various ceramic powders by liquid-phase sintering. Loosely packed powders were heated in a vacuum of  $10^{-4}$  Pa to 1723 K in approximately 5 ks, held, unless otherwise stated, for 900 s at this temperature, and furnace cooled. The powder sizes, the values assumed for the densities of the powders, and the predicted theoretical densities of composites made from them are listed in Table 1.

**Table 1** Densities of iron aluminide, selected ceramics, and iron aluminide composites containing 50 vol.% ceramic.

Material	Powder Size	Density ( $\text{Mg/m}^3$ )	Theoretical Density of FeAl Composite with 50 vol.% Ceramic ( $\text{Mg/m}^3$ )
Fe-40 at. %Al	-325 mesh (<45 $\mu\text{m}$ )	6.06	
$\text{TiB}_2$	-325 mesh (<45 $\mu\text{m}$ )	4.5	5.3
$\text{ZrB}_2$	-325 mesh (<45 $\mu\text{m}$ )	6.08	6.07
$\text{TiC}$	3 $\mu\text{m}$	4.93	5.49
WC	5 - 8 $\mu\text{m}$	15.6	10.8

From the liquid-phase sintered samples bend specimens as well as chevron-notched specimens were fabricated by electro-discharge machining and grinding. Bend strengths and fracture toughnesses were measured in three-point bending with a span of 20 mm and a crosshead speed of 10  $\mu\text{m/s}$ ; fracture toughnesses  $K_Q$  were determined from the energy  $W$  absorbed during the tests, the area  $A$  swept out by the crack, and the plane strain Young's modulus  $E'$  as

$$K_Q = [(W/A) E']^{1/2}.$$

The  $E'$  values for the composites were calculated from the Young's moduli of the iron aluminide matrix,  $E=180$  GPa, and the respective ceramics, using Ravichandran's (1994) eqn. (8) and a value  $\nu=0.2$  for Poisson's ratio.

## III. Experimental Results and Discussion

Figure 1 illustrates the liquid-phase sintering process for iron aluminides. The alumina boat in this figure was initially loosely filled to the top with a

FeAl/TiB<sub>2</sub> powder mixture. The finished product illustrates the significant shrinkage occurring during liquid-phase sintering.

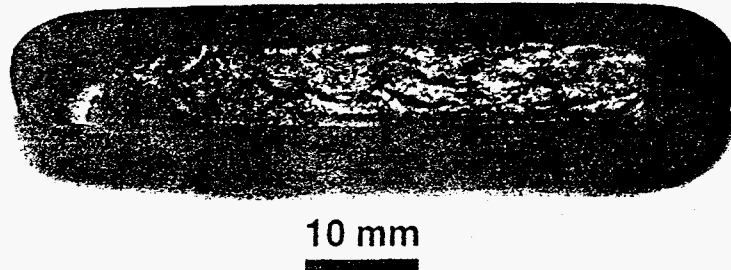


Fig. 1 Liquid-phase sintered Fe-40 at. % Al + 40 vol. % TiB<sub>2</sub> composite.

Figures 2-5 show scanning electron micrographs of mechanically polished sections of FeAl liquid-phase sintered with TiB<sub>2</sub>, ZrB<sub>2</sub>, TiC, or WC. The micrographs reveal only little porosity and suggest the absence of significant interfacial reactions in these systems. The rounded appearance of the WC particles is quite different from the angular shapes typically found in the WC/Co system. Since the latter shapes are the result of significant dissolution of the WC in Co, the FeAl/WC microstructure may indicate a low solubility of WC in FeAl. Since iron aluminides may be alloyed with many ternary elements (Schneibel et al., 1995) it is conceivable that the solubility of the investigated ceramics in liquid iron aluminides may be improved by alloying.

Figure 6 shows the results of immersion density measurements for FeAl/TiB<sub>2</sub> containing different volume fractions of TiB<sub>2</sub>. The composites containing ZrB<sub>2</sub>, TiC, or WC showed qualitatively similar behavior. In the case of 40 vol. % TiB<sub>2</sub>, sintering for 14.4 ks resulted in a significantly higher density as compared to sintering for 900 s. It is presently not clear why the measured density was in this case slightly higher than the theoretical density. One possible explanation might be preferential evaporation of aluminum. The processing aspects of these types of materials have so far not been addressed in great detail and will require more work.

Table 2 shows selected values for the bend strengths, the plane strain Young's moduli assumed in the fracture toughness calculations, the fracture toughnesses  $K_{Ic}$ , and the hardnesses of the composites fabricated with TiB<sub>2</sub>, ZrB<sub>2</sub>, TiC, and WC. The mechanical properties of FeAl/WC are shown in more detail in Figs. 7 and 8. Figure 7 shows the room temperature bend strength of FeAl/WC for different WC volume fractions. Above 60 vol. % the bend strengths start to decrease. This finding is consistent with significant porosity found above 60 vol. % WC. The fracture toughnesses of FeAl/WC composites decrease monotonically as the WC volume fraction increases (Fig. 8). The fracture toughness of polycrystalline Fe-40 at. % Al without ceramic



Fig. 2 Liquid-phase sintered Fe-40 at. % Al + 40 vol.% TiB<sub>2</sub> composite.

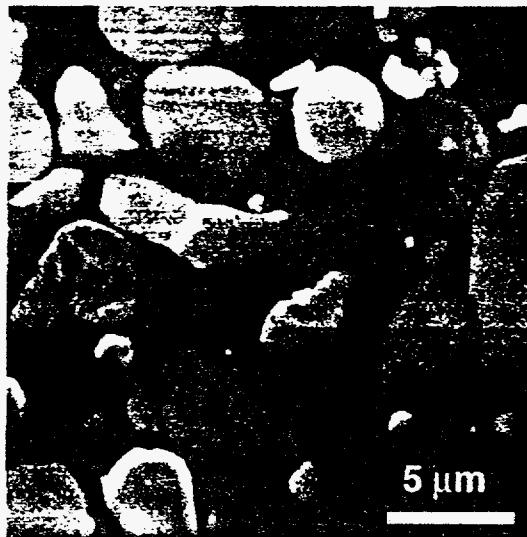


Fig. 3 Liquid-phase sintered Fe-40 at. % Al + 40 vol.% ZrB<sub>2</sub> composite.



Fig. 4 Liquid-phase sintered Fe-40 at. % Al + 50 vol.% TiC composite.

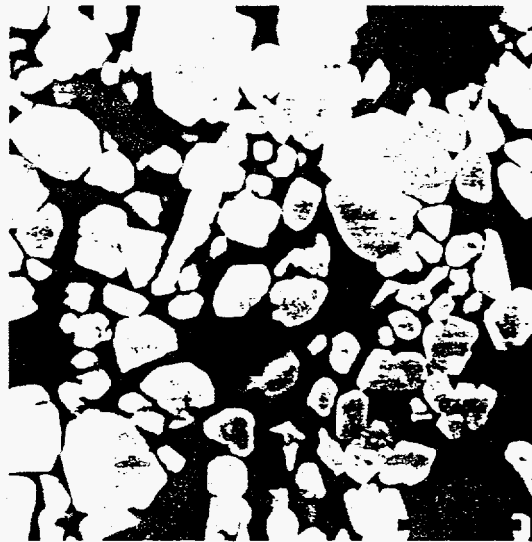


Fig. 5 Liquid-phase sintered Fe-40 at. % Al + 60 vol.% WC composite.

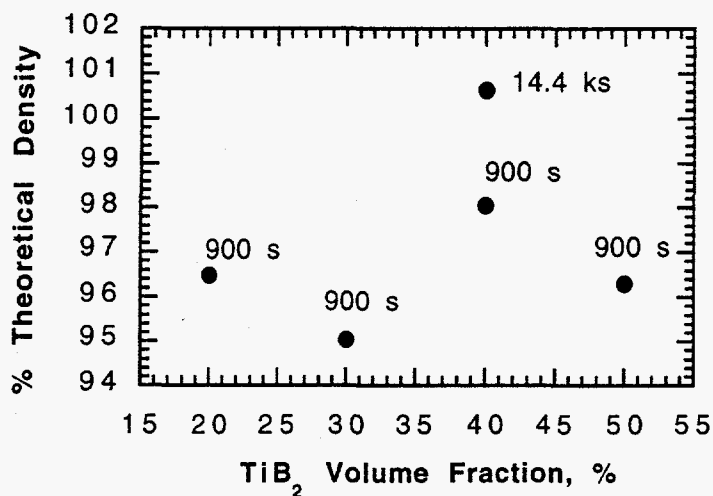


Fig. 6 Percentage of theoretical density for liquid-phase sintered Fe-40 at. % Al + TiB<sub>2</sub>. The sintering times at 1723 K are indicated.

Table 2 Selected mechanical properties of iron aluminide-bonded ceramics.

Fe-40 at. % Al with	Bend Strength (MPa)	E' (GPa)	K <sub>Q</sub> (MPa m <sup>1/2</sup> )	Hardness (Rockwell A)
40 vol.% TiB <sub>2</sub> *	1230	276	27	75
40 vol.% ZrB <sub>2</sub> *	1300	276	28	75
60 vol.% TiC	1050	303	15	84
50 vol.% WC	1385	333	24	77

\*isothermally forged

reinforcements determined at a crack propagation velocity similar to that used in this research is approximately 30 MPa m<sup>1/2</sup> (Schneibel, 1993). Surprisingly, Fig. 8 shows that the incorporation of 50 vol.% WC carbide particles into iron aluminide does not degrade the fracture toughness significantly.

Although the mechanical properties of cobalt-bonded WC are significantly better than those of iron aluminide-bonded WC, the present results are encouraging. Even if it may not be possible to achieve the mechanical properties of WC/Co, some of the special properties of iron aluminides such as their oxidation and sulfidation resistance may make them of interest in those applications, where cobalt binders would degrade too quickly.

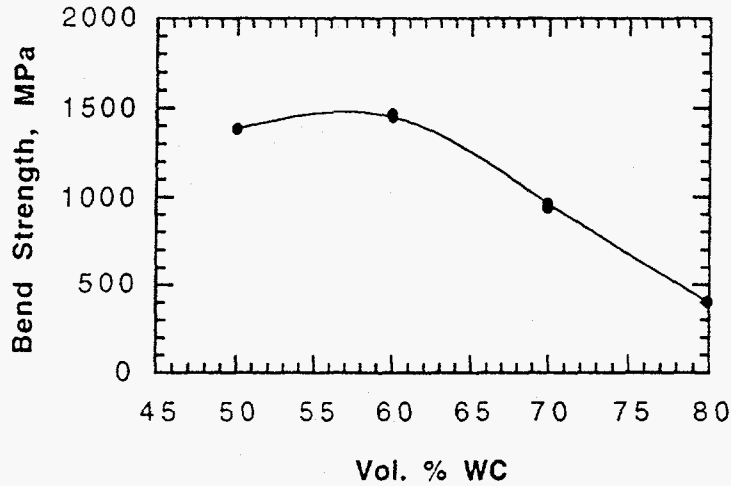


Fig. 7 Three-point bend strength of Fe-40 at. % Al + WC.

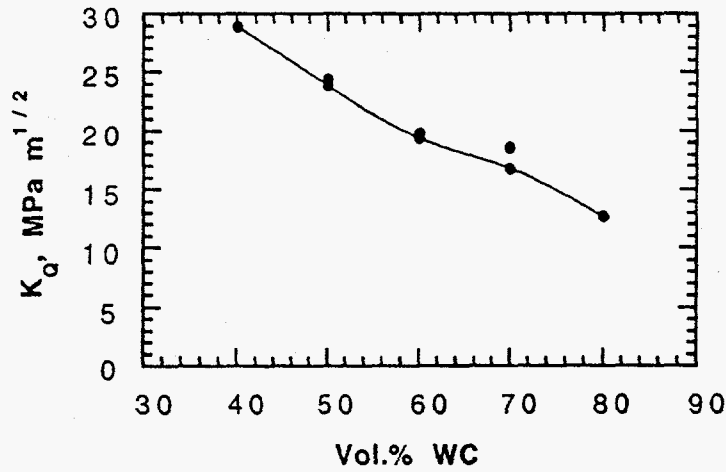


Fig. 8 Fracture toughness of Fe-40 at. % Al + WC.

#### Conclusions

It has been shown that ceramics such as TiB<sub>2</sub>, ZrB<sub>2</sub>, TiC, and WC may be liquid-phase sintered with iron aluminides. Close-to-theoretical densities have been found for ceramic volume fractions up to approximately 60 vol.%. Since the iron aluminide binder has special properties such as excellent oxidation and sulfidation resistance, composites or hard metals based on such binders may find

uses in some applications. Although the processing of the composites was carried out in a very simple manner, useful mechanical properties have been found. It is anticipated that further improvements in the mechanical properties will be realized by improved processing coupled with suitable alloying modifications of the iron aluminide binder.

#### Acknowledgments

This research was sponsored by the Division of Materials Sciences, and the Assistant Secretary for Energy Efficiency and Renewable Energy, Office of Industrial Technologies, Advanced Industrial Materials (AIM) Program, U.S. Department of Energy, under contract DE-AC05-84OR21400 with Lockheed Martin Energy Systems, Inc.

#### References

- J. A. Horton, I. Baker, S. Hanada, R. D. Noebe, and D. S. Schwartz (eds.), "High Temperature Ordered Intermetallic Alloys VI," Vol. 364, MRS, Boston, MA (1995).
- C. G. McKamey and P. J. Maziasz, "Effect of Heat Treatment Temperature on Creep-Rupture Properties of Fe<sub>3</sub>Al-Based Alloys," in J. H. Schneibel and M. A. Crimp (eds.), "Processing, Properties, and Applications of Iron Aluminides," TMS, Warrendale, PA, pp. 147-58 (1994).
- C. G. McKamey, J. H. DeVan, P. F. Tortorelli, and V. K. Sikka, "A Review of Recent Developments in Fe<sub>3</sub>Al-Based Alloys." *J. Mater. Res.* **6**, 1779-1805 (1991).
- A. K. Misra, "Identification of Thermodynamically Stable Ceramic Reinforcement Materials for Iron Aluminide Matrices," *Metall. Trans. A* **21A**, 441-6 (1990).
- K. P. Plucknett, T. N. Tiegs, K. B. Alexander, P. F. Becher, J. H. Schneibel, B. Waters, P. A. Menchhofer, "Intermetallic Bonded Ceramic Matrix Composites," to be published in "Ceramics for Structural and Tribological Applications," Vancouver, B.C., Aug. 21-23, CIM, 1995.
- K. S. Ravichandran, "A simple Model of Deformation Behavior of Two Phase Composites," *Acta Metall. Mater.* **42**, 1113-23 (1994).
- J. H. Schneibel and M. G. Jenkins, "Slow Crack Growth at Room Temperature in FeAl," *Scr. Metall. Mater.* **28**, 389-93 (1993).
- J. H. Schneibel and M. A. Crimp (eds.), "Processing, Properties, and Applications of Iron Aluminides," TMS, Warrendale, PA (1994).
- J. H. Schneibel, E. P. George, E. D. Specht, and J. A. Horton, "Strength, Ductility, and Fracture Mode in Ternary FeAl Alloys," in J. A. Horton, I. Baker, S. Hanada, R. D. Noebe, and D. S. Schwartz (eds.), "High Temperature Ordered Intermetallic Alloys VI," Vol. 364, MRS, Boston, MA, pp. 73-78 (1995).
- T. N. Tiegs, P. A. Menchhofer, K. P. Plucknett, K. B. Alexander, P. F. Becher, and S. B. Waters, "Hardmetals Based on Ni<sub>3</sub>Al as the Binder Phase," to be published in Proceedings 4th International Conference on Powder Metallurgy in Aerospace, Defense, and Demanding Applications, Anaheim, CA, May 8-10, 1995.

#### DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.