# IRON ALUMINIDE-TITANIUM CARBIDE COMPOSITES - MICROSTRUCTURE AND MECHANICAL PROPERTIES

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## ABSTRACT

Composites of intermetallics and carbides (with binder contents less than 50 vol. %) are considered as potential candidates for applications requiring high wear resistance in corrosive environments. Intermetallics, especially aluminides, provide the corrosion resistance, and the high hardness of the carbide phase contributes to increased wear resistance of the composites. In this study, cost effective and simple processing techniques to obtain FeAl-TiC composites, over a wide range of binder volume fractions, are demonstrated. Binder volume fractions range from 0.15 to 0.7 (18 to 75 wt % binder). Two techniques - liquid phase sintering of mixed powders and pressureless melt infiltration of TiC preforms - were used to process fully dense composites. Melt infiltration of TiC preforms was found to be very successful for obtaining fully dense composites with binder volume fractions from 0.15 to 0.3 (18 to 34 wt. %), whereas for higher binder contents liquid phase sintering of mixed powders was the best approach. Mechanical properties of these composites including the 3-point bend strength, fracture toughness and hardness are presented.

## INTRODUCTION

TiC-based cermets have been investigated for use in cutting tool and wear applications since the 1930's (1). Metallic binders such as Ni-based alloys, iron and steel were selected due to their excellent wetting of the TiC surfaces and desirable combination of ductility and toughness (2). With increasing demands for the use of TiC-based cermets in applications requiring excellent corrosion and wear resistance, investigation of new binder materials is essential. In this study, an intermetallic alloy, iron aluminide (Fe-40 at. % Al, abbreviated as Fe40Al in the text) is used as a binder. Iron aluminides are known to be extremely resistant to corrosion under sulfidizing atmospheres, oxidizing atmospheres and in molten salts (3-5). Iron aluminides can form an impervious aluminum oxide layer not only in oxygen and air atmospheres but also in a variety of mixed gas and salt conditions and thus impart corrosion resistance in a wide range of corrosive environments (3,5). Additionally, binders such as Ni- and Co-based alloys could, due to their carcinogenic nature, pose problems during processing and machining. In this respect, iron aluminides are significantly more environmentally benign. Also from an economic point of view, new binder materials with lower raw material costs than Co or Ni need to be identified.

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At first glance, an intermetallic alloy, traditionally thought to be brittle, may not seem to be the appropriate choice for a binder material. But, recently, it has been shown that the brittle nature of intermetallic aluminides arises due to an extrinsic environmental effect (6,7) and an inherent weakness of grain boundaries (7-9). The detrimental extrinsic effect arises due to a reaction between the moisture in air and aluminum in the alloy to yield alumina and atomic hydrogen, which induces classic hydrogen embrittlement at crack tips. Hence, intermetallic aluminides, when tested in controlled environments such as dry oxygen or under high vacuum show high ductility (6-9). Microalloying with just 300 wt. ppm boron increases the strength of the grain boundaries and reduces the degree of environmentally induced embrittlement (7-9). Reductions in grain size have also led to significant improvements in the ductility of iron aluminides (10).

With a better understanding of the mechanical properties of intermetallic aluminides, composites of intermetallics and ceramics may yield an unique combination of properties. Thus a number of intermetallic-carbide and intermetallic-boride composites with more than 40 vol % ceramic phase have been processed by liquid phase sintering to nearly full density - $Fe40Al-TiB_2$ (11,12), $Fe40Al-ZrB_2$ (11,12), Fe40Al-WC (11,12), $Ni_3Al-TiC$ (13) and $Ni_3Al-WC$ (14). In this study, the processing of fully dense Fe40Al-TiC composites with Fe40Al volume fractions from 0.15 to 0.70 (18 to 75 wt. %) is discussed. For composites with binder (Fe40Al) contents from 40 to 70 volume percent (45 to 75 wt. %) liquid phase sintering of a mixture of powders was successful (11,12). For lower contents of the binder phase - 15 to 30 vol. % (18 to 34 wt. %) pressureless infiltration of TiC preforms was found to be necessary to obtain fully dense composites. Typical microstructures are shown and preliminary mechanical properties are reported.

## EXPERIMENTAL DETAILS

The starting materials used for processing of the composites were TiC powder (2-3 μm, Kennametal, Inc) and pre-alloyed Fe-40 at. % Al (-325 mesh, Homogenous Metals Inc.).

For processing of composites by conventional liquid phase sintering, TiC and Fe40Al powders were mixed and placed in an alumina crucible. The mixture was held at 1200 °C for 30 minutes for degassing in a Ti-gettered dynamic vacuum of about $10^{-4}$ Pa. The mixture was allowed to sinter at 1450 °C for an hour.

For composites processed by melt infiltration, TiC powder was uniaxially cold pressed at 140 MPa to about 67 % of the theoretical density. The green pellet was then placed in an alumina dish with an appropriate amount of the iron aluminide powder, corresponding to the ultimate desired volume fraction, on the top surface of the pellet. The furnace was maintained under a Ti-gettered dynamic vacuum of about $10^{-4}$ Pa. After degassing at 1200 °C for 30 minutes, the infiltration was carried out at a temperature of 1450 °C. The sample was held at this temperature for an hour allowing sufficient time for sintering after the initial infiltration. After cooling, specimens were cut, polished and examined with an optical microscope and a Hitachi-S4100 scanning electron microscope (SEM) equipped with a light element energy-dispersive spectrometer (EDS).

Specimens for bend testing and fracture toughness measurements were electro-discharge machined and ground into bars with dimensions of 3 x 4 x 21 mm³ and polished with 3 μm diamond paste. Room temperature flexure strengths were performed using a 3-point bending fixture witha 20 mm span. Loads were applied in an Instron 4501 testing machinewith a cross-head speed of 10 μm/sec. Chevron-notched specimens were also tested in the same set-up and the fractured surfaces were observed using a SEM. Rockwell hardness ($R_A$) of the composites was determined using a Wilson hardness tester.

# RESULTS AND DISCUSSION

Figures 1(a, b, and c) show typical microstructures for the composites containing 50, 30 and 15 vol. % Fe40Al, respectively. The dark regions are TiC and the bright regions represent the intermetallic phase. An almost continuous intermetallic phase is observed. This suggests excellent wetting of the TiC particles by liquid iron aluminide. The presence of a thin film of the intermetallic phase around all particles would result in a very low TiC contiguity, a measure of the TiC-TiC contact in the composite. This has been observed to be a beneficial microstructural feature which lead to higher bend strengths and improved fracture toughness (15). Composites with binder contents below 30 % were processed by melt infiltration of TiC preforms. Further details of the melt infiltration technique and the advantages of the technique for processing the composites will be discussed in a later publication.

The compositions of the alloy and TiC particles were determined in the SEM by EDS analysis. Standards of pure Fe40Al and TiC were used for the quantitative analysis. Titanium concentrations in the alloy were determined to be about 1-2 at. % and Fe and Al concentrations in TiC were below the detectability limit. The Fe/Al ratio in the binder phase was determined to be 1.5, consistent with the starting powder composition. No secondary phases were observed during examination in the SEM.

Chevron-notched specimens were tested in a 3-point bend setup to obtain fracture toughness values for the composite with 40 vol. % intermetallic. Since fracture occurred in a stable manner, a continuous load-displacement curve was obtained and the area under this curve was integrated to obtain the energy, per unit crack area (denoted as G), dissipated during the fracture of the sample. With the elastic modulus calculated for a composite as given by Eq. 8 in Ref. (16), one obtains the fracture toughness from the following equation:

$$K_q = [E \cdot G/ (1-v^2)]^{1/2}$$

where E is the calculated elastic modulus for the composite containing 40 vol % intermetallic (303 Gpa) and $v$ is Poisson's ratio (taken to be 0.2 in our case). An average value of 21.4 MPa·m$^{1/2}$ was obtained after testing two specimens. The composite with 30 and 20 vol. % intermetallic had fracture toughness values of 18 and 10 MPa·m$^{1/2}$, respectively.

The fracture surface of a chevron-notched test specimen is shown in Fig. 2. The distinctive features of the fracture surface are indicated - significant ductilility of the intermetallic (A), a few surfaces due to cleavage fracture of the TiC particles (B), and debonding at the TiC/intermetallic interface (C).

The apparent ductility of the intermetallic phase can be rationalised on the basis that the iron aluminide ligaments between individual TiC grains are expected to be much smaller than the Fe40Al grain size. Investigations of the fracture of polycrystalline Fe40Al have shown that failure is predominantly intergranular (7). However, in the composite, since each ligament essentially acts as a single crystal during the ligament deformation process, contribution from intergranular failure is absent. In addition, these thin single crystalline intermetallic ligaments, constrained between the TiC particles, will respond to applied stress in a different manner than the bulk, unconstrained intermetallic (17). Fracture of bulk Fe40Al single crystals occurs by cleavage on {001} (18). Present observations of the fracture surfaces in these composites did not show typical features of crystallographic cleavage fracture. The deformation behavior of the Fe40Al ligaments is likely to help in the bridging in the wake of the cracks, resulting in a high fracture toughness of the composite (for comparison $K_{IC}$ of TiC = 4.4 MPa·m$^{1/2}$). Further systematic studies are required to better understand the role of the intermetallic phase in improving the toughness of a composite. It is also known that addition of ternary elements to bulk polycrystalline iron aluminides increases the yield stress and change their mode of fracture (19). Detailed studies of the role of ternary alloying additions to modify the mechanical
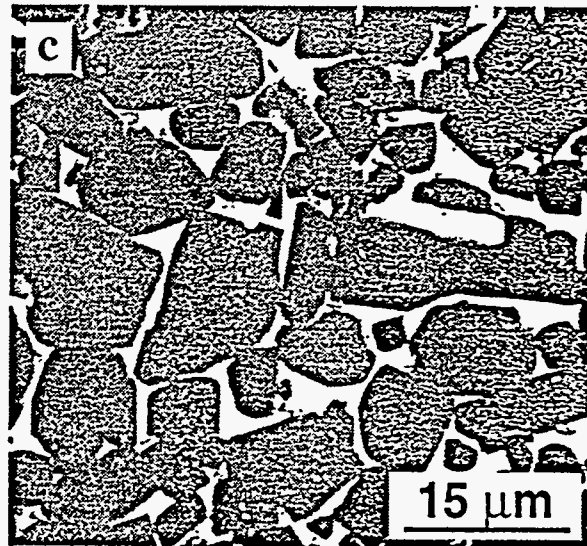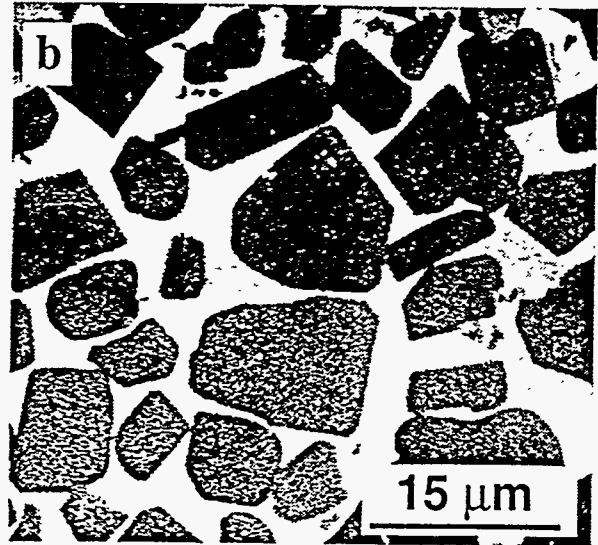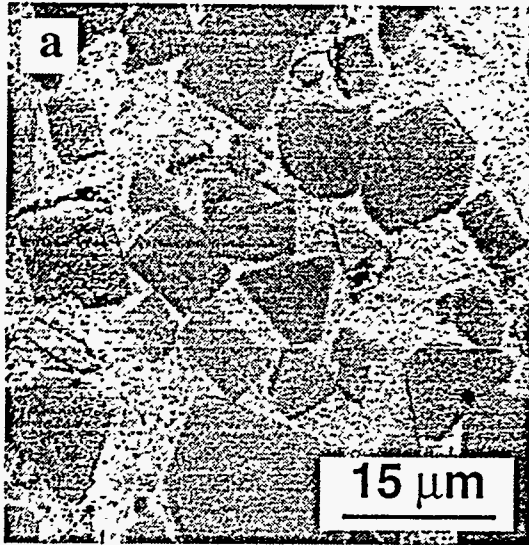
Fig. 1: Fe40Al-TiC composite with (a) 50 vol. % , (b) 30 vol. %, and (c) 15 vol. % intermetallic
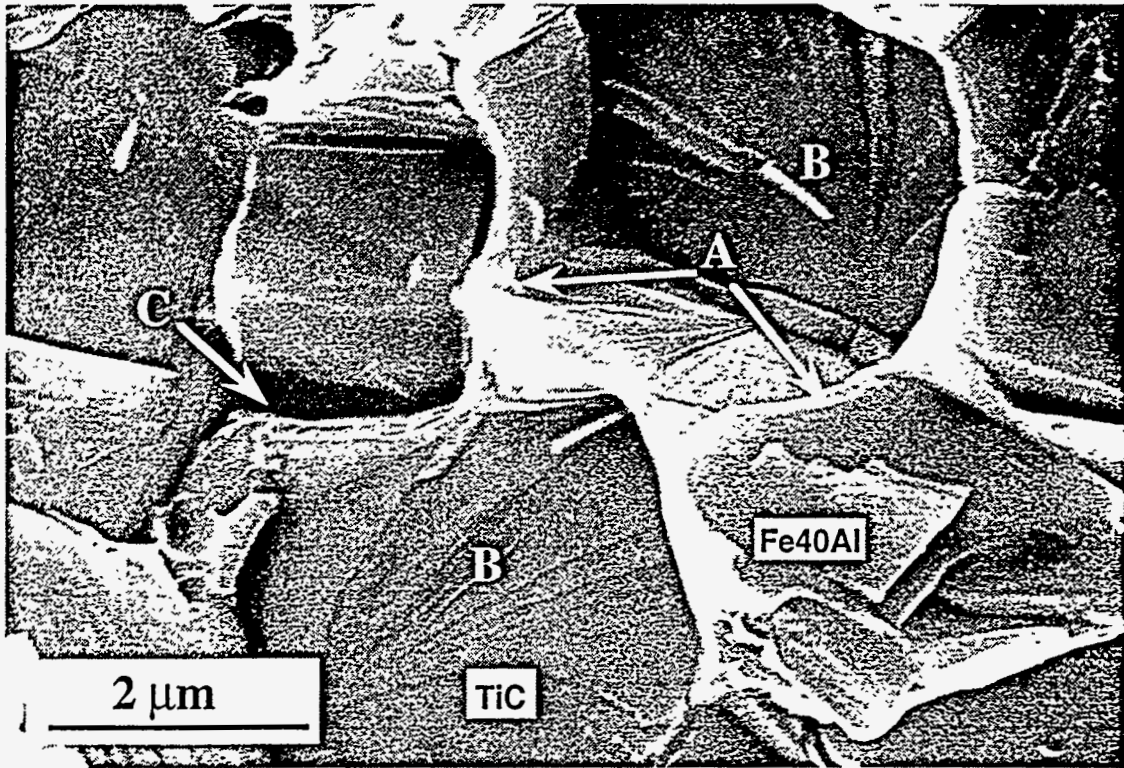
Fig. 2: Fracture surface of a Fe40Al-TiC composite with 30 vol % intermetallic phase

properties of these composites and tailor the properties for different engineering requirements are underway.

The room temperature bend strengths and Rockwell hardness values for composites with different volume fractions of the intermetallic phase (15 - 40 vol. %) are reported in Table 1. For comparison purposes, the mechanical properties of other cermets are also shown in Table 1.

### TABLE 1

Selected mechanical properties of Fe40Al-TiC cermet and another commercially available cermet.

| Binder composition (at. %) | Binder vol. % (wt %) | Carbide composition (in wt. %) | Hardness | Bend Strengths (in MPa) | Fracture Toughness (MPa$\cdot$m$^{1/2}$) |
|---|---|---|---|---|---|
| Fe-40Al | 40 (45) | TiC - 55 | 81 $R_A$ | 942 MPa (3 tests) | 21.4 (2 tests) |
| Fe-40Al | 30 (35) | TiC - 65 | 83.5 $R_A$ | 1034 MPa (3 tests) | 18 (3 tests) |
| Fe-40Al | 20 (23.5) | TiC - 76.5 | 87 $R_A$ | 750 MPa (3 tests) | 12.7 (3 tests) |
| Fe-40 Al | 15 (17.8) | TiC - 82.2 | 90 $R_A$ | - | - |
| [a]Ni-16Mo | 21 (31) | TiC - 58 NbC - 6 WC - 3 | 89 $R_A$ | 1700 MPa | 10.6 |

a - Ref. 20

The Fe40Al-TiC cermets have hardness values (81 - 90 $R_A$) and fracture toughness values (10-21 MPa·m$^{1/2}$) almost on par with those reported for commercially available cermets. Cermets with 30 and 40 vol. % intermetallic phases showed high fracture toughness values (18 and 21 MPa·m$^{1/2}$, respectively) with possible reasons as discussed previously. All the specimens were sintered to near full density (> 99.5 % theoretical density) but the composite with 85 volume % TiC retained a semicircular crack. This was possibly introduced during the uniaxial cold-pressing of the TiC pellets, due to an inhomogeniety in the green density. Infiltration of only 15 vol. % intermetallic was probably not sufficient to allow rearrangement of the TiC particles to eliminate the crack. Thus bend strength and fracture toughness could not be determined due to the lack of sufficiently large defect-free specimens. Improvement in the processing technique is underway. It should be mentioned that the average grain size of the carbide particles in the commercially available cermets listed in the table are in the range of 1-3 μm. The carbide grain size shown in Fig. 1 is of the order of about 10 μm. For a given binder volume fraction, bend strengths are known to increase significantly with decreasing grain size (21) and therefore the comparison in Table 1 is intended only to provide an idea of the potential of these composites. Improvements in the mechanical properties are expected on optimization of the volume fractions and grain sizes in these composites. Additionally, if the bonding at the Fe40Al-TiC interface can be improved by additions of ternary alloying elements, higher interface strengths will lead to a better load transfer between the ceramic and intermetallic. Such modifications are likely to increase the bend strength, possibly at the expense of fracture toughness.

## SUMMARY

In this investigation, fully dense TiC-iron aluminide (Fe-40 at. % Al) composites with binder contents from 15 to 70 volume percent (17.8 - 75 wt. %) were obtained. Conventional lqiuid phase sintering was successful for binder volume fractions between 0.4 and 0.7 (45 - 75 wt. %). Pressureless melt infiltration of TiC preforms was found necessary for processing fully dense composites with binder volume fractions less than 0.3 (34 wt. %). Specimens with 30 vol % intermetallic exhibited bend strengths of 1034 MPa, fracture toughness of 18 MPa·m$^{1/2}$

and a Rockwell ($R_A$) hardness of 83.5. Further improvements in bend strengths may be possible by controlling the grain size and by modifications of the Fe40Al/TiC interface strengths.

## ACKNOWLEDGMENTS

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