

IRON ALUMINIDE COMPOSITE AND METHOD OF MANUFACTURE THEREOF

Field of the Invention

The invention relates generally to iron aluminide composites and method of manufacture thereof.

Background of the Invention

Iron base alloys containing aluminum can have ordered and disordered body centered crystal structures. For instance, iron aluminide alloys having intermetallic alloy compositions contain iron and aluminum in various atomic proportions such as Fe_3Al , FeAl , FeAl_2 , FeAl_3 , and Fe_2Al_3 . Fe_3Al intermetallic iron aluminides having a body centered cubic ordered crystal structure are disclosed in U.S. Patent Nos. 5,320,802; 5,158,744; 5,024,109; and 4,961,903. Such ordered crystal structures generally contain 25 to 40 atomic % Al and alloying additions such as Zr, B, Mo, C, Cr, V, Nb, Si and Y.

An iron aluminide alloy having a disordered body centered crystal structure is disclosed in U.S. Patent No. 5,238,645 wherein the alloy includes, in weight %, 8-9.5 Al, ≤ 7 Cr, ≤ 4 Mo, ≤ 0.05 C, ≤ 0.5 Zr and ≤ 0.1 Y, preferably 4.5-5.5 Cr, 1.8-2.2 Mo, 0.02-0.032 C and 0.15-0.25 Zr. Except for three binary alloys having 8.46, 12.04 and 15.90 wt % Al, respectively, all of the specific alloy compositions disclosed in the '645 patent include a minimum of 5 wt % Cr. Further, the '645 patent states that the alloying elements improve strength, room-temperature ductility, high temperature oxidation resistance, aqueous corrosion resistance and resistance to pitting. The '645 patent does not relate to electrical resistance heating elements and does not address properties such as thermal fatigue resistance, electrical resistivity or high temperature sag resistance.

Commonly owned U.S. Patent Nos. 5,595,706 and 5,620,651 disclose iron base alloys containing aluminum which are useful for electrical resistance heating elements. Examples of heating element configurations can be found in commonly owned U.S. Patent

Nos. 5,530,225 and 5,591,368. Other examples of electrical resistance heating elements can be found in commonly owned U.S. Patent Nos. 5,060,671; 5,093,894; 5,146,934; 5,188,130; 5,224,498; 5,249,586; 5,322,075; 5,369,723; and 5,498,855.

5 A 1990 publication in *Advances in Powder Metallurgy*, Vol. 2, by J.R. Knibloe et al., entitled "Microstructure And Mechanical Properties of P/M Fe₃Al Alloys", pp. 219-231, discloses a powder metallurgical process for preparing Fe₃Al containing 2 and 5% Cr by using an inert gas atomizer. This publication explains that Fe₃Al alloys have a DO₃ structure at low temperatures and transform to a B2 structure above about 550°C. To make sheet, the powders were canned in mild steel, evacuated and hot extruded at 1000°C to an
10 area reduction ratio of 9:1. After removing from the steel can, the alloy extrusion was hot forged at 1000°C to 0.340 inch thick, rolled at 800°C to sheet approximately 0.10 inch thick and finish rolled at 650°C to 0.030 inch. According to this publication, the atomized powders were generally spherical and provided dense extrusions and room temperature ductility approaching 20% was achieved by maximizing the amount of B2 structure.

15 A 1991 publication in *Mat. Res. Soc. Symp. Proc.*, Vol. 213, by V.K. Sikka entitled "Powder Processing of Fe₃Al-Based Iron-Aluminide Alloys," pp. 901-906, discloses a process of preparing 2 and 5% Cr containing Fe₃Al-based iron-aluminide powders fabricated into sheet. This publication states that the powders were prepared by nitrogen-gas atomization and argon-gas atomization. The nitrogen-gas atomized powders
20 had low levels of oxygen (130 ppm) and nitrogen (30 ppm). To make sheet, the powders were canned in mild steel and hot extruded at 1000°C to an area reduction ratio of 9:1. The extruded nitrogen-gas atomized powder had a grain size of 30 μm. The steel can was removed and the bars were forged 50% at 1000°C, rolled 50% at 850°C and finish rolled 50% at 650°C to 0.76 mm sheet.

25 A paper by V.K. Sikka et al., entitled "Powder Production, Processing, and Properties of Fe₃Al", pp. 1-11, presented at the 1990 Powder Metallurgy Conference Exhibition in Pittsburgh, PA, discloses a process of preparing Fe₃Al powder by melting constituent metals under a protective atmosphere, passing the metal through a metering

nozzle and disintegrating the melt by impingement of the melt stream with nitrogen atomizing gas. The powder had low oxygen (130 ppm) and nitrogen (30 ppm) and was spherical. An extruded bar was produced by filling a 76 mm mild steel can with the powder, evacuating the can, heating 1 1/2 hr at 1000°C and extruding the can through a 25 mm die for a 9:1 reduction. The grain size of the extruded bar was 20 μ m. A sheet 0.76 mm thick was produced by removing the can, forging 50% at 1000°C, rolling 50% at 850°C and finish rolling 50% at 650°C.

Oxide dispersion strengthened iron-base alloy powders are disclosed in U.S. Patent Nos. 4,391,634 and 5,032,190. The '634 patent discloses Ti-free alloys containing 10-40% Cr, 1-10% Al and $\leq$ 10% oxide dispersoid. The '190 patent discloses a method of forming sheet from alloy MA 956 having 75% Fe, 20% Cr, 4.5% Al, 0.5% Ti and 0.5% Y_2O_3 .

A publication by A. LeFort et al., entitled "Mechanical Behavior of $FeAl_{40}$ Intermetallic Alloys" presented at the Proceedings of International Symposium on Intermetallic Compounds - Structure and Mechanical Properties (JIMIS-6), pp. 579-583, held in Sendai, Japan on June 17-20, 1991, discloses various properties of FeAl alloys (25 wt % Al) with additions of boron, zirconium, chromium and cerium. The alloys were prepared by vacuum casting and extruding at 1100°C or formed by compression at 1000°C and 1100°C. This article explains that the excellent resistance of FeAl compounds in oxidizing and sulfidizing conditions is due to the high Al content and the stability of the B2 ordered structure.

A publication by D. Pocchi et al., entitled "Production and Properties of CSM FeAl Intermetallic Alloys" presented at the Minerals, Metals and Materials Society Conference (1994 TMS Conference) on "Processing, Properties and Applications of Iron Aluminides", pp. 19-30, held in San Francisco, California on February 27 - March 3, 1994, discloses various properties of $Fe_{40}Al$ intermetallic compounds processed by different techniques such as casting and extrusion, gas atomization of powder and extrusion and mechanical alloying of powder and extrusion and that mechanical alloying has been employed to reinforce the material with a fine oxide dispersion. The article states that FeAl alloys were prepared

having a B2 ordered crystal structure, an Al content ranging from 23 to 25 wt % (about 40 at %) and alloying additions of Zr, Cr, Ce, C, B and Y_2O_3 . The article states that the materials are candidates as structural materials in corrosive environments at high temperatures and will find use in thermal engines, compressor stages of jet engines, coal gasification plants and the petrochemical industry.

A publication by J. H. Schneibel entitled "Selected Properties of Iron Aluminides", pp. 329-341, presented at the 1994 TMS Conference discloses properties of iron aluminides. This article reports properties such as melting temperatures, electrical resistivity, thermal conductivity, thermal expansion and mechanical properties of various FeAl compositions.

A publication by J. Baker entitled "Flow and Fracture of FeAl", pp. 101-115, presented at the 1994 TMS Conference discloses an overview of the flow and fracture of the B2 compound FeAl. This article states that prior heat treatments strongly affect the mechanical properties of FeAl and that higher cooling rates after elevated temperature annealing provide higher room temperature yield strength and hardness but lower ductility due to excess vacancies. With respect to such vacancies, the articles indicates that the presence of solute atoms tends to mitigate the retained vacancy effect and long term annealing can be used to remove excess vacancies.

A publication by D.J. Alexander entitled "Impact Behavior of FeAl Alloy FA-350", pp. 193-202, presented at the 1994 TMS Conference discloses impact and tensile properties of iron aluminide alloy FA-350. The FA-350 alloy includes, in atomic %, 35.8% Al, 0.2% Mo, 0.05% Zr and 0.13% C.

A publication by C.H. Kong entitled "The Effect of Ternary Additions on the Vacancy Hardening and Defect Structure of FeAl", pp. 231-239, presented at the 1994 TMS Conference discloses the effect of ternary alloying additions on FeAl alloys. This article states that the B2 structured compound FeAl exhibits low room temperature ductility and unacceptably low high temperature strength above 500°C. The article states that room temperature brittleness is caused by retention of a high concentration of vacancies following

high temperature heat treatments. The article discusses the effects of various ternary alloying additions such as Cu, Ni, Co, Mn, Cr, V and Ti as well as high temperature annealing and subsequent low temperature vacancy-relieving heat treatment.

Summary of the Invention

5 The invention provides an iron aluminide composite comprising iron aluminide, an oxide filler and an additive which improves metallurgical bonding of the oxide filler to the iron aluminide. The oxide filler can comprise alumina, zirconia, yttria, rare earth oxide and/or beryllia. The additive can comprise a refractory carbide such as TiC, HfC and/or ZrC. A preferred ratio of oxide: additive is 1 to 3. The composite can be used for various
10 devices such as tool bits, structural components or electrical resistance heating elements in devices such as heaters. According to a preferred embodiment, the composite comprises a liquid phase sintered composite.

 The iron aluminide preferably comprises a binary alloy of iron and aluminum or an alloy. For instance, the iron aluminide alloy can comprise, in weight %, 14-32% Al, $\leq$
15 2.0% Ti, $\leq$ 2.0% Si, $\leq$ 30 % Ni, $\leq$ 0.5 % Y, $\leq$ 15 % Nb, $\leq$ 1% Ta, $\leq$ 3% W, $\leq$ 10% Cr, $\leq$ 2.0% Mo, $\leq$ 1% Zr, $\leq$ 1% C and $\leq$ 0.1% B. The oxide filler preferably comprises alumina which can be present in any desired amount such as $\leq$ 40%. The additive preferably comprises $\leq$ 40% TiC.

 According to various preferred aspects of the invention, the composite can be Cr-free, Mn-free, Si-free, and/or Ni-free. The composite can include non-oxide filler ceramic
20 particles such as SiC, Si₃N₄, AlN, etc. Preferred iron aluminide alloys include 20.0-31.0% Al, 0.05-0.15% Zr, $\leq$ 3% W, $\leq$ 0.1% B and 0.01-0.2% C; 14.0-20.0% Al, 0.3-1.5% Mo, 0.05-1.0% Zr, $\leq$ 3% W and $\leq$ 0.2% C, $\leq$ 0.1% B and $\leq$ 2.0% Ti; and 20.0-31.0% Al, 0.3-0.5% Mo, 0.05-0.3% Zr, $\leq$ 0.2% C, $\leq$ 2% W, $\leq$ 0.1% B and $\leq$ 0.5% Y.

25 The electrical resistance heating element can be used for products such as heaters, toasters, igniters, heating elements, etc. wherein the composite has a room temperature resistivity of 80-400 $\mu\Omega \cdot \text{cm}$, preferably 90-200 $\mu\Omega \cdot \text{cm}$. The composite preferably heats

to 900°C in less than 1 second when a voltage up to 10 volts and up to 6 amps is passed through the alloy. When heated in air to 1000°C for three hours, the composite preferably exhibits a weight gain of less than 4%, more preferably less than 2%. The composite preferably exhibits thermal fatigue resistance of over 10,000 cycles without breaking when pulse heated from room temperature to 1000°C for 0.5 to 5 seconds.

With respect to mechanical properties, the composite has a room temperature flexure strength of at least 300 MPa in the liquid phase sintered condition and at least 1000 MPa in the hot forged condition.

The invention also provides a powder metallurgical process of making an iron aluminide composite by forming a mixture of iron aluminide powder, oxide powder and an additive which promotes adhesion of the oxide powder to the iron aluminide, forming the powder mixture into a body and sintering the body. According to various aspects of the method, the body can be formed by hot or cold pressing and the sintering can comprise solid state, partial liquid or liquid phase sintering. For example, the forming can be carried out by placing the powder in a metal can, sealing the metal can with the powder therein, and hot pressing or hot extruding the metal can. Alternatively, the body can be made by liquid phase infiltration of an iron aluminide matrix into a mass of oxide filler particles. In order to densify and/or shape the sintered body, the sintered body can be hot forged or subjected to other working steps such as cold working, extrusion, rolling, etc. If desired, the powder mixture can be cold pressed prior to sintering and/or annealed subsequent to sintering.

Brief Description of the Drawings

Figure 1 shows an X-ray diffraction pattern for an FeAl/Al₂O₃ composite in accordance with the invention;

Figure 2 shows an X-ray diffraction pattern for an FeAl/ZrO₂ composite in accordance with the invention;

Figure 3 shows a scanning electron microscope image of an FeAl/ZrO₂ composite in accordance with the invention;

Figure 4 shows exudation of FeAl during liquid phase sintering of an FeAl/Al₂O₃ composite which did not include a TiC additive in accordance with the invention;

5 Figure 5 shows the effect of TiC on improving liquid infiltration of Al₂O₃ of iron aluminide;

Figure 6 shows a scanning electron microscope image of a polished section of an FeAl/TiC/Al₂O₃ composite in accordance with the invention;

10 Figure 7 shows a hot forged coupon of Fe-15TiC-15Al₂O₃ (vol. %) in accordance with the invention wherein the interior of the coupon is sound and some edge cracking is evident around the exterior of the coupon;

Figure 8 is an optical micrograph of a liquid phase sintered composite of FeAl-16.5TiC-16.5Al₂O₃ (vol. %) in accordance with the invention;

15 Figure 9 is an optical micrograph of a hot forged composite of FeAl-15TiC-15Al₂O₃ (vol. %) in accordance with the invention;

Figure 10 is a graph of stress versus crosshead displacement produced during a flexure stress test of a composite of FeAl-15TiC-15Al₂O₃ (vol. %) in accordance with the invention; and

20 Figure 11 is a graph of load versus crosshead displacement produced during a fracture toughness test of a composite of FeAl-15TiC-15Al₂O₃ (vol. %) in accordance with the invention.

Detailed Description of the Preferred Embodiments

25 The present invention is directed to iron aluminide composites including iron aluminide, an oxide filler and an additive which improves metallurgical bonding of the oxide filler to the iron aluminide. According to one aspect of the invention, the iron aluminide can include an iron concentration ranging from 4 to 32% by weight (nominal) and the oxide filler can comprise one or more oxides such as alumina, zirconia, yttria, rare

earth oxide and/or beryllia. The additive preferably comprises at least one refractory carbide, refractory nitride or refractory boride such as TiC, HfC, ZrC, TiN, HfN, ZrN, TiB₂, HfB₂ and/or ZrB₂.

The concentration of the alloying constituents used in forming the iron aluminide is expressed herein in nominal weight percent. However, the nominal weight of the aluminum essentially corresponds to at least about 97% of the actual weight of the aluminum in the iron aluminide. For example, in a preferred composition, a nominal 18.46 wt % may provide an actual 18.27 wt % of aluminum, which is about 99% of the nominal concentration.

The iron aluminide can be processed or alloyed with one or more selected alloying elements for improving properties such as strength, room-temperature ductility, oxidation resistance, aqueous corrosion resistance, pitting resistance, thermal fatigue resistance, electrical resistivity, high temperature sag or creep resistance and resistance to weight gain. The iron aluminide composite can be used to make heating elements for various devices such as described in commonly owned U.S. Patent No. 5,530,225 or 5,591,368. However, the composite can be used for other purposes such as in thermal spray applications wherein the composite could be used as coatings having oxidation and corrosion resistance. Also, the composite can be used as oxidation and corrosion resistant electrodes, furnace components, chemical reactors, sulfidization resistant materials, corrosion resistant materials for use in the chemical industry, pipe for conveying coal slurry or coal tar, substrate materials for catalytic converters, exhaust pipes for automotive engines, porous filters, etc.

According to one aspect of the invention, in the case where the composite is used for heating elements of electrical smoking articles, the geometry of the composite can be varied to optimize heater resistance according to the formula: $R = \rho (L/W \times T)$ wherein R = resistance of the heater, ρ = resistivity of the heater material, L = length of heater, W = width of heater and T = thickness of heater. The resistivity of the heater material can be varied by adjusting the iron aluminide alloy composition and/or the amount and/or type

of filler material in the composite. The composite can optionally include filler such as ceramic particles to enhance creep resistance and/or thermal conductivity. The composite may also incorporate particles of electrically insulating material for purposes of making the composite creep resistant at high temperature and also enhancing thermal conductivity and/or reducing the thermal coefficient of expansion of the composite. The electrically insulating/conductive particles/fibers can be added to a powder mixture of Fe, Al or iron aluminide or such particles/fibers can be formed by reaction synthesis of elemental powders which react exothermically during manufacture of the composite.

The composite can be made in various ways. For instance, the iron aluminide of the composite can be made from a prealloyed powder or by mechanically alloying the alloy constituents. The mechanically alloyed powder can be processed by conventional powder metallurgical techniques such as by canning and extruding, slip casting, centrifugal casting, hot pressing and hot isostatic pressing. Another technique is to use pure elemental powders of Fe, Al and optional alloying elements and mechanically alloying such ingredients. In addition to the above, the above mentioned electrically insulating and/or electrically conductive particles can be incorporated in the powder mixture to tailor physical properties and high temperature creep resistance of the composite.

The composite is preferably made by powder metallurgy techniques. For instance, the composite can be produced from a mixture of powder having different fractions but a preferred powder mixture comprises particles having a size smaller than minus 100 mesh. According to one aspect of the invention, the iron aluminide powder can be produced by gas atomization in which case the powder may have a spherical morphology. According to another aspect of the invention, the iron aluminide powder can be made by water atomization in which case the powder may have an irregular morphology. The iron aluminide powder produced by water atomization can include an aluminum oxide coating on the powder particles and such aluminum oxide can be broken up and incorporated in the composite during thermomechanical processing of the powder to form shapes such as sheet, bar, etc. The alumina particles are effective in increasing resistivity of the iron aluminum

alloy and while the alumina is effective in increasing strength and creep resistance, the ductility of the alloy is reduced.

When molybdenum is used as one of the alloying constituents of the iron aluminide it can be added in an effective range from more than incidental impurities up to about 5.0% with the effective amount being sufficient to promote solid solution hardening of the iron aluminide alloy and resistance to creep of the alloy when exposed to high temperatures. The concentration of the molybdenum can range from 0.25 to 4.25% and in one preferred embodiment is in the range of about 0.3 to 0.5%. Molybdenum additions greater than about 2.0% detract from the room-temperature ductility due to the relatively large extent of solid solution hardening caused by the presence of molybdenum in such concentrations.

Titanium can be added to the iron aluminide in an amount effective to improve creep strength of the iron aluminide alloy and can be present in amounts up to 3%. When present, the concentration of titanium is preferably in the range of $\leq 2.0\%$.

When carbon and the carbide former are used in the iron aluminide alloy, the carbon is present in an effective amount ranging from more than incidental impurities up to about 0.75% and the carbide former is present in an effective amount ranging from more than incidental impurities up to about 1.0% or more. The carbon concentration is preferably in the range of about 0.03% to about 0.3%. The effective amount of the carbon and the carbide former are each sufficient to together provide for the formation of sufficient carbides to control grain growth in the iron aluminide alloy during exposure thereof to increasing temperatures. The carbides may also provide some precipitation strengthening in the iron aluminide alloy. The concentration of the carbon and the carbide former in the iron aluminide alloy can be such that the carbide addition provides a stoichiometric or near stoichiometric ratio of carbon to carbide former so that essentially no excess carbon will remain in the finished alloy.

Zirconium can be incorporated in the iron aluminide alloy to improve high temperature oxidation resistance. If carbon is present, an excess of a carbide former such as zirconium in the iron aluminide alloy is beneficial in as much as it will help form a

spallation-resistant oxide during high temperature thermal cycling in air. Zirconium is more effective than Hf since Zr can form oxide stringers perpendicular to the exposed surface of the iron aluminide alloy which pins the surface oxide whereas Hf forms oxide stringers which are parallel to the surface.

5 The carbide formers include such carbide-forming elements as tungsten, titanium, zirconium, niobium, tantalum and hafnium and combinations thereof. The carbide former is preferably in a concentration sufficient for forming carbides with the carbon present within the iron aluminide alloy. The concentrations for tungsten, niobium, tantalum, titanium, zirconium and hafnium when used as carbide formers can be present in amounts
10 up to 3 wt % each.

In addition to the aforementioned alloy elements the use of an effective amount of a rare earth element such as about 0.05-0.25% cerium or yttrium in the iron aluminide alloy composition is beneficial since it has been found that such elements improve oxidation resistance of the alloy.

15 The oxide filler can be in the form of particles such as powder, fibers, etc. For example, the composite can include up to 40 wt % of oxide particles such as Y_2O_3 , Al_2O_3 , rare earth oxide, beryllia or combinations thereof. The oxide particles can be added to a melt or powder mixture of Fe, Al and other alloying elements. Alternatively, the oxide can be created *in situ* by water atomizing a melt of an aluminum-containing iron-based alloy
20 whereby a coating of alumina or yttria on iron-aluminum powder is obtained. During processing of the powder, the oxides break up and are arranged as stringers in the final product. Incorporation of the oxide particles in the iron aluminide alloy is effective in increasing the resistivity of the alloy. For instance, by incorporating about 0.5-0.6 wt % oxygen in the alloy, the resistivity can be raised from around $100 \mu \Omega \cdot \text{cm}$ to about
25 $160 \mu \Omega \cdot \text{cm}$.

The additive for promoting bonding between the iron aluminide and oxide filler can comprise any element or compound which improves wetting of the iron aluminide, i.e. lowers surface tension and/or contact angle. For instance, the additive can comprise a

carbide which is stable in molten iron aluminide. A preferred additive is a refractory carbide such as TiC, HfC and/or ZrC. During liquid phase sintering wherein the iron aluminide is partially or fully melted, the refractory carbide remains solid and promotes bonding of the oxide filler to the molten iron aluminide matrix.

5 In order to improve thermal conductivity and/or resistivity of the iron aluminide alloy, particles of electrically conductive and/or electrically insulating metal compounds can be incorporated in the alloy. Such metal compounds include oxides, nitrides, silicides, borides and carbides of elements selected from groups IVb, Vb and VIb of the periodic table. The carbides can include carbides of Zr, Ta, Ti, Si, B, etc., the borides can include
10 borides of Zr, Ta, Ti, Mo, etc., the silicides can include silicides of Mg, Ca, Ti, V, Cr, Mn, Zr, Nb, Mo, Ta, W, etc., the nitrides can include nitrides of Al, Si, Ti, Zr, etc., and the oxides can include oxides of Y, Al, Si, Ti, Zr, etc.

Additional elements which can be added to the iron aluminide alloy include Si, Ni and B. For instance, small amounts of Si up to 2.0% can improve low and high
15 temperature strength but room temperature and high temperature ductility of the alloy may be adversely affected with additions of Si above 0.25 wt %. The addition of up to 30 wt % Ni can improve strength of the iron aluminide alloy via second phase strengthening but Ni adds to the cost of the alloy and can reduce room and high temperature ductility thus leading to fabrication difficulties particularly at high temperatures. Small amounts of B can
20 improve ductility of the alloy and B can be used in combination with Ti and/or Zr to provide titanium and/or zirconium boride precipitates for grain refinement.

The invention is now described with reference to the following examples which provide exemplary details of how to make low-cost FeAl-based composites.

FeAl-based composites reinforced with insulating oxide filler can be prepared by a
25 variety of techniques including conventional casting and powder metallurgical processes. However, because oxides are oxidation resistant and have poor electrical conductivity, their presence in iron aluminide composites can be used to increase the electrical resistivity of the composite which is an advantage in resistance heater applications. In the following

examples, fabrication of iron aluminide-oxide composites was carried out using powder metallurgical techniques.

In the following examples, iron aluminide composites were prepared using Al_2O_3 and/or ZrO_2 as the oxide particulates. ZrO_2 , in particular, exhibits a high coefficient of thermal expansion, and has therefore a relatively small thermal mismatch with the iron aluminide matrix. The composites were made by hot-pressing as well as low-cost techniques such as liquid phase sintering.

In order to fabricate FeAl/oxide composites, the following three issues were addressed: (a) the thermodynamic compatibility between the oxides and the iron aluminide matrix, (b) the degree by which oxide particles are wetted by liquid iron aluminides, and (c) the extent to which the wetting behavior can be modified by alloying additions to the iron aluminide. It has been found that Al_2O_3 is thermodynamically compatible with FeAl whereas ZrO_2 is not. Further, while liquid iron aluminide does not wet Al_2O_3 adequately, additions of TiC to FeAl/ Al_2O_3 powder mixtures improves wetting and fabricability. Hot forging of FeAl-15 vol. % TiC-15 vol. % Al_2O_3 composites improved the room temperature flexure strength more than threefold. For instance, room temperature flexure strengths exceeding 1000 MPa can be obtained with the hot-forged composites. Such improvement in mechanical properties may be due to reduction in residual porosity in the composites. In addition, a dramatic improvement of the liquid phase sintering behavior can be obtained by incorporating an additive (e.g., TiC) which promotes wetting of the oxide filler.

Experiments were carried out with FeAl/ Al_2O_3 and FeAl/ ZrO_2 specimens prepared by mixing Fe-40 at. % Al, Al_2O_3 or Y_2O_3 -stabilized ZrO_2 powders and liquid phase sintering them in vacuum at 1450°C or 1500°C. In the following discussion, "FeAl" is intended to denote Fe-40 at. % Al. As a result of X-ray diffraction data, it has been determined that the FeAl/ Al_2O_3 composite included alpha- Al_2O_3 and FeAl and the FeAl/ ZrO_2 composite included cubic stabilized ZrO_2 as well as FeAl. However, there was also evidence of substantial amounts of alpha alumina suggesting a displacement reaction of the type: $3 \text{ZrO}_2 + 24 \text{FeAl} \rightarrow 2 \text{Fe}_3\text{Al} + 3 \text{Fe}_6\text{Al}_6\text{Zr} + 2 \text{Al}_2\text{O}_3$, where $\text{Fe}_6\text{Al}_6\text{Zr}$ is a

ternary intermetallic phase. Consistent with the proposed reaction, electron dispersive spectroscopy (EDS) in a scanning electron microscope (SEM) verified the presence of FeAl, FeAlZr intermetallic and Al_2O_3 .

5 A hot-pressed FeAl/ZrO₂ specimen including 10% Al_2O_3 and 10% ZrO₂ was tested to determine flexure strength. Optical microscopy of the sample revealed that a reaction occurred in the material and chipped edges of flexure bars ground from the material indicated that the material was brittle in nature. The flexure bars fractured in a brittle manner indicating that the iron aluminide had reacted to form more brittle phases. The material exhibited a room temperature flexure strength of 215 ± 29 MPa. As a result of
10 the tests it was determined that ZrO₂ is not thermodynamically stable in contact with FeAl.

In the following experiments, prealloyed iron aluminide powders were mixed with oxide powders. The powder mixtures were then poured into alumina crucibles which were covered with an alumina lid. In most cases, the crucibles had an inner diameter and inner height of 38 mm and 8 mm, respectively. Although the powder mixtures were not cold
15 pressed prior to sintering, cold pressing prior to sintering is expected to improve the fabricability significantly. The filled crucibles were usually pumped overnight to an indicated vacuum better than 10^{-5} Torr. Subsequently, the specimen was ramped to 1450 or 1500°C over a period of 2 h, held for 0.2 to 0.3 h at that temperature, followed by furnace cooling. At 1450 or 1500°C, the iron aluminide melted and liquid phase sintering
20 occurred. Attempts were also made to infiltrate oxide powders with liquid iron aluminide alloys. In a number of cases, elemental Ti or C was added to the binary iron aluminides to enhance the wetting. The best coupons were obtained when a fraction of the oxide powders was replaced by TiC powders. The metal alloy and oxide powders employed in the examples are summarized in Table 1. Table 2 summarizes data obtained from the
25 specimens. Tables 1 and 2 will be used to discuss the various processing experiments carried out.

Figures 1 and 2 show powder X-ray diffraction patterns for specimens A003 (FeAl/ Al_2O_3) and A004 (FeAl/ZrO₂). Consistent with thermodynamic stability, the

diffraction pattern for the FeAl/Al₂O₃ composite indicates mostly α -Al₂O₃ and FeAl. Two small peaks at 21 and 30° could not be identified. The diffraction pattern for the FeAl/ZrO₂ composite indicates cubic stabilized ZrO₂ as well as FeAl. However, there is evidence for substantial amounts of α -alumina suggesting a displacement reaction of the type:



where Fe₆Al₆Zr is a ternary intermetallic phase. The X-ray result is substantiated by Figure 3, in which electron dispersive spectroscopy (EDS) in a scanning electron microscope (SEM) verifies the presence of FeAl, FeAlZr intermetallic, and Al₂O₃. Clearly, ZrO₂ is not thermodynamically stable in contact with liquid FeAl. Once this was found out, processing with ZrO₂ was discontinued.

In iron aluminide composites containing carbides and borides, wetting by liquid iron aluminides is so effective that porous preforms made from these ceramics are readily infiltrated. The applicability of this approach to oxides was investigated (Specimens A005, A006, A011, A012). Iron aluminide powder was placed on a bed of Al₂O₃ or ZrO₂, followed by heating to 1450°C in vacuum in order to melt the iron aluminide. As expected from the literature on the wetting of oxides by liquid metals, infiltration did not occur. A possible solution to this might be addition of a reactive element such as Ti. However, infiltration did also not occur when Ti was added to the iron aluminide powder (A005, A006). Additions of TiC particulates are expected to improve infiltration behavior during liquid phase sintering of FeAl/TiC/Al₂O₃ mixtures.

Experiments were carried out with alumina powder A002, which had a particle size less than 38 μm . Liquid phase sintering of iron aluminides with alumina resulted usually in porous coupons and large amounts of exuded FeAl, which was expelled because of its poor wetting. This is illustrated by Figure 4. When the volume fraction was on the order of 30 wt (specimens A020 and A041) the coupons were very fragile. When the content was lowered to values on the order of 20 wt (A014), the coupons tended to be stronger. An iron aluminide powder A040 gave poor results (specimen A044) apparently because the

powder had a larger particle size than the $<45\ \mu\text{m}$ powder used for other samples (A032). This larger size may have contributed to the poor sinterability. Additions of Ti or C (A007, A016, A018) did not cause noticeable improvements. These results are consistent with the infiltration experiments. However, as shown below, additions of TiC improved the fabricability dramatically.

Partial replacement of Al_2O_3 by TiC improved the fabricability substantially. In coupons A021, A022, and A023 the TiC/ Al_2O_3 ratio was systematically increased. Once the TiC content was increased to sufficiently high levels ($\geq 18\ \text{wt}\%$), the specimens appeared dense and exhibited no or only few surface cracks. Figure 5 shows a successfully processed coupon containing TiC and Al_2O_3 . The raised patches on this coupon appear to be exuded iron aluminide. However, as compared to Figure 4 the wetting is dramatically improved. The microstructure of an FeAl/TiC/ Al_2O_3 coupon is depicted in Figure 6. Although there is still some porosity, many Al_2O_3 particles, such as that in the center of Figure 6, are fully surrounded by FeAl.

Surprisingly, additions of Ti were detrimental to the fabricability (A025, A026, A027). However, small amounts of C (0.3 wt%, specimens A028 and A030) did not degrade the fabricability. Thus, optimized additions of C have the potential to improve the fabricability.

In summary, Al_2O_3 was found to be a suitable reinforcement in iron aluminide cermets. ZrO_2 , on the other hand, was unstable in contact with liquid FeAl, and brittle Fe-Al-Zr intermetallics formed instead. As expected, Al_2O_3 was poorly wetted by liquid iron aluminide. Surprisingly, additions of either Ti or C to the iron aluminide did not improve the wetting of the Al_2O_3 . However, the combined addition of Ti and C, in the form of TiC particulates, improved the wettability dramatically and resulted in much denser coupons.

Various changes and modifications can be made to the process according to the invention. For instance, cold pressing of the powder mixtures can be used to reduce the porosity of the final product. Optimization can be achieved by conducting quantitative density and porosity measurements to determine the concentrations of alloying additions

such as carbon. Further, it is expected that niobium additions will have a beneficial influence on the wetting and bonding of Al_2O_3 by iron aluminides. Instead of prealloyed FeAl powders, elemental Fe and Al powders can also be used as well. In fact, the exothermic reaction between the elemental Fe and Al may be beneficial. Also, elemental powders are softer than prealloyed FeAl powder (which is strongly hardened by frozen-in thermal vacancies) and will therefore result in higher green density. High green densities will lead to higher final densities associated with improved strength and oxidation resistance.

Table 1: Raw materials used in this research

Designation	Composition	Size
A001 ZrO_2 Powder	$\text{ZrO}_2\text{-Y}_2\text{O}_3$ (93-7)	-325 mesh ($\leq 45\mu\text{m}$)
A002 Al_2O_3 Powder	Al_2O_3	-400 mesh ($\leq 38\mu\text{m}$)
A019 Graphite Powder	C	μm range
A024 TiC Powder	TiC	$1.9\mu\text{m}$
A032 FeAl Powder	Fe-40 at. % Al	-325 mesh ($\leq 45\mu\text{m}$)
A033 Ti Powder	Ti, 99.5 % pure	-200 mesh ($\leq 75\mu\text{m}$)
A040 FeAl Powder		
A045 TiC Powder	TiC, 99% metal basis	$2.5\text{-}4\mu\text{m}$

Table 2: Summary of processing experiment

Specimen Number	Composition	Powders used (see Table 1)	Purpose	Findings
A003	Fe40Al-22 wt% Al_2O_3	A032, A002	powder x-ray diffraction, metallography	estimated porosity 20%

Specimen Number	Composition	Powders used (see Table I)	Purpose	Findings
A004	Fe40Al-30 wt% ZrO ₂		powder x-ray diffraction, metallography	estimated porosity 20%
A005	Fe40Al-11 wt% Ti/Al ₂ O ₃	A032, A033, A002	Infiltration attempt	no infiltration found
A006	Fe40Al-11 wt% Ti/Al ₂ O ₃	A032, A033, A001	Infiltration attempt	no infiltration found
A007	(Fe40Al-11 wt% Ti)/20 wt% Al ₂ O ₃	A032, A033, A002	Liquid phase sintering with Ti addition	Porous pellet, exuded FeAl, Pellet electrically conductive
A008	(Fe40Al-11 wt% Ti)/28 wt% ZrO ₂	A032, A033, A001	Liquid phase sintering with Ti addition	Porous pellet, exuded FeAl, Pellet electrically conductive
A009	Fe40Al/15 wt% TiC/12 wt% Al ₂ O ₃	A032, A045, A002	Liquid phase sintering with TiC addition	Dense appearance, exuded patches on top (see macrograph)
A010	Fe40Al/14 wt% TiC/14 wt% ZrO ₂	A032, A045, A001	Liquid phase sintering with TiC additions	Dense appearance, large surface cracks
A011	Fe40Al/ZrO ₂	A032, A001	Infiltration attempt	No infiltration found
A012	Fe40Al/Al ₂ O ₃	A032, A002	Infiltration attempt	No infiltration found

Specimen Number	Composition	Powders used (see Table I)	Purpose	Findings
A013	Fe40A1/30 wt% ZrO ₂	A032, A001	Liquid phase sintering	Porous, fragile pellet, exuded FeAl.
A014	Fe40A1/22 wt% Al ₂ O ₃	A032, A002	Liquid phase sintering	Porous pellet, exuded FeAl
A015	(Fe40A1-11 wt% Ti)/30 wt% ZrO ₂	A032, A033, A001	Liquid phase sintering	Porous pellet, exuded FeAl
A016	(Fe40A1-11 wt% Ti)/22 wt% Al ₂ O ₃	A032, A033, A002	Liquid phase sintering	Porous pellet, exuded FeAl
A017	(Fe40A1-2.9 wt% C)/30 wt% ZrO ₂	A032, A019, A001	Liquid phase sintering	Porous pellet, black and silver areas, no exuded FeAl
A018	(Fe40A1-2.9 wt% C)/22 wt% Al ₂ O ₃	A032, A019, A002	Liquid phase sintering	Porous pellet, exuded FeAl
A020	FeAl/33 wt% Al ₂ O ₃	A032, A002	Liquid phase sintering	Porous pellet, fragile, exuded FeAl
A021	Fe40A1/9 wt% TiC/22 wt% Al ₂ O ₃	A032, A024, A002	Liquid phase sintering	Dense appearance, but many surface cracks. Some exuded FeAl

Specimen Number	Composition	Powders used (see Table 1)	Purpose	Findings
A022	Fe40A1/18 wt% TiC/14 wt% Al ₂ O ₃	A032, A024, A002	Liquid phase sintering	Dense appearance, a few surface cracks, no exuded FeA1
A022B	Fe40A1/18 wt% TiC/14 wt% Al ₂ O ₃	A032, A024, A002	Liquid phase sintering	Dense appearance, a few surface cracks, no exuded FeA1
A023	Fe40A1/27 wt% TiC/7 wt% Al ₂ O ₃	A032, A024, A002	Liquid phase sintering	Dense appearance, a few surface cracks, no exuded FeA1
A025	(Fe40A1-5 wt% Ti)/18 wt% TiC/14 wt% Al ₂ O ₃	A032, A033, A024, A002	Liquid phase sintering	Dense appearance, several surface cracks, exuded FeA1
A026	(Fe40A1-1.4 wt% C)/18 wt% TiC/14 wt% Al ₂ O ₃	A032, A019, A024, A002	Liquid phase sintering	Dense appearance, many surface cracks, exuded FeA1
A027	(Fe40A1-1.1 wt% Ti)/18 wt% TiC/14 wt% Al ₂ O ₃	A032, A033, A024, A002	Liquid phase sintering	Dense appearance, many surface cracks, exuded FeA1

Specimen Number	Composition	Powders used (see Table 1)	Purpose	Findings
A028	(Fe40A1-0.3 wt%C)/18 wt% TiC/14 wt% Al ₂ O ₃	A032, A019, A024, A002	Liquid phase sintering	Dense appearance, a few surface cracks, exuded FeAl
A029	Fe40A1/18 wt% TiC/14 wt% Al ₂ O ₃	A032, A024, A002	Liquid phase sintering	Dense appearance
A030	Fe40A1-0.3 wt%C/18 wt% TiC/14 wt% Al ₂ O ₃	A032, A019, A024, A002	Liquid phase sintering	Dense appearance
A031	Fe40A1/18 wt% TiC/14 wt% Al ₂ O ₃	A032, A045, A002	Liquid phase sintering	Dense appearance, a few surface cracks, exuded FeAl
A041	Fe40A1/30 wt% Al ₂ O ₃	A040, A024, A002	Liquid phase sintering	Fragile coupon, exuded FeAl
A042A	Fe40A1/18 wt% TiC/14 wt% Al ₂ O ₃	A040, A024, A002	Liquid phase sintering	Porous coupon with many surface cracks, exuded FeAl
A043A	Fe40A1/24 wt% Al ₂ O ₃	A032, A002	Liquid phase sintering	Porous coupon, a few surface cracks, exuded FeAl
A043B	Fe40A1/24 wt% Al ₂ O ₃	A032, A004	Liquid phase sintering	Porous coupon, very fragile, exuded FeAl

Specimen Number	Composition	Powders used (see Table 1)	Purpose	Findings
A044	FeAl/24 wt% Al_2O_3	A040, A002	Liquid phase sintering	Porous coupon, very fragile, exuded FeAl

As a result of the above experiments it was determined that FeAl did not wet Al_2O_3 sufficiently well to fabricate FeAl/ Al_2O_3 composites by liquid phase sintering. In order to improve the sintering behavior, some of the Al_2O_3 powder was replaced by TiC powder. For example, specimen A009 was fabricated from Fe-40 at. % Al powder (-325 mesh or $<45\mu\text{m}$), TiC powder ($2.5\text{--}4\mu\text{m}$), and Al_2O_3 powder ($\leq 38\mu\text{m}$), the sample having a nominal composition of FeAl-16.5 vol. % TiC- 16.5 vol. % Al_2O_3 . Compositions and preparation techniques for specimen A009 and additional specimens are set forth in Table 3. The same size powders used for specimen A009 were also used for A046. Specimen A062C was made from powders having the following sizes: $1\text{--}5\mu\text{m}$ Fe, $10\mu\text{m}$ Al, $2.5\text{--}4\mu\text{m}$ TiC and $\leq 38\mu\text{m}$ Al_2O_3 . The liquid phase sintering was carried out as follows: 0.3 h in vacuum for specimen A009, 0.2 h in vacuum for specimen A046, 0.2 h in vacuum for specimen A047, 0.2 h in vacuum for specimen A050, and 0.2 h in vacuum for specimen A062C.

Table 3.

Specimen Number	Composition	Powders Used For Iron Aluminide	Processing Technique
A009	FeAl-16.5vol%TiC-16.5vol% Al_2O_3	Prealloyed FeAl, TiC and Al_2O_3	Liquid phase Sintering at 1450°
A046	FeAl-16.5vol%TiC-16.5vol% Al_2O_3	Prealloyed FeAl, TiC and Al_2O_3	Liquid phase Sintering at 1500°C
A047	FeAl-16.5vol%TiC-16.5vol% Al_2O_3	Fe and Al	Liquid phase Sintering at 1500°C
A050	FeAl-9wt%Nb-16.5vol%TiC-16.5vol% Al_2O_3	Prealloyed FeAl and Nb	Liquid phase Sintering at 1500°C

A055	FeAl-10%Al ₂ O ₃ ZrO ₂	Prealloyed FeAl	Hot Pressed
A062C	FeAl-15vol%TiC- 15vol%Al ₂ O ₃	Fe, Al, TiC and Al ₂ O ₃	Liquid phase Sintering at 1500°C and hot forging at 1000°C from 20 to 8 mm

Table 4.

5	Specimen	Composition at. %	Flexure Strength MPa
	A046E-1	FeAl-15vol%TiC- 15vol%Al ₂ O ₃	304
	A050A-1	FeAl-9wt%Nb- 16.5vol%TiC- 16.5vol%Al ₂ O ₃	189
	A050A-2	FeAl-9wt%Nb- 16.5vol%TiC- 16.5vol%Al ₂ O ₃	185
	A055#1	FeAl-10vol%Al ₂ O ₃ - 10vol%ZrO ₂	212
10	A055#1	FeAl-10vol%Al ₂ O ₃ - 10vol%ZrO ₂	217
	A055#1	FeAl-10vol%Al ₂ O ₃ - 10vol%ZrO ₂	249
	A055#2	FeAl-10vol%Al ₂ O ₃ - 10vol%ZrO ₂	169
	A055#2	FeAl-10vol%Al ₂ O ₃ - 10vol%ZrO ₂	226
	A062C#1	FeAl-15vol%TiC- 15vol%Al ₂ O ₃	996
15	A062C#1	FeAl-15vol%TiC- 15vol%Al ₂ O ₃	1081
	A062C#1	FeAl-15vol%TiC- 15vol%Al ₂ O ₃	1160

A062C#2	FeAl-15vol%TiC- 15vol%Al ₂ O ₃	1099
A062C#2	FeAl-15vol%TiC- 15vol%Al ₂ O ₃	1202
A062C#2	FeAl-15vol%TiC- 15vol%Al ₂ O ₃	1173
A062C#3	FeAl-15vol%TiC- 15vol%Al ₂ O ₃	1056
A062C#3	FeAl-15vol%TiC- 15vol%Al ₂ O ₃	981

Specimens with the nominal composition FeAl-16.5vol%TiC-16.5vol%Al₂O₃ were also fabricated by cold-pressing and subsequent sintering for 12 minutes at 1500°C in vacuum. Similar results were achieved using prealloyed FeAl (specimen A046) or elemental Fe and Al powders (specimen A047). However, the composite fabricated from elemental powders may have a slightly lower porosity level. In specimen A050, elemental Nb was added to the composite with the expectation that Nb would bond well to the Al₂O₃ and improve fracture toughness.

In the experiments, it was found that fully dense material was not produced during liquid phase sintering even when TiC was added to the composite material. As such, secondary processing was utilized to remove the pores. Specimen A062C was made by mixing 60 g of Fe, Al, TiC and Al₂O₃ and liquid phase sintering the mixture in an Al₂O₃ crucible to provide a FeAl-15TiC-15Al₂O₃ (vol. %) composite. The sintered cylinder was hot forged at 1000°C from a height of 20 mm to approximately 8mm. The hot forged coupon is shown in Figure 7 wherein edge cracking can be seen around the periphery of the coupon and the interior of the coupon is sound.

Figure 8 is an optical micrograph of specimen A046 fabricated with prealloyed Fe40Al powder. The bright TiC particles, dark Al₂O₃ particles, with black pores surrounded by gray iron aluminide matrix are clearly visible. Processing with elemental

Fe and Al powders, instead of prealloyed FeAl powder, gave similar results except that the porosity levels may have been lower. Figure 9 shows the microstructure of a hot forged coupon (A062C) wherein there is an absence of porosity.

Specimens for room temperature flexure tests were prepared by grinding samples having a cross section of approximately 3x4 mm. The flexure tests were carried out with a span of 20 mm and a cross head speed of 10 $\mu\text{m/s}$. The fracture stress σ_f was calculated from the linear-elastic equation: $\sigma_f = 1.5 L \cdot P / (wt^2)$, where L is the span, P is the load at fracture, w is the specimen width and t is the specimen thickness.

The strength of liquid phase sintered FeAl-16.5TiC-16.5Al₂O₃ (vol. %) exceeded 300 MPa (specimen A046E-1). Fracture occurred not catastrophically, but in a gradual manner by controlled crack propagation. The reason for the gradual fracture is thought to be the porosity of the material which did not permit sufficient storage of elastic energy to result in catastrophic fracture. The Nb-alloyed material A050 exhibited fracture in a gradual manner and had a much lower strength of 187 MPa which is presumably due to its higher porosity. Although the Nb may have strengthened the interfacial Al₂O₃/FeAl bonding, this could not be verified because of the negative effect of the high porosity levels.

Hot forging resulted in a pronounced strength increase. Figure 10 shows three stress displacement curves for bend bars machined from coupon A062C (FeAl-15TiC-15Al₂O₃, vol. %). The curves demonstrate not only a high strength, but also a small amount of ductility. The beneficial effect of the hot forging is attributed to the removal of porosity. Some specimens were annealed for 1 day at 500°C in order to remove thermal vacancies which were presumably frozen in during the hot forging. The removal of excess vacancies in iron aluminides results in a reduction of the high yield strength and an increase in ductility. Although the anneal was expected to reduce the flaw sensitivity and increase fracture strength, it was found that the anneal did not affect the fracture strength significantly.

The room temperature fracture toughness of the hot forged FeAl-15TiC-15Al₂O₃ composite was determined from the controlled fracture of chevron-notched specimens. Figure 11 shows a measured load-displacement curve. The fracture toughness was evaluated from the equation: $K_Q = [(W/A)E']^{1/2}$ where W is the absorbed energy (which corresponds to the area under the load-displacement curve), A is the area traversed by the crack, and E' is the plane strain Young's modulus, namely $E/(1-\nu^2)$. A value of 0.25 was assumed for ν . The Young's modulus E was estimated from the following equation: $E = [(cE_p E_m + E_m^2)(1+c)^2 - E_m^2 + E_p E_m] / [(cE_p + E_m)(1+c)^2]$ where $c = (1/V_p)^{1/3} - 1$. V_p is the volume fraction of the ceramic particles, E_p and E_m are the moduli of the ceramic phases (estimated to be 410 GPa) and the matrix (180 GPa). Using the above equations, the Young's modulus for FeAl-15TiC-15Al₂O₃ (vol. %) is estimated to be 228 Gpa.

The fracture toughness of two specimens evaluated in this manner are listed in Table 5. Considering the relatively low fracture toughness of monolithic iron aluminides (30-50 MPa m^{1/2}), the composites exhibited satisfactory fracture toughnesses.

Table 5. Fracture Toughness of Hot Forged FeAl-15TiC-15Al₂O₃ (vol. %)

Sample	W	H	W	A	G _i	E	K _Q
A062C#	6.59	1.66	2.67	2.216	2973.7	228.0	26.9
A062C	7	1.7	2.8	2.38	2941.2	228.0	26.7

From the foregoing discussion it can be appreciated that Al₂O₃ is not wetted well enough by liquid FeAl to allow the processing of composites by liquid phase sintering. In contrast to Al₂O₃, ZrO₂ is thermodynamically not stable in contact with iron aluminides. Since brittle intermetallic phases form during the reaction between ZrO₂ and FeAl, ZrO₂ is less desirable as a filler in FeAl/ceramic composites. On the other hand, TiC promotes wetting of Al₂O₃ by FeAl. Moreover, instead of prealloyed FeAl, elemental Fe and Al powders may be used for liquid phase sintering of FeAl/TiC/Al₂O₃ composites. Additions of refractory metals such as Nb may improve the properties of the composites provide porosity can be reduced to acceptable levels. Room temperature flexure strengths of

approximately 300 MPa can be achieved for liquid phase sintered iron aluminide composites containing TiC and Al_2O_3 . Hot forging of liquid phase sintered FeAl-TiC- Al_2O_3 composites can increase the room temperature flexure strengths to approximately 1000 MPa as well as provide fracture toughness on the order of $27 \text{ MPa m}^{1/2}$.

5 The foregoing has described the principles, preferred embodiments and modes of operation of the present invention. However, the invention should not be construed as being limited to the particular embodiments discussed. Thus, the above-described
embodiments should be regarded as illustrative rather than restrictive, and it should be appreciated that variations may be made in those embodiments by workers skilled in the art
10 without departing from the scope of the present invention as defined by the following claims.

WHAT IS CLAIMED IS:

1. An iron aluminide composite comprising iron aluminide, an oxide filler, and an additive present in an amount which improves metallurgical bonding between the oxide filler and the iron aluminide.

2. The iron aluminide composite of claim 1, wherein the iron aluminide composite comprises a liquid phase sintered composite which is Cr-free, Mn-free, Si-free and/or Ni-free.

3. The iron aluminide composite of claim 1, wherein the additive comprises 2 to 40% titanium carbide and the oxide comprises 2 to 40% alumina.

4. The iron aluminide composite of claim 1, wherein the iron aluminide composite includes $\leq 40\%$ by weight of oxide filler in the form of particles or fibers, the oxide filler being present in an amount equal to 1 to 3 times the amount of the additive.

5. The iron aluminide composite of claim 1, wherein the oxide filler comprises 10 to 25 vol.% alumina and the additive comprises 10 to 25 vol.% TiC.

6. The iron aluminide composite of claim 1, wherein the iron aluminide includes $\leq 2\%$ Mo, $\leq 2\%$ Ti, $\leq 1\%$ Zr, $\leq 2\%$ Si, $\leq 30\%$ Ni, $\leq 0.5\%$ Y, $\leq 0.1\%$ B, $\leq 15\%$ Nb, $\leq 1\%$ Ta, $\leq 3\%$ Cu and $\leq 3\%$ W.

7. The iron aluminide composite of claim 1, wherein the iron aluminide consists essentially of 20.0-31.0% Al, $\leq 1\%$ Mo, 0.05-0.15% Zr, $\leq 0.1\%$ B, 0.01-0.2% C, $\leq 3\%$ W, balance Fe.



8. The iron aluminide composite of claim 1, wherein the iron aluminide consists essentially of 14.0-20.0% Al, 0.3-1.5% Mo, 0.05-1.0% Zr, $\leq 0.1\%$ B, $\leq 0.2\%$ C, ≤ 2.0 Ti, $\leq 3\%$ W and balance Fe.

9. The iron aluminide composite of claim 1, wherein the iron aluminide consists essentially of 20.0-31.0% Al, 0.3-0.5% Mo, 0.05-0.3% Zr, $\leq 0.2\%$ C, $\leq 0.1\%$ B, $\leq 0.5\%$ Y, $\leq 2\%$ W and balance Fe.

10. The iron aluminide composite of claim 1, wherein the iron aluminide composite is in the form of an electrical resistance heating element having a room temperature resistivity of $80 - 400\mu\Omega \cdot \text{cm}$.

11. The iron aluminide composite of claim 10, wherein the electrical resistance heating element heats to 900°C in less than 1 second when a voltage up to 10 volts and up to 6 amps is passed through the composite.

12. The iron aluminide composite of claim 10, wherein the electrical resistance heating element exhibits a weight gain of less than 4% when heated in air to 1000°C for three hours.

13. The iron aluminide composite of claim 10, wherein the electrical resistance heating element has a resistance of 0.5 to 7 ohms throughout a heating cycle between ambient and 900°C .

14. The iron aluminide composite of claim 1, wherein the oxide comprises alumina, yttria, rare earth oxide and/or beryllia, and the additive comprises at least one refractory carbide, refractory nitride or refractory boride.



15. The iron aluminide composite of claim 1, wherein the iron aluminide comprises, in weight %, over 4% Al, $\leq$ 1% Cr.

16. A powder metallurgical process of making an iron aluminide composite comprising steps of:

mixing a powder of iron and aluminum with an oxide powder and an additive present in an amount which increases metallurgical bonding of the oxide powder to the iron aluminide;

forming a mass of the powder into a body; and

sintering the body sufficiently to form a composite of the iron aluminide and oxide powder.

17. The process of Claim 16, wherein forming comprises hot or cold pressing.

18. The process of Claim 16, wherein the sintering comprises solid state sintering, partial liquid phase sintering wherein part of the iron aluminide is melted or liquid phase sintering wherein all of the iron aluminide is melted.

19. The process of Claim 16, wherein the forming comprises placing the powder in a metal can and hot extruding the metal can into a rod, bar, tube, or other shape.

20. The process of Claim 16, wherein the iron aluminide is a binary alloy.

21. The process of Claim 16, wherein the oxide powder comprises alumina, zirconia, rare earth oxide and/or beryllia powder and the additive comprises at least one refractory carbide, refractory nitride or refractory boride.



22. The process of Claim 16, wherein the powder of iron and aluminum comprises prealloyed FeAl powder or elemental powders of at least iron and aluminum.

23. The process of Claim 16, wherein the oxide powder is present in an amount equal to 1 to 3 times the amount of the additive.

24. The process of Claim 16, wherein the oxide powder consists essentially of Al_2O_3 and the additive consists essentially of TiC.

25. The process of Claim 16, wherein the oxide powder has particle sizes of 0.01 to 10 μm .

26. The process of Claim 16, further comprising forming the body into an electrical resistance heating element.

27. The process of Claim 16, wherein the body is formed into a shaped body by placing elemental powders of Fe and Al in a metal can, sealing the can and heating the sealed metal can such that the powders undergo reaction synthesis and form the iron aluminide during the extruding.

28. The process of Claim 16, wherein the sintering is carried out in a vacuum or an inert gas atmosphere.

29. The process of Claim 28, wherein the inert gas atmosphere comprises hydrogen.

30. The process of Claim 16, wherein the body is formed into an electrical resistance heating element having a room temperature resistivity of 80 - 400 $\mu\Omega\text{-cm}$.

