# DEVELOPMENT OF A HIGH SPECIFIC STIFFNESS MECHANICALLY MILLED FeAl INTERMETALLIC ALLOY

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## ABSTRACT

Ordered FeAl intermetallic alloys are attractive materials for medium and high temperature industrial applications but their use has been restricted until now by their room temperature brittleness and their poor creep resistance. In this study, powder metallurgy (P/M) techniques such as gas atomization and mechanical milling have been used to develop a FeAl alloy with enhanced ductility and strength at both low and high temperature. The improvement method combines ductilization by grain boundary strengthening, grain size reduction and oxide dispersion strengthening.

This material has been characterized and tested in the form of extruded bars. Microstructure, order and texture of as-extruded and heat treated samples have been studied by TEM, X-ray diffraction and Mössbauer spectroscopy. Physical and mechanical properties of the material are compared to some conventional engineering alloys in order to discuss the conceivable applications in aeronautical and automotive industries.

## INTRODUCTION

Iron aluminides based on FeAl (ordered bcc-type B2 structure) exhibit an excellent corrosion resistance in oxydizing, sulfurizing and carburizing environments up to 1000°C, good erosion resistance, high strength and elastic modulus coupled with a relatively low density ($< 6$ g.cm$^{-3}$ i.e. 25% lower than that of conventional intermediate temperature materials, steels and nickel base alloys) and a low raw material cost [1-2]. Because of their high specific strength and stiffness (i.e. referred to density), these intermetallics stand as candidates for the fabrication of reactors or engines parts up to 600°C; their excellent corrosion resistance up to 1000°C allows to imagine their use at higher temperature in corrosive environments for the fabrication of components for engines or turbines, heat exchangers... Although these alloys have been studied for many years [14], their use has been impeded until now by two major drawbacks: room temperature brittleness and poor creep resistance.

Over the years, considerable effort has been expended to ductilize FeAl alloys at low temperature. As opposed to stoichiometric Fe-50 at.%Al alloys which are completely brittle [3-5], it has been determined that a limited plasticity can be obtained by increasing the iron content. Nevertheless, fracture remains predominantly intergranular [4-9]. Since these alloys are known to deform by <111>{110} slip at low temperature [10-13], intergranular fracture does not result from failure to satisfy Von Mises criterion of five independent slip systems. Based upon these observations, initial efforts to ductilize iron-rich FeAl intermetallics were aimed at increasing the grain boundary cohesive strength.

Heat treatments promoting the formation of carbides have been found to enhance the ductility of iron-rich FeAl alloys by reducing carbon segregation at grain boundaries. Sainfort and al. [14] have shown that additions of up to 0.1 wt.%Zr also promote carbide formation and increase ductility. As for $Ni_3Al$, boron doping enhances the grain boundary strength of iron-rich FeAl alloys and promotes transgranular cleavage fracture [3-6, 8]. However, this concentration must be optimized [3-4] because, at large B concentrations, interstitial strengthening elevates the yield strength and cleavage occurs before plastic elongation, thus the benefits of enhanced grain boundary strengthening are not observed.

Room temperature ductility of iron-rich FeAl can also be enhanced by slow cooling the alloys from elevated temperatures in order to reduce the point defect concentration [4-5]. The result is a decrease of the yield strength which permits yielding at stresses lower than that required for grain boundary decohesion. Although similar behaviour has been observed for B containing alloys, the effect is less pronounced than for non-B containing alloys.

The poor grain boundary strength is not the only cause of low temperature embrittlement of iron-rich FeAl. Recent investigations have revealed significant increase in the ductility when moisture is excluded from the test environment [1, 5, 8-9]. Liu and al. [8-9] have proposed that this embrittlement results from an extrinsic reaction between the water vapor and Al atoms at the surface resulting in the formation of $Al_2O_3$ and atomic hydrogen, the latter of which subsequently embrittles the material.

Nagpal and Baker [15] have shown that the elongation of Fe-45 at.%Al tested in air is independent of strain rate up to $10^{-2}$ $s^{-1}$ but increases with increasing strain rate at faster rates up to 1 $s^{-1}$. Webb and Lefort [16] have studied the coupled effects of strain rate and environment on the room temperature ductility of a FeAl alloy with a composition corresponding to optimum grain boundary strengthening (Zr and B doping). They have observed that ductility is dramatically enhanced with the increase of strain rate when tensile tests are performed in air. On the contrary, when testing under vacuum, plastic elongation is not influenced by strain rate. Observation of fracture surfaces indicates that embrittlement occurs as the result of premature cleavage fracture in affected grains at the periphery of the specimens. Thus, it has been proposed that environmental embrittlement of iron-rich FeAl at ambient temperature results from a time-dependent weakening of cleavage planes in affected grains probably due to the absorption of hydrogen produced by the decomposition of atmospheric water vapor. Removing the environment (or increasing the strain rate) enhances the ductility by eliminating (or reducing the number of) affected grains.

## DEVELOPMENT OF A FeAl ALLOY WITH ENHANCED ROOM TEMPERATURE DUCTILITY

Webb and Lefort [16] have combined the known concepts on the room temperature brittleness of iron-rich FeAl to build a schematic model taking into consideration material and experimental variables (composition and environment) and illustrating the grain size dependence of the yield strength, the grain boundary strength and the cleavage fracture strength. This model has been used to design an iron-rich FeAl intermetallic with enhanced ductility at ambient temperature/environment [17]. This has been obtained by simultaneously maximizing the grain boundary cohesive strength (B and Zr additions) and increasing the cleavage fracture strength (refinement of the grain size).

The composition of this alloy, designated FeAl40 Grade 3, has been chosen to provide maximum grain boundary strengthening (Fe bal., 24 wt.%Al, 0.11 wt.%Zr, 15 wppm B). The grain size reduction has been obtained by mechanical milling, a powder metallurgy technique. In this technique, prealloyed metallic powders are milled very energetically with steel balls under controlled atmosphere. Due to repeated shocks with the steel balls, the powders are repeatedly flattened, fractured and rewelded. The accumulation of dislocations in the material induces the formation of ultrafine crystallites [19], typically a few tens of nanometers. Mechanical milling has also been used to introduce a nanometer size oxide dispersion ($Y_2O_3$) within the microstructure in order to inhibit grain growth during heating of the material by pinning of grain boundaries. Consequently, after hot extrusion of the milled powder, the microstructure remains very fine. Another benefit of the dispersed oxide particles is to improve the high temperature strength of the material by trapping dislocations at dispersoid/matrix interfaces [20]. Once trapped, the dislocations must bow out then break away from the particles to continue moving. The total line length (and thus energy) of the dislocations increases due to the interaction with the dispersoids requiring higher stresses and/or temperature.

Recent work has lead to optimize the elaboration of FeAl40 Grade 3 alloy [21]. In a first step, master alloy ingots with the above-mentioned composition are cast from pure metals. Then, this material is argon atomized and sieved to produce a fine spherical FeAl intermetallic powder. Dry milling is performed under pure argon atmosphere in a pilot scale rotating ball-mill (up to 10 kg of powder per batch) charged with 100 kg of steel balls (fig. 1). Yttrium oxide (1 wt.%) is added in the form of powder with granulometry < 1 μm. All handling operations are realized in very clean conditions to limit atmospheric contamination and avoid inclusions. Chemical analysis of initial and milled powers have shown that the milling operation brings about 1000 wppm of oxygen. A slight carbon contamination (about 200 wppm) is due to the wear of the steel balls.

After milling, oxide dispersion strengthened FeAl powders are canned and degassed then extruded at 1100°C to produce bars (fig. 2) with diameters ranging from 10 to 30 mm. About 100 kg of material have been produced.

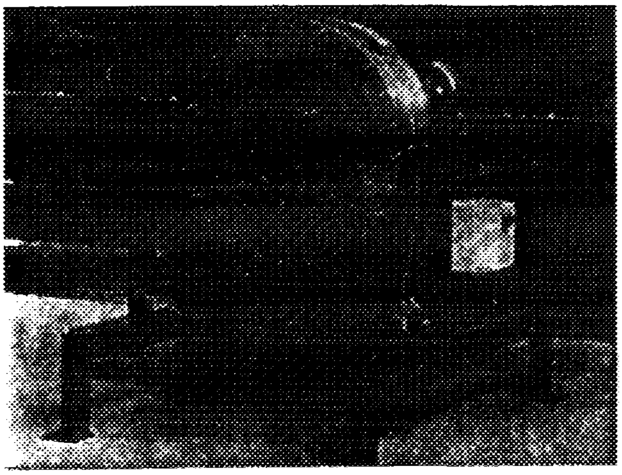

Figure 1. Pilot scale rotating ball-mill (up to 10 kg per batch) used to produce FeAl40 Grade 3

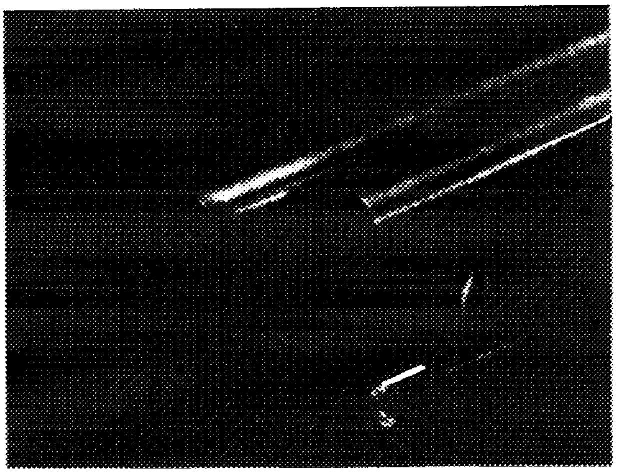

Figure 2. Extruded bar of FeAl40 Grade 3 alloy

As-extruded FeAl40 Grade 3 (fig. 3-4) exhibits a very fine grain size (approximately 1 μm). Two types of dispersoids have been observed in the microstructure: very fine yttrium oxide particles (20-30 nm) and coarser particles containing Al, Y and O identified as $(AlY)_2O_3$.

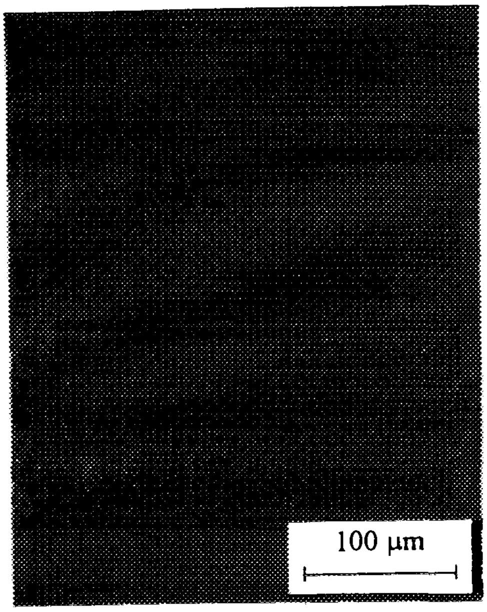


Figure 3: Microstructure of as-extruded FeAl40 Grade 3

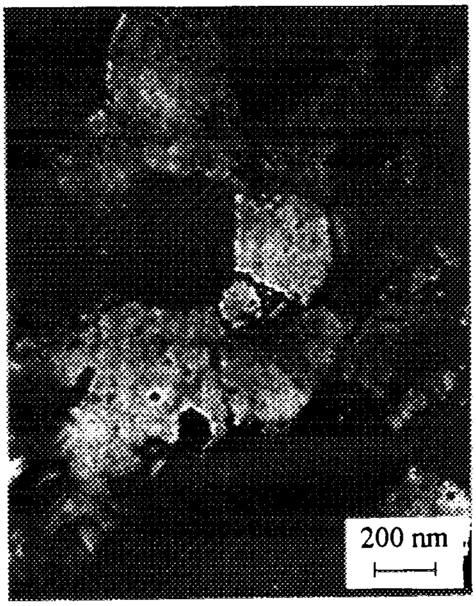


Figure 4: TEM micrograph of FeAl40 Grade 3

Samples annealed at temperatures lower than 1200°C during 1 to 10 hours keep a fine structure, similar to that of as-extruded material. On the contrary, after 1250°C-1 h annealing, FeAl40 Grade 3 exhibits a bimodal grain size distribution with a majority of fine grains and a small percentage of coarse grains (about 100 µm). Stout and Crimp [22] have previously observed such an abnormal grain growth in an extruded prealloyed Fe-40 at.%Al after annealing at 1050°C. The very higher temperature in the case of FeAl40 Grade 3 alloy is due to the pinning of grain boundaries by the homogeneous oxide dispersion. After annealing at 1300°C, the material is fully recrystallized and the structure is composed mainly of large grains (200 µm in tranverse direction, > 500 µm in extrusion direction).

Determination of texture in as-extruded and annealed materials was made by pole figures examination. As-extruded material exhibits a 110-oriented fiber texture in the extrusion direction. No modification is observed after annealing at 1000°C-1 hour. On the contrary, the primary recrystallization obtained after annealing at 1100°C and 1200°C induces a strengthening and a homogenization of this 110 fiber texture. At 1250°C, pole figure still shows an intense 110 fiber texture but azimutal reinforcements traducing the presence of coarse disoriented grains are also observed. This is in good agreement with the bimodal grain size induced by the abnormal recrystallization phenomenon.

Magnetic properties of FeAl alloys with Al content close to 50 at.% are very sensitive to the order degree. In the perfectly ordered state, the Curie temperature $T_C$ is below room temperature and the alloy has a zero average hyperfine field while when disorder increases, $T_C$ and the average magnetic moment of Fe atoms (which is strongly dependent on the number of first Al neighbours) increase. Consequently, the study of order in FeAl40 Grade 3 has been made by Mössbauer spectroscopy, a magnetic technique that gives access to the local environment of iron atoms. Mössbauer spectrum of initial atomized powder is composed of a single line characterizing the non-magnetic B2 ordered phase. On the contrary, as-milled powder appears to be fully disordered. After extrusion, the Mössbauer spectrum is very similar to that of atomized powder indicating that re-ordering occurs during preheating and consolidation steps.

## PROPERTIES AND APPLICATION PROSPECTS

FeAl40 Grade 3 specimens have been simply machined from the extruded bars and tensile tests have been conducted in the less favourable conditions (in air, non polished samples, low initial strain rate $3.10^{-4}\ s^{-1}$). Room temperature tensile properties are shown in Table I and compared with those of an unreinforced FeAl alloy with the same matrix composition produced by extrusion of atomized powder [16]. These figures are very close to the ones obtained by Strothers and Vedula [18] on an O.D.S. FeAl elaborated by similar P/M techniques (grain size about 5 µm) but it must be noticed that, in this case, tensile tests were conducted on electropolished samples what is very favourable and skew the comparison (Lefort and al. [1,16] have demonstrated that electropolishing of specimens can artificially increase the room temperature ductility of FeAl alloys).

Table I. Room temperature tensile properties of FeAl40 Grade 3 compared to unreinforced FeAl alloy with the same composition

| | Unreinforced FeAl | FeAl40 Grade 3 (as-extruded) |
|---|---|---|
| Yield strength *(MPa)* | 518 | 894 |
| Rupture strength *(MPa)* | 532 | 1147 |
| Plastic elongation *(%)* | 0.6 | 6.4 |

In Table II, physical and mechanical properties (at 20°C and 500°C) of FeAl40 Grade 3 are compared to those of some conventional metallic alloys. Very few creep data are available today. Test conducted at 500°C, in air, on as-extruded material indicate a 100-h rupture stress of 440 MPa. Lefort and al. [17] have studied the effect of an yttrium oxide dispersion on the creep behavior of a FeAl alloy. Both dispersion strengthened and unreinforced materials were prepared by mechanical milling and extrusion. Constant load creep tests were performed at 700°C under a constant stress of 55 MPa. They have observed that oxide dispersion dramatically increases the creep resistance of the FeAl matrix by almost 3 orders of magnitude as the steady state creep rate of the unreinforced FeAl was $3\times10^{-3}$/hr compared to $8\times10^{-6}$/hr for the material with $Y_2O_3$ dispersion.

From the currently known properties, the strong points of this alloy appear to be:
- a low density and high specific strength compared to steels and nickel alloys,
- a high specific stiffness compared to light alloys (Al, Ti), steels and nickel alloys,
- a high ductility compared to other intermetallics (TiAl, NiAl),
- a high mechanical strength up to 600°C compared to aluminium alloys and polymer matrix composites,
- a high dry corrosion resistance compared to most stainless steels and nickel alloys.

Although properties data are today very scarce on numerous points (toughness, fatigue, machinability, weldability, wear resistance, corrosion in salted spray ...), it is however possible to consider FeAl40 Grade 3 as a candidate for the substitution of light alloys, steels or nickel alloys in industrial applications taking advantage of its specificities:

- its density 25% lower than that of steels and nickel alloys can be favourably used to lighten some structural parts in aeronautical industry in order to reduce the total weight of aircrafts or helicopters and thus increase performances, reduce fuel consumption and limit atmospheric pollution. Examples of applications can be aeronautical nuts and bolts, landing gears, brakes...

- thanks to its high specific strength, FeAl40 Grade 3 can also be considered for the substitution of some critical high speed moving/rotating components of reactors or engines generally fabricated with high strength materials regardless of their density (steels and nickel alloys). The mass reduction of such moving parts generally diminishes problems of inertia, friction and vibration thus reducing overheating and power loss. Consequently, this substitution can allow to reduce dramatically the number or the size of annex components such as bearings, fixing or clamping systems, springs, cooling systems... which can induce a large weight reduction by "snow ball effect". Examples of such critical parts are clack valves, poppet valves, axles, piston axles, transmission shafts, turbine blades.

- a very particular interest of the alloy is the high specific stiffness $\sqrt{E/\rho}$ (10 to 20% higher than that of light alloys, steels and nickel alloys). This property can be used to improve the efficiency and the performances of reactors, for instance, by increasing the critical rotating speed of a shaft to avoid resonance or allowing to increase the flow in injection nozzles without vibration problems.

- taking into account the excellent corrosion resistance of the material, components such as furnace resistors or heat exchanger tubes can be considered.

Table II. Physical and mechanical properties of FeAl40 Grade 3 (as-extruded) compared to some conventional industrial alloys (typical values).

| | FeAl40 Grade 3 | Aluminium alloy 2024 (T6) | Titanium alloy TA6V (P/M) | Stainless steel 316L | Nickel alloy 625 | Superalloy IN100 |
|---|---|---|---|---|---|---|
| Density | 5.9 | 2.77 | 4.43 | 8 | 8.44 | 7.75 |
| Melting temp. *(°C)* | 1310 | 502 | 1600 | 1375 | 1290 | |
| Thermal expansion coeff. *($10^{-6}$/°C)* | 25 | 22 | 9 | 15 | 12.8 | 13 |
| Thermal conductivity *(W/m.K)* | 12 | 151 | 7 | 16 | 10 | 17 |
| <u>20°C</u> | | | | | | |
| Yield strength *(MPa)* | 894 | 393 | 860 | 170-310 | 517 | 850 |
| Rupture strength *(MPa)* | 1147 | 476 | 930 | 480-620 | 930 | 1010 |
| Elongation *(%)* | 6.4 | 10 | 13 | 30-40 | 43 | 9 |
| Young modulus *(GPa)* | 200 | 72 | 114 | 190-215 | 207 | 215 |
| Specific stiffness $\sqrt{E/\rho}$ *$(MPa/g.cm^{-3})^{1/2}$* | 184 | 161 | 156 | 154-164 | 157 | 167 |
| Specific strength YS/ρ *$(MPa/g.cm^{-3})$* | 188 | 142 | 191 | 21-39 | 61 | 110 |
| <u>500°C</u> | | | | | | |
| Yield strength *(MPa)* | 663 | | 500-550 | 108 | 405 | 885 |
| Rupture strength *(MPa)* | 704 | | 600-770 | | 745 | 1090 |
| Elongation *(%)* | 32 | | 22 | | 50 | |
| Young modulus *(GPa)* | 110 | | 91 | 155 | | 190 |
| Specific stiffness $\sqrt{E/\rho}$ *$(MPa/g.cm^{-3})^{1/2}$* | 139 | | 143 | 140 | | 158 |
| Specific strength YS/ρ *$(MPa/g.cm^{-3})$* | 112 | | 111-122 | | | 114 |

## CONCLUSION

Using the known concepts on the room temperature brittleness of FeAl intermetallics, a new iron-rich FeAl alloy with enhanced ductility and strength has been developed by powder metallurgy techniques, in particular gas atomization and mechanical milling. Maximization of the grain boundary strength by optimization of composition has been combined with an increase of the fracture strength by grain size reduction. According to a previous work performed in the same laboratory by Lefort and Webb, this improvement results from a decrease of the sensitivity of the material towards environment. In addition, a nanometer size oxide dispersion has been introduced in the matrix to stabilize the small grain size and improve the high temperature mechanical properties.

The obtained material (FeAl40 Grade 3) tested in the form of extruded bars exhibits a dramatic increase of both yield strength, rupture strength and ductility compared to conventional FeAl intermetallic. Such mechanical properties allow to imagine industrial applications, in particular in aeronautical industries, for the manufacturing of components working at temperatures up to about 600°C. Because of the high specific properties of the material (strength and stiffness referred to density), the substitution of steels and nickel alloys for the fabrication of high speed moving/rotating parts of reactors and engines could be of particular interest.

FeAl40 Grade 3 alloy has been selected as a reference material in the scope of CEASI (Concerted European Action on Structural Intermetallics), a four year programme approved by the EEC. In this programme, several industrial and university laboratories are now characterizing and testing this material to increase the amount of properties data. Subjects of particular interest are high temperature properties, creep, fatigue and friction welding.

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