PRODUCTION AND PROPERTIES OF CSM FeAl INTERMETALLIC ALLOYS

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ABSTRACT

Fe40Al intermetallic compounds have been investigated in order to assess their potential applications. These alloys show excellent corrosion resistance and good mechanical properties at high temperature. The first phase of the research carried out at CSM was focused on fabrication of such alloys by both conventional and innovative technologies. This paper gives an overview of the work performed at CSM: several processing and characterization results are described and compared with other well known commercial alloys.

Some automotive and mechanical components demonstrators, manufactured by different processes, are also showed.

INTRODUCTION

The research efforts in the intermetallic compositions field have risen in the last ten years, with particular emphasis on the Ni, Ti and Fe based aluminides.

The Centro Sviluppo Materiali (CSM) interests were mainly concerned with Fe-Al alloys. B2 FeAl ordered alloys were investigated for their potential moderate-high temperature applications combined with a relative low density, (d = 5.6 g/cm³), and low raw materials costs [1]. Two factors limit the application of these alloy: poor room temperature ductility and inadequate high temperature creep resistance. For this reason a special powder technology, Mechanical Alloying, was employed to reinforce the material with a fine oxide dispersion. The aim of this work is to give an overview of the outstanding results obtained at CSM and the details of fabrication technology are discussed. The process investigated include conventional melting and casting technology and powder processing. Mechanical and corrosion properties [2-7], on products made by various processes will be also presented and compared. Fe40Al mechanical alloyed displays room temperature elongation A% up to 13,5%. The high temperature sulphidation resistance of FeAl is more than 4 time the resistance of MA956 commercial alloy. Determination of brittle to ductile transitions versus strain rates and temperatures permits hot forging of FeAl: some components are shown.

The results obtained show the potential for FeAl application as replacement of both austenitic and ferritic steels in high temperature aggressive environments [8].

Materials and Processing Technologies

FeAl alloys were prepared by two main preparation routes: conventional melting and casting technologies and powder processing. The processing technology effort has been focused on four FeAl alloys, reported in Tab. I.

Tab. I - *Chemical analysis (%wt) of four of the FeAl alloys manufactured and studied at CSM.* (G.A. = Gas Atomization; M.A. = Mechanical Alloyed).

Alloy	Al	Zr	Cr	Ce	C	B	Fe	Y_2O_3	Process
A	24,0	0,11	—	—	0,0023	0,0014	Bal	—	Ingot + Extrusion
B	25,0	—	2,0	0,1	—	0,0030	Bal	—	Ingot + Extrusion
C	23,0	0,15	—	0,4	0,0078	0,0013	Bal	—	G.A. + Extrusion
D	24,0	0,10	—	—	0,0100	0,0010	Bal	0,5	M.A. + Extrusion

All the alloys have B2 ordered crystal structure, with Al content ranging from 23 to 25%wt, (about 40%at). The composition of the FeAl alloys resulted from the optimization of the workability and the mechanical properties, [8]. Alloy D was mechanically alloyed with Y_2O_3 to evaluate the potential for oxide dispersion strengthening of FeAl alloy.

Ingots and Processing Technologies

Iron-aluminides ingots have been produced by a vacuum induction melting (V.I.M.) furnace. They showed a coarse grained structure, Fig. 1, with a pipe that reached about one third of the total length and was surrounded by spreaded porosities. The ingot break-down was not possible from these starting conditions. An improved microstructure could be obtained by controlling the cooling rate after casting or by re-melting the V.I.M. ingots by a vacuum arc remelting furnace, (VAR) (Fig. 2), [8].

Several tenths of FeAl ingots were cast in dies with diameters of about 80-120 mm and length from 300 to 400 mm. Specimens taken from these ingots were hot-compressed by an industrial hydraulic press, (400 tons), taking care of

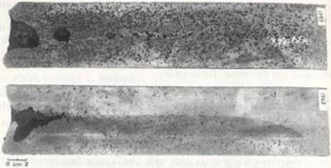

Fig. 1 - Microstructure of a vacuum arc remelted FeAl ingot.

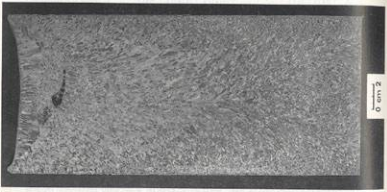

Fig. 2 - Examples of FeAl vacuum induction melted ingots with pipe like central porosities.

avoiding thermal shocks and controlling strain rates, (from 0.2 to 10 s^{-1}), and temperature accurately, (from 950°C up to 1150°C).

Reduction in thickness of about 70%, was obtained in at least two steps at two different temperatures, (the height of samples was reduced from 50 mm to 15 mm without cracks). Two typical microstructures were obtained, illustrated in Fig. 3a and 3b respectively: an equiaxed structure with large grains (150 to 250 μm), and a bimodal structure with unrecrystallized grains surrounded by small size grains, (less than 20 μm), [2].

Some ingots were canned in a mild steel containers and then hot-extruded to bars and tubes. The extrusion were performed between 950°C and 1100°C with extrusion ratio ranging between trom 8:1 to 18:1, by controlling the extrusion ram speed and lubrication conditions. In spite of the extrusion parameters the bars microstructures, Fig. 4 were full recristallized, homogeneous and fine grain sized, (20 - 25 μm).

Tensile and creep tests specimens were cut from these extruded bars for mechanical testing and other samples were taken for the corrosion/oxidation characterizations. The results are summarized in the property sections.

Rolling

The ingot break-down tests by hot-rolling performed on laboratory scale were not fully satisfactory. As-cast material samples. (thickness 35mm, length 100mm, width 40mm), were cut from VIM and VAR ingots.

These samples were canned in stainless steel containers in order to avoid thermal shocks that could cause micro cracking on the outer surtaces.

The rolling process is not simple: the rolling temperature ranges from 900°C up to 1200°C and at least every two passes it was necessary to warm up the samples: it was possible to obtain a 40:1 reduction in height in about ten passes. The scaling-up from laboratory to semi-industrial plant need more dedicated efforts: heated rollers, fine rolling speed control, cooling rate control device.

a) 200 μm b) 200 μm

Fig. 3 - Typical microstructure FeAl compressed samples taken from vacuum induction melted ingots: a) equiaxed structure, b) bimodal structure.

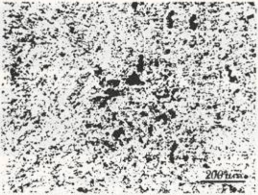

Fig. 4 - Typical microstructure of a FeAl extruded bar.

Investment casting

Some FeAl ingots were machined for subsequent investment casting. Components of simple geometry and shape were produced, (creep and tensile test specimens), after having solved some reaction problems between the molten metal and the ceramic shell. In a second step the investment casting process was also used to produce some mechanical component as: automotive valves and rockers, (Fig. 5).

Hot Forging

The materials used for hot-torging tests carried out with an industrial plant were cut from extruded bars. The laboratory tests permitted to determine brittle to ductile transition in temperature and strain rate [2], but these data must be adjusted because of a lot of other parameters play important role in the scaling-up. The process procedure and tooling were as close as possible to that common for standard materials: stainless steels and nimonic alloys.

Cracking problems due to thermal shocks and grain boundary weakness were overcome by deformation rate control and by heating up tooling. Some bolts and some nuts manufactured are showed in fig. 6. Several process were used to manufacture automotive valves, i.e.: direct extrusion, investment casting and electro-upsetting followed by forging.

The electro-upsetting is an optimum deformation process for brittle or difficult to form material like FeAl. In fact the deformation rate is well controlled, the thermal shocks are overcome so this process is almost "isothermal". A prototype of FeAl automotive valve. obtained by elettro-upsetting plus torging, is shown in Fig. 7.

POWDER ROUTE

Powders of iron-aluminides were prepared by two different techniques: Gas Atomization and Mechanical Alloving.

The gas atomized powders were used for coating experiments or consolidated by extrusion.

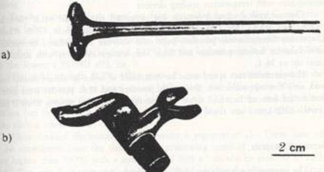

Fig. 5 - FeAl components: a) automotive inlet valves; b) a rocker.

Fig. 6 - Bolts in FeAl.

Fig. 7 - FeAl inlet valve obtained by: extrusion + electro-upsetting and forging.

The mechanical alloyed powders, starting from elemental powders, were processed in an industrial high energy dry ball mill under vacuum or controlled atmosphere with temperature cooling devices.

The mechanical alloying process has permitted also to add oxide particles, (Y_2O_3 and Al_2O_3), for the strengthening of the matrix. These ODS (Oxide Dispersion Strengthened) powders were cold and hot consolidated in medium steel cans by hot-compression and high temperature extrusion with reduction ratio up to 36:1.

The extrusion ram speed must be controlled in order to obtain sound bars with very fine microstructure. Both G.A. powders and M.A. powder were extruded in the form of bars and tubes with resulting grain size ranging from 2 μm. (FeAl + ODS) to 7 μm (FeAl without ODS).

ALLOYS PROPERTIES

The outstanding results on FeAl alloys obtained in the characterization tests carried out at CSM (or with its collaboration) will be described in this section.

MECHANICAL PROPERTIES

Machinability Tests

Various machinability tests have been performed on FeAl extruded bars obtained from both ingots and powders. The machinability strongly depends on surface finish. In machining tests it was also determined that the surface quality improved with decreasing grain size and it is related to the tendency to the intergranular fracture. The optimized FeAl alloys reported in tab I are fully machinable by conventional techniques, (Fig. 6).

Tensile Tests

Tensile specimens were machined from FeAl compressed samples, (alloys A and B, Tab I), or extruded bars, (alloy C and D Tab. I).

Alloy A and B: tensile tests were carried out at different strain rate in the range: 0.05 s^{-1} to 5 s^{-1}, at two temperature 950°C and 1100°C, [2]. Fig. 8 are shows the graphs of elongation. (A%). versus strain rate, (ε s^{-1}), the transition strain rate is the maximum rate in which fracture occurs in plastic conditions.

The transitions from brittle to ductile behaviour for boths alloy and both temperatures are evident: at 950°C for alloy A happens at strain rate between 0.5 s^{-1} and 0.8 s^{-1}, at 1100°C the transition is shifted to higher strain rate, between 4.6 s^{-1} and 5.3 s^{-1}. For allov B the transition is between 0.2 s^{-1} and 0.5 s^{-1} at 950°C and it moved at higher strain rates at 1100°C but it was not determined. The existence of a transition strain rate, depending upon temperature and alloy composition, was an input to define the secondary process conditions.

Tensile tests specimens were cut from extruded bars of alloy C and strained to failure at temperature ranging from 25°C to 1100°C, [5]. The critical temperature, (maximum temperature for brittle behaviour in tensile tests), depending on strain rate is about 400°C for the highest ε (0.03 s^{-1}) and 500°C for the lowest ε (0.0003 s^{-1}), fig. 9.

The yield strength (0,2% offset method), reached a maximum close to the critical temperatures then keeps values higher than 300 MPa up to 750°C. For temperatures higher than 750°C the yield strength, (YS), drops and high elongations (up to 130%), are reached with low stable loads, indicating grain boundary sliding phenomena.

More detailed discussion of these results is reported in [5]. These data are to be considered from the technological processing point of view: at temperature higher then 750°C with a strain rate of 0,03 s^{-1} should be possible to form FeAl. The very fine grain size obtained by M.A. together the dispersion of cera-

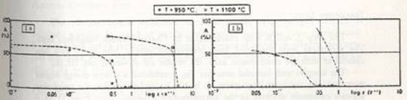

Fig. 8 - Transition curves brittle to ductile behaviour for FeAl at 950°C and 1100°C: on the left alloy A, on the right alloy B.

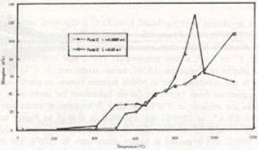

Fig. 9 - Elongation (A%) versus temperature (°C) for FeAl alloy C.

mic particles is a effective way to achieve materials with relative high ductility at room temperature (up to 13,5%), and a very high mechanical strength, (UTS up to 1250 MPa and YS up to 785 MPa), [12. 13 and 14]. FeAl room temperature tensile data with (alloy D) and without (alloy A) ceramic particles are compared in Tab. II.

Tab.II: *Comparison between FeAl alloy A, (without ceramic dispersoids) and FeAl alloy D (with ceramic dispersoids) in RT tensile tests.*

Alloy	YS (MPa)	UTS (MPa)	A (%)
A	585	620	2,8
D	785	1250	13,5

Toughness Tests

The alloy A and B were characterized by Charpy V-notch toughness tests. The specimens were machined from compressed sample, (the notch axis was coincident with the compression axis). the results are shown in tab. III [6].

The samples with bimodal structure Fig. 3 exhibited a charpy energy higher than that with equiaxed large grains. It had to be underlined that the toughness appears extremely sensitive to the microstructure (dramatic drop from 55 to 6 J/cm²) and that the microstructure itself is controlled by very narrow deformation temperatures and strain rates windows.

Tab. III - *Charpy V-notch tests on FeAl, the hammer has a maximum impact energy of 150 J mean energy on four tests.*

Alloy	Structure	Grain Size (μm)		Mean Energy
		Large	Small	J/cm²
A	Bimodal	240	< 1	55 ± 3
A	Equiaxed	74	—	39 ± 4
B	Bimodal	280	1	53 ± 5
B	Equiaxed	162	—	6 ± 1

Creep Tests

The FeAl alloys have been investigated also in terms of creep resistance. In Tab. IV some comparative results of tests carried out at 600 °C and 200MPa are shown. The grain size and oxide dispersion affect the time to rupture.

Tab. IV - *Creep tests for three FeAl alloys. Tests condition*: Temperature T = 600°C, Stress σ = 200 MPa.

Alloy	Grain Size (μm)	Rupture Time (hr)	Process
B	20	92,5	extruded ingot
C	7	27	G.A. powder
D	2	408	M.A. + ODS

Environmental Tests FeAl exhibit good resistance to the oxidation and sulphidation due to the formation of slowly growing Al_2O_3 scales [9, 12].

Aqueous Corrosion Tests

The electrochemical behaviour of FeAl in the range of pH = 0,3-13 in sulphate solution by mean of anodic polarization curves has been investigated: FeAl displays better performances than the elemental metals in alkaline and neutral solutions, being wider the pH application field, (pH > 8 for Fe, pH 4-10 for Al), [16] and [17].

Oxidation/Sulphidation Tests

In order to evaluate the behaviour of FeAl in corrosive environments, specimens were subjected to exposure in oxidizing and sulphidizing atmosphere at temperatures of 600°C up to 2500 hours and exposure in oxidizing atmosphere at 800°C up to 1500 hours. Tests specimens were weighed at 500 h intervals in order to evaluate the weight gain/loss per unit of area resulting from environmental attack. In addition, the depth of oxidation/corrosion layer was determined by optical microscopy and electron microscopy (SEM).

Reference materials for these experiments were AISI 316L and 800H stainless steels [18]. Results after 500 hours testing are presented in tab. V.

Tab. V - *Test results after 500 h or testing*, (sulphuridizing condition: $PO_2 = 10^{-24}$ bar, $PS = 3 \times 10^{-9}$ $a_s = 0{,}5$, Ptot = 70 bar, T = 600°C).

Material	Oxidation 800°C	Sulphidation 600°C	
	Weight change	Attack depth	degradation
316L Steel (18Cr-10Ni-2Mo)	0,72 mg/cm²	25 μm	100%
800H 20Cr-30Ni	0,30 mg/cm²	15 μm	60%
FeAl alloy A	0.11 mg/cm²	5 μm	20%

These results demonstrate the typical trends in the relative ranking of the different materials, being the FeAl superior to all other reference alloys tested.

The very promising behaviour of FeAl alloy in a sulphidizing atmosphere is confirmed in laboratory coal gasification environment tests.

FeAl behaviour is compared with MA956 alloy, one of the best performing materials for the coal gasification plants. In Fig. 10 are described some tests results (700°C, $H_2-2\%H_2S-7\%CO_2$).

The sulphidation resistance of FeAl is higher than that of MA956 also in the preoxidated condition. More details about this tests will be discussed in a forthcoming conference [19].

Hot Corrosion in Molten Salts

Some FeAl specimens. alloy C, were coated with Na_2SO_4-NaCl and V_2O_5 and exposed in combustion gases at 600°C. After a first phase the corrosion in Na_2SO_4-NaCl slows down significantly as shown in Fig 11, while the corrosion in $Na_2SO_4-V_2O_5$ mixture is longer lasting.

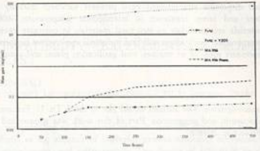

Fig. 10 - Comparative tests in sulphidizing atmosphere between FeAl and MA956 at 700°C, $H_2 - 2\% H_2S - CO_2$).

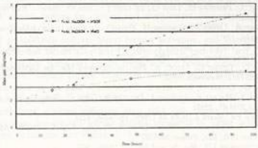

Fig. 11 - Hot corrosion at 600°C in molten salts of FeAl.

Conclusions

FeAl alloys display good mechanical strength and corrosion resistance combined with a low density, (Fe40Al = 6 g/cm³). B2 alumunides are currently being investigated for potential application as competitors of several stainless steels.

This paper shows that it is possible the production of hot workable FeAl alloys on a semi-industrial scale (100Kg per batch). The competitive advantage of these alloys results from their lower density and raw materials cost. In addition, oxide dispersion strengthened FeAl presents sufficient ductility ad room temperature and creep resistance at intermediate temperature, (600-750°C). These materials are candidate as structural materials in corrosive environments at high temperature. Such alloys will find industrial applications in thermal engines, compressors stage of jet engines, coal gasification plants and petro-chemical industry.

Acknowledgments. The authors would like to thank Dr. U. Franzoni for the helpful discussion and suggestions. Part of this work was sponsorized by EEC BRITE Project n. 2038/87.

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