



EFFECT OF ALLOYING ELEMENTS ON PROPERTIES
OF ALUMINIDE-BASE INTERMETALLIC MATERIAL
DEVELOPED VIA POWDER METALLURGY ROUTE

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**Effect of Alloying Elements on Properties
of Aluminide-Base Intermetallic Material
Developed via Powder Metallurgy Route**

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Abstract

The present study deals with the effect of additive element on structure and properties of Fe-Al intermetallic. FeAl-based intermetallic alloys with chromium or molybdenum addition were fabricated by sintering of mechanically alloyed powders. The nominal compositions of Fe-Al intermetallic alloys used in the present study were Fe-25Al, Fe-24.375Al-2.5X, Fe-23.75Al-5X, Fe-23.125Al-7.5X and Fe-22.5Al-10X (X = Cr, Mo) in wt% with the addition of 0.5% n-heptane and 0.5% zinc stearate. Bulk samples were prepared by mechanical alloying in a planetary mill for 4 hours, pressing at 360 MPa and sintering at 1000 °C for 2 hours. For compression tests, specimens were cut into 2mm x 2mm x 5 mm size and tested at room temperature using Instron machine. The phase identification and microstructure of the consolidated material was examined by x-ray diffraction and scanning electron microscope correspondingly. Density, hardness and corrosion behaviour of the Fe-Al based intermetallic alloy were studied as well. Results showed that in compressive mode, the fracture strength and ductility of FeAlCr₂ sintered alloys increase with an increase in Cr composition due to solid solution strengthening. However, the addition of Mo more than 2.5wt% was accompanied by a reduction in both properties due to the presence of Mo-rich precipitate particles in FeAl microstructure which may act as stress concentrator. Corrosion potential of Fe-Al-Cr intermetallic alloy in 3.56wt% NaCl solution was found to increase with increasing of Cr content. These alloys exhibited typical passive behavior in NaCl solutions. SEM observation of the corroded surface showed that the aluminides were subjected to pitting corrosion in 3.56wt% NaCl media. It is revealed that the presence of chromium has enhanced passivation behavior of FeAl₂Cr in NaCl solution.

1.0 Introduction

1.1 Literature Review

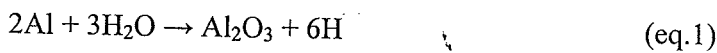
Intermetallic compounds have been the subject of scientific interest for more than 50 years because their attractive physical and mechanical properties. In recent years research mostly focused on the use of monolithic intermetallic materials based on aluminides as replacements for denser structural materials such as steels or superalloys for high temperature service. Among the intermetallics, iron aluminides are attractive for elevated temperature applications due to their low density, low material cost and good high temperature mechanical properties (Stoloff, 1998). In addition they exhibit excellent corrosion resistance in oxidizing and sulphidizing atmospheres. Of the iron aluminides, FeAl, has a B2 structure and exists over a wide range of Al concentrations at room temperatures (36-50at%). Iron aluminides based on FeAl exhibit better oxidation resistance than Fe₃Al alloys, have lower densities compared to the steels and commercial iron based alloys and exhibit higher electrical resistivity comparable to the commercial metallic heating elements. These properties allow them to be considered as high temperature structural materials, gas filters, heating elements, furnace fixtures, heat-exchanger piping, catalytic converter substrates, automotive exhaust systems and chemical production system (Stoloff, 1998; Saigal et al., 2003).

In recent years, there has been considerable interest in the synthesis and processing of intermetallic compounds by means of mechanical alloying. Mechanical alloying (MA) is a widely used process for synthesis of variety of alloys. The MA process leads to an alloy formation by solid state reactions assisted by severe plastic deformation that occurs during ball milling of elemental powders. The MA technique allows one to overcome problems such as, e.g., large difference in melting points of the alloying

components as well as unwanted segregation or evaporation that could occur during melting and casting. Usually MA products possess nanocrystalline or amorphous structure. Nanocrystalline materials are potentially attractive for many applications since the reduction of the grain size to nanometer scale can improve their physical and mechanical properties (Krasnowski and Kulik, 2006).

Iron aluminides were previously excluded from the realm of structural materials because of their brittleness at ambient temperatures and poor strength at elevated temperatures. Recent research and development activities have demonstrated that adequate engineering ductility can be achieved in the aluminides through the control of microstructure and alloy additions (Stoloff et al., 2000). Both tensile and creep strengths of the aluminides are substantially improved by alloying with refractory elements, which results in solution hardening and particle strengthening. The high-temperature strength and fabricability of FeAl is improved by alloying this material with molybdenum, carbon, chromium, and vanadium (Stoloff, 1998; Stoloff et al., 2000).

Schneibel et. al, (1996) has explained that the poor room temperature ductility of binary iron aluminides is due to their susceptibility to hydrogen embrittlement (HE). This type of embrittlement is thought to be due to the reduction of water vapor in the environment surrounding the material via reactions such as:



and



Several methods have been proposed to minimize it like oxide coatings (to minimize hydrogen pick-up from the environment), heat treatments (to produce a partially recrystallized microstructure) and alloying with passivity-inducing elements (which provide a passive layer on the surface of the intermetallic that reduces the hydrogen liberation rates on the surfaces and thereby minimizes hydrogen entry). The effects of ternary additions, such as Ti, V, Cr, Mn, Ni, Nb, Mo, Ta, Cu and Si on room temperatures ductility and strength have been extensively investigated. Even Cr additions as low as 2% have been reported to be effective in providing improved ductilities (Balasubramaniam, 1997; Huang et. al, 1999)

Though, these materials are mainly developed for high temperature applications, ambient temperature aqueous corrosion can also be a serious problem for its durability. This can happen when the alloys are possibly exposed to humid environment containing sulphates and/or chlorides, either during alloy processing stage or during idling time of the finished components. Much of the previous research on the iron aluminides focused on alloy development, processing and microstructural control and improving mechanical properties. Only a few investigations have reported to date the corrosion behavior of the iron aluminide and its alloys. Extensive studies have been published for oxidation, sulfidation and chloridation, whereas an aqueous corrosion behavior has been less explored.

Investigations of mechanically alloyed FeAl intermetallic material have mostly concentrated on synthesizing and thermodynamic studies of intermetallic powder. Commercialization of FeAl has been limited due to the fabrication difficulty and poor

mechanical properties both at ambient and elevated temperature (Charlott et.al, 1999). It is, therefore, important to develop optimized processing methods to utilize the important attributes of intermetallic in developing high performance products. The capability of converting finely divided intermetallic powders into useful, precise and high engineering components efficiently and economically has prompted widespread research in the field of powder metallurgy.

A systematic investigation has been conducted by the present authors to fabricate tribological Fe-Al intermetallic alloys by a mechanical alloying approach. The main goal of this study is to investigate the effect of chromium and molybdenum alloying element on the microstructural and properties of FeAl. Cr was chosen as the ternary alloying element because it has been reported that addition of Cr responsible for improving ductility, offering passivity, and add up to localized corrosion resistance. While, Mo was chosen because other than improving mechanical properties, it also gives beneficial effects on sulphidation resistance.

1.2 Objectives:

1. To characterize mechanical properties and corrosion resistant of Fe-Al bulk material which produced by powder compaction and sintering process.
2. To compare the effect of different alloying elements on the brittleness and corrosion behavior of FeAl

2.0 Research Methodology

Generally, research method in this project can be simplified as shown in Figure 1.

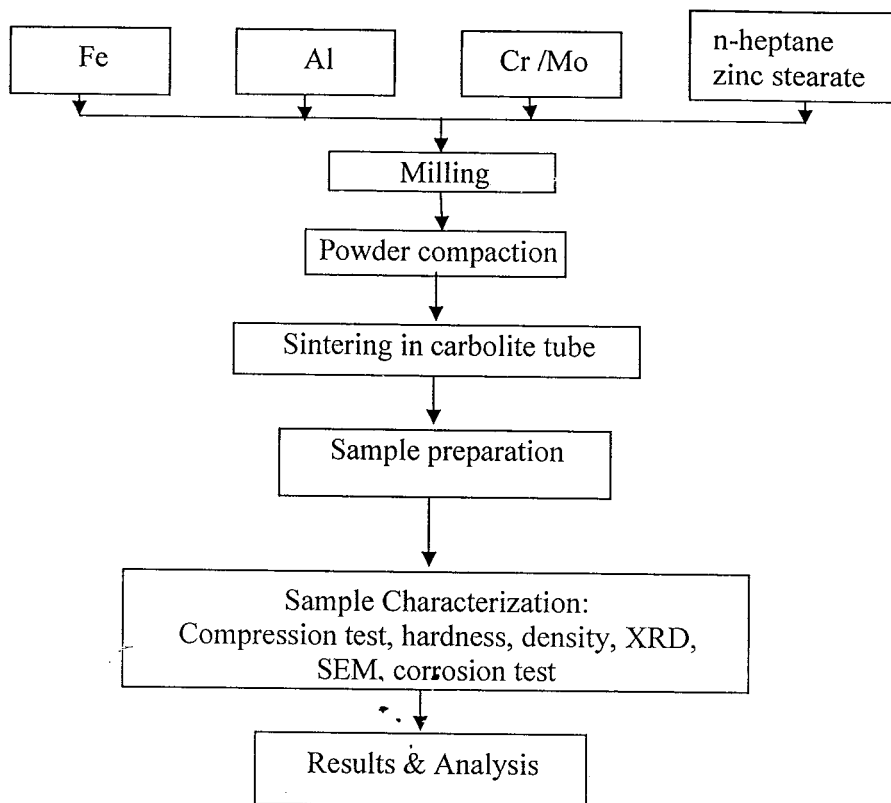


Figure 1: Flowchart of research work

The nominal compositions of Fe-Al-Cr alloys used in the present study were Fe-25Al, Fe-24.375Al-2.5Cr, Fe-23.75Al-5Cr, Fe-23.125Al-7.5Cr and Fe-22.5Al-10Cr (in wt%) with the addition of 0.5% n-heptane and 0.5% zinc stearate. While for Fe-Al-Mo alloys, the investigated compositions were Fe-25Al, Fe-24.375Al-2.5Mo, Fe-23.75Al-5Mo, Fe-23.125Al-7.5Mo, Fe-22.50Al-10.0Mo.

Mechanical alloying of the mixture powder was carried out in a planetary mill for 4 hours. Consolidation of the mechanically alloyed powders was performed through cold

pressing under a pressure of 360 MPa followed by sintering at 1000 °C for a hold time of 2 hours. Sintering profile of the sample is shown in Figure 2.

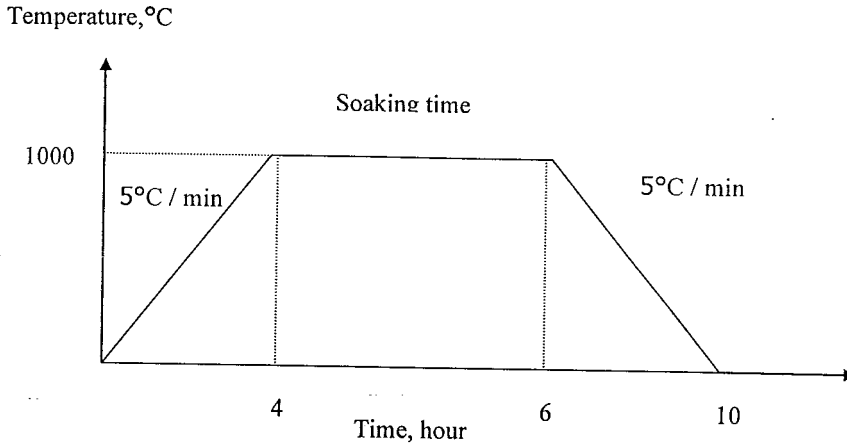


Figure 2: Temperature profile for sintering of Fe-Al based intermetallic alloys.

Mechanical properties of the sintered compacts were evaluated by compression test. For compression tests, rectangular specimens with dimensions of $2 \times 2 \times 5 \text{ mm}^3$ sizes were sectioned from the compacts by diamond cutting. All surfaces of the specimens were polished with SiC paper. These specimens were tested at room temperature using Instron Machine. The structure of the sintered compacts was characterized by X-ray diffraction (XRD). The specimens were mechanically polished to $1 \mu\text{m}$ grade diamond powder and etched with 5% Nital before observed under scanning electron microscope. Density of the sample was measured using Archimedes method and hardness of the sample was measured using Vickers microhardness tester.

Before commencement of corrosion experiment, iron aluminides sample were mounted using cold setting resin with one end soldered to a copper wire. Mounted specimens were polished in a similar manner that was done for optical microscopy. These samples

were further rinsed with distilled water and methanol and dried. The anodic polarization measurements were conducted at room temperature in 3.56wt% NaCl solution. Passivation behaviour and pitting resistance of iron aluminides were studied using potentiodynamic polarization technique with a scan rate of 0.167mV/s.

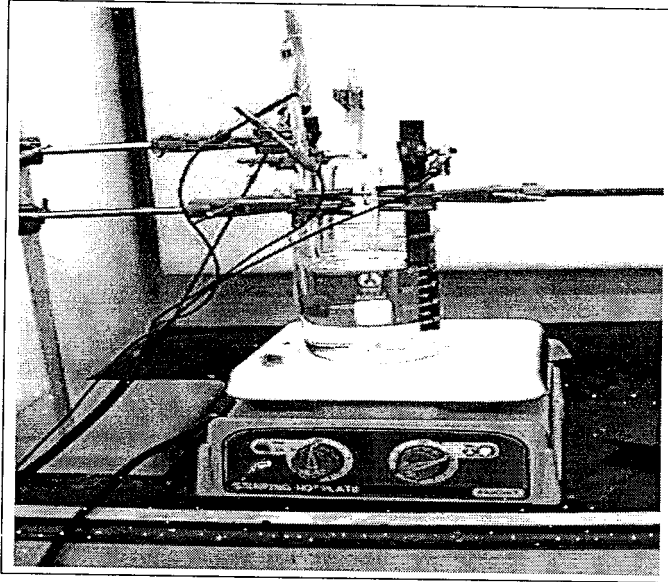


Figure 3: A set up for corrosion test using a potentiostat

The set-up consisted of a potentiostat driven by Gamry Instrument software and an electrochemical corrosion cell (Figure 3). The cell used for the polarization studies was a beaker with 500ml capacity, a graphite bar as counter electrode, a copper sulphate electrode as a reference electrode and iron aluminides sample as a working electrode. After corrosion test, specimen was rinsed with distilled water and dried. The sample was observed under a scanning electron microscopy (SEM) to examine the nature of corrosion.

3.0 Results and Discussion

This research is purposely to study the effect of chromium and molibdenum addition on intermetallic Fe-Al behaviour prepared via mechanical alloying method. This research involves milling process of ferum, aluminium and chromium/molibdenum powder mixture powder, followed by c ompaction, s intering and characterization including x-ray diffraction, microstructure, density, hardness, compression and corrosion tests. Results and discussion for each analysis are given in the following subtopics.

3.1 X-ray Diffraction Analysis

X-ray diffraction analysis (XRD) was carried out on five samples of sintered Fe-Al intermetallic which contains different percentage of chromium or molibdenum content. The main objective to do x-ray diffraction analysis is to identify the intermetallic phases exists in the sample after sintering.

Figure 4 illustrates the diffraction patterns correspond to the five samples consist of different compositions of Fe-Al-Cr. The phase was identified by comparing the peak positions and intensities with those listed in the Intenational Committee of Diffraction Data (ICDD) files. Figure 4(a) of the X-ray diffraction patterns shown by FeAl mixture sample is corresponding to the FeAl (File Number: 33-20) and FeAl₂ (File Number: 33-19) phases but the intensity of FeAl₂ peak is very low relative to FeAl.

Figure 4(b) to (e) illustrates the pattern from the Fe-Al mixtures with different amount of Cr composition. For Cr containing FeAl mixtures, only single phase of Fe₂AlCr (File Number: 42-1486) is detected. Another feature in the XRD pattern is that the peaks of (220) approximately at $2\theta=44^\circ$ are shifted to lower angles with increasing Cr addition

which indicate that Cr present in solid solution in the alloyed intermetallics of Fe-Al-Cr. The atomic radius, r , of Fe, Al and Cr are 0.1241 nm, 0.1431 nm and 0.1249 nm respectively. Therefore the decrease in lattice parameter of Fe_2AlCr is due to the replacement of larger Al atom by smaller Cr atom.

Figure 5 shows the diffraction pattern correspond to the five different Fe-Al-Mo intermetallic alloys. Figure 5 (a) of the X-ray diffraction pattern shown by FeAl mixture sample is corresponding to the FeAl (File Number: 33-0020). From FeAl binary diagram the expected major phase to exist at composition of 25% Al with sintering temperature of 1000°C is FeAl phase. From the ICDD file the lattice structure of FeAl intermetallic compound is BCC structure.

Figure 5(b) to (e) shows the patterns from the FeAl with different amount of Mo addition. For Mo containing Fe-Al mixtures, single phase of FeAl (File Number: 33-0020) is also detected. XRD studies indicate that the Mo present in solid solution in the alloyed intermetallics of Fe-Al-Mo. Ternary diagram of Fe-Al-Mo shows that for the addition of Mo in range of 2.5-10% with sintering temperature of 1000°C , the expected phase to exist is FeAl.

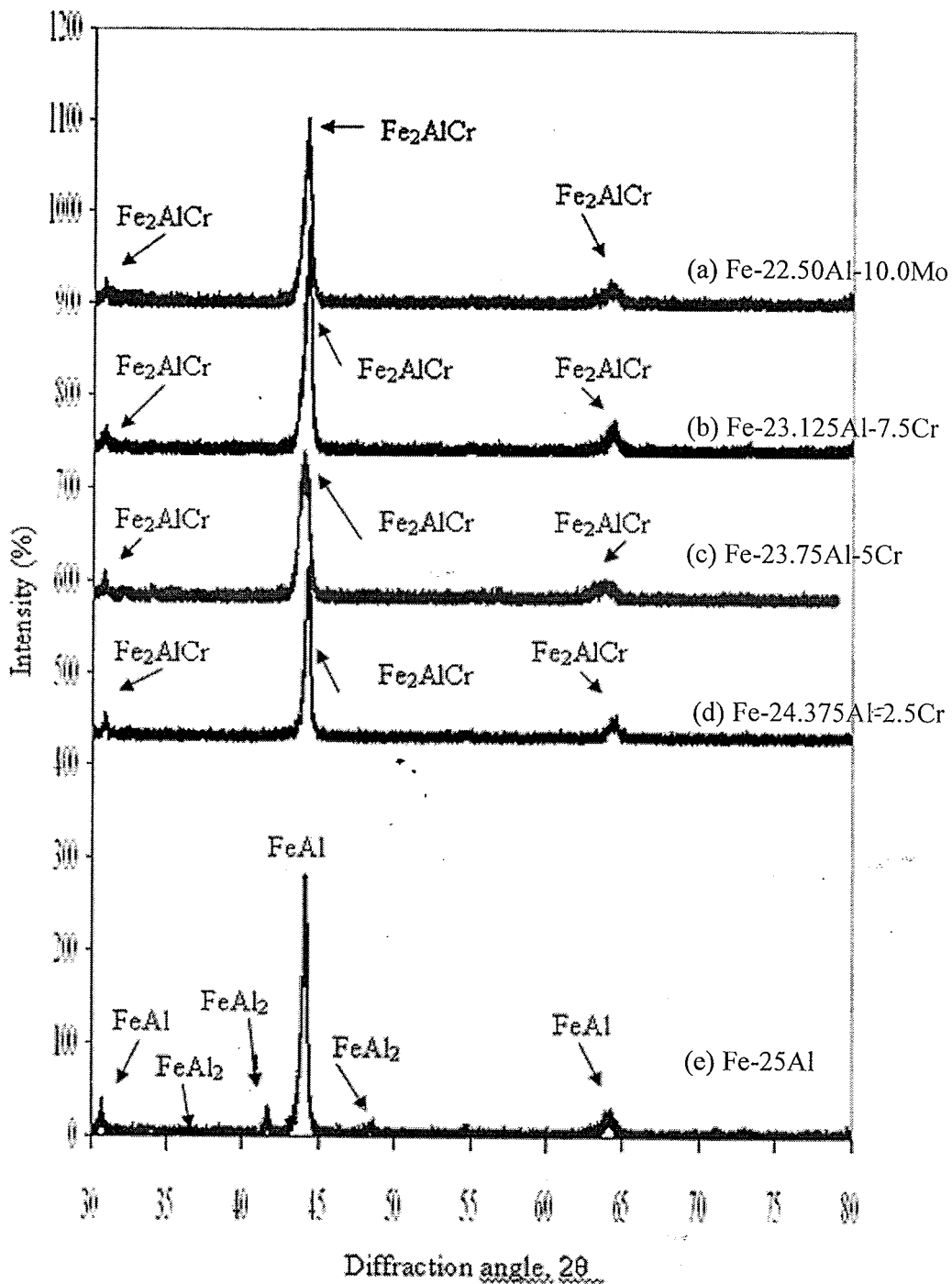


Fig. 4: XRD pattern of the Fe-Al-Cr intermetallic alloy with different compositions: (a) Fe-22.50Al-10.0Cr, (b) Fe-23.125Al-7.5Cr, (c) Fe-23.75Al-5Cr, (d) Fe-24.375Al-2.5Cr and (e) Fe-25Al

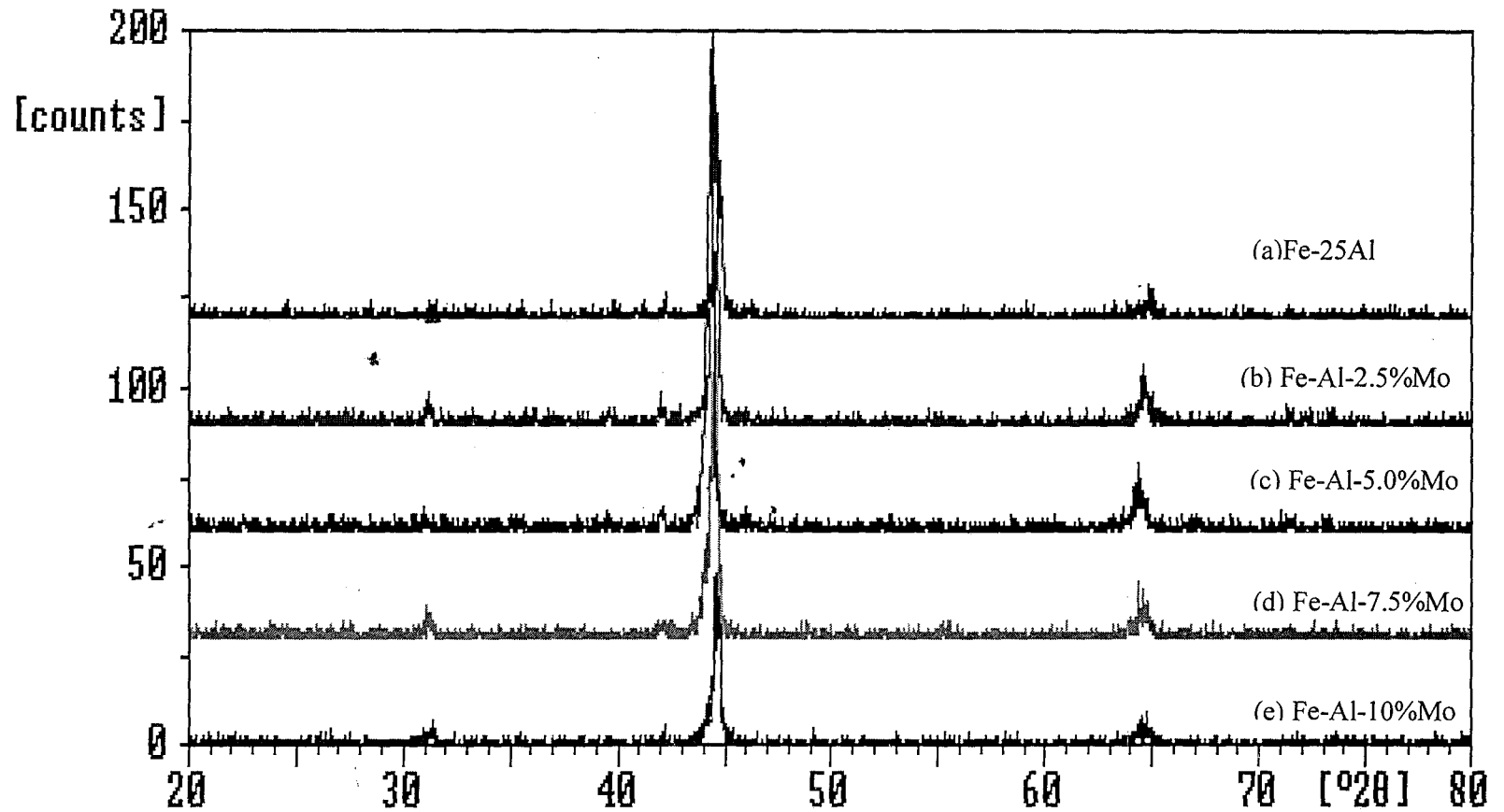
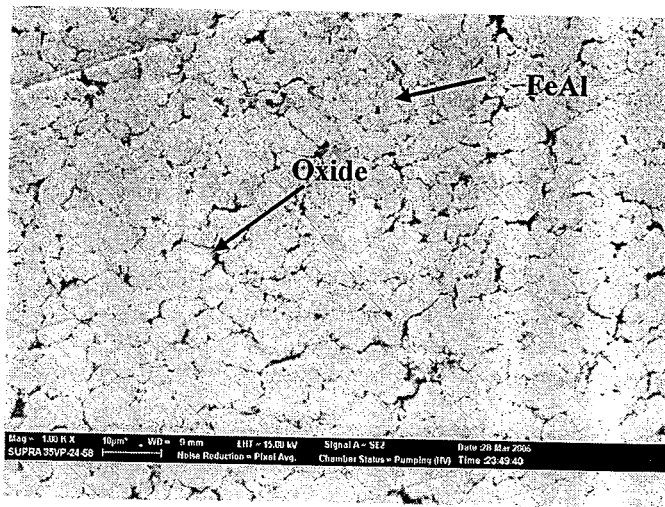


Fig. 5: XRD pattern of the Fe-Al-Mo intermetallic alloy with different compositions: (a) Fe-25Al, (b) Fe-24.375Al-2.5Mo, (c) Fe-23.75Al-5Mo, (d) Fe-23.125Al-7.5Mo and (e) Fe-22.50Al-10.0Mo.

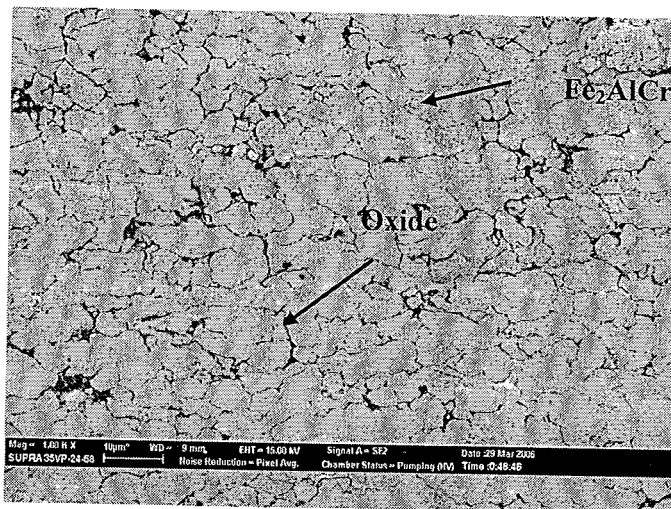
3.2 Microstructure Observation

In this research, scanning electron microscope (SEM) was used to observe the microstructure of sintered Fe-Al intermetallic compound with different content of chromium/molibdenum. Fig.6 shows the observed microstructures for sintered Fe-Al-Cr alloys with different Cr contents.

Fig.7 shows the observed microstructures for sintered Fe-Al-Mo alloys with different Mo contents. White precipitates can be observed mainly on the grain boundaries for sample containing Mo. The amount of this precipitates obviously increases with increasing Mo content. EDX analysis was performed to determine elemental composition in the white precipitate (spot X) and the dark matrix (spot Y). Analysis on spot X shows high content of Mo, followed by Fe and Al. While for spot Y, the major element presence is Fe, followed by Al and Mo. C and O are also traced as contamination. This results show that the white precipitate is Mo rich particle which is not fully dissolved in the FeAl intermetallic phase.



(a)

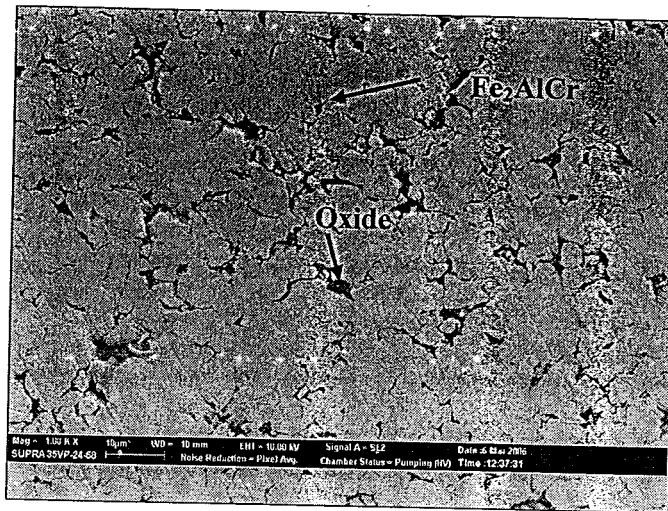


(b)

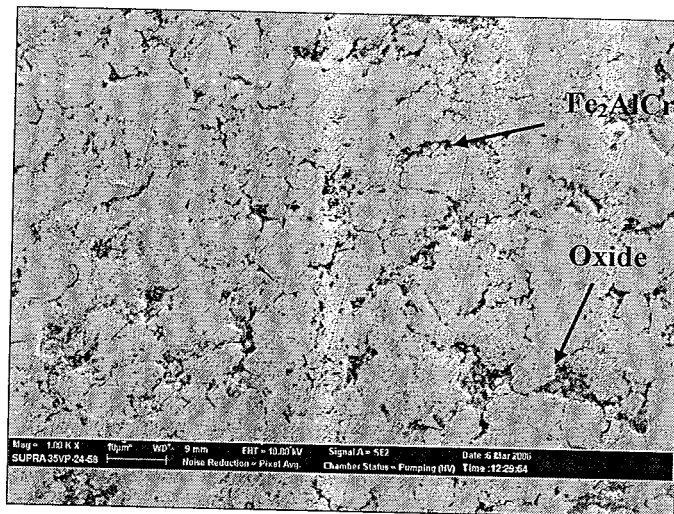
Figure 6: SEM of sintered Fe-Al-Cr intermetallic alloy with different compositions:

(a) Fe-25Al (b) Fe-24.375Al-2.5Cr (c) Fe-23.75Al-5Cr (d) Fe-23.125Al-7.5Cr

(d) FeAl-7.5Cr (e) FeAl-10Cr

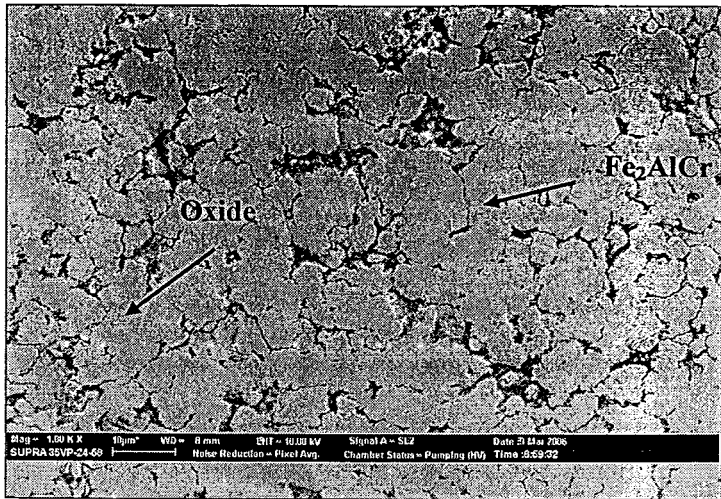


(c)



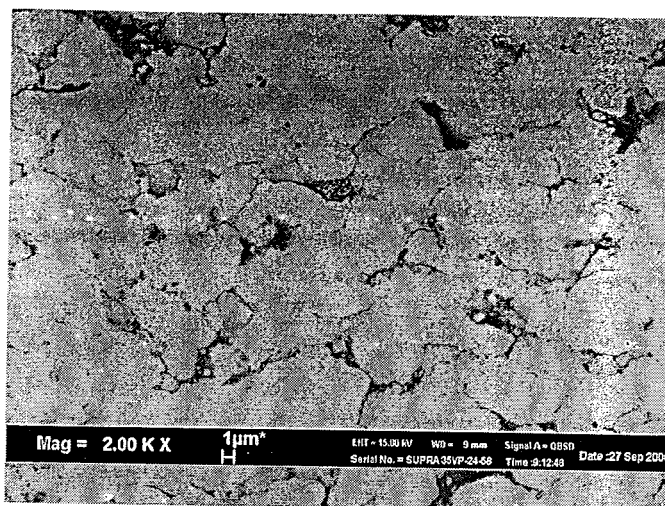
(d)

Figure 6 (continue)

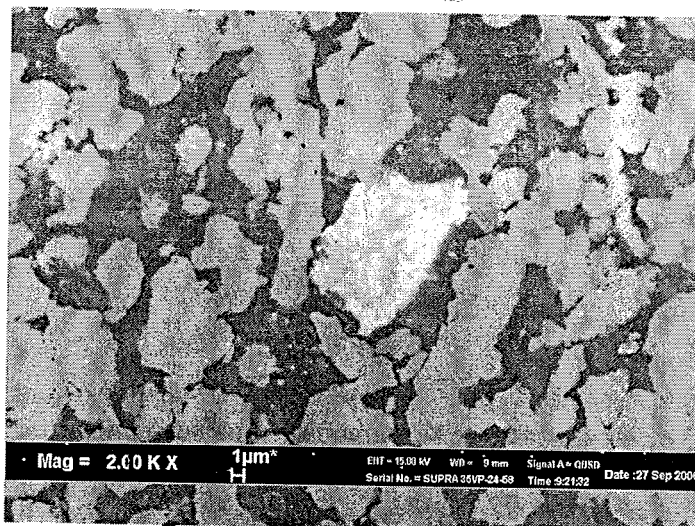


(e)

Figure 6 (continue)

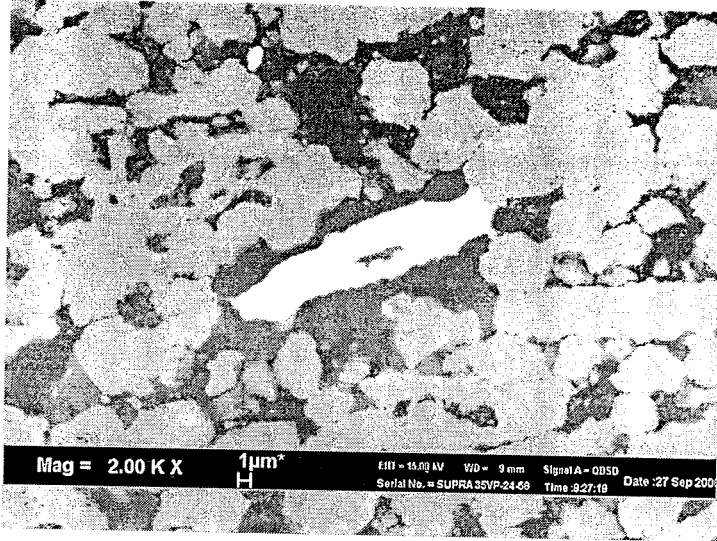


(a)

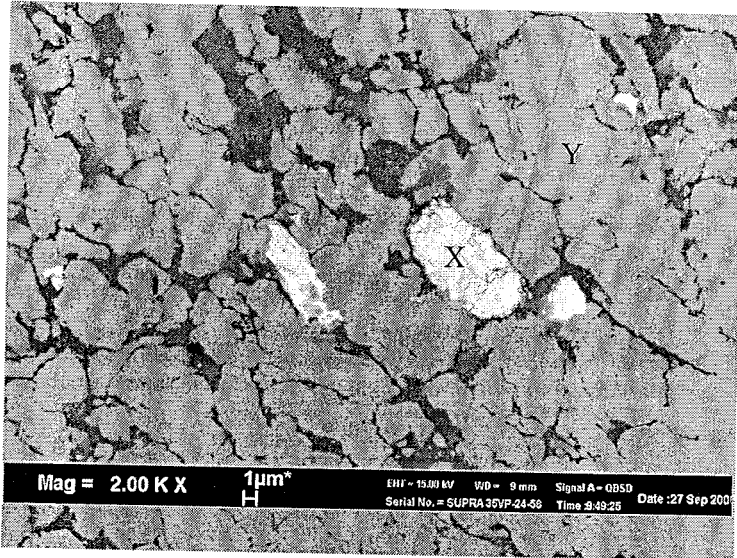


(b)

Figure 7 : SEM of sintered Fe-Al-Mo intermetallic alloy with different compositions:
(a) Fe-25Al, (b) Fe-24.375Al-2.5Mo, (c) Fe-23.75Al-5Mo, (d) Fe-23.125Al-7.5Mo and
(e) Fe-22.50Al-10.0Mo.

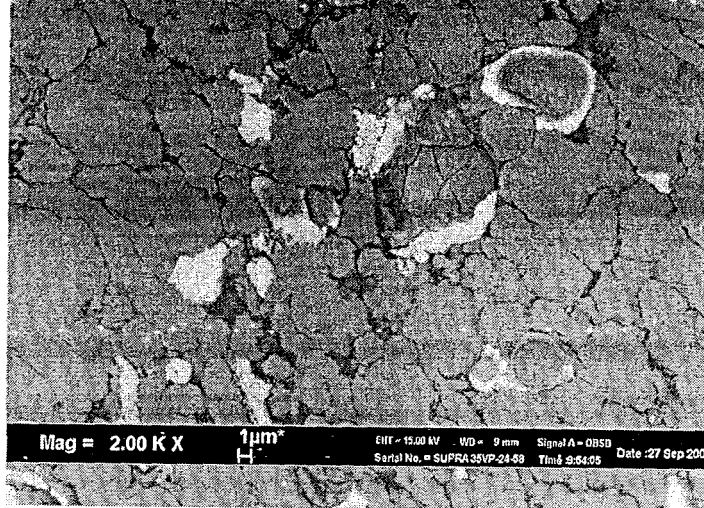


(c)



(d)

Figure 7 (continue)



(e)

Figure 7 (continue)

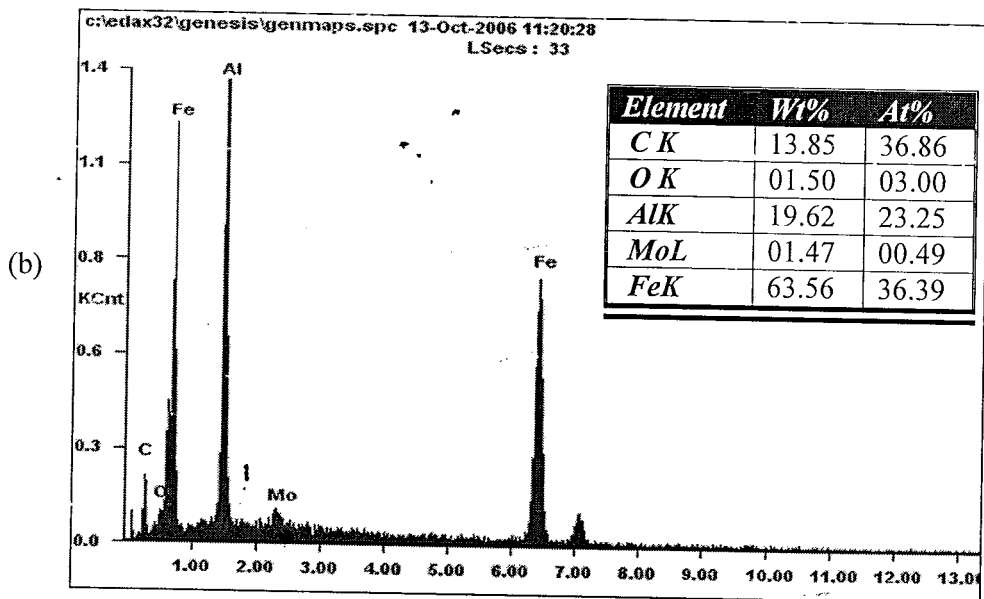
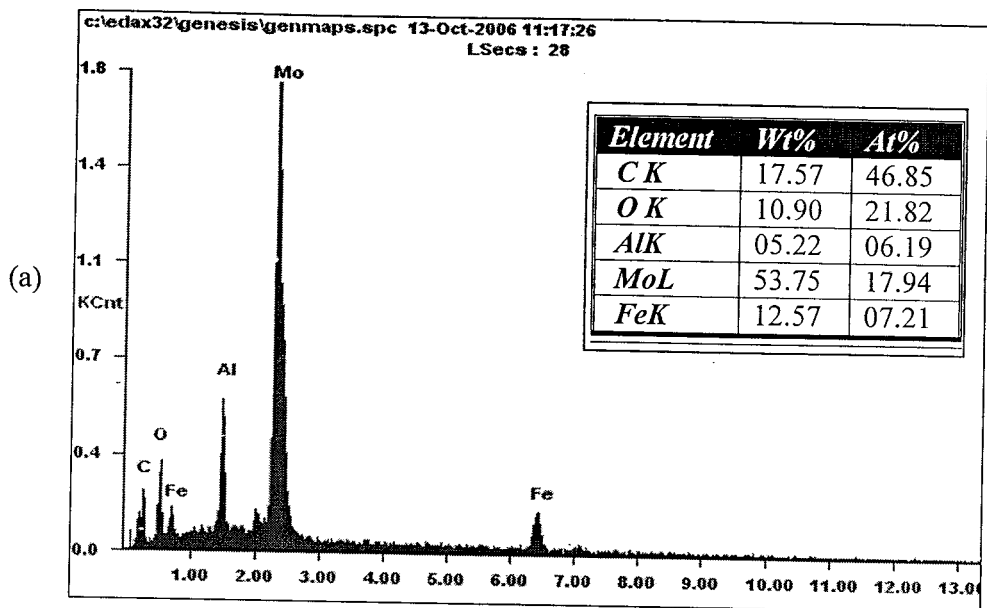


Figure 8 EDX on a) spot X and b) spot Y in Figure 7c.

3.3 Density Analysis

Archimedes method was carried out to measure density of intermetallic alloy bulk sample. In this test, four samples were used for each composition to ensure data consistency.

Figure 9 shows the green and sintered densities of Fe-Al-Cr intermetallic with different amount of Cr. It is observed that the addition of Cr has increased the density by eliminating pores in Fe-Al intermetallic compound.

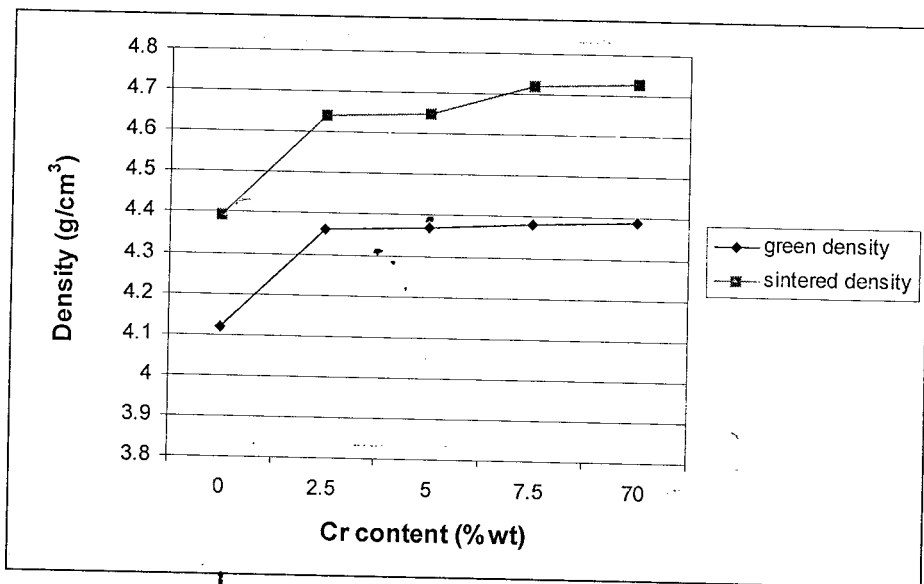


Figure 9: Densities of green and sintered body Fe-Al-Cr with different amount of Cr

Figure 10 is the average sintered density of Fe-Al-Mo intermetallic compound with different Mo composition. As shown in Figure 10, the sintered density increases when the percentage of Mo has been increased. Sample with 10% molybdenum shows the highest density while sample with 2.5% molybdenum shows the lowest density.

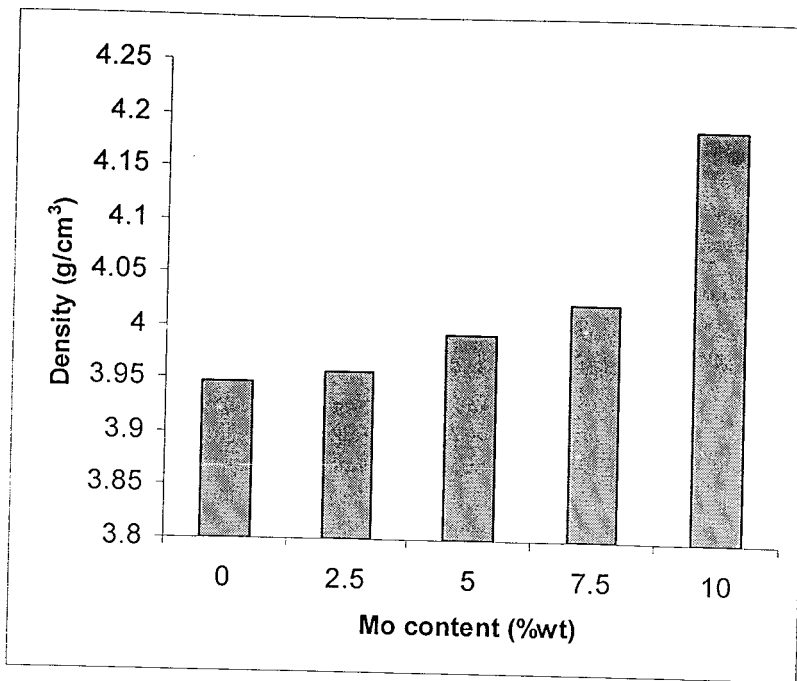


Figure 10: Sintered density of Fe-Al-Cr with different amount of Mo

3.4 Mechanical Properties

In order to investigate mechanical properties of the Fe-Al-Cr and Fe-Al-Mo intermetallic alloys, compression test were carried out. Each test was repeated for three different specimens from the same alloy. The compression test results for each of the alloys were obtained from the average of the three tests. The important results of the compressive stress-strain curves of Fe-Al-Cr alloys are shown in Figure 11 and Figure 12.

Figure 11 shows the dependence of the compressive fracture stress (fracture strength) of FeAl alloys on the presence of Cr alloying elements. The increased fracture strength of FeAl with 2.5-10% of Cr is most likely attributed to solid solution strengthening of Cr as confirmed by XRD analysis. Solid solution strengthening occurs when the

dislocation mobility in a solid is restricted by the introduction of solute atoms (Husni, 2005). The presences of Cr solute atoms in FeAl alloys introduce discrete barriers which restrict the motion of the dislocation glide that would lead to strengthening.

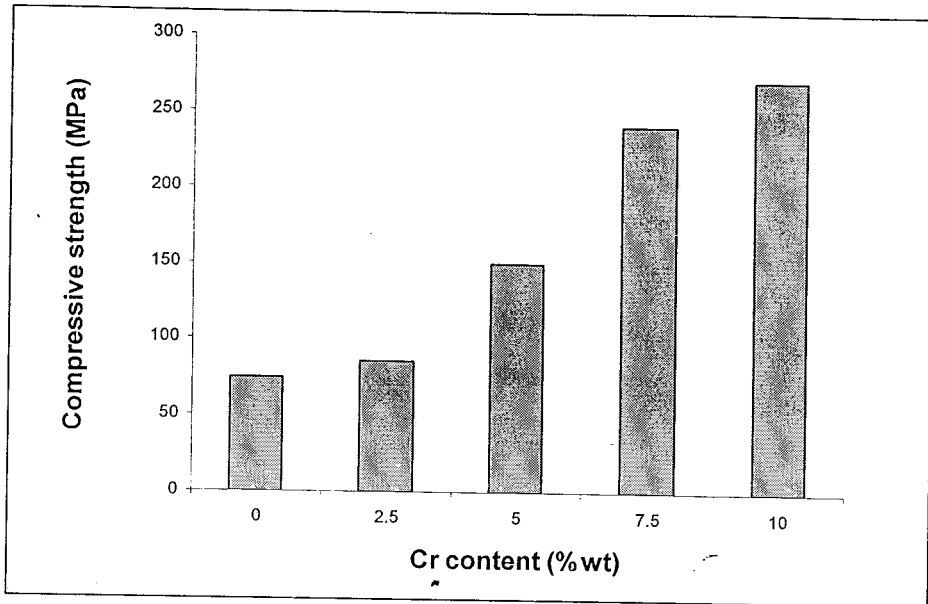


Fig. 10: Compressive strength of the Fe-Al-Cr intermetallic

Alloying with Cr also produces a higher strain compared to the base intermetallic which indicates the improvement in ductility (Figure 12). The ductility results of the present study are in agreement to earlier report where it was shown that Cr additions provide a passive layer on the surface of the intermetallic (Balasubramaniam, 2002). He has explained the effect of Cr addition on embrittlement of FeAl by mixed potential analysis. He concluded that the rate of hydrogen liberation on the passivated surface of the Cr-alloyed iron aluminide is much lower than that surface of the binary aluminide. Addition of higher content of Cr is beneficial to increase ductility by inducing thicker passive film which delay hydrogen entry into FeAl alloys. Therefore, ductility is improved because hydrogen embrittlement of iron aluminides has been minimized.

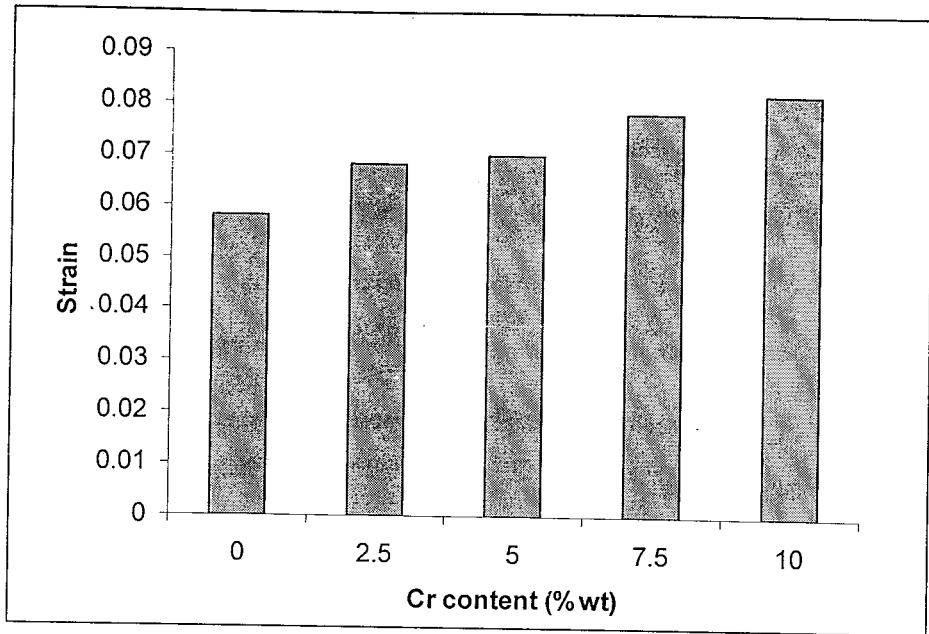


Fig. 12: Strain of the Fe-Al-Cr intermetallic

The important results of the compressive stress-strain curves of Fe-Al-Mo alloys are shown in Figure 13 and Figure 14. As reported by (Sun et. al, 1995), Mo are found to be effective in strengthening aluminides through solid solution effect. On the other hand, room temperature ductility decreases with addition of this element. However, in the present work it was found that the addition of 2.5% Mo has given the result that compressive strength and compressive strain are the highest value.

The decrease in compressive strength in the present work probably is due to the presence of Mo-rich precipitates in Fe-Al-Mo intermetallic. As discusses previously by (Banerjee and Balasubramaniam, 1998), these precipitates can be assumed as the 'flaw' which act as stress concentrators, thereby becoming the sources of premature failure.

When stress is applied, crack tends to first initiate around the precipitates, then grows, and propagate leading to fracture. It can be said that after the onset of the plastic deformation, due to the presence of more stress concentrators, the applied stress for Fe-Al-5.0%Mo, Fe-Al-7.5%Mo and Fe-Al-10.0%Mo is lower than Fe-Al-2.5%Mo.

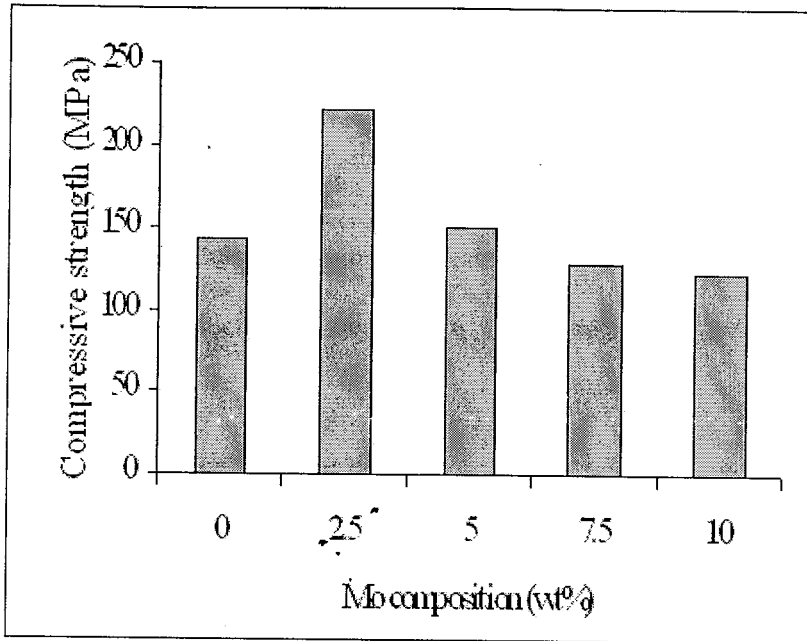


Fig. 13: Compressive strength of the Fe-Al-Mo intermetallic

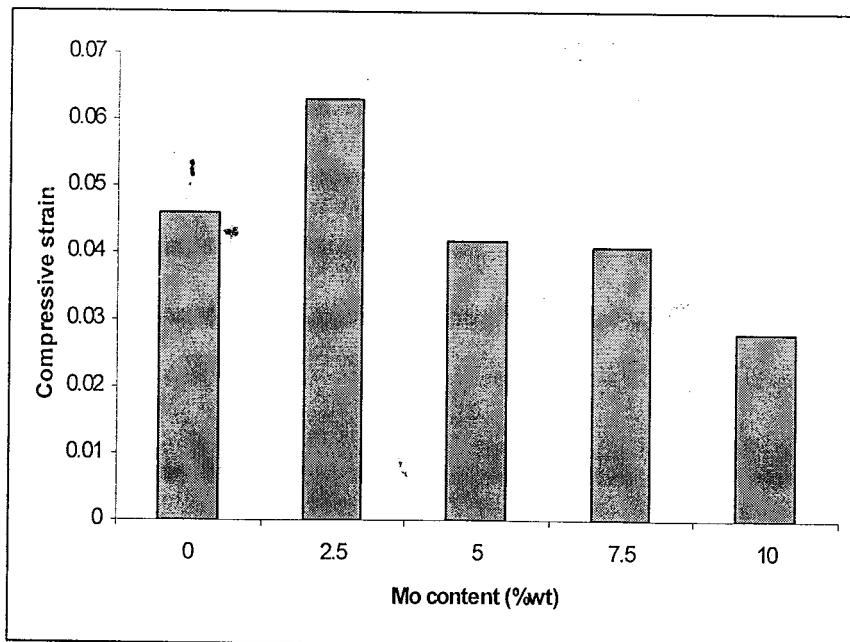


Fig. 14: Strain of the Fe-Al-Mo intermetallic

3.5 Corrosion Test

Potentiodynamic polarization curves of all the four Fe-Al-Cr alloys and Fe-Al binary are presented in Figure 15. These Fe-Al-Cr alloys exhibit a broad passive-pitting behavior. The potentiodynamic polarization curve shows the passive-pitting behavior of Fe-Al-Cr in 3.56wt% NaCl media. The electrochemical kinetic parameters which determine the passive behavior of these alloys namely are corrosion potential, E_{corr} , passive current density (i_{passive}) which is equal to corrosion current density, and pitting potential E_{pitt} are derived from the curves. Polarization curves in Figure 15 shows that the addition of Cr tends to slightly increase the passive pitting breakdown potential of the alloys and thus slightly increases pitting resistance of the alloy.

After the corrosion test, the samples were observed under SEM in order to study the nature of corrosion behavior of the aluminides in NaCl media. Fig. 2 shows that all samples are preferentially attacked by pitting corrosion after they are polarized higher than their pitting potentials. In addition, it may naturally occur on passive metal or alloys due to passive film breakdown at potential $\geq E_{\text{pitt}}$ or due to mechanical action which can damage the passive film at potential $< E_{\text{pitt}}$. Pitting corrosion is initiated at locations at which the passive film is weaker than other areas due to less Cr content. Results also confirm that aluminides without Cr addition are more susceptible to pitting corrosion compared to other aluminides with the Cr addition.

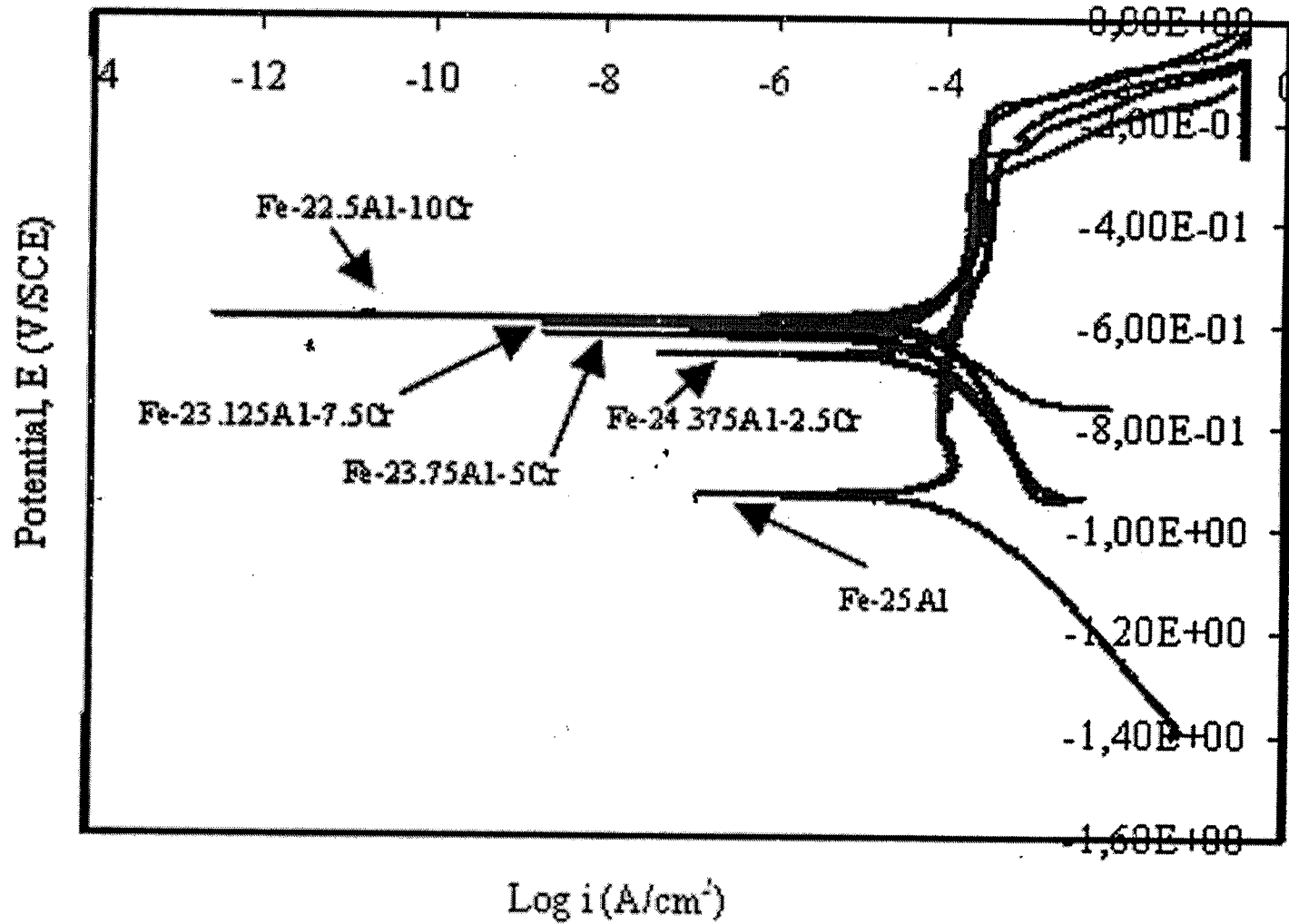


Figure 15: Potentiodynamic polarization curves of Fe-Al-Cr in 3.56wt% NaCl media

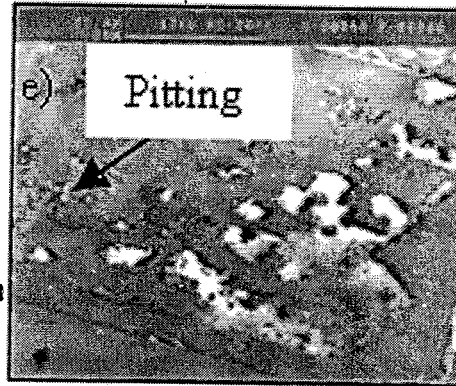
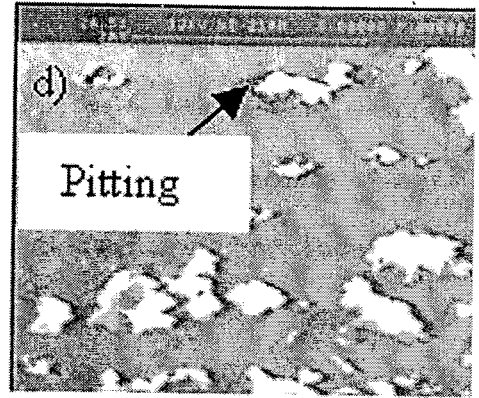
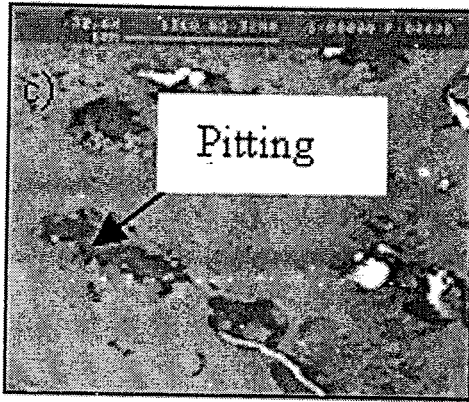
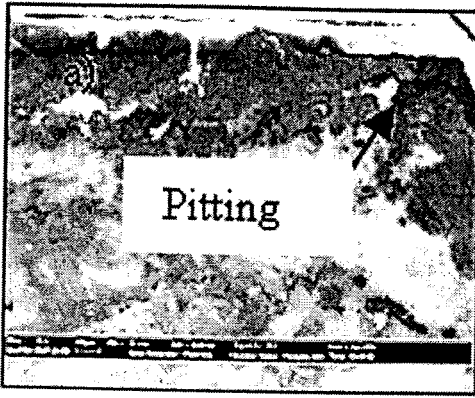


Figure 16: SEM image of aluminides after polarization showing pitting attack

(a) Fe-25Al (b) Fe-24.375Al-2.5Cr (c) Fe-23.75Al-5Cr (d) Fe-23.125Al-7.5Cr (e) Fe-22.5Al-10Cr

4.0 Conclusion

1. XRD results shows that with composition of Fe-25wt%Al and sintering temperature of 1000°C, high intensity peak of FeAl phase observed. Low intensity peak of FeAl₂ is detected as well indicating that this phase exist with minor iron aluminide intermetallic compound which is FeAl. This results suggest that intermetallic compound produced contains FeAl as major phase and FeAl₂ as minor phase.
2. For Fe-Al-Cr mixtures, a single phase of Fe₂AlCr is detected. When more Cr content is added, a decrease in the lattice parameter value of Fe₂AlCr occurs indicating that Cr atom substitutes Al atom in Fe₂AlCr crystal structure.
3. For Fe-Al-Mo mixtures, a single phase of FeAl is observed in XRD pattern which suggest that Mo exist as substitutional atom in FeAl solid solution.
4. Room temperature ductility of iron aluminide can be enhanced by the addition of passivity-inducing element of Cr to the base iron aluminide. Cr exists in solid solution in the iron aluminide which provide additional fracture strength to the base metal.
5. Results show that 2.5wt%Mo addition significantly increased the ultimate stress and ultimate strain in compressive mode due to solid solution hardening. However, addition of Mo more than 2.5wt% is associated with a reduction in both properties caused by the presence of Mo-rich precipitate particles.
6. Potentiodynamic polarization of iron aluminides in NaCl media shows the Fe-Al-Cr are susceptible to pitting corrosion. The pitting corrosion resistance and the passivation potential range of Fe-Al-Cr alloys increases with increasing of Cr content.

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APPENDIXES

LIST OF PUBLICATION

Zuraidah Mohd Yasin, **Zuhailawati Hussain** And Sunara Purwadaria
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Mechanical properties studies on Cr alloyed FeAl.
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STUDIES ON CORROSION BEHAVIOR OF FE-AL INTERMETALLIC IN NATRIUM CHLORIDE SOLUTION

Zuraidah Mohd Yasin, Zuhailawati Hussain and Sunara Purwadaria

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Introduction

Iron aluminides based on Fe_3Al and $FeAl$ possess unique properties and have attracted considerable attention as potential candidates for warm structural application and as promising substitutes for stainless steels and cast irons at room temperature due to their excellent oxidation and corrosion resistance [1]. The aluminides also offer low material cost, and low density (as compared with Fe- and Ni-based alloys). In recent years, there has been considerable interest in the synthesis and processing of intermetallic compounds by means of mechanical alloying. Mechanical alloying is a high energy ball milling process that involves repeated welding and fracturing of powder particles. This technique can produce fine powders with a nanoscale grain size and a homogeneous distribution of dispersoid, which is expected to improve the ambient temperature ductility and high temperature strength of intermetallic compounds. Although these materials are mainly developed for high temperature applications, ambient temperature aqueous corrosion can also be a serious problem for its durability. This can happen when the alloys are possibly exposed to humid environment containing sulphates and/or chlorides, either during alloy processing stage or during idling time of the finished components [2]. Where the corrosion resistance of the metal has to be enhanced, passivity-inducing elements should be added.

A systematic investigation has been conducted by the present authors to fabricate tribological $FeAl$ -Cr intermetallic alloys by a mechanical alloying approach. Cr was chosen as the ternary alloying element because it has

been reported that addition of Cr responsible for offering passivity, and add up to localized corrosion resistance. The present work is undertaken to investigate the electrochemical corrosion behavior of the iron aluminides in a natrium chloride solution to understand the effect of Cr addition on the electrochemical corrosion behaviour of iron aluminides.

Experimental

Materials

The nominal compositions of alloys used in the present study are Fe-25Al, Fe-24.375Al-2.5Cr, Fe-23.75Al-5Cr, Fe-23.125Al-7.5Cr and Fe-22.5Al-10Cr (in wt%) with the addition of 0.5% n-heptane and 0.5% zinc stearate. Bulk samples were prepared by mechanical alloying in a planetary mill for 4 hours, pressing at 360 MPa and sintered at 1000°C for 2 hours before cut into 10mm x 10mm x 5mm dimension. The sample was mounted with one end soldered to a copper wire.

Apparatus and Procedures

Passivation behaviour of iron aluminides was studied using potentiodynamic polarization technique with a scan rate of 0.167mV/s in 3.56wt% NaCl solution at room temperature. The set-up consisted of a potentiostat driven by Gamry Instrument software and an electrochemical corrosion cell. The cell used for the polarization studies was a beaker with 500ml capacity, graphite bar as counter electrode, copper sulphate electrode as a reference electrode and iron aluminides sample as a working electrode. After corrosion test, the sample was observed under a scanning electron microscope.

Results and discussion

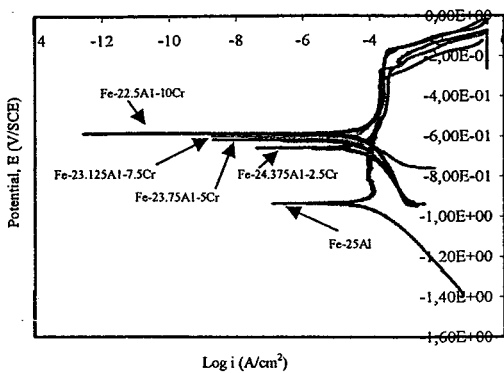


Figure 1: Potentiodynamic polarization curves of Fe-Al-Cr in 3.56wt% NaCl media

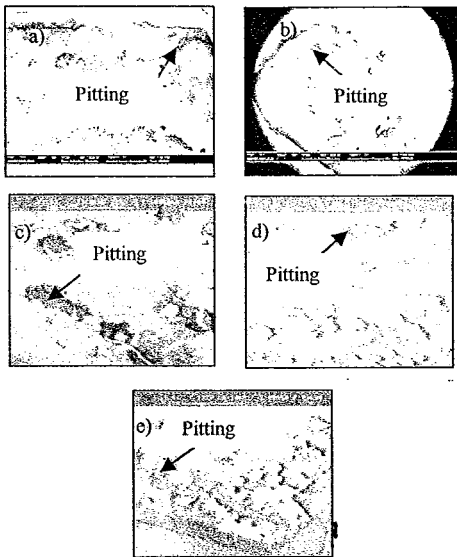


Figure 2: SEM image of aluminides after polarization showing pitting attack (a) Fe-25Al (b) Fe-24.375Al-2.5Cr (c) Fe-23.75Al-5Cr (d) Fe-23.125Al-7.5Cr (e) Fe-22.5Al-10Cr

Potentiodynamic polarization curves of all the four Fe-Al-Cr alloys and Fe-Al binary are presented in Figure 1. These Fe-Al-Cr alloys exhibit a broad passive-pitting behavior. The addition of Cr tends to slightly increase the passive pitting breakdown potential of the alloys and thus slightly increase pitting

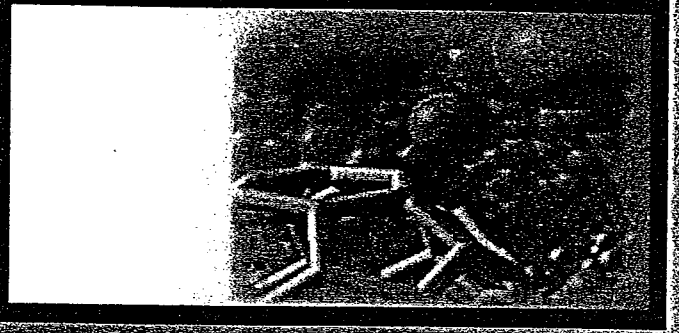
resistance of the alloy. Fig. 2 shows that all samples are preferentially attacked by pitting corrosion after they are polarized higher than their pitting potentials. In addition, it may naturally occur on passive metal or alloys due to passive film breakdown at potential $\geq E_{pitt}$ or due to mechanical action which can damage the passive film at potential $< E_{pitt}$. Pitting corrosion is initiated at locations at which the passive film is weaker than other areas due to less Cr content. Results also confirm that aluminides without Cr addition are more susceptible to pitting corrosion compared to other aluminides with the Cr addition.

Conclusion

Potentiodynamic polarization of iron aluminides in NaCl media shows the Fe-Al-Cr are susceptible to pitting corrosion. The pitting corrosion resistance and the passivation potential range of Fe-Al-Cr alloys increases with increasing of Cr content.

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Mechanical Properties of Iron Aluminides Intermetallic Alloy with Molybdenum Addition

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Abstract

Iron aluminides are attractive for elevated temperature applications due to their low density, low material cost and good high temperature mechanical properties. In addition, they exhibit excellent corrosion resistance in oxidizing and sulphidizing atmospheres. However, the main drawback concerning their possible technological applications is its ductility in the cast form at room temperatures.

In this work, FeAl-based alloys, with and without molybdenum addition were fabricated by sintering of mechanically alloyed powders in order to investigate the effect of molybdenum on iron aluminide mechanical properties. The nominal compositions of alloys used in the present study were Fe₂₅Al, Fe_{24.38}Al_{2.5}Mo, Fe_{23.75}Al_{5.0}Mo and Fe_{23.13}Al_{7.5}Mo (in wt%) with the addition of 0.5% n-heptane and 0.5% zinc stearate. Bulk samples were prepared by mechanical alloying in a planetary mill for 4 hours, pressing at 360 MPa and sintering at 1000 °C for 2 hours. For compression tests, specimens were cut into 2mm x 2mm x 5 mm size and tested at room temperature using Instron machine. The phase identification and microstructure of the consolidated material was examined by x-ray diffraction and scanning electron microscope correspondingly.

Table 1 shows the important results of the compressive stress-strain curves of FeAl sintered materials. It can be seen that Mo addition up to 2.5wt% significantly increased the ultimate stress and ultimate strain in compressive mode. The improvement in these properties is due to solid solution hardening [1] as confirmed by XRD analysis. However, the addition of Mo more than 2.5wt% was accompanied by a reduction in both properties. The decrease in these properties is probably caused by the presence of Morich precipitate particles as observed in the microstructure of the FeAl which may act as stress concentrator [2].

Table 1. Compression properties of alloy investigated.

Alloy Composition (wt%)	Ultimate Stress (MPa)	Ultimate Strain (mm/mm)
Fe-25Al	142	0.046
Fe-24.8Al-2.5Mo	221	0.063
Fe-23.75Al-5Mo	151	0.042
Fe-23.13Al-7.5Mo	128	0.041
Fe-22.5Al-10Mo	123	0.028

Keywords: Iron-aluminide Intermetallic/Alloying Element/ Mechanical Properties.

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Mechanical Properties Of Iron Aluminides Intermetallic Alloy With Molybdenum Addition

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Abstract:

In this work, FeAl-based alloys with and without molybdenum addition were fabricated by sintering of mechanically alloyed powders in order to investigate the effect of molybdenum on iron aluminide mechanical properties. Bulk samples were prepared by mechanical alloying for 4 hours, pressing at 360 MPa and sintering at 1000 °C for 2 hours. The specimens were tested in compression at room temperature using Instron machine. The phase identification and microstructure of the consolidated material was examined by x-ray diffraction and scanning electron microscope correspondingly. Results show that 2.5wt%Mo addition significantly increased the ultimate stress and ultimate strain in compressive mode due to solid solution hardening. However, the addition of Mo more than 2.5wt% was accompanied by a reduction in both properties caused by the presence of Mo-rich precipitate particles.

1. Introduction

Intermetallic compounds have been the subject of scientific interest for more than 50 years because their attractive physical and mechanical properties. In recent years research mostly focused on the use of monolithic intermetallic materials based on aluminides as replacements for denser structural materials such as steels or superalloys for high temperature service. Among the intermetallics, iron aluminides are attractive for elevated temperature applications due to their low density, low material cost and good high temperature mechanical properties (1). In addition they exhibit excellent corrosion resistance in oxidizing and sulphidizing atmospheres. Of the iron aluminides, FeAl, has a B2 structure

and exists over a wide range of Al concentrations at room temperatures (36-50at%). Iron aluminides based on FeAl exhibit better oxidation resistance than Fe₃Al alloys, have lower densities compared to the steels and commercial iron based alloys and exhibit higher electrical resistivity comparable to the commercial metallic heating elements. These properties allow them to be considered as high temperature structural materials, gas filters, heating elements, furnace fixtures, heat-exchanger piping, catalytic converter substrates, automotive exhaust systems and chemical production system (1,2).

Iron aluminides were previously excluded from the realm of structural materials because of their brittleness at

ambient temperatures and poor strength at elevated temperatures. Recent research and development activities have demonstrated that adequate engineering ductility (6) can be achieved in the aluminides through the control of microstructure and alloy additions. Both tensile and creep strengths of the aluminides are substantially improved by alloying with refractory elements, which results in solution hardening and particle strengthening. The high-temperature strength and fabricability of FeAl is improved by alloying this material with molybdenum, carbon, chromium, and vanadium (1,6).

Thus, in the present study, FeAl intermetallic powder will be synthesized using mechanical alloying and consolidated via powder metallurgy route, particularly cold pressing and sintering. The main goal of this study is to investigate the effect of alloying element of molybdenum, on the microstructural and properties of FeAl.

2. Methodology

In this work, FeAl-based alloys with and without molybdenum addition were fabricated by sintering of mechanically alloyed powders in order to investigate the effect of molybdenum on iron aluminide mechanical properties. The nominal compositions of alloys used in the present study were Fe-25Al, Fe-24.38Al-2.5Mo, Fe-23.75Al-5.0Mo, Fe-23.13Al-7.5Mo and Fe-22.50Al-10Mo (in wt%) with the addition of 0.5% n-heptane and 0.5% zinc stearate.

Bulk samples were prepared by mechanical alloying in a planetary mill for 4 hours, pressing at 360 MPa and sintering at 1000 °C for 2 hours. For compression tests, specimens were cut into 2mm x 2mm x 5 mm size and tested at room temperature using Instron machine. The specimens was mechanically polished to 1 μ m grade diamond powder and etched with 5% Nital

before observed under scanning electron microscope. The phase composition of the consolidated material was examined by x-ray diffraction analysis.

3. Results & Discussion

Figure 1 shows the diffraction pattern correspond to the five different alloys. The phase was identified by comparing the peak positions and intensities with those listed in the International Committee of Diffraction Data (ICDD) files. Figures 1 (a) of the X-ray diffraction pattern shown by FeAl mixture sample is corresponding to the FeAl (File Number: 33-0020). From FeAl binary diagram the expected major phase to exist at composition of 25% Al with sintering temperature of 1000°C is FeAl phase. From the ICDD file the lattice structure of FeAl intermetallic compound is BCC structure.

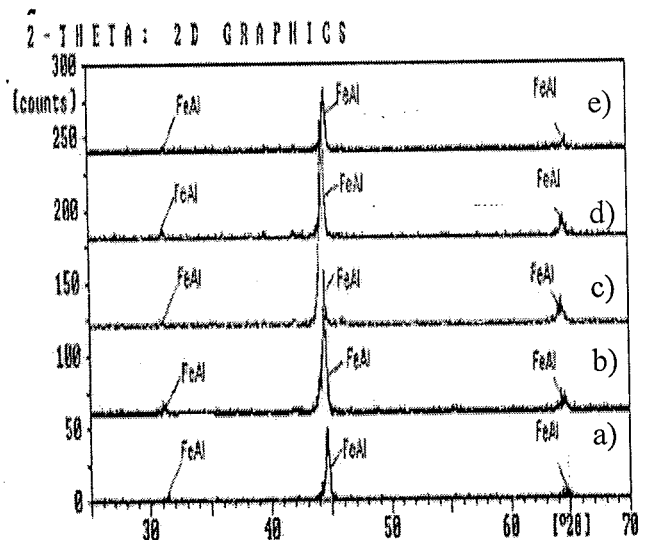


Figure 1: The XRD pattern of the alloy formed with different composition of Mo (a) 0%Mo (b) 2.5%Mo (c) 5.0%Mo (d) 7.5%Mo (e) 10.0%Mo

Figure 1(b) to (e) shows the patterns from the FeAl with different amount of Mo addition. For Mo containing Fe-Al mixtures,

single phase of FeAl (File Number: 33-0020) is also detected. XRD studies indicate that the Mo present in solid solution in the alloyed intermetallics of Fe-Al-Mo. The ternary diagram of Fe-Al-Mo show the addition of Mo in range of 2.5-10% with sintering temperature of 1000°C, the expected phase to exist is FeAl.

Fig.2 shows the obtained microstructures for sintered Fe-Al-Mo alloys with different Mo contents. White precipitates can be observed mainly on the grain boundaries for sample containing Mo. The amount of this precipitates obviously increases with increasing Mo content. The compositions of the precipitates determined by EDX is shown in Fig. 3, which indicates the presence of a large concentration of Mo.

As reported by [7], Mo are found to be effective in strengthening aluminides through solid solution effect. On the other hand, room temperature ductility decreases with addition of this element. However, in the present work it was found that the addition of 2.5% Mo has given the result that tensile strength and tensile strain are the highest value (Fig. 4 and Fig. 5).

The decrease in compressive strength in the present work probably is due to the presence of Mo-rich precipitates in Fe-Al-Mo intermetallic. As discussed previously by [8], these precipitates can be assumed as the 'flaw' which act as stress concentrators, thereby becoming the sources of premature failure. When stress is applied, crack tends to first initiate around the precipitates, then grows, and propagate leading to fracture.

It can be said that after the onset of the plastic deformation, due to the presence of more stress concentrators, the applied stress for Fe-Al-5.0%Mo, Fe-Al-7.5%Mo and Fe-Al-10.0%Mo is lower than Fe-Al-2.5%Mo.

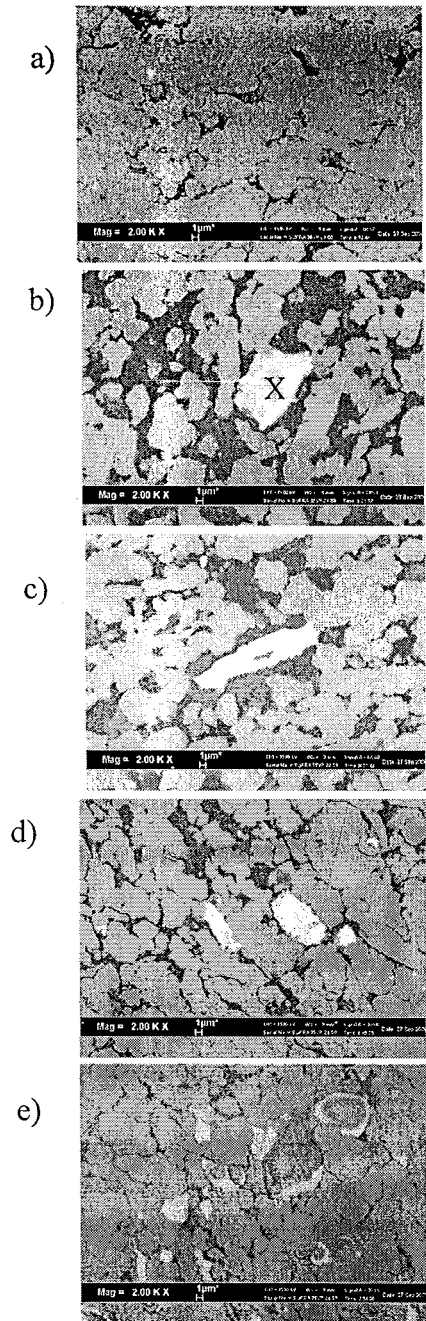


Figure 2: SEM image for sample Fe-Al with different composition of Mo (a) 0%Mo (b) 2.5%Mo (c) 5.0%Mo (d) 7.5%Mo (e) 10.0%Mo 2000X

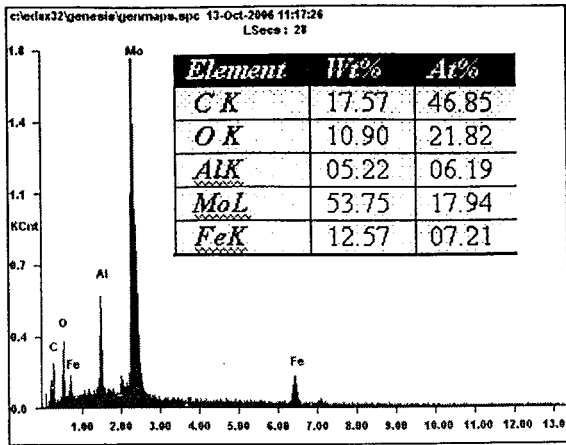


Figure 3: EDX for Fe-Al-Mo sample at region X

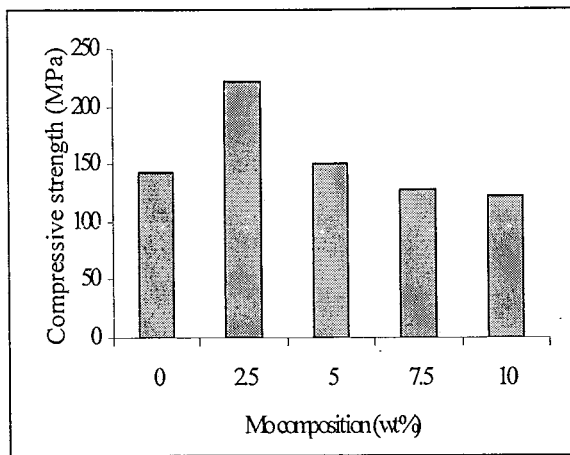


Fig. 4: Compressive strength of the Fe-Al-Mo intermetallic

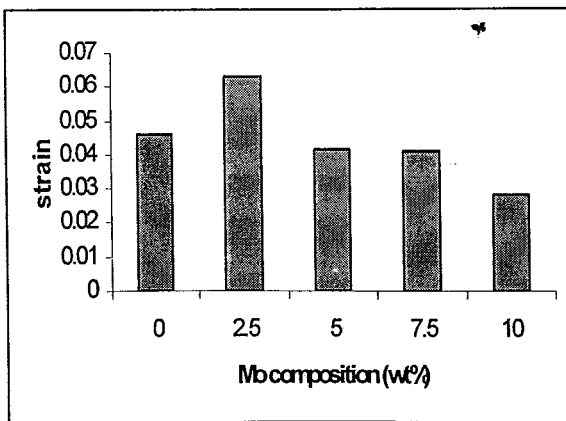


Fig. 5: Strain of the Fe-Al-Mo intermetallic

4. Summary

Bulk samples of FeAl-based intermetallic alloys with w molybdenum addition were prepared by mechanical alloying, pressing and sintering. Results show that 2.5wt%Mo addition significantly increased the ultimate stress and ultimate strain in compressive mode due to solid solution hardening. However, the addition of Mo more than 2.5wt% was accompanied by a reduction in both properties caused by the presence of Mo-rich precipitate particles.

5. ACKNOWLEDGEMENT

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MECHANICAL PROPERTIES STUDIES ON Cr ALLOYED FeAl

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Abstract: Phase identification and mechanical properties of FeAl intermetallic alloys with addition of Cr were obtained. The alloys were produced with powder metallurgy technique through mechanical alloying method. Compression test was carried out and some insights on the alloys mechanical properties were withdrawn. The main effect of Cr element on the FeAl structure was related with the improvement of the compressibility ductility and fracture strength. The results showed that in compressive mode, the fracture strength and ductility of FeAl sintered alloys were increased with an increase in Cr composition due to solid solution strengthening and formation of passive layer..

Key Words: Iron-aluminide Intermetallic/Alloying Element/ Mechanical Properties

1. INTRODUCTION

Intermetallic compounds have been the subject of scientific interest for more than 50 years because their attractive physical and mechanical properties. In recent years research mostly focused on the use of monolithic intermetallic materials based on aluminides as replacements for denser structural materials such as steels or superalloys for high temperature service. The main goal of these developments are weight reduction, properties optimization or the tailoring of properties for specific applications for instance high-temperature superconducting aluminum alloys with Nb additions, low-density alloys with Mg additions and good corrosion resistance with Fe additions.

Among the intermetallics, iron aluminides base on FeAl and Fe₃Al are attractive for elevated temperature applications due to their low density, low material cost and good high temperature mechanical properties [1]. In addition they exhibit excellent corrosion resistance in oxidizing and sulphidizing atmospheres. Of the iron

aluminides, FeAl, has a B2 structure and exists over a wide range of Al concentrations at room temperatures (36-50at%). Iron aluminides based on FeAl exhibit better oxidation resistance than Fe₃Al alloys, have lower densities compared to the steels and commercial iron based alloys and exhibit higher electrical resistivity comparable to the commercial metallic heating elements. These properties allow them to be considered as high temperature structural materials, gas filters, heating elements, furnace fixtures, heat-exchanger piping, catalytic converter substrates, automotive exhaust systems and chemical production system [2].

However, in the FeAl compound, likewise in the other intermetallic aluminides, the main drawback concerning their possible technological applications is its ductility in the cast form at room temperatures and processing problems due to hydrogen embrittlement (HE). Several methods have been proposed to minimize HE like oxide coatings (to minimize hydrogen pick-up from the environment), heat treatments (to produce a partially recrystallized microstructure) and alloying with passivity-inducing elements [3].

Considerable studies have been done to improve iron aluminide mechanical properties throughout control of microstructure and alloy composition. Nine passivity-inducing elements were alloyed to Fe₃Al to produce intermetallic composition Fe-24Al-5M where M=Ti, Zr, V, Nb, Ta, Cr, Mo, W and Si [4]. Only the Cr- and Ti-alloyed intermetallic exhibited ductility. The other intermetallics were brittle due to the precipitation of additional brittle phase

The objective of the present work was to investigate effect of Cr alloying element on the mechanical properties and structure of Fe-Al intermetallic.

2. EXPERIMENTAL PROCEDURE

Commercial metal powders of Fe, Al and Cr were employed to produce FeAl-based alloys in the present study. The nominal compositions of alloys used in the present study were Fe-25Al, Fe-24.38Al-2.5Cr, Fe-23.75Al-5.0Cr and Fe-23.13Al-7.5Cr (in wt %) with the addition of 0.5% n-heptane and 0.5% zinc stearate. Mechanical alloying of the mixture powder was carried out in a high energy planetary-type ball milling system at a rotational speed of 300rev/min for 4 hours. Consolidation of the mechanically alloyed powders was performed through cold pressing under a pressure of 360 MPa followed by sintering at 1000 °C for a hold time of 2 hours.

Mechanical properties of the sintered compacts were evaluated by compression test. For compression tests, rectangular specimens with dimensions of 2 x 2 x 5 mm³ sizes were sectioned from the compacts by diamond cutting. All surfaces of the specimens were polished with SiC paper. These specimens were tested at room temperature using Instron Machine. The structure of the sintered compacts was characterized by X-ray diffraction (XRD).

3. RESULTS AND DISCUSSION

One of the common approaches used recently, to enhance the ductility of the FeAl intermetallic alloy has been the alloying with elements. In this investigation, the effect of the addition of Cr as well as the effect of Cr content to the FeAl intermetallic was explored.

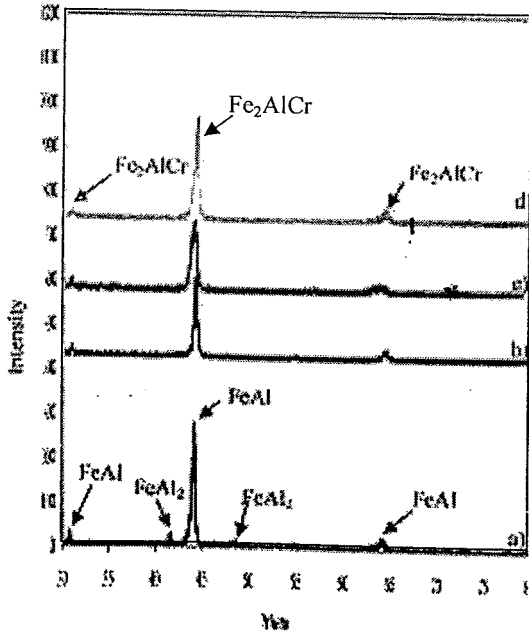


Fig. 1: The XRD pattern of the alloy (a) Fe-25Al (b) Fe-24.375Al-2.5Cr (c) Fe-23.75Al-5Cr (d) Fe-23.125Al-7.5Cr

Figure 1 illustrates the diffraction patterns correspond to the five different prepared alloys. Figure 1(a) of the X-ray diffraction patterns shown by FeAl mixture sample is corresponding to the FeAl (File Number: 33-20) and FeAl₂ (File Number: 33-19) phases but the intensity of FeAl₂ peak is very low relative to FeAl. Figure 1(b) to (e) illustrates the pattern from the Fe-Al mixtures with different amount of Cr composition. For Cr containing FeAl mixtures, only single phase of Fe₂AlCr (File Number: 42-1486) is detected. Another feature in the XRD pattern is that the peaks of (220) approximately at 2θ=44° are shifted to lower angles with increasing Cr addition which indicate that Cr present in solid solution in the alloyed intermetallics of Fe-Al-Cr. The atomic radius, *r*, of Fe, Al and Cr are 0.1241 nm, 0.1431 nm and 0.1249 nm respectively. Therefore the decrease in lattice parameter of Fe₂AlCr is due to the replacement of larger Al atom by smaller Cr atom.

Compression tests were carried out from each of the obtained alloys. Each test was repeated for three different specimens from the same alloy. The compression results from each of the alloys were obtained from the average of the three tests. The important results of the compressive stress-strain curves of FeAl alloys were shown in Figure 2 and Figure 3. Figure 2 shows the dependence of the fracture stress (fracture strength) of FeAl alloys on the presence of Cr alloying elements. The increased fracture strength of FeAl with 2.5-10% of Cr is most likely attributed to solid solution strengthening of Cr as confirmed by XRD analysis. Solid solution strengthening occurs when the dislocation mobility in a solid is restricted by the introduction of solute atoms [5]. The presences of Cr solute atoms in FeAl alloys introduce discrete barriers which restrict the motion of the dislocation glide that would lead to strengthening.

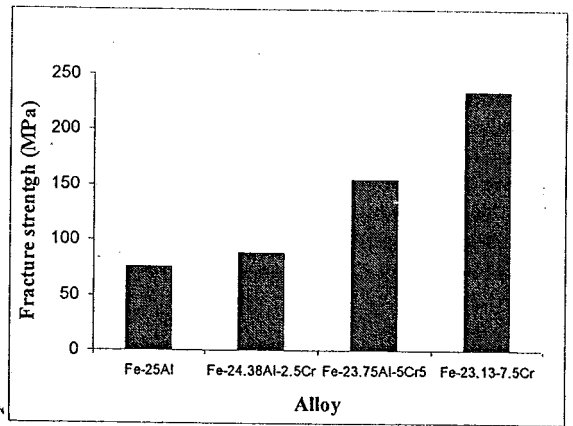


Fig. 2: Fracture strength of the Fe-Al-Cr intermetallic

Alloying with Cr also produced a higher strain compared to the base intermetallic which indicate the improvement in ductility. The ductility results of the present study are in agreement to earlier report where it was shown that Cr additions provide a passive layer

MECHANICAL PROPERTIES STUDIES ON Cr ALLOYED FeAl

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Abstract:

Phase identification and mechanical properties of FeAl intermetallic alloys with addition of Cr were obtained. The alloys were produced with powder metallurgy technique through mechanical alloying method. Compression test was carried out and some insights on the alloys mechanical properties were withdrawn. The main effect of Cr element on the FeAl structure was related with the improvement of the compressibility ductility and fracture strength. The results showed that in compressive mode, the fracture strength and ductility of FeAl sintered alloys were increased with an increase in Cr composition due to solid solution strengthening and formation of passive layer..

Key Words:

Iron-aluminide Intermetallic/Alloying Element/ Mechanical Properties

Effects of Superheated Steam and Hot Air on the Mechanical Properties of Rubberwood

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Abstract:

A pilot-scale rubberwood dryer was constructed and injected with superheated steam and hot air to study the effect of the hybrid system on the drying rate and mechanical properties of the wood. A total of 300 pieces of rubberwood boards each with dimensions of 1 m long x 3 in. wide x 1 in. thick were stacked in 1 m x 1 m 1.7 m (60 ft³) pallet. The stack was impinged with alternating cycles of superheated steam and hot air. The time required for conventional drying was 129 hours and the time schedule for the hybrid drying system was only 64 hours resulting in a 66.7% reduction. After being dried, the rubberwood boards were mechanically tested for static bending, compression strength, hardness and shear strengths. From the mechanical tests, hybrid drying system using superheated steam and hot air had no significant effect on the mean shear strength parallel to grain; however; the mean compression strength parallel to grain was reduced by 24.23% and the mean MOR by 21.35%. Nonetheless, the mean MOE was increased by 30.41% and the mean of hardness by 16.4%.

Key Words:

Rubberwood drying, Drying, Superheated Steam, Stack Temperature

on the surface of the intermetallic [3]. [3] has explained the effect of Cr addition on embrittlement of FeAl by mixed potential analysis. He concluded that the rate of hydrogen liberation on the passivated surface of the Cr-alloyed iron aluminide is much lower than that surface of the binary aluminide. Addition of higher content of Cr is beneficial to increase ductilities by inducing thicker passive film which delay hydrogen entry into FeAl alloys. Therefore, ductility is improved because hydrogen embrittlement of iron aluminides has been minimized.

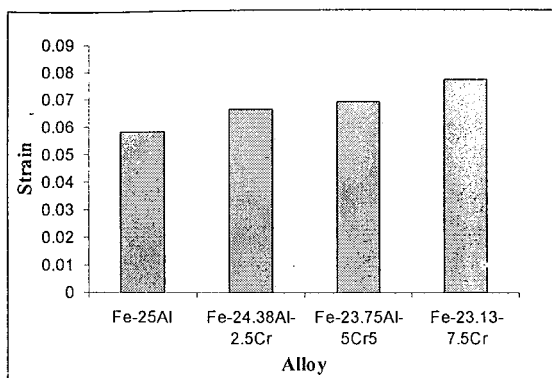


Fig. 3: Strain of the Fe-Al-Cr intermetallic

4. CONCLUSION

The room temperature ductility of iron aluminide can be enhanced by the addition of passivity-inducing element of Cr to the base iron aluminide. Cr exists in solid solution in the iron aluminide which provide additional fracture strength to the base metal.

5. ACKNOWLEDGEMENT

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