DEVELOPMENT OF POWDER METALLURGY ROUTE FOR PRODUCTION OF NOVEL FE-AL INTERMETALLICS FOR HIGH TEMPERATURE APPLICATIONS

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To my mom; i want to thank you for all you have given to me and all you have done for me. Your love and enthusiasm for my pursuits gave me energy and encouragement when i needed it most.

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ABSTRACT

FeAl based intermetallic alloys are being proposed as engineering materials for high temperature applications due to their low density, low materials cost, low content of strategic elements and oxidation resistance. These intermetallic alloys are suitable for applications in aggressive and corrosive environments up to 900°C. However they may fail through loss of strength or gradually deteriorate through reaction with the surrounding atmosphere when exposed to temperature higher than 900°C. Therefore, the formation of a stable protective oxide scale or alumina on the surface is required to protect the underlying materials when exposed to high temperature. In this research, the FeAl alloys were produced by using powder metallurgy route which consisted of mechanical alloying process, cold compaction, sintering, hot compaction and surface treatment via ion implantation. The addition of reactive elements or their oxides such as Y, Y_2O_3 and CeO_2 by mechanical alloying or ion implantation method may improve their oxidation resistance through the enhancement of the alumina scale adhesion to the underlying alloys. Characterizations by using SEM and XRD were carried out before and after each process to investigate the microstructure, phase change and formation of the oxide layer. Cyclic oxidation tests were performed at 900°C and 1100°C to study the oxidation behavior of these intermetallic alloys. The results showed that the FeAl intermetallic alloys were successfully produced by mechanical alloying, hot compaction and surface treatment via ion implantation. The FeAl intermetallic with 3×10^{15} ion/cm^2 doses of yttrium implanted exhibited the lowest oxidation kinetics at 900°C while FeAl intermetallic with 1 wt% yttria and 9×10^{15} ion/cm^2 doses of yttrium implanted exhibited the lowest oxidation kinetics at 1100°C.

ABSTRAK

Aloi intermetalik berasaskan FeAl telah dicadangkan sebagai bahan kejuruteraan untuk aplikasi suhu tinggi kerana mempunyai ketumpatan dan kos bahan yang rendah, kandungan elemen strategik yang rendah dan ketahanan pengoksidaan yang baik. Walaubagaimana pun bahan ini berkemungkinan akan gagal melalui kehilangan kekuatan atau menjadi semakin lemah melalui tindak balas dengan persekitaran yang terdedah kepada suhu yang tinggi daripada 900°C. Oleh itu, pembentukan satu lapisan oksida atau alumina sebagai pelindung adalah sangat diperlukan untuk memelihara bahan dasar apabila terdedah kepada suhu tinggi. Dalam projek ini, intermetalik FeAl telah dihasilkan dengan menggunakan kaedah metalurgi serbuk yang terdiri daripada proses pengaloian mekanikal, proses pemadatan sejuk, pensinteran, pemadatan panas dan rawatan permukaan melalui implantasi ion. Penambahan unsur-unsur reaktif atau oksidanya seperti Y, Y_2O_3 dan CeO_2 secara pengaloian mekanikal atau implantasi ion boleh menguatkan lagi lekatan antara lapisan oksida dengan logam dasar dan seterusnya meningkatkan ketahanan pengoksidaan. Pencirian dengan menggunakan SEM dan XRD telah dijalankan sebelum dan selepas setiap proses untuk mengkaji perubahan mikrostruktur, perubahan fasa dan pembentukan lapisan oksida. Ujian pengoksidaan berkitar telah dilakukan pada 900°C dan 1100°C untuk mengkaji kelakuan pengoksidaan bahan ini. Hasil kajian menunjukkan bahawa intermetalik FeAl telah berjaya dihasilkan secara pengaloian mekanikal, pemadatan panas dan rawatan permukaan melalui implantasi ion. Intermetalik FeAl yang diimplan dengan 3×10^{15} ion/cm^2 menunjukkan kadar pengoksidaan terendah pada suhu 900°C manakala intermetalik FeAl dengan 1 wt% yttria dan diimplan dengan 9×10^{15} ion / cm^2 mempamerkan kadar pengoksidaan terendah pada suhu 1100°C.

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LIST OF SYMBOLS

Fe	-	Ferum/Iron
Al	-	Aluminium
FeAl	-	Ferum-Aluminium
Y_2O_3	-	Yttria
CeO_2	-	Ceria
Al_2O_3	-	Aluminium Oxide/ Alumina
Cr_2O_3	-	Chromia
SiO_2		Silicon Oxide
Y	-	Yttrium
Ce	-	Cerium
La	-	Lanthanum
XRD	-	X-Ray Diffraction
SEM	-	Scanning Electron Microscope
EDX	-	Energy Dispersion X-ray
DTA	-	Differential Thermal Analysis
PM	-	Powder Metallurgy
SHS	-	Self-propagation High temperature Synthesis
RE	-	Reactive Element
II	-	Ion Implantation
TRIM	-	Transport of Ion in Matter
h	-	Hour
Wt%	-	Weight Percentage
At%	-	Atomic Percentage

FCC	-	Face Cubic Centre
BCC	-	Body Cubic Centre

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CHAPTER I

INTRODUCTION

1.1 Background of Study

The need for materials with high temperature capability in industries such as electric power generation, transportation and materials production/processing has increased dramatically since early 1900s. Process efficiency increases with operating temperature and early attempts to improve efficiency by raising temperatures were not always successful. Materials with the necessary capability were rarely available and the importance of high temperature materials in determining equipment performance and reliability was gradually appreciated. From the mid 1900s accelerating effort has been directed towards increasing the temperature capability of existing materials systems and developing new materials types. Understanding the material behavior and control of component manufacture to ensure the desired behavior have been key elements of these activities for all materials systems.

The requirement to operate at progressively higher temperatures will remain an ongoing need for the foreseeable future. Many industries will benefit from increased operating temperatures. As for example, in electricity generation, the efficiency of ultra-supercritical pulverized coal power plant can be increased from the current 47% to 50% if steam parameters can be increased from 290-bar/580°C to 325-bar/625°C. This will give major saving in fuel and consequent environmental benefits. Materials with higher temperature capability are essential if these, and many other objectives are to be met [1].

High temperature materials research in the metals and alloys area is still an extremely important field, and new alloy and composite systems are continually being developed for new applications. High temperature materials can be defined in several different ways, all of which are somewhat arbitrary. The least arbitrary definition is based on the maximum temperature used as a proportion of the melting temperature. In materials science and technology, high temperature can be defined as a temperature equal to, or greater than, approximately two-thirds of the melting point of a solid. High-temperature intermetallics have been vigorously studied since the early 1950s for various applications such as for the aerospace and power-generation industries. In this study, high temperature materials are taken to be those materials that are used specifically for their heat-resisting capabilities, such as strength or resistance to oxidation above 900°C. Many efforts have been made to improve materials high temperature oxidation resistance. There are two methods in improving the oxidation resistance, one is alloying and the other is surface treatment.

Intermetallic is a class of materials which formed by the combination of two or more metal elements, generally (but not always) falling at or near a fixed stoichiometric ratio and ordered on at least two or more sublattices. Aluminides, like NiAl, TiAl and FeAl alloys are one of the most materials considered for high temperature applications. Serious research on high temperature intermetallics began in the early 1950's and increased significantly around 1970 because of their perceived potential in aerospace. With weight saving being a key requirement, early work concentrated on the aluminide intermetallics based on nickel and titanium. Subsequent intermetallics such as Fe_3Al have been developed because of their potential benefits in replacing steels in various high temperature applications. They show excellent oxidation resistance, which is achieved by the formation of a protective Al_2O_3-scale.

Iron aluminides or FeAl are of great interest because of their many unique properties. The most exciting of these properties include low density, lower cost because iron and aluminium are the most abundantly available elements and excellent resistant to high-temperature oxidation [2]. They contain enough aluminium to form thin films of aluminium oxides in the oxidizing environments that are often protective. They posses relatively high specific strengths and suitable mechanical properties at elevated temperatures. However, these alloys only suitable for applications in aggressive and corrosive environments up to 900°C. Besides, the commercial uses of these compounds have been seriously hindered by deficiencies in their mechanical properties, mainly the poor ductility at room temperature and the inadequate creep resistance at high temperature. Therefore, the FeAl intermetallics alloys have undergone extensive development in the recent years exclusively for high temperature applications [3]. Powder metallurgy is one of the method used to introduce a dispersion of Y_2O_3 (1% in weight) via mechanical alloying process which can increase the mechanical properties of the FeAl intermetallics alloys at room temperature and improve the alloys creep resistance and high temperature strength.

The oxidation resistance might become a limiting factor for component design in the application at high temperature. These had become the main problem for heat-resistant application [4]. Metallic materials are protected against high-temperature oxidation by the formation of protective oxide scales such as Cr_2O_3, SiO_2 or Al_2O_3, which possess sufficiently low growth rates to prevent rapid component degradation. In this study, FeAl based alloys had been developed by using the powder metallurgy methods. Powder metallurgy methods have an advantage with respect to microstructure control, materials used, product homogeneity and mass production. The exploring and using of powder processing techniques will lead to improve mechanical properties due to their smaller grain size [5]. Decreasing the grain size will increase the material ductility [6][7].

Mechanically alloyed materials also have excellent oxidation and hot corrosion resistance. The increased resistance to oxidation-sulfidation attack is due to the homogeneous distribution of the alloying elements and the improved scale adherence due to the dispersoid itself. While concerning improvements in alloy corrosion resistance at high temperature, the most interesting chemical elements to incorporate into iron-base alloys are oxygen-active elements, such as yttrium, or rare earths, such as cerium or hafnium [8].

Surface treatment via ion implantation technique can modify the corrosion behavior and surface mechanical properties such as hardness and fatigue resistance of the alloys. Besides, this ion implantation will also lowers the coefficient of sliding friction and modifies the surface chemistry [9]. The using of the ion implantation technique will allows the introducing of a controlled yttrium concentration into the sample. Cyclic oxidation test and characterization of the samples are carried out to study the effect of each processes used in the powder metallurgy methods to the materials microstructure, phase transformation, and material properties or performance by using Scanning Electron Microscope (SEM), Energy Dispersion X-Ray (EDX) and X-Ray Diffraction analysis (XRD).

1.2 Rational of Using Powder Metallurgy Route

Intermetallic alloys are invariably made of alloying constituents having large difference in melting temperature and density leading to segregation of components during formation. Such alloys are apt to have poor room temperature ductility. Efforts were made in various laboratories to prepare these materials by using conventional melting and powder processing approaches. Both these techniques employ pure components for melting. Conventional melting is usually carried out either by arc melting or by induction melting techniques under air, vacuum or controlled atmosphere. However, conventional melting encounters a host of problems related to melting. Firstly, due to large difference in melting temperature and density of iron and aluminium, homogeneous melting is not achieved. Secondly, adding aluminium to molten iron causes a large temperature rise of the molten bath. Such a sudden rise in temperature is dangerous for operators and results in melt oxidation, longer holding time prior to pouring and a potential for missing the target chemistry because of oxidation of alloying elements. Therefore, in this work the mechanical alloying process in the powder metallurgy route had been used to produce the FeAl intermetallics materials since this process capable of producing many high temperature melting intermetallics with homogenous structure and composition easily.

1.3 Problem Statement

Iron aluminides or Fe-Al based intermetallic alloys suitable for applications in aggressive and corrosive environments up to 900°C. However they may fail through loss of strength or gradually deteriorate through reaction with the surrounding atmosphere at temperature higher than 900°C. Therefore, the formation of a stable protective oxide scale on the surface is required to protect the underlying materials. The addition of 0.1 wt% to 2 wt% reactive elements or its oxide such as Y, Y_2O_3 and CeO_2 by mechanical alloying or treating the surface by implantation methods may improve their oxidation resistance through the enhancing of the alumina scale adhesion to the underlying metals. The oxide layer or scale formed tends to spall easily and the oxidation rate will increase if the amount is more than 2 wt% or less than 0.1 wt% [10].

1.4 Objectives of Study

The objectives of this research are:

i. To develop FeAl based intermetallics alloys for high-temperature applications by using powder metallurgy method.
ii. To study the effect of reactive elements addition such as yttria (Y_2O_3) and cerium dioxide (CeO_2) on the FeAl microstructure and oxidation behavior at 900°C and 1100°C .
iii. To study the effect of yttrium ion implantation on the FeAl oxidation behavior at 900°C and 1100°C.
iv. To determine of the optimum way of developing novel FeAl based alloy for high temperature application based on the results analyzed.

1.5 Scopes of Study

The scopes of this research are:

i. Development of high temperature material FeAl based alloy with and without addition of reactive elements such as Y_2O_3 and CeO_2 by using mechanical alloying process and followed by powders consolidation.
ii. Characterization of the as milled powders and as consolidated powders by using the Scanning Electron Microscope (SEM), Energy Dispersion X-ray (EDX), X-Ray Diffraction analysis (XRD) and Differential Thermal Analysis (DTA).

iii. Implementation of surface treatment via ion implantation with different doses of yttrium ion, 3×10^{15} ion/cm^2, 6×10^{15} ion/cm^2 and 9×10^{15} ion/cm^2.

iv. Implementation of cyclic oxidation at 900°C and 1100°C for 200 hours oxidation time.

v. Microstructure and phase analysis before and after the implementation of ion implantation and cyclic oxidation by using SEM and XRD.

vi. Determination of the optimum way of developing FeAl based alloy with and without reactive elements addition and yttrium ion implantation for high temperature application based on the results analyzed.

CHAPTER II

LITERATURE REVIEW

2.1 Introduction to Powder Metallurgy

It is well-known that materials with the same nominal composition can be produced in different ways, from classical melting and casting, practiced by conventional metallurgy, via powder consolidation by powder metallurgy methods, mechanical alloying, combustion synthesis with thermo-mechanical treatment, etc. Each technological route produces material having different microstructure, different concentrations and types of defects and therefore totally different properties [11].

Generally, powder metallurgy (PM) applications fall into two main groups. The first group consists of components difficult to make by any other production method, such as those components made from tungsten, molybdenum, or tungsten carbide. Porous bearings, filters, and many types of hard and soft magnetic components are also produced exclusively by the PM process. The second group of applications consists of components for which PM produces near-net shape components and is a cost-effective alternative to machined components, castings and forgings. Examples are automotive components such as clutch plates, connecting rods, cam shafts, and planetary gear carriers. PM components are used in a variety of

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