REGIONAL COOPERATION ON RESEARCH OF ADVANCE MATERIAL AND FAILURE ANALYSIS

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Regional Cooperation on Research of Advance Material and Failure Analysis

- 6 Years of Research Cooperation Between Universiti Tun Hussein Onn Malaysia (UTHM) and Badan Tenaga Nuklir Indonesia (BATAN)

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ABSTRACT

The aim of the regional cooperation was started with a simple aim of sharing the expertise and promoting a scientist exchange program. The cooperation focused on the development of an integrated and multidiscipline project with expectation of creating a snowball effect on human resources development and networking environment at both national and international level. Currently, the Cooperation is on going to renew its present goal to a new qualitative level, in which both partner will complement each other in a profound interrelation of experiment, characterization and modeling and also to prepare the basis for the technological progress as desired. The future cooperation, however, will not directed to the technological application, but it will more focus on the development realization, hopefully it will give a great importance in development of technology by self.

1. Introduction

Universiti Tun Hussein Onn Malaysia was, formely, known as Kolej Universiti Tun Hussein Onn is currently, inviting expertise from Badan Tenaga Nuklir Indonesia (Batan) to cooperate in supervising to the postgraduate students and also to create a research project which could contribute to the human resources development for both site and in addition to develop an integrated multidiscipline approach and research based an oriented product. Initially the cooperation was started on exploring the application of a high sophisticated equipment: Scanning Electron Microscope and X- Ray Diffraction Equipment for a microstructure characterization over various samples which are provided by BATAN. Sequentially a seminar was conducted to review the status of joint research cooperation between UTHM and BATAN. The seminar also propose the possibility of the cooperation between Asean and European Link [1], and [2]. UTHM supported this cooperation through 2 (two) short term research grant projects with entitled [1] " Development of Catalytic Converter for Automotive Application (Vot 0153)" and [2] "Development of Fe-Al based alloy using Nano Technology (Vot 0157)". The projects were then also supported by The Malaysian Education Ministry through approval on the projects : "Development of Elemental Powder Metallurgy Route for Producing of Novel Nanostructure FeAl Intermetallic for High Temperature Application (Vot 0265) for the period 2006 to 2009 and "Improvement of Ferritic Stainlesss Steel for High Temperature Fuel Cell (Vot 0361)" for the period of 2007 to 2009. Universiti Tun Hussein Onn Malaysia (UTHM) supported this cooperation work through providing a short grant research entitled "Develop an Innovative Spiral Spring Form for Catalytic Carrier of Catalytic Converter (Vot 0311)" for a tutor too.

During the running out project mentioned with a concrete user-specific question, especially to the user which employed catalyst substrates in auto mobile, had suggested that the goal of the project more directed to create a comprehensive material-scientific and technology-oriented authority with both partners.

2. Current Status of Cooperation

2.1 Current Status R &D on Catalytic Converter [3], [5], [6], [7]

A multidiscipline approach, high and sophisticated technologies are required to develop a catalytic converter. University Tun Hussein Onn Malaysia (UTHM) and Badan Tenaga Nuklir Indonesia (BATAN)-Indonesia in which has a limited expertise and infrastructure in such technology agree to cooperate in developing an innovative catalytic converter aimed for finding an appropriate technology by using human and natural resources in both countries. The efforts involved in conducting a conceptual design and preliminary design based on Pahl/Betz approached as well.

The increasing number of vehicles around the world had increased the air pollution due to the harmful engine emissions. The first strict regulation to control automobile emissions was introduced through a US Clean Air Act,1970, in the United States of America. Since that time, continuous stringent regulations had been forced to the automobile companies to accelerate their research and development to fulfill these regulations. One of the technological advances was concentrated on the removal of pollutants in the exhaust system and it was inspired by the invention of modern catalytic converter in 1965. Since then, exhaust after treatment technology had become a main focus of among researchers and it had been implemented by the automobile industries around the world. Proton installed the catalytic converter for its new type Proton such as in Gen 2 and Persona.

Catalytic converter is a device installed in the exhaust system to reduce the harmful emissions from the engine. In a gasoline engine, the main harmful gases are CO, HC and NO_x. These gases are eliminated by the basic reactions occur inside a catalytic converter i.e. i) oxidation for CO and HC and ii) reduction for NO_x. Simultaneous reaction of oxidation and reduction gives it which called in term : Three Way Catalyst (TWC) and it had become the most common type of catalytic converter.

There are four basic components of catalytic converter i.e. i) washcoat ii) catalyst iii) casing and iv) substrate. Washcoat is a thin layer of alumina (Al_2O_3) coating, typically 20-150 μm thick with a high surface area on the top of substrate. It also called as a catalyst carrier, and acts as an intermediate layer in order oxidation and reduction process to occur. In TWC, washcoat contains ceria compound which playing role in providing oxygen storage capacity (OSC). They absorb oxygen in lean conditions and release it as oxygen under rich conditions. This will enhance the conversion to all three pollutants as far as the engine condition is buffered around stoichiometric point. An effort to develop a washcoat using difference deposition technique of SiC and Alumina on FeCrAl is on going [4].

Catalyst accelerates the chemical reaction of oxidation and reduction but neither products nor reactants of the reaction. The most common metals used Platinum (Pt) and Palladium (Pd) for its oxidation and Rhodium (Rh) for its reduction. The combination of Pd/Rh and Pt/Rh are commonly used in the Three Way Catalyst (TWC). However, intensive research on several potential catalyst including Cu-ZSM-5, and metal impregnated zeolite are necessary. The project started with deposition of natural zeolite into the existing substrate and the developed a corrugated aluminium to test the influence of zeolite on reducing of harmfull gases (Figure 1 and Figure 2). The research concentrated now on using nickel because it is relatively cheap and easy to get it [4].

Casing is a physical enclosure designed to protect the substrate from the external dirt; flow, thermal shock and vibration related load. Several types of materials are available for the selection purposes, such as structural steel and cast iron, however the most common type in use is stainless steel with the thickness of 1 to 2 mm. Another criterion is the shape of the casing including circle, ellipse and square. Design of diffuser also affects the flow distribution which is important to improve flow uniformity and enhance the performance of catalytic converter.

There is a continuous research on the substrate development for a catalytic converter. Two types of substrates dominate in the market are metallic and ceramic. The earliest structure was in the porous beads or pellets before being replaced with the extruded honeycomb. Extruded ceramic honeycomb had been used for a catalytic converter since its first patent in 1965. The honeycomb use cordierite ($2MgO.2Al_2O_3.5SiO_2$) which has a high thermal shock resistance, high melting and softening points, high attrition resistance and low pressure losses. However, there is a continuous effort to use porous ceramic as an alternative substrate. Porous ceramics are brittle materials with closed, fully open or partially interconnected porosity. In the automotive application, open cell ceramic foams are commonly used in the exhaust system. Some of the advantages are due to its high geometrical surface area, great hardness, strong oxidation resistance and small thermal expansion coefficient. An effort of producing a porous ceramic combined with a zeolite was conducted [8], [9], [10] and product was appreciated by Malaysian scientist as innovative product. The product was awarded some medals in different exhibition.

Metallic substrate has been used in certain market and it shows opportunities for further development of products and systems to treat the exhaust gases. The monolith is made from Ferum Chromium Aluminium ultra thin foil, corrugated and laid up to form a honeycomb structure. It has several advantages including higher thermal and shock resistance properties compared to cordierite, more robust and able to develop higher cell density. The wall thickness of metal foil is much less than the ceramic which produce higher frontal area and lower the pressure drop. These criteria are definitely needed by the exhaust system in order to improve the emissions without sacrificing the engine performance.

The substrate technology has a rapid progress for both ceramic and metallic. For ceramic; 400, 600, 900 and 1200 cpsi with the wall thickness has been reduced from 12 mil (0.305 mm) to 2 mil (0.05 mm). The advantage of metallic substrate is the ability to reduce the thickness ranged from 0.05 mm to 0.02 mm (2 mil to 0.8 mil) and able to produce up to 1600 cpsi. Hence, the pressure loss is greatly reduced; the heat capacity is 20% lower than the traditional 400 cpsi and finally improve the cold start behaviour. It also can be located closer to the engine (with higher operating temperature i.e 1100 °C) to treat the gases during the cold start.

Determination of the substrate dimension is a vital part to obtain a high conversion efficiency of catalytic converter. Correlations between geometrical dimensions to the performance had been investigated over some parameters including length, substrate diameter, wall thickness and cell shape of the substrate. The substrate length was investigated from 4 to 16 cm. Generally, the emission decrease with the increase of substrate length but it differs for different species. However, it can be concluded that 14-16 cm substrate length was a suitable lenght; longer substrate will increase the thermal substrate, large volume and lastly increase the cold start emission. While for the cross sectional area, a large cross section is not effective compared to a similar volume with larger length and smaller cross sectional area. Other important criteria of geometrical selection are cell density and wall thickness. Higher cell density will increase surface reaction area per unit volume. Hence the decrease of emission is significant for the cell density between 200-800 cpsi. The same trend was seen on the wall thickness where its reduction will improve the overall emission due to the decrease of substrate heat capacity.

This cooperation results in an innovative tool to produce a spiral corrugated foil with length 150 cm, width 100 cm and the thickness 0,11 mm, 0, 065 and 0, 045 mm (Figure 3, Figure 4 and Figure 5). The foil was supplied by Thysenkrupp.

Besides that this project worked on application of Transmission Electron Microscope. It revealed that the commercial FeCrAl alloys in which contains minor Zr and Hf elongated often form inside the scale consisting of Zr/Hf carbides for (Figure 6). This carbides incorporated at first into the Al_2O_3 scale does not occur locally or randomly, but is rather a typical phenomenon. The carbide precipitates in the alloy initially remain stable as long as they are embedded inside the scale where the oxygen partial pressure is extremely small. Upon further scale thickening by inward growth, the oxygen partial pressure becomes higher. This leads to the transformation of carbides to oxides [13].

2.2 Research on Development of Fe Al based alloy [14],[15],[16],[17]

When materials are subjected under high temperature condition such as occurred in gas turbines, rockets engines and furnace components, they may fail through loss of strength or they may gradually deteriorate through reaction with the surrounding atmosphere. Oxygen is very corrosive to the most materials, especially at high temperatures. High temperature oxidation is basically the reaction of a metal with oxygen to form an oxide. Most high-temperature-resistant alloys oxidize to form an external alumina layer, or scale, whose slow growth protects the underlying alloy from continued aggressive oxidation. Thus, the formation of a stable protective oxide scale on the surface is required to protect the component below.

Fe-Al based intermetallic alloys are great of interest because it offers a lot of exciting properties including a low density, lower cost material since iron and aluminium are the most abundantly available elements on earth, and excellent resistant to high-temperature oxidation, sulfidation, carburization and molten salts . However, this Fe-Al alloys show some drawbacks such as low room-temperature ductility and lack of high-temperature strength especially at higher aluminium content. Therefore, efforts to improve their ductility at room temperature have been made by appropriate processing, such as a micro alloying, and oxide dispersion strengthening (ODS), which led to enhancement of grain boundary cohesions, grain structure refinement, control of grain shape and adjusting the recrystallization condition.

In this work, the alloys was produced by using powder metallurgy routes which include the mechanical alloying process, cold compaction, sintering, hot compaction and surface treatments via ion implantation. Each route process produced materials having different microstructures, phases and properties. The addition of reactive elements or their oxides such as Y, Y_2O_3 and CeO_2 through a mechanical alloying or ion implantation method may improve their oxidation resistance through the enhancing 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 the formation of the oxide layer. Cyclic oxidation tests were performed at 900°C and 1100°C to study the oxidation behavior of this material. The results showed that the FeAl intermetallic had successfully produced by mechanical alloying, hot compaction and surface treatment via ion implantation. The FeAl intermetallics with 3×10^{15} ion/cm^2 doses of yttrium implanted exhibits the lowest oxidation kinetics at 900°C while FeAl intermetallics with 1 wt% yttria and 9×10^{15} ion/cm^2 doses of yttrium implanted exhibits the lowest oxidation kinetics at 1100°C. The yttrium implantation to the FeAl alloyed with 1 wt% CeO_2 had increase the oxidation kinetic of the alloy at both oxidation temperature except for alloy implanted with 3×10^{15} ions/cm^2 at 900°C and alloy implanted with 9×10^{15} ions/cm^2 at 1100°C.
Figure 7 shows the SEM images at the cross section of un-implanted and implanted Fe-45 at% Al samples after oxidized at 900°C. From the figures, it can be seen clearly see clearly the oxide scale or layer formed on the substrate surface. No scale spallation occurred in both of the samples. Figure 8 shows that the scale spallation tends to occurr seriously in the un-implanted Fe-45 at% Al after oxidation at 1100°C. The implanted Fe-45 at% Al showed better scale adherence compared to the un-implanted one. Figure 9a and Figure 9b show the effect of yttrium ion implantation and ion doses to the oxidation kinetics of Fe-45Al alloys with 1 wt% CeO_2 addition at 900°C and 1100°C respectively The current research continued with developing FeCr for intermetallic for fuel cell and some result was presented [18], [19], [20].

2.3 Research on Failure Analysis

The cooperation on failure analysis related to systematic investigation a Superheater Tube of Coal Fired Power Plant [21] , Corrosion Issue on Water Gate in Batu Pahat, Malaysia [22] and Systematic Investigation of Failure Analysis on a Steam Trap Bypass Tube in a Coal-Fired Power Plant [23]. The Systematic Investigation of Failure Analysis on a Steam Trap Bypass Tube in a Coal-Fired Power Plant related to a coal fired power plant with its normal operation temperature of 540°C in which its steam trap bypass tube in that power plant was totally fractured. The aim of this study is to explore the evidence related to the steam trap bypass tube failure and to determine the failure mechanism and the root cause of the failure and to give an appropriate recommendation. This study consists of failure mode inventory of the steam trap bypass tube failure, collection of background information about the process, component function and operating conditions. Detailed investigation is carried out by visual examination, nondestructive testing (NDT), metallurgical testing which consists of microstructure examination, chemical testing and mechanical testing. Optical Microscopy (OM), Scanning Electron Microscopy (SEM) combined with Energy Dispersive X-ray Spectroscopy (EDS), Glow Discharge Spectrometer (GDS) and Energy

Dispersive X-ray Diffraction (XRD) experiments were used throughout the investigation on the sample obtained. From the evidence with considering the contribution factors such as temperature, pressure and environment, a fault analysis was made and it can be concluded that the cause of failure to the steam trap bypass is due to multi causes which consists of creep failure and hydrogen damage. The root cause of high temperature creep and hydrogen damage which occurred at the steam trap bypass tube is due to material properties that are inadequate for the actual operating conditions of a steam trap bypass tube which is not according to the specification. The material must be actually ASTM SA-335-P22 (2.25Cr-1Mo) with 490 MPa minimum tensile strength and 320 MPa minimum yield strength. However from the investigation found that the material used was ASTM SA-192 (low strength carbon steel) with 324 MPa minimum tensile strength and 180 MPa minimum yield strength.

3. Conclusion

Co-operation is planning to set a qualitative new level as its goals now. The partners complement each other in a profound interrelation of experiment, characterization and modeling and prepare the basis for the technological progress desired. In respect to differently than with the current project, the in coming work is slightly different, it will not be aimed to a direct technological application., but more on the development of fundamental realizations.

4. Acknowledgment

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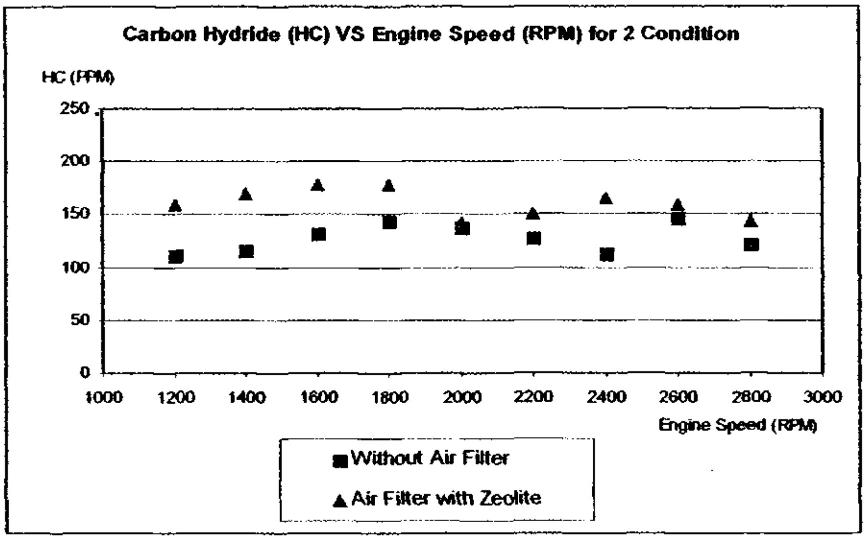

Figure 1. Comparison of HC content with and without zeolite

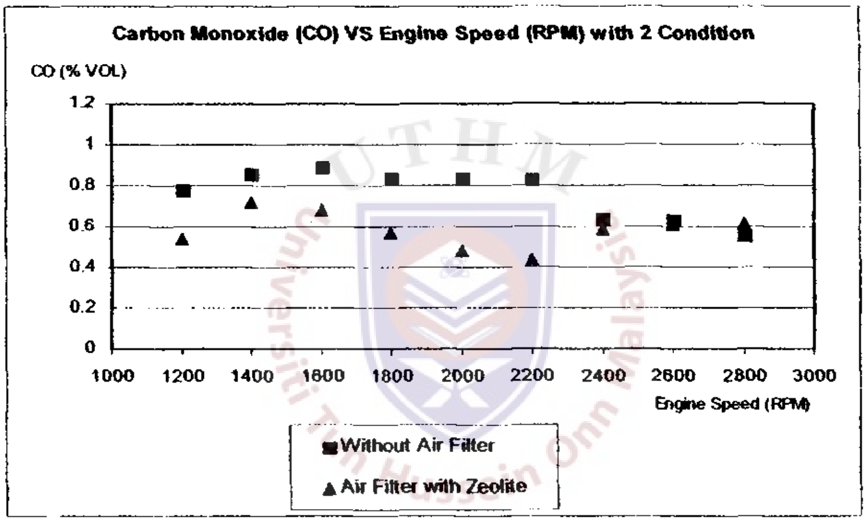

Figure 2. Comparison of CO content with and without zeolite

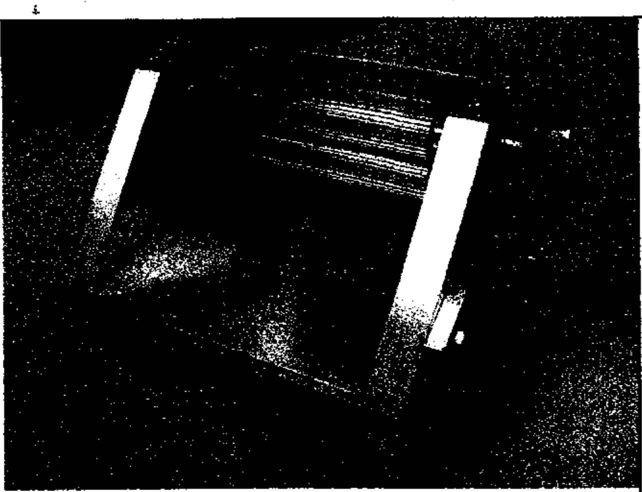

Figure 3. Apparatus for producing a corrugated sheet metal foil

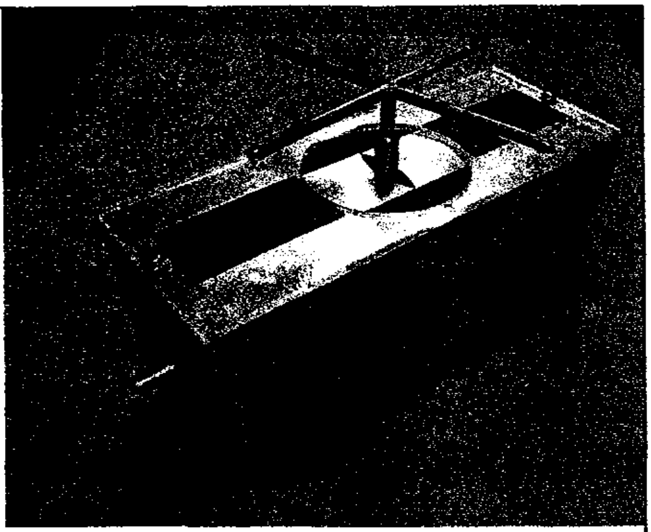

Figure 4. Apparatus for producing a spiral form of subtrate

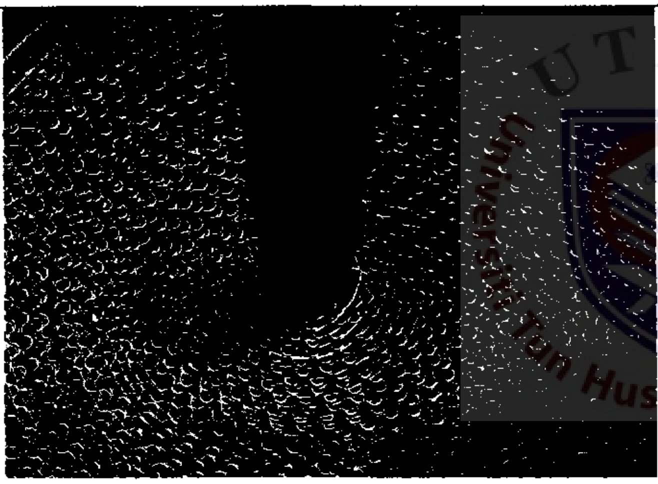

Figure 5. Corrugated spiral form fabricated by an innovative apparatus

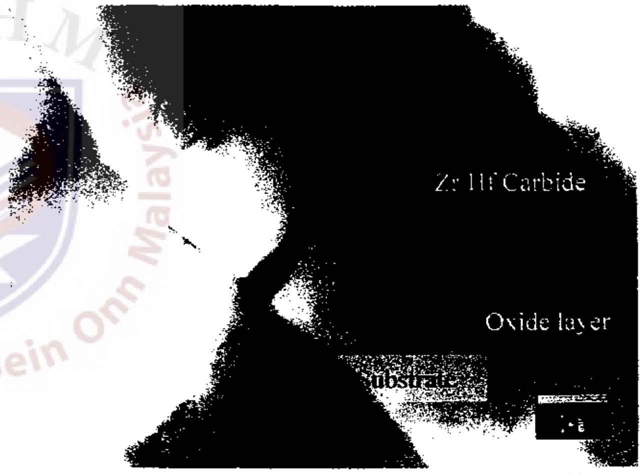

Figure 6. TEM micrographs of FeCrAl alloy revealing the presences of carbide precipitates like an anchor type at the oxide and substrate interface.

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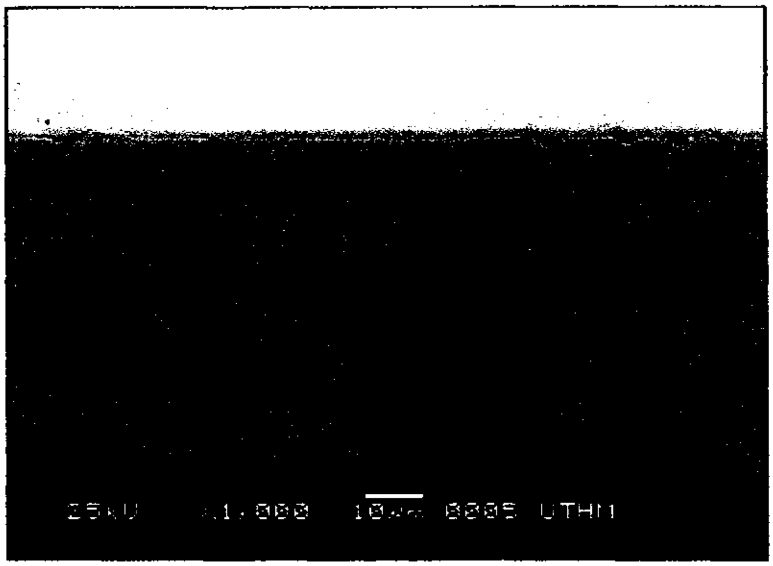
(a)



(b)

Figure 7: The SEM images at the cross-section area for (a) un-implanted FeAl, and (b) implanted FeAl after oxidation at 900°C.

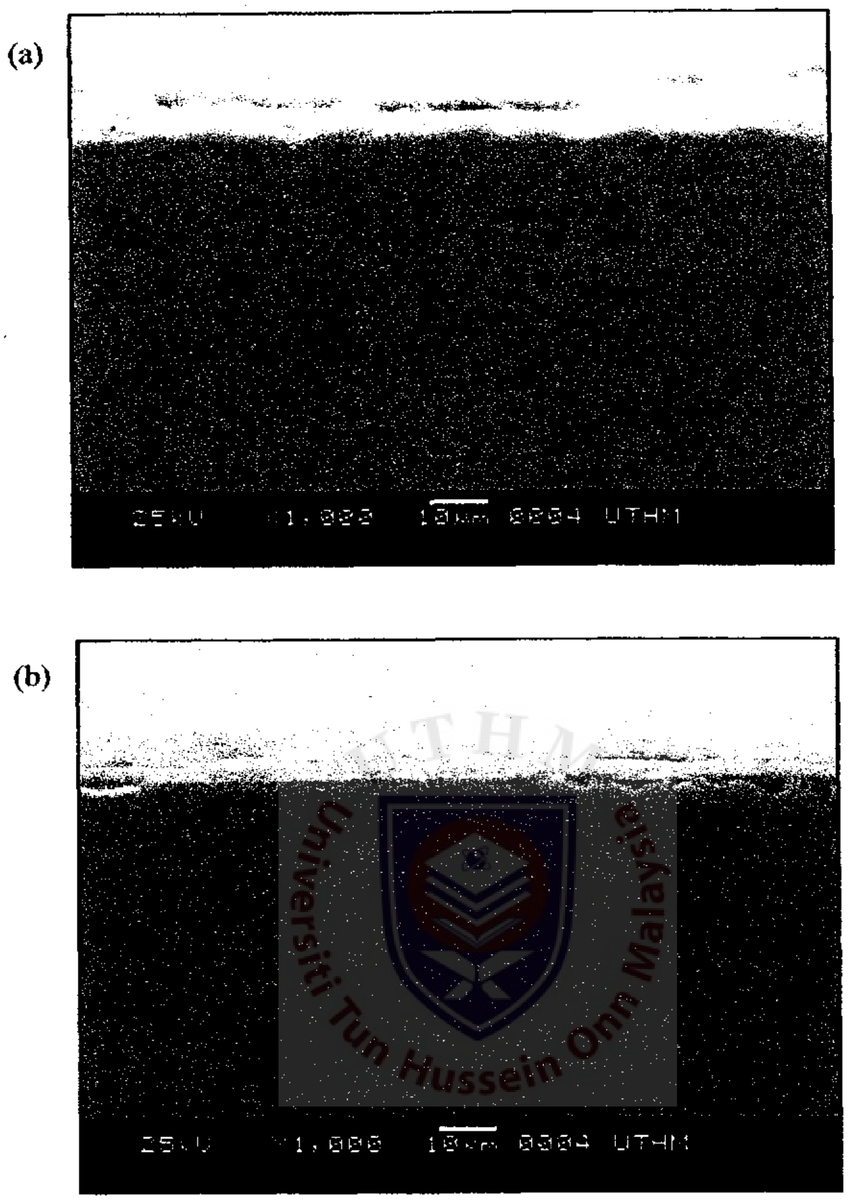
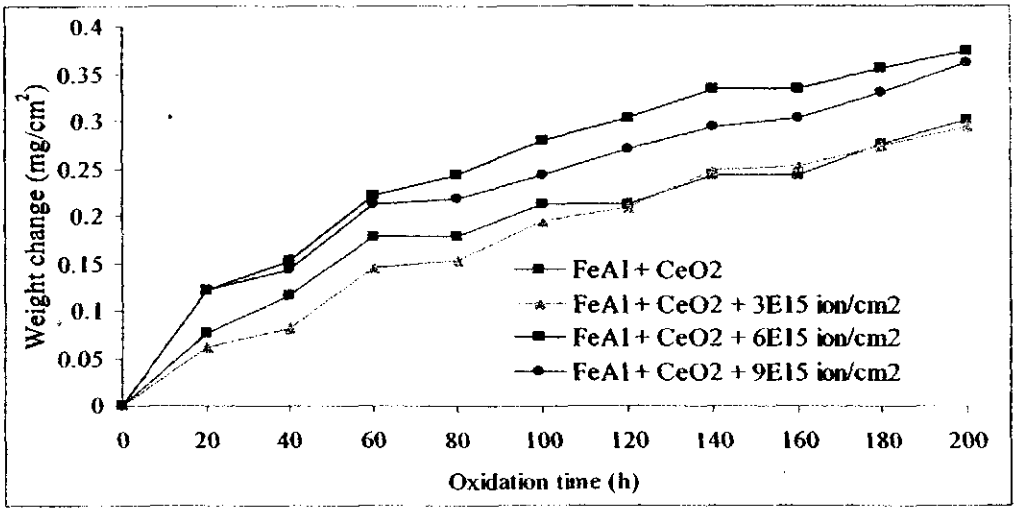
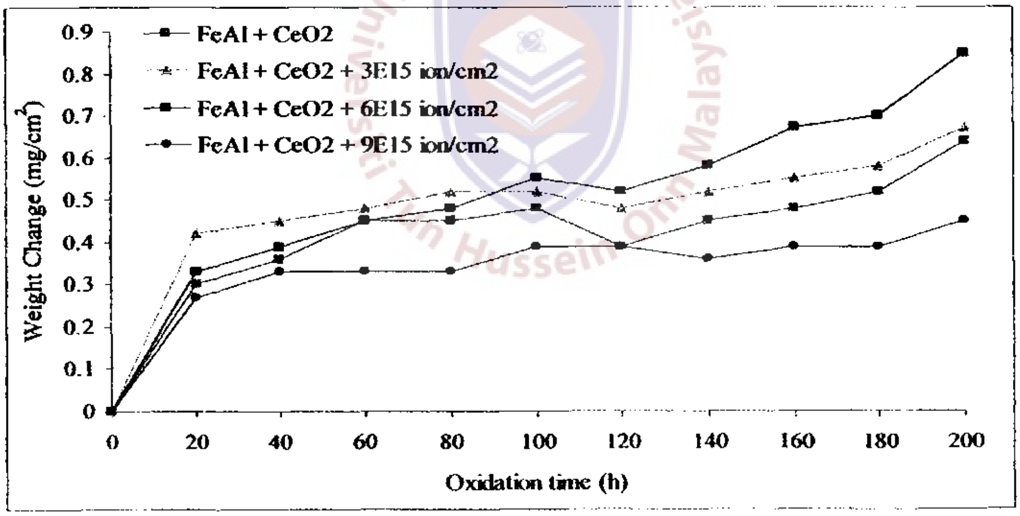


Figure 8: The SEM images at the cross-section area for (a) un-implanted FeAl, and (b) implanted FeAl after oxidation at 1100°C.



(a) The effect of yttrium ion implantation doses on the oxidation behavior of FeAl based alloy with 1 wt% CeO2 addition at 900°C.



(b) The effect of yttrium ion implantation doses on the oxidation behavior of FeAl based alloy with 1 wt% CeO2 addition at 1100°C.

Figure 9: Variation of mass gain as a function of exposure time during oxidation.