CARBOCHLORINATION REDUCTION OF ILMENITE (FETI03)

RIZAL ASTRAWINATA

UNIVERSITI SAINS MALAYSIA
KAMPUS KEJURUTERAAN
2008

UNIVERSITI SAINS MALAYSIA

Laporan Akhir Projek Penyelidikan Jangka Pendek

Carbochlorination Reduction of Ilmenite ($FeTiO_3$)

by

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PEJABAT PENGURUSAN & KREATIVITI PENYELIDIKAN
RESEARCH CREATIVITY AND MANAGEMENT OFFICE [RCMO]

LAPORAN AKHIR PROJEK PENYELIDIKAN JANGKA *PENDEK*
FINAL REPORT OF SHORT TERM RESEARCH PROJECTS

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1) **Nama Ketua Penyelidik** :
Name of Research Leader :

Ketua Penyelidik *Leader*	PTJ *School/Centre*
Rizal Astrawinata	School of Materials & Mineral Resources Engineering

Nama Penyelidik Bersama
(Jika berkaitan) :
Name/s of Co-Researcher/s
(if applicable)

Penyelidik Bersama *Co-Researcher*	PTJ *School/Centre*
Professor Dr. Kamarudin Hussin	(*Formerly*) School of Materials & Mineral Resources Engineering
Prof. Madya Dr. Azizan Aziz	School of Materials & Mineral Resources Engineering

2) **Tajuk Projek** : ....
Title of Project:

Carbochlorination Reduction of Ilmenite ($FeTiO_3$)

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3) **Abstrak untuk penyelidikan anda**

(Perlu disediakan di antara 100 – 200 perkataan di dalam **Bahasa Malaysia dan Bahasa Inggeris**. Ini kemudiannya akan dimuatkan ke dalam Laporan Tahunan Bahagian Penyelidikan & Inovasi sebagai satu cara untuk menyampaikan dapatan projek tuan/puan kepada pihak Universiti & luar).

Abstract of Research

(Must be prepared in 100 – 200 words in Bahasa Malaysia as well as in English. This abstract will later be included in the Annual Report of the Research and Innovation Section as a means of presenting the project findings of the researcher/s to the university and the outside community)

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Thermogravimetric analysis (TGA) has been proven to be an effective tool in the studies of carbochlorination kinetics of oxide minerals. Using this technique effects of the reaction parameters, such as gas flow rate, temperature and partial pressure of the carbochlorinating gas mixture on the rate of reaction have been examined by earlier investigators. Experimental results indicated that the rate of reaction, in some instances, increases as the carbon mass fraction increases.

Particularly for oxides of metallic titanium, e.g., ilmenite ($FeTiO_3$) and rutile (TiO_2), the chlorination of metallic oxide in the presence of carbon (carbochlorination) has displayed the potential to set off two or more consecutive reaction stages as indicated by the different activation energy values for each reaction stage.

In this research investigation, which was financed by the short-term grant, unfortunately, the intended carbochlorination experimentation could not be carried out because the limited budget was not enough to provide an experimental rig which abides the safety & health regulation on the usage of toxic chlorine gas. Without the hazardous gas monitoring and safety devices, however, the experimental rig can and has been utilized to conduct thermogravimetric analysis (TGA) using non-toxic gases, such as : air, oxygen, nitrogen, etc.

The objective of the investigation that has been carried out with the existing experimental rig was to study the reaction mechanism of titanium aluminides in air and in nitrogen gas environment at high temperatures. Although this alloy has superior high-temperature mechanical properties, its high-temperature application is limited by its relatively poor resistance against hot gases.

As the outcome of this study, the experimentally obtained activation energies of the scale formation reaction on the surface of Ti-48Al-2Cr-2Nb alloy exposed in air and in nitrogen gas are 277.5 kJ/mol and 224.8 kJ/mol, respectively. The obtained result is in accord with the quantitative observation of scale thickness on the surface of the alloy which is thicker in nitrogen gas than in air. At higher temperatures, the rate of growth and hence the scale thickness will increase. The scale formation on the surface of the alloy may eventually limit the alloy's service life-time.

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Activation energy determination for reaction mechanism on titanium aluminides exposed in air and in nitrogen gas at 800° – 1000° C.

Introduction

Gamma TiAl based alloy which contains 45-55 at-% aluminum is one of the titanium aluminides having great potential for high-temperature applications, such as jet engine components and exhaust valves in car engines, owing to their specific strength, stiffness, creep and fatigue resistance at elevated temperatures (700-1000 °C). As structural materials, the alloy will have not only to withstand mechanical stresses but also be exposed to aggressive atmospheres and/or hot gases.

The purpose of this investigation was to study the reaction kinetics of T1-48Al-2Cr-2Nb alloy exposed in air and in nitrogen gas at elevated temperatures through the use of the well-known empirical Arrhenius relationship for activated processes.

Experimental

Small ~4g button-shaped castings of Ti-48Al-2Cr-2Nb alloy were prepared by arc-melting. The castings were then cut with diamond cutting tool into small (10 x 10 x 2 mm) square coupons and polished to a 2000 grit finish using SiC paper followed by 0.1 μm and 0.05 μm alumina powder.

Kinetic behavior was studied by thermogravimetric analysis (TGA) in 10 (ten) hours tests at 800 , 900 and 1000 °C under flowing air and under flowing nitrogen gas environments. Exposed coupons were etched with *Kroll reagent* (10 vol-% HF , 4 vol-% HNO_3 , 86 vol-% H_2O) and characterized by X-ray diffraction (XRD) , secondary mode scanning electron microscopy (SEM) , and energy dispersive X-ray analysis (EDX).

Results

The activation energies of the reaction product, appearing as scales on the surface of the Ti-48Al-2Cr-2Nb alloy exposed in air and in nitrogen, have been determined to be 277.5 kJ/mol and 224.8 kJ/mol, respectively. Measured scale thickness on the alloy exposed in air and in nitrogen gas at 1000 °C for 10 (ten) hours were 11.4 μm and 15.3 μm, respectively. Thicker scale developed on the surface of the alloy exposed in nitrogen further substantiates the lesser value of the activation energy under this environment. At higher temperatures the thickness of the resulted scale increases accordingly.

Exposed in air, the outermost layer of the scale was made up of TiO_2, followed by an intermixedAl_2O_3/TiO_2 scale directly beneath it. This observation suggests that the titanium atom has higher diffusion rate toward the alloy surface than the aluminum atom even though aluminum thermodynamically has higher affinity to oxygen than titanium. Similarly, for the coupons exposed in nitrogen gas, an outermost layer of TiN contaminated with minute amount of TiO_2 impurity spread over the inner layer of Ti_2AlN contaminated with TiO_2 and Al_2O_3 .

XRD analysis revealed the existence of γ-TiAl and α_2-Ti_3Al phases in the Ti-48Al-2Cr-2Nb alloy produced by arc-melting. Microstructural observation using SEM/EDX analysis indicated the presence of duplex microstructure consisting of single-phase γ-TiAl grains and two-phase grains with lammelar structure of alternating α_2-Ti_3Al and γ-TiAl plates. It is common knowledge that the single-phase gamma and duplex structure give rise to high strength and some ductility but relatively poor creep resistance, lowfatigue strength and low fracture toughness.

4) Sila sediakan Laporan teknikal lengkap yang menerangkan keseluruhan projek ini. [Sila gunakan kertas berasingan]
Kindly prepare a comprehensive technical report explaining the project (Prepare report separately as attachment)

Senaraikan Kata Kunci yang boleh menggambarkan penyelidikan anda :
List a glosssary that explains or reflects your research:

Bahasa Malaysia	Bahasa Inggeris
Analisis Permeteran graviti haba.	Thermogravimetric analysis............................
Titanium aluminida	Titanium aluminides
Tenaga pengaktifan	Activation energy
....	
....	....
....	....

5) **Output Dan Faedah Projek**
Output and Benefits of Project

(a) * Penerbitan *(termasuk laporan/kertas seminar)*
Publications (including reports/seminar papers)
(Sila nyatakan jenis, tajuk, pengarang, tahun terbitan dan di mana telah diterbit/dibentangkan).
(Kindly state each type, title, author/editor, publication year and journal/s containing publication)

....
........................— N O N E —...
....

(b) **Faedah-Faedah Lain Seperti Perkembangan Produk, Prospek Komersialisasi Dan Pendaftaran Paten atau impak kepada dasar dan masyakarat.**
Other benefits such as product development, product commercialisation/patent registration or impact on source and society

Fundamental research for academic & educational purposes only.

* Sila berikan salinan
* *Kindly provide copies*

(c) **Latihan Gunatenaga Manusia**
Training in Human Resources

i) Pelajar Siswazah : ...**None**...
Postgraduate students:
(perincikan nama, ijazah dan status)
(Provide names, degrees and status)
....

....

....

ii) Pelajar Prasiswazah *:* **1 (one) completed...**
Undergraduate students:
(Nyatakan bilangan)
(Provide number)
....

Ooi Tze Shian, *"Determination of Activation Energy and Oxidation Mechanism of Titanium Aluminides Alloy in Compressed Air and Nitrogen Gas at Temperatures 800 – 1000 °C".*

iii) Lain-Lain : **3 (three) to be carried out this academic year....**
Others:
....

Aisha bt Mohd. Ripin (matric no. 73455), *"Factors Affecting the Oxidation Nature of Magnesium Alloys".*

Ooi Mey Ling (matric no. 73517), *"The High-temperature Oxidation Resistance of Copper-Aluminum Alloy".*

Ng Siew Nee (matric no. 73504), *"High-temperature Oxidation Kinetics of Copper"*

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6. **Peralatan Yang Telah Dibeli :**
Equipment that has been purchased:

Vertical tube furnace
High-precision analytical balance
Data acquisition system
Experimental cell arrangement

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KOMEN JAWATANKUASA PENYELIDIKAN PUSAT PENGAJIAN
Comments of the Research Committees of Schools/Centres

Laporan akhir projek ini didapati memuaskan. Hasil yang diperolehi juga telah memenuhi hasrat objektif project ini.

Projek ini telah menghasilkan seorang pelajar sarjana dan satu artikel juga berjaya di jana.

TANDATANGAN PENGERUSI
JAWATANKUASA PENYELIDIKAN PUSAT PENGAJIAN
Signature of Chairman
[Research Committee of School/Centre]

PROF.MADYA DR. AZIZAN BIN AZIZ
Timbalan Dekan
(Pengajian Siswazah dan Penyelidikan)
P.P. Kejuruteraan Bahan & Sumber Mineral
Universiti Sains Malaysia

22-2-07
TARIKH
Date

Updated : 16MAC2006

Activation energy determination for reaction mechanism on titanium aluminides exposed in air and in nitrogen gas at 800° – 1000° C.

Ooi Tze Shian , R. Astrawinata , O.J.P. Manalu

Abstract

Oxidation and nitridation experiments have been conducted on Ti-48Al-2Cr-2Nb (at.-%) alloy in flowing laboratory air and in flowing nitrogen gas at atmospheric pressure in the temperature range of 800 to 1000 °C for 10 (ten) hours by thermogravimetry (TGA). Parabolic rate law was used to describe the growth of scales in both cases. The activation energy, Q , was found to be 277.4 kJ/mol for oxidation and 224.8 kJ/mol for nitridation. The activation energy for oxidation obtained in this work falls within the range of the activation energy for oxidation reported in the literature.

1. Introduction

The combination of low density with high strength and creep resistance at elevated temperatures offered by the gamma alloys based on TiAl intermetallic compound are currently challenging conventional Ni-based superalloys. The attractive properties possessed by these gamma alloys has led to the development of numerous structural materials for the last 15 years that can be used for both aerospace and automobile industrial applications. The alloys under development contain several alloying elements to optimize thermally stable microstructures and/or to improve high temperature mechanical properties. Optimized mechanical properties can be obtained with alloys consisting of γ-TiAl with 3 to 15 vol.-% α_2-Ti_3Al. The chemical composition of commercially available γ/α_2–TiAl alloys with optimized high-temperature strength can be described as follows: Ti , (45-48 at.-%) Al , (1-3 at.-%) X , (2-5 at.-%) Y , and (< 1 at.-%) Z , where X = Cr , Mn, and V ; Y = Nb, Ta, Mo, and W ; and Z = Si, B, and C. With respect to mechanical properties, the maximum operating temperature for γ-TiAl alloys is considered to be approximately 750 °C while the maximum target application temperature for this type of material is up to 900 °C.

In spite of the efforts to improve the oxidation resistance of γ-TiAl alloys by element additions, there seems to be no single alloying element which can improve mechanical properties and oxidation resistance simultaneously. For instance, Nb, Ta or W are known to improve the oxidation resistance of the alloy to some degree, however, a large amount addition results in embrittlement of the alloy and an increase of the alloy density. The alloys thus developed do not have sufficient oxidation resistance for a long cumulative operation at temperatures higher than 800 °C. One important hindrance for the use of γ-TiAl alloys at their upper allowable temperature limit is the insufficient oxidation resistance at temperatures beyond 700 °C and their sensitivity to environmentally induced embrittlement caused by oxygen dissolution. It is well known that the oxidation behavior of TiAl alloys is affected by the atmosphere. Usually, its oxidation resistance is better in pure oxygen than in air (21 % O_2 , 79% N_2), however, this is not true in some cases, because of the contradictory effect of nitrides.

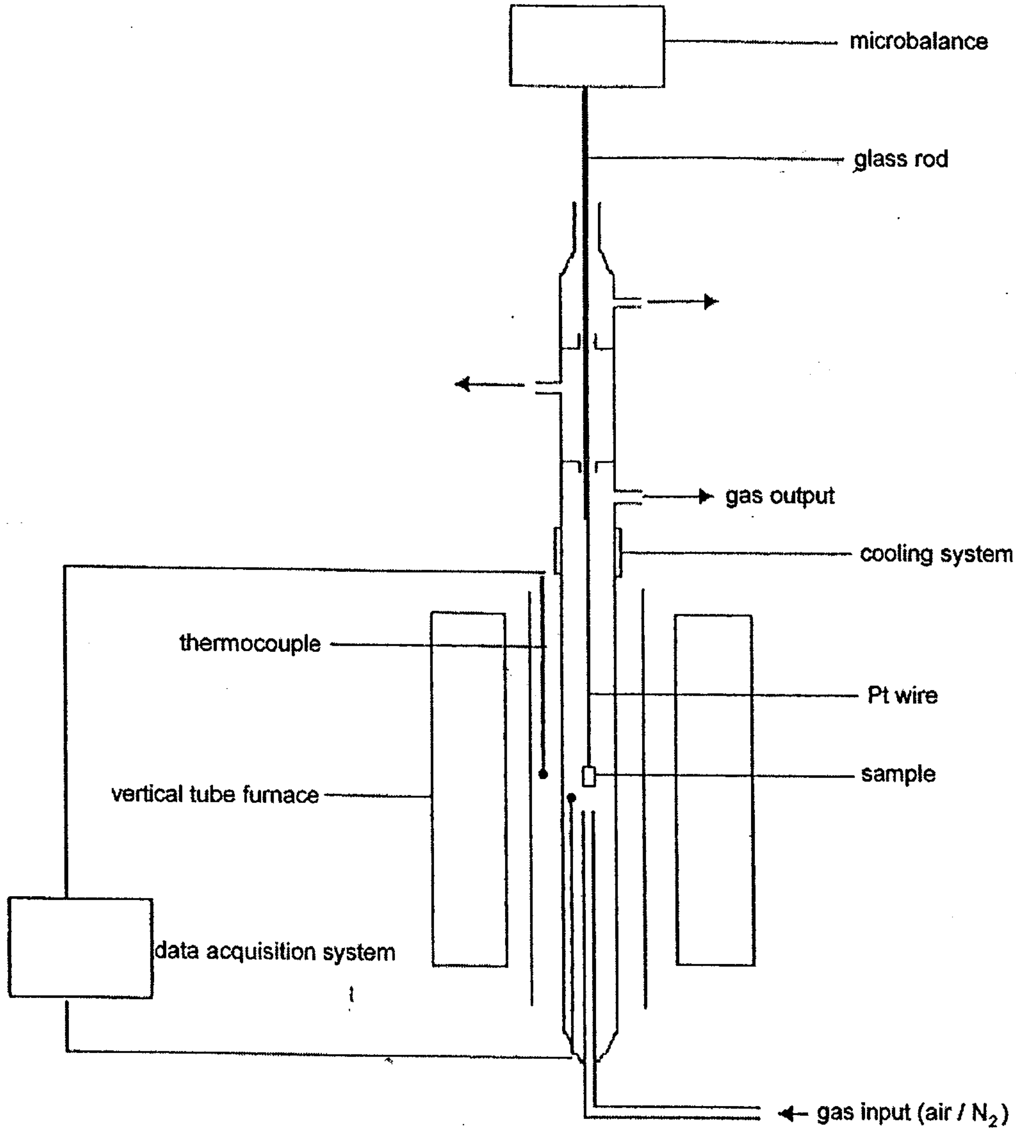

Figure 1 : Schematic diagram of the thermogravimetry arrangement with microbalance

The protection of the alloys at service temperature is provided by the formation of surface oxides that are slow growing, stable and adherent to the substrate. Unfortunately, the scale on gamma alloys consists of a mixture of Al_2O_3 and TiO_2 without forming a continuous and protective Al_2O_3 layer, and shows considerable growth at high temperatures. This problem can principally be overcome by ensuring the successful formation of a continuous aluminum oxide scale on the surface of the substrate. Therefore a number of recent studies have been aimed at the development of alumina-forming TiAl-based alloys. Results have shown that alumina formation γ-TiAl-based alloys can, in principle, be obtained by suitable Cr addition, Another possible solution to achieve the formation of a continuous layer of Al_2O_3 on the surface is a combination alloying addition of Nb, W, Si, Mo, and Ta with niobium being the most effective alloying element. Although the addition of Nb is effective to improve the oxidation resistance, the complex and synergistic effects of the alloying elements still remains unsolved.

This report presents the oxidation and nitridation behavior of an intermetallic alloy Ti-48Al-2Nb-2Cr exposed isothermally in air and in nitrogen gas with emphasis on the determination of each individual activation energy by thermogravimetric analysis (TGA).

2. Experimental Procedures

2.1. Materials and Sample Prepaeation

The raw materials used in this research were powder of titanium (99.7% pure, STREM Chemicals), chromium (99% pure, STREM Chemicals), niobium (99.8% pure, STREM Chemicals), and flakes of aluminum (98.7% pure, BDH Lab.). The materials were initially prepared to the desired composition of Ti-48Al-2Cr-2Nb (all in at.-%) and mechanically mixed for 10 (ten) hours. The alloy samples were obtained by arc-melting from pellets of pressed powder mixture under argon gas environment. The initial melting produced ~20 mm in diameter and ~10 mm thick buttons of about 4 g each, and repeated melting of the button was done five times in order to insure the homogeneous distribution of metals. Diamond-coated blade cutting tool was used to prepare cubic specimens of 10x10x2 mm. The specimens were then ground to a surface finish of 2000 grit using SiC paper, followed by 0.1 μm and 0.05 μm alumina paste, and subsequently cleaned ultrasonically in acetone.

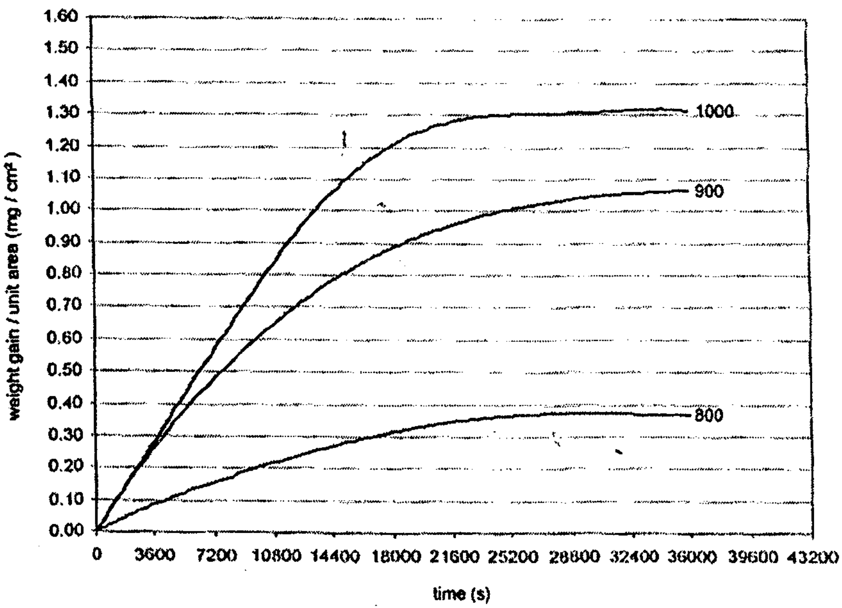

Figure 2 : Weight gain vs. time in flowing laboratory air at different temperatures

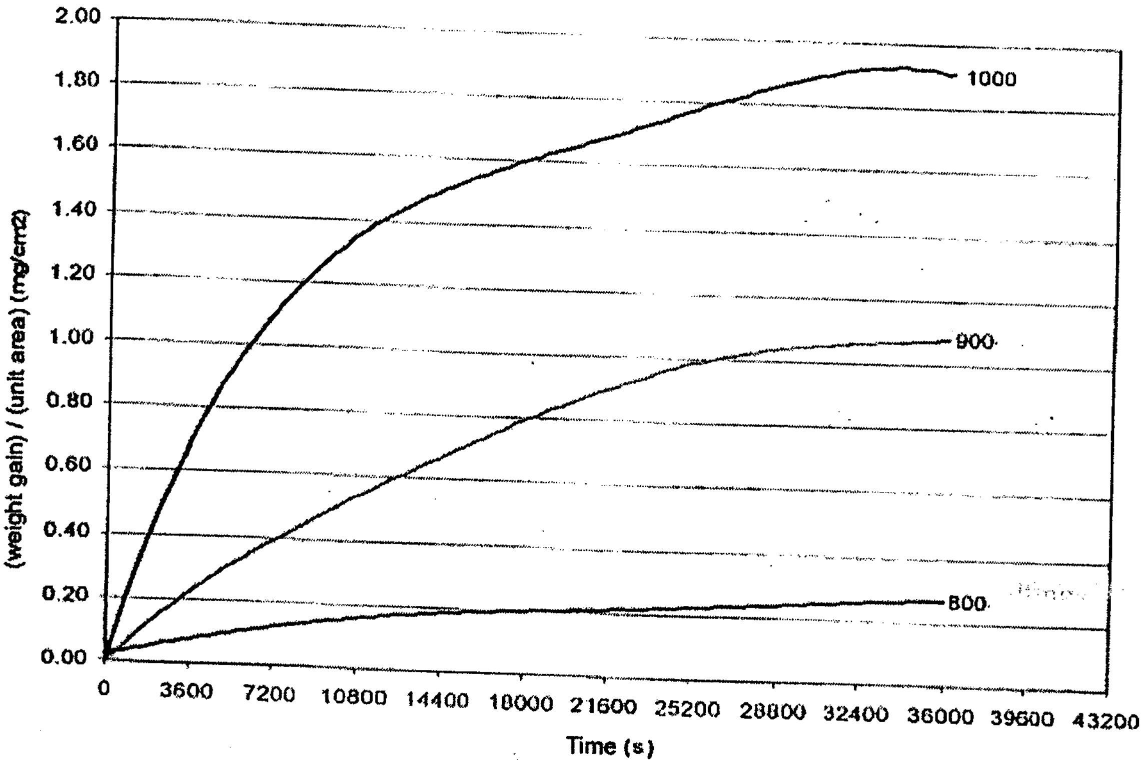

Figure 3 : Weight gain vs time in flowing nitrogen gas at different temperatures

2.2 Oxidation Experiments

Isothermal oxidation tests were performed at 800 . 900 and 1000 °C for 10 (ten) hours employing thermogravimetric method (TGA) for measuring the reaction rates. Tests were carried out in a flow of laboratory air and of nitrogen gas under atmospheric pressure at a rate of 5 (five) SCFH (standard cubic feet per hour). The specimen was attached to the end of the hanging assembly, which was later suspended from the thermobalance. The change in weight during oxidation was collected as a function of time by a data acquisition system. At the end of the test the specimens were furnace cooled, and were etched with Kroll reagent (10 vol-% HF, 4 vol-% HNO_3 , 86 vol-% H_3O) and characterized by X-ray diffraction (XRD), secondary mode scanning electron microscopy (SEM), and energy dispersive X-ray analysis (EDX) .

3, Results and Discussion

A review of earlier investigations on gamma alloys based on TiAl intermetallic compound showed that the parabolic rate law has been, in general, the basis of interpretation of the weight gain *vs.* time data. Hence, the parabolic rate law was considered as the basis of data processing and interpretation of results in this investigation as well. Mass changes per unit surface area against time of Ti-48Al-2Cr-2Nb alloy specimens exposed to flowing laboratory air and to nitrogen gas at 800 , 900 , and 1000 °C are shown in Figure 2 and Figure 3, respectively. From these figures it can

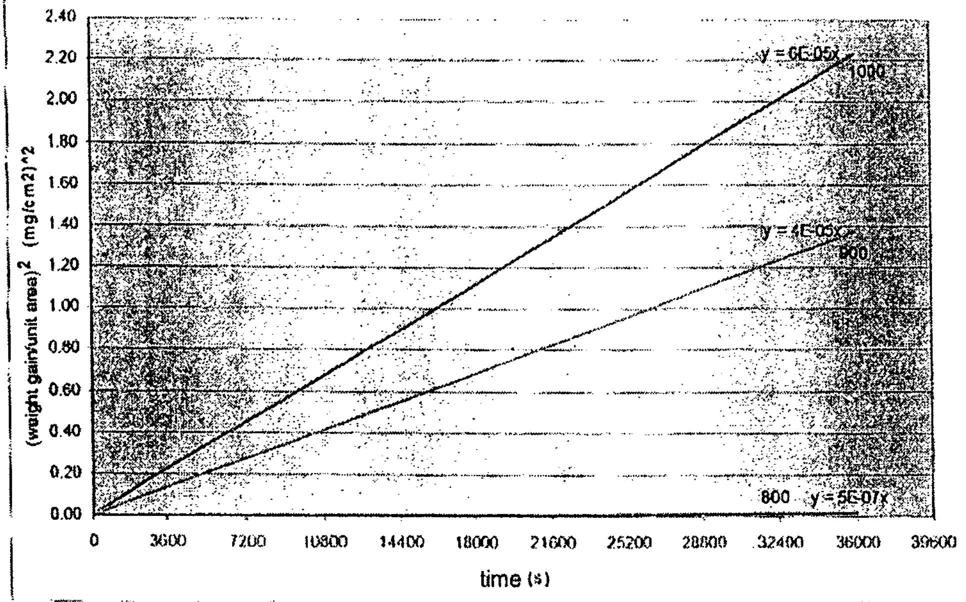

Figure 4 :($\triangle W / A)^2$ vs. time plot for oxidation

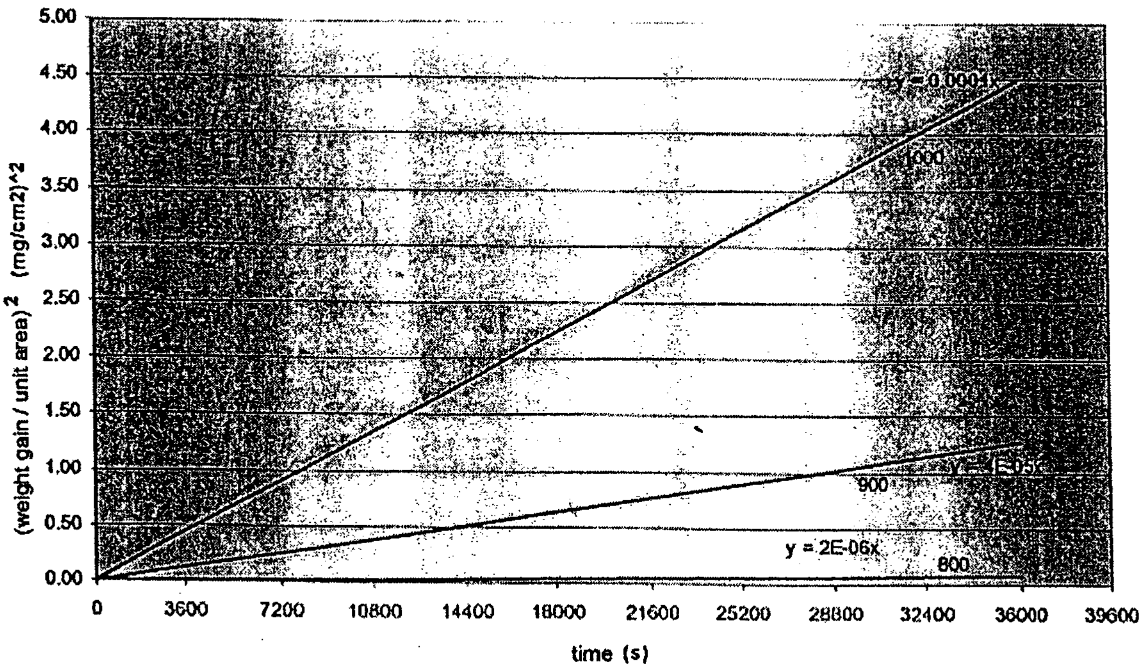

Figure 5 : $(\triangle W / A)^2$ vs time plot for nitridation

be concluded that the mass gain of the specimens in laboratory air and in nitrogen gas during exposure at 800 and 900 °C were similar. However, after exposure at 1000 °C the final mass gain of the specimen under nitrogen gas environment was significantly higher than that in air. Correspondingly, cross-sectional observation revealed that thicker scale was produced on specimen exposed in nitrogen gas (15.3 μm) compared to the one exposed in air (11.4 μm) under identical operational condition of 1000 °C for 10 (ten) hours.

The parabolic rate constant (k_p) is related to the weight gain per unit surface area of a specimen ($\Delta W/A$) and exposure time (t) by :

$$\left(\frac{\Delta W}{A}\right)^2 = k_p t \qquad (1)$$

where k_p is the slope of the linear regression-fitted line of $(\Delta W/A)^2$ *vs.* t data. Figures 4 and 5 show the experimental results for Ti-48Al-2Cr-2Nb specimens exposed in flowing laboratory air and in flowing nitrogen gas, respectively.

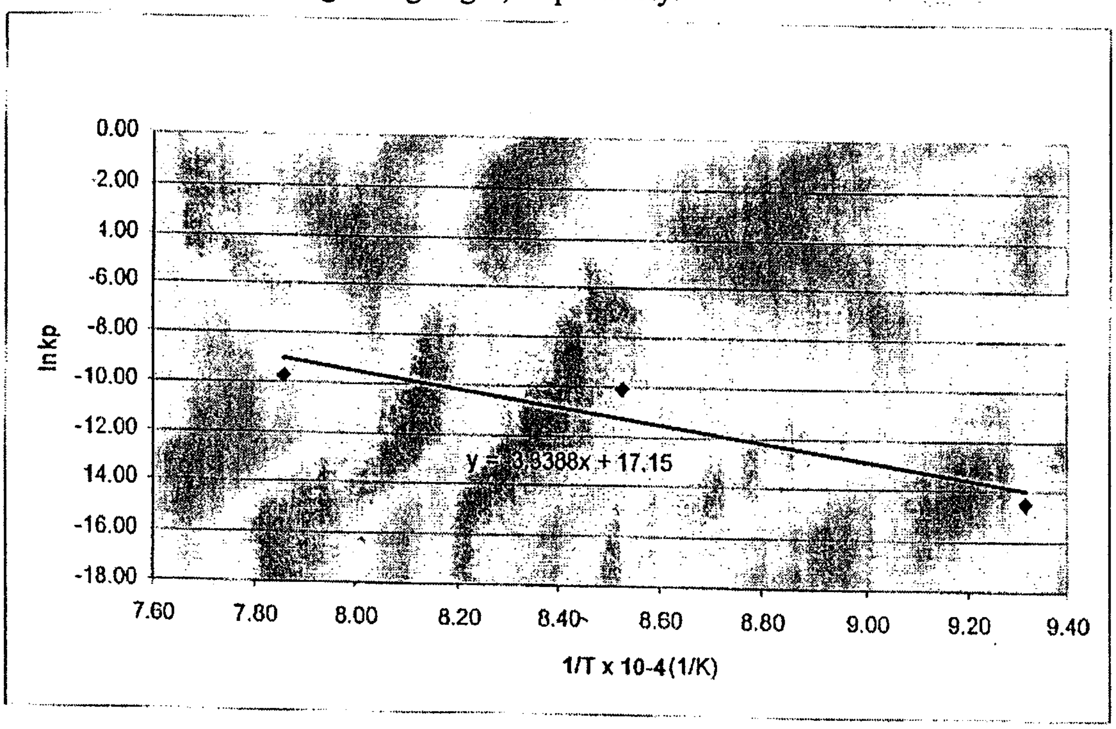

Figure 6 : Arrhenius plot of parabolic rate constants for oxidation

Earlier investigators correlated the overall parabolic rate constant (k_p) and temperature through an Arrhenius-type equation as

$$k_p = k_0 \exp\left(-\frac{Q}{RT}\right) \qquad (2)$$

where k_o is the pre-exponential constant, Q is the activation energy, and T is the absolute temperature. Taking the natural logarithm on both sides of the Arrhenius-type equation and plotting $ln\ k_p$ vs. $(^1/_T)$ would yield a straight line whose slope when multiplied by the universal gas constant R is the activation energy Q. Such plots are shown in Figures 6 and 7 for Ti-48Al-2Cr-2Nb alloy specimens under experimental conditions of flowing laboratory air and of flowing nitrogen gas, respectively. From the slope of the linear regression plots values of Q = 277.4 kJ/mol (oxidation) and Q = 224.8 kJ/mol (nitridation) were obtained.

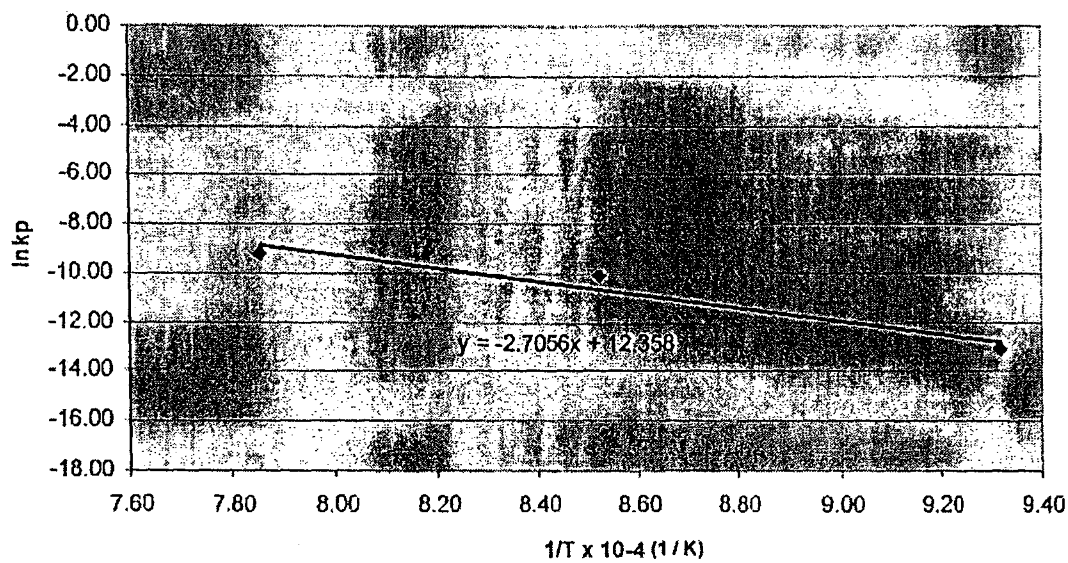

Figure 7 : Arrhenius plot of parabolic rate constants for nitridation

Table I presents a comparison of the activation energies obtained in this work with the results of other researchers. The Q values differ considerably from one research result to the other covering a variety of alloy composition. Our Q-value of 277.4 kJ/mol for oxidation lies within the overall range of Q values of 255-330 kJ/mol for oxidation. It is well documented that the TiN formation interrupts the establishment of a continuous alumina scale, therefore gamma alloys based on TiAl intermetallic compound do not form a protective alumina scale in air. The absence of oxygen in the flowing nitrogen gas environment minimizes the competing reaction between nitridation and oxidation, and results in a much lower activation energy of 224,8 kJ/mol for nitridation.

Table I. Activation Energies (Q) of Oxidation and Nitridation

System	Q (kJ/mol)	Reference
Oxidation of Ti-25Al	255	Welsch & Kahveci, 1988
Oxidation of Ti-25Al	269	Choudhury, et al., 1976
Oxidation of Ti-25Al-11Nb	330	Roy, et al.,1996
Oxidation of Ti-25Al-10Nb-3V-1Mo	248	Liu & Welsch,1988
Oxidation of Ti-25Al - 2Nb-2Cr	277.4	this study
Nitridation of Ti-25Al - 2Nb-2Cr	224.8	this study

One should keep in mind, however, that the experimentally obtained activation energy Q merely represents the best fit parameters to a given set of data measured over some arbitrarily selected temperature range. Consequently, the Q values are best viewed only as useful empirical approximations.

4. Conclusions

The activation energies of the reaction product, appearing as scales on the surface of the Ti-48Al-2Cr-2Nb alloy exposed in air and in nitrogen, have been determined to be 277.5 kJ/mol and 224.8 kJ/mol, respectively. Measured scale thickness on the alloy exposed in air and in nitrogen gas at 1000 °C for 10 (ten) hours were 11.4 μm and 15.3 μm, respectively. Thicker scale developed on the surface of the alloy exposed in nitrogen further substatiate the lesser value of the activation energy under this environment. At higher temperatures the thickness of the resulted scale increases accordingly.

5. References

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Pusat Pengajian Kejuruteraan Bahan dan Sumber Mineral

PENENTUAN TENAGA PENGAKTIFAN DAN MEKANISME PENGOKSIDAAN ALOI TITANIUM ALUMIDA DALAM KEADAAN UDARA TERMAMPAT DAN NITROGEN PADA SUHU 800°C-1000 °C

Oleh

OOI TZE SHIAN
Penyelia: Prof. Madya Dr. Rizal Astrawinata

Disertasi ini diserahkan untuk memenuhi sebahagian syarat

keperluan bagi ijazah Sarjana Muda Kejuruteraan dengan Kepujian

(Kejuruteraan Bahan)

Universiti Sains Malaysia

Mei 2006

DETERMINATION OF ACTIVATION ENERGY AND MECHANISME OXIDATION OF TITANIUM ALUMINIDES ALLOY IN COMPRESSED AIR AND NITROGEN GAS AT TEMPERATURE 800°C-1000 °C

ABSTRACT

This project is to study the oxidation mechanism of titanium aluminides alloy in compressed air atmosphera and in nitrogen gases at high temperatures. Although this material has a high temperature capability, but these alloys often limited by relative poor resistance against hot gas. Activation energy for titanium aluminides alloy in air is 277.5kJ/mol and for nitrogen gases is 224.8kJ/mol in this study. This statement is support by the growth of oxides thickness at nitrogen gases is higher then in compressed air. The formation of oxides may eventually limit the service time. When temperature was increased, rate of oxidation and thickness of oxides layer will also increase. During the oxidation process, weight change is recorded by computer system. Morphology surface and cross section analysis was carried out by using optical microscope, scanning electron microscope (SEM), and energy dispersive spectrometers (EDX).

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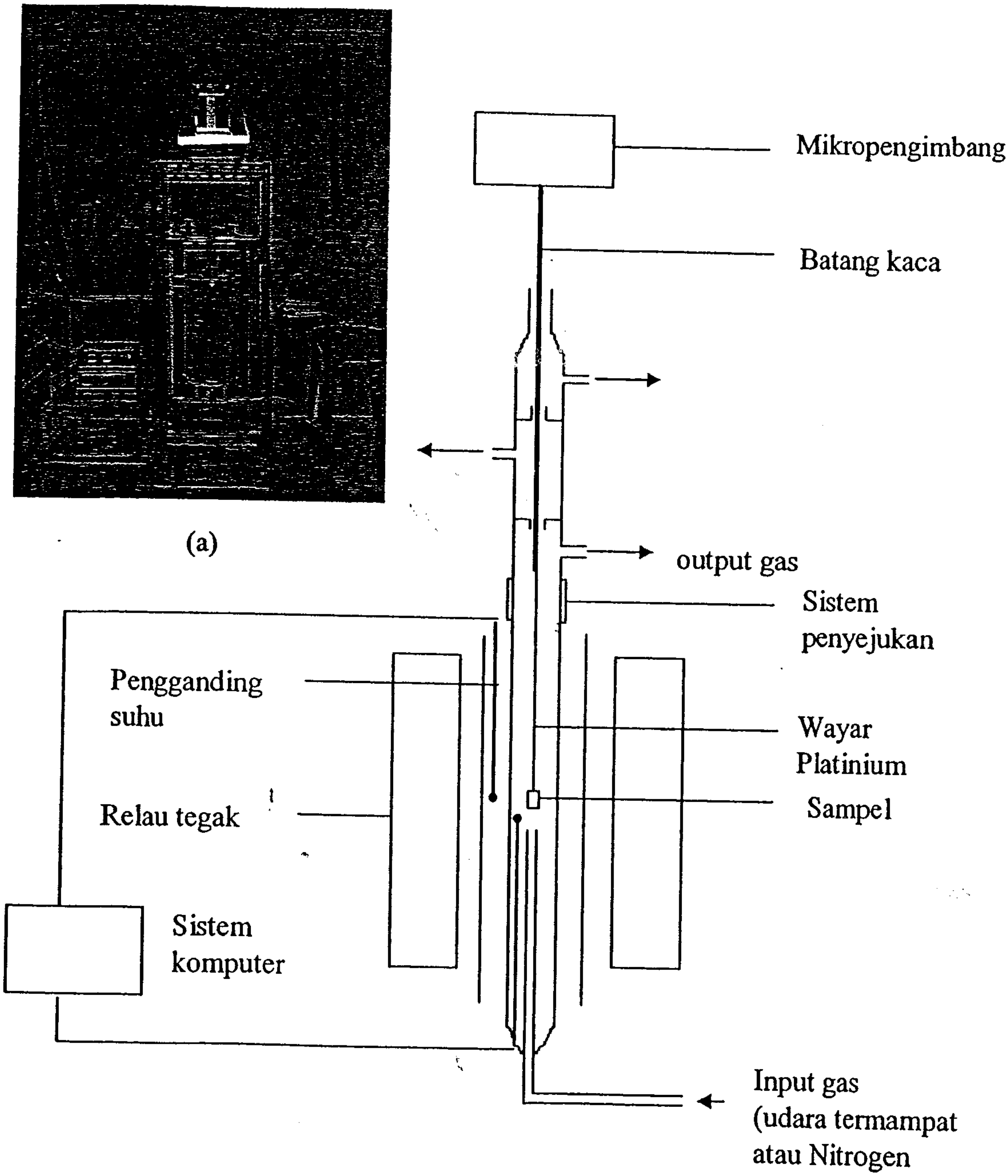

(a)

(b)

Rajah 3.9: (a) menunjukkan peralatan pengoksidaan, (b) skemetik sistem peralatan yang digunakan.