UNIVERSITI TEKNIKAL MALAYSIA MELAKA

EFFECT OF CHROMIUM CARBIDE ($Cr_{23}C_6$) FORMATION ON THE MICROSTRUCTURE AND CORROSION PASSIVITY BEHAVIOR OF AISI 316

This report submitted in accordance with requirement of the Universiti Teknikal Malaysia Melaka (UTeM) for the Bachelor Degree of Manufacturing Engineering (Engineering Materials) with Honours.

by

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APPROVAL

This report is submitted to the Faculty of Manufacturing Engineering of UTeM as a partial fulfillment of the requirements for the degree of Bachelor of Manufacturing Engineering (*Engineering Materials*) with Honours. The member of the supervisory committee is as follow:

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ABSTRACT

AISI 316 is known as austenitic stainless steel which has Face Centre Cubic (FCC) structure. AISI 316 has good corrosion resistance at the ambient temperature. However, the corrosion behavior of AISI 316 is change due to the environment condition. AISI 316 normally used particularly for sour service applications where corrosion resistance is required in chloride environments. While these steels show many superior characteristics, limitations are associated with the welding of these steels, particularly controlling the weld structure and properties and understanding how the weld metallurgy may influence the susceptibility to intergranular attack. The focus of this study is to report on the findings from a detailed study of the various heating time within AISI 316 as a function of heat input and type of sensitization occurred, in terms of the metallurgical structure, composition, and mechanical properties and to assess the susceptibility to intergranular attack. Heating procedure was performed using the manual Oxyacetylene fuel cutter technique for 5 seconds, 10 seconds, 20 seconds, 40 seconds, and 60 seconds as a function indicate that at both long and short heating time. Structural analysis consisted of optical microscopy of heated specimens, chromium carbide ($Cr_{23}C_6$) and ferrite determination by using X-ray diffraction (XRD), including Vickers hardness measurements. Cyclic Potentiodynamic Polarization (CPP) test was used to determine pitting corrosion at susceptible intergranular attack towards AISI 316 structure. The electrolyte solution used was 3.5 % NaCl as alternative of aggressive chloride environments. The test was conducted at ambient temperature. The potential was scanned from -500 mV (SCE) to +200 mV (SCE) and back to -500 mV (SCE) at a rate of 1.67 mV/s and the ratio of the reactivation charge to the passivation charge was calculated. From the results obtained, it can be shown that it was correlated with the local variations in material compositions and microstructure caused by the heat

sensitizing procedure. From the XRD analysis, the $Cr_{23}C_6$ is detected at the XRD graph pattern by following the reference point obtained from International Center for Diffraction Data (ICDD) library. Thus, it indicated the presence of $Cr_{23}C_6$ at the tested samples. The present of $Cr_{23}C_6$ is obtained during microstructure analysis which is found the $Cr_{23}C_6$ structure at the tested samples. CPP analysis indicates that the corrosion passivity behavior of as received material of AISI 316 had noblest pitting nucleation resistance (E_{pit}) with the value of 301 mV and showed the widest immunity zone or protection range which is 192.8 mV. The E_{pit} and immunity zone of AISI 316 is decrease with increasing heating time towards it. The worst corrosion passivity behavior is occurred to specimen heated for 60 s. E_{pit} of this specimen was shifted to active potential by getting -84 mV and present lowest immunity zone with it protection range only 173 mV. Since 60 seconds show the best view of the three heated structure zone, the results show significant value of hardness results. Fully heated zone get 223 $kgfmm^{-2}$ higher value of hardness compare to HAZ with 193 $kgfmm^{-2}$ and base metal 175 $kgfmm^{-2}$. As a conclusion of this study, when heating time towards AISI 316 is increase, the grain boundaries of AISI 316 became broader and the $Cr_{23}C_6$ formation in the grain is also increase makes it hardness properties is increase. However, at the region rich with $Cr_{23}C_6$ is easily attack by environment containing chloride ion.

ABSTRAK

AISI 316 dikenali sebagai keluli tahan karat jenis austenitic yang mempunyai struktur kiub pusat muka (FCC). AISI 316 mempunyai rintangan kakisan yang baik di suhu bilik. Bagaimanapun, tingkah laku kakisan AISI 316 berubah disebabkan keadaan persekitaran. AISI 316 biasanya digunakan terutama untuk aplikasi berasid di mana rintangan kepada kakisan adalah diperlukan dalam persekitaran klorida. Walaupun ia mempunyai banyak ciri-ciri yang sangat bagus, terdapat had-had dikaitkan dengan struktur kimpalan keluli ini, sesetengahnya ialah kawalan struktur dan ciri – ciri kimpalan serta pemahaman bagaimana metalurgi kimpalan boleh mempengaruhi kerintanan untuk serangan kakisan antara butir. Tumpuan kajian ini ialah untuk melaporkan tentang penemuan-penemuan daripada satu kajian terperinci mengenai kepelbagaian waktu pemanasan terhadap AISI 316 sebagai satu fungsi terhadap input kepanasan dan jenis kakisan antara butir yang berlaku dalam soal struktur pelogaman, komposisi, dan sifat-sifat mekanikal dan bagi menilai kerentanan untuk serangan antara butiran. Cara – cara pemanasan telah dilakukan menggunakan teknik pemotong oxyacetylene untuk tempoh 5 saat, 10 saat, 20 saat, 40 saat, dan 60 saat sebagai satu fungsi untuk menunjukkan waktu pemanasan lama dan pendek. Analisis struktur merangkumi aspek mengenalpasti kehadiran Chromium Carbide ($Cr_{23}C_6$) dan juga unsur ferit dengan menggunakan X - Ray Diffraction (XRD). Dan meneropong struktur specimen – specimen yang telah dibakar mengunakan Carl Zeiss Optical Microscope. Ujian Cyclic Potentiodynamic Polarization (CPP) telah digunakan untuk menentukan kakisan bopeng di bahagian yang berlaku serangan antara butiran pada struktur AISI 316 manakala ukuran-ukuran kekerasan menggunakan ujian Vickers microhardness Larutan elektrolit yang dipakai ialah 3.5 % NaCl sebagai gantian kepada persekitaran klorida. Ujian dijalankan pada suhu bilik. Potensi telah diset pada ujian CPP adalah daripada -500 mV (SCE) kepada +200 mV (SCE) dan kembali semula

untuk -500 mV (SCE) pada kadar 1.67 mV / s dan nisbah cas pengaktifan semula dan cas pempasifannya telah dihitung. Daripada keputusan-keputusan diperolehi, ia menunjukkan yang ia boleh dihubung kait dengan perbezaan - perbezaan setempat dalam komposisi bahan dan struktur mikro disebabkan oleh cara – cara pemanasan. Daripada analisis XRD, $Cr_{23}C_6$ telah di kesan pada graf XRD dengan merujuk kepada tanda rujukan yang diperolehi dari perpustakaan International Center for Diffraction Data (ICDD). Oleh itu, ia menunjukkan kehadiran $Cr_{23}C_6$ pada sampel yang di uji. Kehadiran $Cr_{23}C_6$ ketika analisis struktur mikro mendapati struktur $Cr_{23}C_6$ pada sampel yang di uji. Analisis CPP menunjukkan yang keadaan kakisan pasif bahan AISI 316 rintangan penukleusan (E_{pit}) yang sangat tinggi dengan nilai 301 mV dan menunjukkan zon keimunan atau julat perlindungan yang yang sangat luas iaitu 192.8 mV. E_{pit} dan zon keimunan AISI 316 berkurang dengan bertambahnya waktu pemanasan terhadapnya. Tingkah laku kepasifan kakisan paling teruk terjadi untuk spesimen yang dipanaskan untuk 60 saat. E_{pit} spesimen ini berpindah ke potensi aktif dengan mendapat -84 mV dan hadir zon keimunan terendah dengan julat perlindunganya hanya 173 mV. Dengan 60 saat menunjukkan keputusan struktur terbaik, tiga zon iaitu struktur yang dipanaskan menunjukkan keputusan nilai kekerasan. Bahan yang terbakar sepanuhnya mempunyai kekerasan sebanyak 223 $kgfmm^{-2}$ .Nilai ini lebih tinggi berbanding kekerasan bahan separa terbakar dengan 193 $kgfmm^{-2}$ dan logam yang tidak terbakar menunjukkan nilai kekerasan 175 $kgfmm^{-2}$. Kesimpulan bagi kajian ini ialah apabila waktu pemanasan terhadap AISI 316 meningkat, sempadan - sempadan bijian AISI 316 menjadi lebih luas dan pembentukan $Cr_{23}C_6$ dalam bijian juga meningkat membuatkan ciri-ciri kekerasannya meningkat. Bagaimanapun, di rantau yang kaya dengan $Cr_{23}C_6$ akan diserang dengan mudah oleh persekitaran yang mengandungi ion klorida.

DEDICATION

To my beloved family, friends and UTeM

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LIST OF ABBREVIATIONS

AC	-	Alternative Current
AISI	-	American Iron and Steel Institute
Aq	-	Aqueous
ASTM	-	American Standard Test Method
CPP	-	Cyclic Potentiodynamic Polarization
DC	-	Direct Current
EBDS	-	Electron Backscatter Diffraction
EDS	-	Energy Dispersive X-ray Spectroscopy
EW	-	Equivalent Weight
FCC	-	Face Centre Cubic
ICDD	-	International Center for Diffraction Data
MCA	-	Multi Crevice Assembly
PRE	-	Pitting Resistance Equivalent
PREN	-	Pitting Resistance Equivalent by considering Nitrogen
SEM	-	Scanning Electron Microscopy
S/N	-	Sample Number
SSE	-	Standard Silver Electrode
XRD	-	X-ray Diffraction

LIST OF SYMBOLS

A	-	Expose Surface Area
e	-	Electron
E_{corr}	-	Corrosion Potential
E_{rp}	-	Repassivation potential
E_{pit}	-	Pitting potential
E_{oc}	-	Open Circuit Potential
F	-	Faraday's constant = 96485.34 C/mol
h	-	hours
i_{app}	-	Applied Current Density
i_{corr}	-	Corrosion Current Density
I_{oc}	-	Open Circuit Current
i_{oc}	-	Open Circuit Current Density
M	-	Atomic Weight
M_i	-	Atomic Weight of the i^{th} Element of the Alloy
n	-	Number of Electron Element
n_i	-	Number of Electron of Component i^{th} Clement of the Alloy
s	-	Seconds
t	-	Time of Reaction Process
wt %	-	Weight Percentage
β_a	-	Anodic Tafel Slope
β_c	-	Cathodic Tafel Slope
δ	-	Delta
σ	-	Sigma
P	-	Applied Load
d_v	-	Average Length of the Two Vickers Diagonals

CHAPTER 1

INTRODUCTION

1.1 Background

Corrosion is defined as the destruction or deterioration of a material because of reaction with its environment. The serious consequences of the corrosion process have become a problem of worldwide significance, where due this problem, more cost had been use in research and development of corrosion engineering as to control corrosion on the material. Material such as stainless steel is suit with construction, mechanical and chemical industries because its alloy elements of stainless steel promote good corrosion resistance. The example stainless steel that had been applied in corrosive construction structure is stainless steel grade AISI 316 which is known as austenite stainless steel. According to the literature, the austenitic grades performed much better than martensitic and ferritic grades (Al-Fozan *et. al.*, 2008). Special properties, including corrosion resistance, oxidation resistance, and strength at high temperatures can be incorporated into austenitic stainless steels by adding certain alloy like molybdenum. Corrosion testing is important aspects of corrosion control because it is used to determine and to evaluate the most effective corrosion prevention. The studies are mainly directed toward mechanistic aspects which subsequently provide information useful for the development of the role of Chromium Carbide ($Cr_{23}C_6$) formation on the microstructure and corrosion passivity behavior of AISI 316. Electrochemical techniques have been the principal tool for characterizing corrosion rate. The resulting microstructure changes and intermetallic phases in surface and subsurface were studied with optical microscope, Vickers micro hardness, and XRD analysis (Kumar *et. al.*, 2009).

1.2 Problem Statement

There are many research proposed that the conventional austenitic stainless steels are commonly used to continuous and intermittent high temperature services because they are creep and corrosion resistant materials (Frangini *et. al*., 2006). Several theories had been proposed that the use of sensitization of these steels promotes intense chromium carbide precipitation in the grain boundaries (Kina *et. al.,* 2008).

Intergranular chromium carbides increase the creep resistance, but decrease ductility, low temperature toughness and corrosion resistance. Some authors have shown that the sensitized material can undergo a healing or desensitization process with increased aging time, owing to bulk chromium (Cr) diffusion which eliminates the Cr depleted condition. It has been found that some plastic deformation prior to sensitization enhanced the kinetics of desensitization, probably because it increases the diffusivity of chromium into the austenitic matrix (Cristobal A. B., et. al., 2006). According to Domankova *et. al.,* (2007), the degree of sensitization (DOS) is influenced by factors such as the steel's chemical composition, the grain size, the degree of strain or temperature and the time of isothermal annealing. The sensitisation involves both the nucleation and growth of carbides at the grain boundaries. Depending on the state or the energy of the grain boundaries they can provide preferential sites for carbide nucleation and act as a favoured diffusion path for the growth of carbides. However, stainless steels are not immune to corrosion attack. Localized corrosion such as pitting corrosion, intergranular corrosion or stress corrosion cracking can be developed mainly in chloride containing environments (Cristobal *et. al*., 2006). Recently, Bilmes *et. al*., (2006) reported potentiodynamic anodic polarization measurements carried out on weld metal stainless steel in chloride containing solutions in order to assess the influence of applied post weld heat treatments (PWHT) on pitting corrosion resistance.

Therefore, the present work aims to investigate and further understanding of the possible action of the corrosion resistances behavior of the AISI 316 and related to the microstructure features. In this work the objective is to gain a further insight into this question as well as into the thermal effects of welding on parent metal by comparing results obtained from weld metals and AISI 316 exposed with oxy fuel cutter. Safe welding procedures should result in welds with corrosion properties meeting comparable requirements equal to the parent metal.

1.3 Objective

(i) To analyze the microstructure of AISI 316 after heat exposure for 5 seconds, 10 seconds, 20 seconds, 40 seconds, and 60 seconds.

(ii) To analyze the effect of $Cr_{23}C_6$ formation to the corrosion behaviour of AISI 316.

(iii) To investigate the hardness of the sensitized, heat affected zone, and non sensitized region of AISI 316.

1.4 Scope

The scope of this study is to investigate the role of $Cr_{23}C_6$ formation which influences the resistance to corrosion activity in AISI 316. The aspects that should be focus on this study are microstructure analysis of AISI 316 due to the exposure of 3000 °C to 3200 °C blue flames from oxyacetylene fuel cutter for 5 seconds, 10 seconds, 20 seconds, 40 seconds, and 60 seconds. After specimens were heated, the presence of $Cr_{23}C_6$ formation at the grain boundary of the specimens is identified by using X-ray diffraction (XRD) respectively. All changes of the microstructure are observed by using Carl Zeiss AxioCam optical microscope and the type of structure present at the tested material is identified. The corrosion properties of the material are studied by using electrochemical technique where Cyclic Potentiodynamic Polarization scan is applied. The purpose of electrochemical technique is to investigate corrosion behaviour of as received material and the specimens heated for various time as stated above. The result of the corrosion behaviour of AISI 316 is then related to the microstructure and XRD analysis. The minor study is to check the hardness by using Vickers Microhardness testing.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

This chapter discusses the review of study on the AISI 316 material properties, mechanisms for the action of $Cr_{23}C_6$ formation and its finding on AISI 316 microstructure and corrosion passivity behavior.

2.2 Stainless Steel

Steels are said to be stainless when they resist corrosion. This is achieved by dissolving sufficient chromium (Cr) in the iron (Fe) to produce a coherent, adherent, insulating and regenerating chromium oxide protective film on the surface. It is not surprising therefore that they are used in the harsh environments of the chemical, oil production and power generation industries, and in utility goods such as furniture, automotive trims and cutlery, where both aesthetic appearance and corrosion resistance are important design criteria. The stainless character occurs when the concentration of Cr exceeds about 12 wt %. However, even this is not adequate to resist corrosion in acids such as hydrochloric acid (HCl) or sulphuric acid (H_2SO_4). Higher Cr concentrations and the sensible use of other solutes such as molybdenum (Mo), nickel (Ni), and nitrogen (N) is then needed to ensure a robust material (Sourmail *et. al*., 2003). Three main types of microstructures exist in stainless steels, for example ferritic, austenitic and martensitic. These microstructures may be obtained by properly adjusting steel chemistry. Out of these three main