

UNIVERSITI TEKNIKAL MALAYSIA MELAKA

STUDY THE EFFECT OF HIGH TEMPERATURE CYCLE EXPOSURE TO EVALUATION ON EMBRITTLEMENT OF WELDED PRESSURE VESSEL STEEL

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

By

NURULHUDA BT ARIFIN

Faculty of Manufacturing Engineering April 2009



UNIVERSITI TEKNIKAL MALAYSIA MELAKA

BORANG PENGESAHAN STATUS TESIS*

TAJUK: STUDY THE EFFECT OF HIGH TEMPERATURE CYCLE EXPOSURE TO EVALUATION ON EMBRITTLEMENT OF WELDED PRESSURE VESSEL STEEL

SESI PENGAJIAN: 2008/2009 Semester 2

Saya NURULHUDA BT ARIFIN

mengaku membenarkan tesis (PSM/Sarjana/Doktor Falsafah) ini disimpan di Perpustakaan Universiti Teknikal Malaysia Melaka (UTeM) dengan syarat-syarat kegunaan seperti berikut:

- 1. Tesis adalah hak milik Universiti Teknikal Malaysia Melaka.
- 2. Perpustakaan Universiti Teknikal Malaysia Melaka dibenarkan membuat salinan untuk tujuan pengajian sahaja.
- 3. Perpustakaan dibenarkan membuat salinan tesis ini sebagai bahan pertukaran antara institusi pengaijan tinggi

SULIT

(Mengandungi maklumat yang berdarjah keselamatan atau kepentingan Malaysia yang termaktub di dalam AKTA RAHSIA RASMI 1972)

TERHAD

Alamat Tetap:

31910, Kampar,

Perak

Tarikh:

(Mengandungi maklumat TERHAD yang telah ditentukan oleh organisasi/badan di mana penyelidikan dijalankan)

 \checkmark

TIDAK TERHAD

No 307, Kampung Bukit Pekan,

(TANDATANGAN PENULIS)

Disahkan oleh:

(TANDATANGAN PENYELIA)

Cop Rasmi: MOHAMAD HAIDIR BIN MASLAN Pensyarah Fakulti Kejuruteraan Pembuatan Universiti Teknikal Malaysia Melaka

14 April 2009

Tarikh: 27/5/09

* Tesis dimaksudkan sebagai tesis bagi Ijazah Doktor Falsafah dan Sarjana secara penyelidikan, atau disertasi bagi pengajian secara kerja kursus dan penyelidikan, atau Laporan Projek Sarjana Muda (PSM).
** Jika tesis ini SULIT atau TERHAD, sila lampirkan surat daripada pihak berkuasa/organisasi berkenaan dengan menyatakan sekali sebab dan tempoh tesis ini perlu dikelaskan sebagai SULIT atau TERHAD.

APPROVAL

This thesis submitted to the senate of UTeM and has been accepted as partial fulfillment of the requirements for the degree of Bachelor of Manufacturing Engineering (Manufacturing Process). The members of the supervisory committee are as follow:

a - 02

Main Supervisor (Official Stamp and Date) MOHAMAD HAIDIR BIN MASLAN Pensyarah Fakulti Kejuruteraan Pembuatan Universiti Teknikal Malaysia Melaka

DECLARATION

I hereby, declared this thesis entitled "Study the Effect of high Temperature Cycle Exposure to Evaluation on Embrittlement of Pressure Vessel Steel" is the results of my own research except as cited in references

Signature Author's Name Date

:

:

:

higher

ABSTRACT

The research of the effect of high temperature cycle exposure to evaluation on embrittlement of welded pressure vessel steel has been developed purposely in order to study the effect of embrittlement behavior of pressure vessel steel after expose to elevated temperature. The temper embrittlement has been usually observed in pressure vessel steel that serving at high temperature, due to the segregation of trace elements at the grain boundaries and/or carbide interface. This impurity elements segregation will cause deterioration hardness of the steel based on the theory. These pressure vessel steel have a potential for temper embrittlement. Therefore, the research and observation in the mechanical properties of tempered welded area are very important in order to support on-going steel development for services temperature and also useful to avoid the embrittlement during the services. The pressure vessel steel (ASTM A516) have been chosen to be used as the type of specimens in this research. The welded area of pressure vessel steel is on service temperature of pressure vessel ($450^{\circ}C$) at different exposure time. The welded area for all specimens after exposure was characterized by microstructure observation, EDS, and hardness measurement. Based on these results, evaluation on embrittlement is discussed in order to determine the approximate effect of high cycle temperature to embrittlment behavior. Experimental results revealed that temper embrittlement hardly affect the hardness value for welded area and the microstructure characteristics of pressure vessel steel are significantly changed.

ABSTRAK

Kajian tentang kesan pendedahan suhu tinggi kepada penaksiran sifat kerapuhan bagi kimpalan pengandang yang diperbuat daripada besi telah di jalankan bagi tujuan untuk mengkaji kesan terhadap sifat kerapuhan pengandang selepas terdedah kepada penigkatan suhu. Pada kebiasaannya, sifat kerapuhan ini selalunya diperhatikan hadir dalam pengandang besi yang beroperasi dalam suhu yang tinggi,; dimana fonomena ini berlaku di sebabkan oleh pengasingan unsur-unsur pada butiran sempadan. Berdasarkan kepada teori, pengasingan unsur-unsur kotoran atau cemaran ini akan mengakibatkan kemusnahan serta kerapuhan besi. Oleh yang demikian, kajian dan pemerhatian keatas ciri-ciri mekanikal kawasan kimpalan adalah sangat penting bagi memastikan pengandang mampu beroperasi pada suhu operasi dan untuk mengelakkan daripada berlakunya kerapuhan ketika pengandang sedang beroperasi. Jenis besi pengandang yang telah di gunakan sebagai spesimen dalam kajian ini adalah ASTM A516. Bagi melengkapkan kajian ini, kaedah yang dijalankan adalah dengan melakukan pendedahan spesimen kepada peningkatan suhu sekitar 450°C terhadap setiap spesimen yang mempunyai kawasan kimpalan. Sifat-sifat mekanikal bagi kawasan kimpalan (kesemua spesimen) selepas melalui proses peningkatan kekerasan dianalisis melalui pemerhatian mikrostrukturnya, ujian pengesahan unsure-unsur serta ujian takat kekerasan yang dilakukan pada suhu bilik. Berdasarkan keputusan ujian-ujian yang telah dijalankn, hasil analysis di bincangkan bagi tujuan mengenal pasti kesan pendedahan suhu yang tinggi terhadap sifat kerapuhan pengandang. Keputusan kajian jelas menunjukkan bahawa pendedahan pengandang kepada suhu yang tinggi sangat mempengaruhi keputusan ujian kekerasan bagi setiap zone kimpalan dan sifat-sifat mikrostruktur besi pengandang tersebut turut berubah dengan nyata sekali.

DEDICATION

This thesis is gratefully dedicated to my parents, Mr. Arifin Abdullah and Sara Ahmad, my beloved family, my supervisor lecturer Mohamad Haidir B. Maslan, and my supported friends towards accomplishing this research.

ACKNOWLEDGEMENTS

Firstly, I want to gratitude to god, most gracious and merciful to show me guidance in accomplishing this research. Without that, I could not finish this project. I would also like to state my appreciation also to those individuals who involved whether directly or indirectly in assisting and aiding me throughout completing this project. Million of thanks to my project supervisor Mr. Mohamad Haidir Bin Maslan and my examiner Mr. Edeerozey Abd. Manaf for their time spending, encouragement, teaching, guidance and ideas for my research. Their contributions will not been forgotten. In this opportunity also, I wish to express my appreciation to the technicians involved in helping to complete this project. The most important is my appreciation to my parents and family. Lastly to all my friends who give their morale support and sharing this challenging time and beautiful life together.

TABLE OF CONTENTS

Abstract	i
Abstrak	ii
Dedication	iii
Acknowledgement	iv
Table of Contents	V
Appendices	viii
List of Figures	ix
List of Tables	xi
List of Abbreviations, Symbols, Specialized Nomenclature	xii
1. INTRODUCTION	1
1.1 Research Background	1
1.2 Problem Statement	2
1.3 Objectives of Project	3
1.4 Scope of Project	3
1.5 Hypothesis	3
2. LITERATURE REVIEW	4
2.1 Metallurgy	4
2.1.1 Body-Centered Cubic Crystal	4
2.1.2 Faced-Centered Cubic Crystal	5
2.2 The Iron-Carbon Diagram	6
2.3 Phase In Steel	7
2.3.1 Austenite	7
2.3.2 Martensite	7
2.3.3 Ferrite	8
2.3.4 Cementite (Fe $_{3}$ C)	8
2.3.5 Pearlite	9

2.3.6 Bainite	9
2.4 Welded	9
2.4.1 Submerged Arc Welding (SAW)	9
2.4.2 Post Weld Heat Treatment (PWHT)	11
2.5 Welding Metallurgy	12
2.5.1 Weld Ability	12
2.5.2 Heat Affected Zone	13
2.5.3 Weld Metal	16
2.5.4 Base Metal	17
2.6 Material Factors	18
2.7 Embrittlement	19
2.7.1 Intergranular Embrittlement	19
2.7.2 Temper Embrittlement	20
2.7.2.1 Temper Martensite Embrittlement	22
2.7.2.2 Reversible Temper Brittleness	23
2.7.2.3 Factor of Temper Embrittlement	25
2.8 Carbide and Nitride Precipitation	26
2.9 Intermetallic Phase	26
2.10 Principle Effect Element on Steel	27
2.10.1 Chromium	27
2.10.2 Molybdenum	27
2.11 Previous Research on Temperature Embrittlement in Pressure Vessel	28
2.11.1 Microstructure and Properties of Post Weld Heat Treated	28
2.25Cr-1Mo weld Metal	
2.11.2 Critical Assessment of the Degree of Temper Embrittlement	29
in 2.25Cr-1Mo Steel	
2.11.3 Cracking of 2.25Cr-1Mo steel Tube/ Stationary Tube-Sheet	31
Weldment of Heat-Exchanger	

3. RESEARCH METHODOLOGY 3.			
3.1 Introduction			
3.2 Research Design	34		
3.3 Material	35		
3.3.1 ASTM A516	35		
3.4 Sample Preparation	36		
3.4.1 Welding Process (SMAW)	36		
3.4.2 Cutting Process	37		
3.4.3 Milling Process	38		
3.5 Exposure	39		
3.5.1 Tempering	39		
3.6 Metallurgy Technique	40		
3.6.1 Grinding and Polishing Process			
3.6.2 Etching Process	41		
3.7 Microstructure Observation	42		
3.8 Testing			
3.8.1 Hardness Test			
3.8.2 Energy Dispersive X-ray Spectroscopy (EDS) Test			
3.8.3 Tensile Test			
4. RESULT & DISCUSSION	46		
4.1 Introduction	46		
4.2 GDS Data Analysis	46		
4.3 Material Factor of Pressure Vessel Steel (ASTM A516)	47		
4.4 Tensile Properties	48		
4.5 Physical Observation after Tempering	49		
4.6 Microstructure Analysis	50		
4.6.1 Microstructure of Base Metal	51		
4.6.2 Microstructure of HAZ Area	52		
4.7 EDS Analysis			
4.8 Hardness Result			

4.8.1 Hardness Comparison	61
4.9 Yield Strength Analysis	65
5. CONCLUSION & RECOMMENDATION	67
5.1 Conclusions	67
5.2 Recommendation	68

REFERENCES

69

APPENDICES

- A Gantt Charts of PSM 1 and PSM 2
- B Grinding Process Procedures
- C Polishing Process Procedures
- D Etching Process Procedures
- E Microstructure Observation Procedures
- F Tensile Test Procedures
- G Hardness Test Procedures
- H EDS Composition Results
- I Tensile Test Results
- J Material Safety Data Sheet (MSDS)

LIST OF FIGURES

2.1	The structure of a body-centered cubic metal	4		
2.2	The structure of a faced-centered cubic unit cell5			
2.3	The Iron-Carbon phase diagram	6		
2.4	The iron- carbon diagram	8		
2.5	Submerged Arc Welding	10		
2.6	J impact transition temperature of welded and tempered weld metals	11		
2.7	Hardness profile through an autogenously weld	13		
2.8	Schematic of a fusion weld in steel, presenting proper terminology for the various region and interfaces.	14		
2.9	Schematic showing the temperature gradient during welding along HAZ	15		
2.10	Schematic representation of microstructures local to the HAZ of welds	16		
2.11	Grain structure and various zones in a fusion weld.	17		
2.12	Characteristic typical weld zone.	18		
2.13	Schematic effect of the temperature on impact toughness of alloy steel	20		
2.14	Room-temperature Charpy V-notch impact energy versus	23		
	Tempering temperature			
2.15	The schematic phenomenon of temper embrittlement	24		
2.16	AES spectra transcrystalline and intercrystalline fracture of	26		
	2.25Cr-1Mo steel.			

3.1	The process flow of research design	34
3.2	Raw Material	36
3.3	View of HAZ Area	37
3.4	Flow of cutting Process	38
3.5	Milling Process	39

3.6	Placing Sample into Rear Furnace			
3.7	Tempering Profile	40		
3.8	Polishing Process	41		
3.9	Etching Process	41		
3.10	Axial Zeiss Optical Microscope	42		
3.11	Rockwell Hardness Machine	43		
3.12	Indents Part during Hardness Measurement for Welded area; Heat	43		
	Affected Zone, and Base Metal			
3.13	Scanning Electron Microscope (SEM) Machine	44		
3.14	Standard Rectangular Tensile Test Specimens	45		
	Source: American Standard Testing Material (2003)			
3.15	Universal Testing Machine (UTM)	45		
4.1	Stress versus Strain Result for Raw Material (ASTM A516)	48		

4.2	Sample of pressure vessel steel after tempering process; 1 Hours (A);	50
	10 Hours (B); and 100 Hours (C).	
4.3	Ferrite and Pearlite Microstructure	51
4.4	Micrograph of Base Metal area; before tempering (A); 1 hour tempering	52
	(B); 10 hours tempering (C); and 100 hours tempering (D)	
4.5	Micrograph of HAZ area; before tempering (A); 1 hour tempering (B);	53
	10 hours tempering (C); and 100 hours tempering (D).	
4.6	Micrograph of base metal area using EDS analysis; 1 hour tempering (A);	54
	10 hours tempering (B); and 100 hours tempering (C).	
4.7	Micrograph of HAZ area using EDS analysis; 1 hour tempering (A);	55
	10 hours tempering (B); and 100 hours tempering (C).	
4.8	Graph of percentage weight of carbon pick-up for base metal area after	56
	tempering	
4.9	Graph of EDS analysis after tempering for base metal; 1 hour tempering	57
	(A); 10 hours tempering (B); and 100 hours tempering (C).	

4.10	Graph of percentage weight of carbon pick-up for HAZ area after	58
	Tempering	
4.11	Graph of EDS analysis after tempering for HAZ area; 1 hour tempering	59
	(A); 10 hours tempering (B); and 100 hours tempering (C).	
4.12	Theory of calculated diffusion distance in ferrite for 1 hour of	60
	tempering time	
4.13	Graph showing the hardness distribution for base metal, HAZ and	61
	welded area; before tempering (A)	
4.14	Graph showing the hardness distribution for base metal, HAZ and	62
	welded area; 1 hours tempering (B); 10 hour tempering (C), and	
	100 hour tempering (D).	
4.15	Graph showing the decreasing of hardness after tempering in base	64
	metal area	
110		<i>с</i> 1

4.16 Graph showing the decreasing of hardness after tempering in HAZ area. 64

LIST OF TABLES

3.1	Physical Data of ASTM A516	
3.2	Chemical Content	36
4.1	GDS Result	47
4.2	Tensile Properties Comparison	49
4.3	Weight (%) of each element in base metal after tempering	56
4.4	Weight (%) of each element in HAZ area after tempering	58
4.5	Vickers hardness result for Base metal	63
4.6	Vickers hardness result for HAZ area	64
4.7	Yield Stress (σ y) of HAZ and base metal element analysis	66

LIST OF ABBREVIATIONS, SYMBOLS, SPECIALIZED NOMENCLATURE

%	-	Percentage
ASTM	-	American Society for Testing and Materials
AISI	-	The American Iron and Steel Institute
HR	-	Rockwell hardness number
${}^{0}C$	-	Degrees Celsius
DBTT	-	Ductile to Brittle Transition
HAZ	-	Heat Affected Zone
BM	-	Base Metal
WM	-	Weld Metal
Cr	-	Chromium
Mo	-	Molybdenum
SEM	-	Scanning Electron Microscope
BCC	-	Body-center cubic
FCC	-	Faced-centered cubic
Fe ₃ C	-	Carbide
CCT	-	Continuous Cooling Transformation
EDX	-	Energy-dispersive X-Ray spectroscopy
TTT	-	Time Temperature Transformation
SEM	-	Scanning Electron Microscope
С	-	Carbon
wt%	-	Weight percentage
Cl	-	Chlorine
Al	-	Aluminium
Si	-	Silicon
Fe	-	Ferum
α	-	Alfa

γ	-	Gamma
Lab	-	Laboratory
FKP	-	Faculty of Manufacturing Engineering
max	-	Maximum
μ	-	Micron
Mpa	-	Mega Paschal
in	-	Inch
mm	-	Millimeter
cm	-	Sentimeter
SAW	-	Submerged Arc welding
PWHT	-	Post Weld Heat Treatment
UE	-	Unembrittlement
LE	-	Light Embrittlement
ME	-	Medium Embrittlement
HE	-	Heavy Embrittlement
EDS	-	Energy Dispersive X-ray Spectroscopy

CHAPTER 1 INTRODUCTION

1.1 Research Background

Towards developing the weld consumables for tempered martensite steels, evaluation on embrittlement is importance in order to prevent catastrophic failure of structure, often originating from brittle fracture of welded joint for engineering system that are operating at high temperature cycle (650 °C - 1100°C) that involve contact of metallic with combustion product gases, such as gas turbines, steam generator, and numerous petrochemical process vessel. Based on that, this research is developing in order to study the effect of high temperature cycle exposure to evaluation on embrittlement of welded pressure vessel steel.

The composition element and structure on base metal are quite different compared to heat affected zone (HAZ) of welded. This is because of the distribution of molybdenum within the alloy parent plate and in the weld heat affected zone. Heavy partition of molybdenum to carbide phases in the metal would result temper embrittlement, as molybdenum retard the segregation of embrittling tramp element to grain boundaries. Therefore, the research and observation in the mechanical properties of tempered welded area are very important in order to support on-going steel development for services temperature > 620° C and also useful to avoid the embrittlement during the services.

Overall part of this study was a consideration of the effect of time tempering on the welded zone (base metal and HAZ area) in a submerged-arc welding. For this purpose, the medium carbon steel (ASTM A516) is used in this research. The 2.25Cr-1Mo steel

has much advantage for both ambient and high temperature steel. The potential uses of this steel are in the electrical power generation plants and petrochemical industries. These pressure vessel steel have a potential for temper embrittlement that lead to toughness degradation and a reduction of a critical flaw size for brittle fracture.

A survey of literature shows the composition to be controlling parameter for temper embrittlement, in-particular the presence of impurity elements such as P and the presence of elements such as Mo which effect of impurity segregation. Much information is available to describe embrittlement phenomenon for Cr-Mo steels. This research also describe the mechanism of temper embrittlement and microstructure characteristic which allows the structural integrity of potential embrittled vessels for the purpose of remaining life assessment and plant life extension

To determine the properties of the steel after tempering process, several tests like Vickers Hardness, EDS analysis and microstructure observation will be done for all samples that tempering at elevated temperature according to ASTM references.

1.2 Problem Statement

Temperature embrittlement is a phenomenon where ductile metal become brittle due to high temperature exposure. This exposure temperature will also affect the hardness of the welded pressure vessel area. Therefore, it is important in order to evaluate embrittlement of material which all of this data could be use for further research to define the fracture of welded pressure vessel steel.

1.3 Objectives of Project

The purpose of this project is:

- i) To study the effect of high temperature cycle exposure to evaluation on embrittlement of welded pressure vessel steel.
- To determine the effect of precipitation elements on microstructure development and hardness of based metal and heat affected zone (HAZ) of welded pressure vessel steel.

1.4 Scope of Project

This study will focus on the effect of high temperature cycle exposure to evaluation on embrittlement of welded pressure vessel steel. This project will include the literature review on the phase transformation in steel, temper embrittlement, the preparation and procedure of heat treatment at elevated temperature, mechanical testing by using hardness measurement and microstructure observation. The microstructure for each specimen will be observed under the Optical Microscope. While the composition of element in welded area (base metal and HAZ) after tempering will be observed using EDS analysis.

1.5 Hypotheses

The tempering process with different exposure time will cause the significant reduction of the hardness of the steel, due to the segregation of trace elements at the grain boundaries and/or carbide interface. The microstructure is also different between Heat affected zones (HAZ), and base metal (BM) when expose at elevated high temperature.

CHAPTER 2 LITERATURE REVIEW

2.1 Metallurgy

2.1.1 Body-Centered Cubic Crystals

Body-centered cubic (BCC) structure can be defined as an atom that lies at each corner of the cube and one in the center. In body –centered cubic structures, each of corner atoms are touch the central atom but the corner atoms do not touch each other, Figure 2.1, [14]. This is one of the common and simplest shapes found in the crystal and minerals. For the other metal with the BCC structure at room temperature include chromium, iron, molybdenum, and vanadium.



Figure 2.1: The structure of a body-centered cubic metal: (**a**) the full solid sphere model, (**b**) the point model shows the location of the atom centered, (**c**) The positions of the centers of the atoms in one unit cell of the BCC [14]

Figure 2.1 (c) shows that nine atoms associate with each cell but some atoms are shared among several cells [11]. Each corner atom in body-centered cubic is shared by eight cells. Since the coordination number is less for BCC than FCC, so also is the atomic packing factor for BCC is lower (0.68 versus 0.74) [15].

2.1.2 Faced-Centered Cubic Crystal

Atom that are located at each of the corner and on the centered of all the faces of cubic unit cell can be defined as face-centered cubic structure (FCC), Figure 2.2, [16].



Figure 2.2: The structure of a faced-centered cubic unit cell: (a) the point model show the atoms location, (b) the full solid sphere that shown all 14 atoms associated with unit cells, (c) the partial solid sphere model shows the fraction of each atom contained within this unit cell. [16]

Metal that has the FCC structure at room temperature are include aluminum, calcium, copper, gold, and lead, nickel, platinum and silver. By referring Figure 2.2, this structure has an atom at each corner plus an additional atom at the center at each face. Since each face is shared by two unit cells, there are four atoms per FCC cell and each atom in the FCC structure has 12 nearest neighbors.