EFFECTS OF FLUCTUATING HEAT TREATMENT OPERATIONS ON CORROSION RESISTANCE OF AUSTENITIC STAINLESS STEEL SAMPLES IN SULPHURIC ACID SOLUTION

BY

OLORUNTOBA DANIEL 'TOYIN

MME/89/1938

A DISSERTATION SUBMITTED TO THE DEPARTMENT OF METALLURGICAL AND MATERIALS ENGINEERING, SCHOOL OF ENGINEERING AND ENGINEERING TECHNOLOGY, FEDERAL UNIVERSITY OF TECHNOLOGY, AKURE.

IN PARTIAL FULFILMENT OF THE REQUIREMENTS FOR THE AWARD OF MASTERS OF ENGINEERING (M. ENG) IN METALLURGICAL AND MATERIALS ENGINEERING

DECEMBER, 2002.

CERTIFICATION

I herby certify that this work was carried out by Oloruntoba Daniel 'Toyin in the Department of Metallurgical and Materials Engineering, School of Engineering and Engineering Technology, Federal University of Technology, Akure, and to the best of my knowledge has not been submitted elsewhere for the award of a degree.

.....

Engr. (Prof.) A. Oni
Supervisor

DEDICATION

This thesis is dedicated to:

Almighty God

The giver of life and peace.

ACKNOWLEDGEMENT

I give the Almighty God the glory, honour, and praises for His care, protection, provision and guidance which I enjoyed through the course of this study. Central to the success of a project is the complimentary efforts of the candidate and his supervisor(s). Consequent upon this, I wish to express my profound gratitude to my supervisor, Engr (Prof.) A. Oni, who showed much interest in the project and for his corrective comments at all times. Without his unflinching supports, my efforts would have proved less fruitful. Thank you Sir, God bless.

I wish to express my appreciations to the Management of the Federal University of Technology, Akure, for sponsoring my M.Eng. Programme through the Staff Development Scheme, the Management of Engineering Materials Development Institute, Akure for offering me the opportunity of using their heat-treatment facilities and the Management of Defence Industries Corporation of Nigeria (DICON), Kaduna for the practical knowledge I have gained from the industries before proceeding for the M.Eng programme.

A paragraph might not be enough to appreciate the person of Engr. (Dr) John Ade Ajayi, infact success in life depends on whom you make contact with; my contact with you along Abuja-Akure road and the subsequent assistance you rendered through the course of this study are in memory.

In getting to this stage of the programme, I have depended on the ability of some lecturers who have most patiently and intelligently impacted the knowledge unto me. I am especially grateful to Engr.(Prof) C. Kamma, Prof O. O. Onyemaobi, Dr. J. Bonode, Engr J. A. Omotoyinbo, Engr B. O. Adewuyi, Engr A. Akinlabi. And to all the staffs of Metallurgical and Materials Engineering Department of the Federal University of Technology, Akure, I register my appreciation.

I wish to thank the authors whose works were consulted, for their efforts in uplifting the body of knowledge. I also benefited from the active assistance of the staff of the Department of Industrial Chemistry Laboratory, Federal University of Technology, Akure, and from constructive review comments of Mr Moses Aderibigbe. Thank you all!

I wish to record my special thanks to my loving parent and brothers: Mrs Oke Jayeoba, Mr G. I. Oloruntoba, Mr Adebola David Oloruntoba for their unalloyed supports and encouragement that never let me down.

The work could not have gotten this far without the support, motivation, inspiration, encouragement, patience and positive ministration of my family during the many hours of toil. Mrs Foluso Eniola Oloruntoba, I very much appreciate you for your continued understanding and whole-hearted co-operation at all times. You are truly a life companion. Your effective fervent prayers at all times provided a dependable fortress and my hope for greater heights. Andrew Otitodara Oloruntoba is equally appreciated for his understanding even when he had to be denied some things for the success of this work. You are all blessed.

I gratefully acknowledge those to whom I am indebted for their roles in the completion of this work. They include: Brothers Olawale Egunlae, Segun Ajayi, Olasupo, Kenneth Alaneme, Tunji Ogunniyi, Ayo Afolabi, Peter Aboginije, Komolafe, Sisters Bola Ajayi, Toyin Olanipekun, Mary Ojo and Mrs Taiwo Oshodin. And special thanks to P. K. concepts on whose system the graphs were produced. To you all, I register my thanks.

The list can continue as there still remain a large number of people that influenced my thinking and academic life to date. I appreciate you all. May the Lord increase you in all your good works.

Conclusively, "To God be the glory! Great things He hath done" and Father, our Creator, hear these our hymns of praise,"..... to Thee alone we give all thanks and all honour, glory combined".

OLORUNTOBA DANIEL 'TOYIN

DECEMBER, 2002.

TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

LIST OF APPENDIXES

ABSTRACT

Corrosion performances of austenitic stainless steel subjected to various cyclic heat treatment operations of normalisation, annealing and solution-quenching have been evaluated. Samples of the as-received material (hot-rolled austenitic stainless steel) were subjected to five cycles of heat-treatment consisting of normalisation soaked for 1 hour and without soaking at 1000°C, annealing soaked for 1 hour and without soaking at 1000°C and solution-quenching soaked for 1 hour and without soaking at 1000°C. Estimation of corrosion rates from weight loss measurements was carried out using chemical weighing balance of model Mettler AE 160.

The results show that corrosion rates decrease with increase in exposure time for as-received and heat-treated samples. A general increase in electrode potentials is observed for all the specimens from –235mV (SCE) to –58 mV (SCE) at the exposure periods above 240 hours. The electrode potentials for all the samples remain within the range of –58mV (SCE) and –8mV (SCE) at the exposure periods between 250 and 750 hours.

It is concluded that corrosion resistance of austenitic stainless steel subjected to normalization, annealing and solufion-quenching at elevated temperature without soaking period at 1000°C is superior to that which was soaked for 1 hour at the temperature. This was attributed to the fact that austenitic stainless steel microstructure remain as austenitic single phase after cooling to room temperature.

CHAPTER ONE

1.1 INTRODUCTION

The corrosion resistance of stainless steel has been a subject of research interest for a long time. The reason for the continued interest on the corrosion of stainless steel is not unconnected with the wide application of stainless steels, especially in chemical processing industries. The rate at which deterioration occurs in the construction industries and in the equipment/machines used in paper industries are very high. This leads to a very high cost of failure, repairs and maintenance of the equipment used in such industries.

Most of the metals and alloys are liable to corrosion, except for a few noble metals such as gold, silver and platinum. The aim to eliminate corrosion has attracted the thought of mankind for ages. Among many metals, stainless steels are used frequently in many applications such as in desalination plants, petrochemicals, pharmaceuticals and automobiles industries. This wide usage of stainless steels is on consequence of their resistance to general corrosion and high strength. However, certain service conditions rendered stainless steel to be highly susceptible to corrosion (Audouard et al 1984, Oldfield, 1989). This can either be chemical or thermal condition. Plumbing or replumbing a large building using stainless steel can cost several million pounds. Replacing a corroded or failing part of a food processing plant is also expensive and the risk of contamination if replacement is not carried out is potentially dangerous. The highly corrosive environments in biomass fuel boilers and waste incinerators still present a great challenge for materials scientist.

The loss of heat efficiency due to severe corrosion of heat exchanger tubes calls for urgent study (Birk, et al 1997). Despites the vast amount of scientific research carried out in the field of corrosion since 1945 (Banerjee, 1985) towards understanding the basic mechanisms of corrosion and the application of this knowledge to its control, the average engineer seems to know very little about the subject; and the wastage of material due to corrosion is consequentially assuming serious proportion, posing a problem to the industries. Although austenitic stainless steel do not undergo hardening effect after heat treatment but its corrosion resistance property can be modified via thermal cycling treatment operation. The enormous use of austenitic stainless steel in production of equipment and machine parts call for critical examination of its corrosion behaviour under thermal fluctuating operations. There is need to improve on the safety of the use of austenitic stainless steel in high temperature application. For these reasons, this work investigates the corrosion characteristics of austenitic stainless steel under fluctuating heat treatment operations. Heat treatment operations such as normalisation, annealing and solution – quenching were carried out at various cycles at 1000°C soaked for 1 hour and without soaking. The effects of fluctuating heat treatments operations on the corrosion performance of austenitic stainless steel samples were determined in sulphuric acid solution. The results obtained showed that austenitic stainless steel (unstabilised grade) can perform credibly well in terms of corrosion resistance under fluctuating heat treatment operations.

1.2 OBJECTIVES OF THE RESEARCH

The general objective of the research is to evaluate the corrosion performances of austenitic stainless steel subjected to repeated slow thermal fluctuations of normalisation, annealing and solution-quenching.

Other specific objectives are to:

(a) Study the variations of electrode potentials with exposure time (hours) of the austenitic stainless steel.

(b) Evaluate the corrosion rates with exposure time (days) of austenitic stainless steel subjected to various cycle of fluctuating heat treatment operations.

1.3 EXPECTED CONTRIBUTION TO KNOWLEDGE.

The research would provide an alternative method for evaluating the corrosion resistance performance of components designed for application in practical engineering systems, whose operations may involve alternate thermal fluctuations.

CHAPTER TWO

LITERATURE REVIEW

2.1 STAINLESS STEELS OVERVIEW

As chromium is added to steels, the corrosion resistance increases progressively due to the formation of a thin protective film of Cr_2O_3, the so – called passive layer. With the addition of about 12% Cr, steels have good resistance to atmospheric corrosion and the popular convention is that this is the minimum level of chromium that must be incorporated in an iron – based material before it can be designated a stainless steel. However, of all steel types, the stainless grades are the most diverse and complex in terms of composition, microstructure and mechanical properties. Given this situation, it is not surprising that stainless steels have found a very wide range of application, ranging from the chemical, pharmaceutical and power generation industries to less aggressive situations in architecture, domestic appliances and steel furniture.

By the late 1800's, iron – chromium alloys were in use throughout the world but without the realization of their potential as corrosion – resistant materials. Harry Brearley, a Sheffield metallurgist, is credited with the discovery of martensitic stainless steels in 1913 when working on the development of improved riffle barrel steels. He found that a steel containing about 0.3 % and 13% Cr was difficult to etch and also remained free from rust in a laboratory environment (Llewellyn, 1992). Such steel formed the basis of the cutlery industry in Sheffield and as type 420 is still used for this purpose to the present day. During the same period, researchers in Germany were responding to pressures for improved steels for the chemical industry. Up until that time, steels containing high levels of nickel were in use as tarnish – resistant materials but had inadequate resistance to corrosion.

Two Krupp employees, Benno Straws and Edward Maurer, are credited with the discovery of Cr – Ni austenitic stainless steels and patents on these materials were registered in 1912. However, workers in France and the United States are also cited as independent discoverers of these steels. During the 1920s and 1930s, rapid developments took place which led to the introduction of most of the popular grades that are still in use today, such as type 302 (18% Cr, 8% Ni), type 316 (18% Cr, 12% Ni, 2.5% Mo), type 410(12% Cr) and type 430 (17% Cr). However even in the 1950s, stainless steels were still regarded as semi – precious metals and were priced accordingly.

In terms of product innovation, perhaps the greatest benefits have been obtained from relatively simple changes, such as the introduction of stainless grades with low carbon contents, i.e. below 0.03% C. This modification has virtually eliminated the risk of intergranular corrosion in unstabilized austenitic grades and has improved the corrosion performance and ductility of austenitic grades (Llewellyn, 1992).. These steels are based on compositions such as 18% Cr, 2% Mo, and 26% Cr 1% Mo and offered the prospect of being a cheaper alternative to an austenitic grade such as type 316 (18% Cr, 12% Ni, 2.5% Mo). Although the low interstitial grades exhibits good corrosion resistance, (particularly with regard to chloride-induced stress corrosion), they tend to retain the problem of conventional ferritic steels in relation to grain coarsening and loss of toughness after welding. On the other hand, high alloy steels involving duplex austenite plus ferrite microstructures are now gaining acceptance, because of their higher strength and better resistance to stress corrosion than conventional austenitic grades(Llewellyn, 1992).

Whereas the consumption of bulk steel products is likely to remain fairly

static, stainless steel is still very much in a growth market. This related to the facts that stainless steel has managed to maintain its traditional image as a decorative material but is now also regarded as an engineering material for use in applications where structural integrity is more important than aesthetic appearance. On the basis of life – cycle costing, stainless steels are also proving to be attractive alternatives to mild steel in structures or components that require frequent painting and maintenance (Llewellyn, 1992). The long - term data obtained in marine locations were also instrumental in convincing operators of off – shore oil and gas platforms of the benefits of using stainless steels for top side architecture in place of traditional painted mild steel. In the North Sea, the environment is particularly aggressive and high corrosion resistance of type 316 is attractive. In the event of fire, stainless steel is also preferred to painted mild steel because of its higher strength at elevated temperature and the elimination of the smoke or toxic hazards associated with burning paint. Because no corrosion allowance is required, stainless steel module walls can be up to 50% thinner than similar components in mild steel, resulting in significant weight savings. Whereas type 316 stainless is considerably more expensive, the cost differential at float out is less than 15% compared with mild steel fabrications (Llewellyn, 1992). A cost analysis by Stone et al (1984) indicates that this initial differential is eliminated after the first five years of operation due to the need for the first repaint on mild steel at that time. Based on the above work type 316 stainless steel has now been used for wall cladding on accommodation modules, ventilation louvers and other architectural features on more than half the oil field in the North Sea(Llewellyn, 1992).Table 1 shows chemical composition for various type of stainless steels. Figures 2.1, 2.2 and 2.3 show Fe-Ni, Fe-Cr and Fe-Cr-Ni-C equilibrium diagrams respectively.

able 1: Compositions of Standard Grades of Wrought Stainless and Heat-Resisting

teels (AISI,1959). (Source Book on Stainless Steels, 1976)

AISI	Nominal composition, %				
Type C	Mn, max	Si, max	Cr	Ni	Others
Austenitic Steels					
201..... 0.15 max	7.50	1.00	16.00 to 18.00	3.50 to 5.50	0.25 max N
202..... 0.15 max	10.00	1.00	17.00 to 19.00	4.00 to 6.00	0.25 max N
301..... 0.15 max	2.00	1.00	16.00 to 18.00	6.00 to 8.00	.....
302..... 0.15 max	2.00	1.00	17.00 to 19.00	8.00 to 10.00	.....
302B... 0.15 max	2.00	3.00	17.00 to 19.00	8.00 to 10.00	.....
303..... 0.15 max	2.00	1.00	17.00 to 19.00	8.00 to 10.00	0.15 min S
303Se.. 0.15 max	2.00	1.00	17.00 to 19.00	8.00 to 10.00	0.15 min Se
304..... 0.08 max	2.00	1.00	18.00 to 20.00	8.00 to 12.00	.....
304L.... 0.03 max	2.00	1.00	18.00 to 20.00	8.00 to 12.00	.....
305..... 0.12 max	2.00	1.00	17.00 to 19.00	10.00 to 13.00	.....
308..... 0.08 max	2.00	1.00	19.00 to 21.00	10.00 to 12.00	.....
309..... 0.20 max	2.00	1.00	22.00 to 24.00	12.00 to 15.00	.....
309S.... 0.08 max	2.00	1.00	22.00 to 24.00	12.00 to 15.00	.....
310..... 0.25 max	2.00	1.50	24.00 to 26.00	19.00 to 22.00	.....
316..... 0.08 max	2.00	1.00	16.00 to 18.00	10.00 to 14.00	2.00 TO 3.00 M
316L.... 0.03 max	2.00	1.00	16.00 to 18.00	10.00 to 14.00	2.00 to 3.00 Mo
Martensitic Steels					
403..... 0.15 max	1.00	0.50	11.50 to 13.00	.....	.....
410..... 0.15 max	1.00	1.00	11.50 to 13.00	.....	.....
414..... 0.15 max	1.00	1.00	11.50 to 13.50	1.25 to 2.50	.....
416..... 0.15 max	1.25	1.00	12.00 to 14.00	.....	0.15 min S
440B....0.75 to 0.95	1.00	1.00	16.00 to 18.00	.....	0.75max Mo
501..... 0.10 min	1.00	1.00	4.00 to 6.00	.....	0.4 to 0.65 Mo
Ferritic Steels					
405.... 0.08 max	1.00	1.00	11.50 to 14.50	.....	0.10 to 0.30 Al
430.... 0.12 max	1.00	1.00	14.00 to 18.00	.....	.....
430F... 0.12 max	1.25	1.00	14.00 to 18.00	.....	0.15 min S
430F Se..0.12 max	1.25	1.00	14.00 to 18.00	.....	0.15 min Se
446..... 0.20 max	1.50	1.00	23.00 to 27.00	.....	0.25 max N

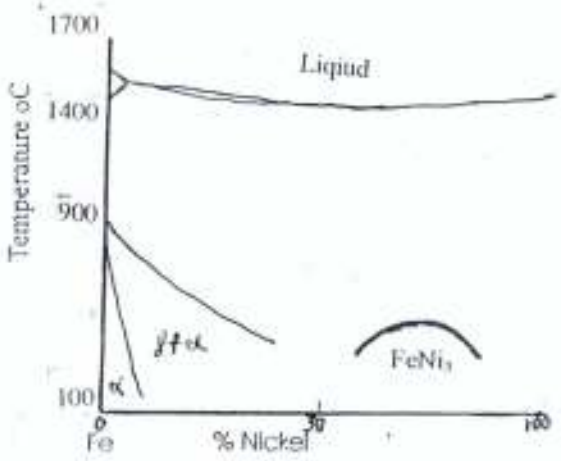


Figure 2.1: Fe-Ni Equilibrium Diagram (Llewellyn, 1992)

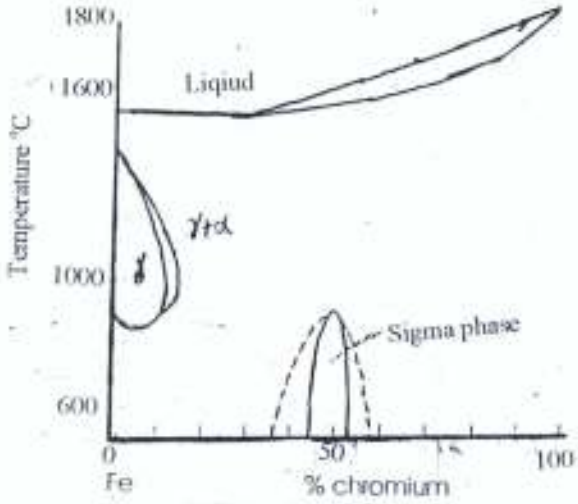


Figure 2.2: Fe-Cr Equilibrium Diagram (Llewellyn, 1992)

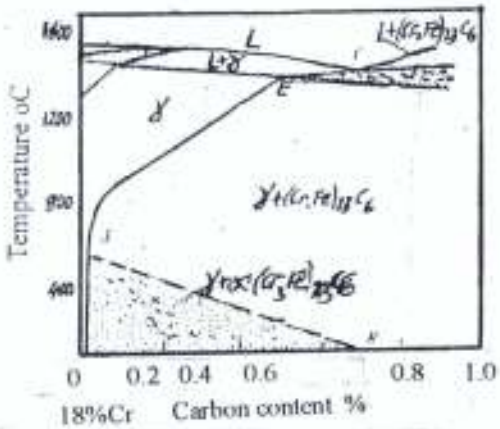


Figure 2.3: Pseoudobinary Equilibrium Diagram of the Fe-Cr-Ni-C System (Lakhtin, 1979)

2.2 CLASSIFICATION OF CORROSION PROCESSES

Corrosion is the gradual deterioration of a material caused by the chemical or electrochemical reaction with its environment, which result to irrecoverable loss. A logical and scientific classification of corrosion processes, although desirable, is by no means simple. This is due to the enormous variety of the surrounding media, varied heterogeneities in metals and the diversity of corrosion reactions However, corrosion may be classified on the basis of (Banerjee, 1985)

2.2.1. The Mechanisms Of Corrosion Processes

(i) Chemical Corrosion (direct combination)

Example: Uniform and non – uniform corrosion

These are generally high temperature metal/gas or metal/vapour reactions (gaseous corrosion) involving non metals like oxygen, halogens, hydrogen sulphide, sulphur vapour.

(ii) Electrochemical corrosion

This type is subjected to basic laws of electrochemical kinetics and generally refers to cases of corrosion of metals in electrolytes with possible generation of galvanic current in the so-called corrosion cells. The corrosion leading to localized attack (localized corrosion) may be classified as follows:

(a) Galvanic corrosion (or bimetallic corrosion) caused by the contact of two or more metals of different electrochemical potential (galvanic metal/metal macro –cell)

(b) The following are initiated owing to the formation of local corrosion cells (galvanic cells) within a metal:

(i) structural selective corrosion (graphitization)

(ii) selective dissolution (dezincification, dealuminification)

(iii) Intercrystalline (intergranular) corrosion (corrosion cracking)

(iv) Stress corrosion cracking

(v) Corrosion fatigue

(vi) Layer (subsurface) corrosion, or exfoliation

(vii) Thermogalvanic corrosion. Example: Steam engine boiler.

(c) The following are caused by the creation of differential aeration cells (oxygen concentration in macro – cells) and are initiated in the environment;

(i) Filiform corrosion

(ii) Crevice corrosion

(iii) Water – line corrosion

(d) Erosion – corrosion (impingement attack) of copper and copper alloys. This results due to formation of metal ion concentration cells (macro – cells)

(e) The following are propagated as a result of conjoint action of active – passive cells and differential aeration cells, and are characteristics of passive metals

(i) Pitting

(ii) Crevice corrosion

(iii) Intercrystalline corrosion

(iv) Stress corrosion cracking

(f) Stray – current corrosion (or, electro – corrosion). This is caused by the formation of electrolytic cells (macro – cells) e.g. the corrosion of underground pipe lines by stray currents

2.2.2 The Nature Of The Environment

Corrosion phenomena can be grouped after environment in the following ways, viz:

(i) Atmospheric corrosion

(ii) Marine corrosion

(iii) Underground corrosion

(iv) Biological corrosion

(v) Hydrogen cracking

(vi) Radiation cracking

(vii) Liquid metal corrosion

(viii) Molten salt corrosion

(ix) Acid corrosion

(x) Corrosion in electrolytes

2.2.3 The Type Of The Corrosion Deterioration

It is customary to subdivide corrosive effects into:

(a) General (or surface) corrosion where the attack is evenly distributed along the metal surface

(b) Local corrosion where attack is concentrated on a very small area, and

(c) Intercrystalline corrosion where attack is centred on the grain boundaries.

If all areas on the metal surface corrode at the same or similar rate, it is known as general (extensive) corrosion, which may be uniform or nor uniform. If certain areas of the metal surface corrode at higher rates than others due to heterogeneities in

the metal, in the environment, or in the geometry of the structure as a whole, then the attack can range from being slightly localized to pitting.

2.2.4 Types Of Corrosion Processes

Shrier (1963) classifies the main types of corrosion processes as follows;

(i) Film –free chemical interaction in which there is direct chemical reaction between a metal and its environment. The metal remains film – free and there is no transport of charge. For example, in the reaction of molybdenum with oxygen, the oxide being volatile, the metal surface remains film – free.

(ii) Electrochemical reactions which involve transfer of charge across an interface. These electrochemical reactions have been further sub – divided into three groups, viz;

(a) Inseparable anode/cathode type. For example, the uniform dissolution of metals in acids, alkalis or neutral aqueous solutions, in organic solvents, or in molten salt. Here the presence of anodes and cathodes, although postulated by theory can not be distinguished by experimental methods.

(b) Separable anode/cathode type. In this case certain areas of the metal can be distinguished experimentally as predominantly anodic or cathodic, although the distances of separation of these areas may be infinitesimally small. Reaction of iron containing a crevice corrosion, water – line corrosion are examples of this type.

(c) Interfacial anode/cathode type. Here the metal surface remains filmed. One entire interface will be anode and the other will be cathode.

In engineering science, corrosion types caused by a conjoint action of corrosion

and mechanical factors deserve special reference. Depending on the mechanical force, the following classifications may be made;

2.2.5 Mechanical Forces Within The Metal

(i) Stress – corrosion cracking (due to combine action between an internal and external tensile stress and local corrosion attack)

(ii) Corrosion fatigue (caused by cyclic stresses in the presence of a corrosive environment).

(iii) Hydrogen cracking (due to combined action between tensile stresses and atomic hydrogen)

2.2.6 Mechanical Wear Effects Or Abrasion On The Metal By A Liquid

(i) Erosion – corrosion (a corrosion reaction accelerated by velocity and abrasion)

(ii) Cavitation corrosion (due to conjoint action of liquid flow rate and corrosion, caused by the formation and collapse of vapour bubbles in a liquid near a metal surface.

2.2.7 Mechanical Action Of A Solid Body On The Metal

(i) Fretting corrosion (localized deterioration at the interface between two contacting surfaces vibrating under pressure.

Corrosion may also be classified either as (Banerjee, 1985):

(a) Low temperature corrosion and high temperature corrosion, or as

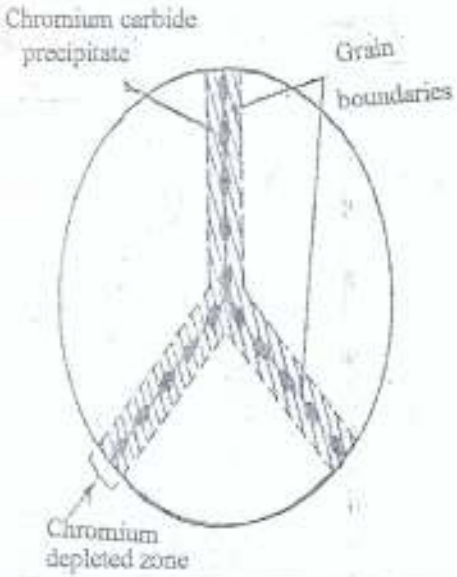
(b) Wet corrosion or dry corrosion

However, the latter only has been somewhat accepted. The term 'wet' includes all reactions in which a liquid is involved in the reaction mechanism while dry corrosion process occurs in the absence of liquid phase, or above the dew point of the environment. The process is frequently made more specific by the use of an adjective that indicates type of environment, for example, bimetallic corrosion, concentration cell corrosion, sea water corrosion, bacteria corrosion.

2.3 INTERGRANULAR CORROSION OF AUSTENITIC STAINLESS STEEL

This particular corrosion was described by Llwellyn (1992), Ross (1977), Lakhtin (1979) and Fontana (1986) as given favourable temperature conditions, solute atoms can segregate to the grain boundaries, causing enrichment in a particular element or the precipitation of metal compounds. Under highly oxidizing conditions, these effects can cause the grain boundaries of stainless steels to become very reactive, leading to the highly localized form of attack known as intergranular corrosion. Both austenitic and ferritic stainless steels are susceptible to intergranular corrosion and, in either case, the problem is caused by the segregation of carbon to the grain boundaries and the formation of the chromium – rich, $Cr_{23}C_6$ carbides.

The concentration of chromium in these carbides is very much higher than that in the surrounding matrix and, at one time, it was postulated that this resulted in galvanic corrosion between the noble carbides and the more reactive matrix. However, currently the most widely accepted theory for intergranular corrosion in stainless steels is that involving chromium depletion. Thus, in forming chromium – rich carbides at the grain boundaries, chromium is drawn out of solid solution and in areas adjacent to the boundaries; the chromium content becomes severely depleted compared to the bulk chromium concentration of the steel. Such areas are then said to have become sensitised in that they no longer contain sufficient chromium to withstand corrosive attack, corrosion can then proceed along grain boundaries. Diagrams illustrating this form of attack in an austenitic stainless steel are shown in Figures 2.4 and 2.5. Table 2 shows the corrosives that induce intergranular corrosion in austenitic stainless steel.



re 2.4: Diagrammatic representation of a grain
ary in sensitized type 304 stainless steel

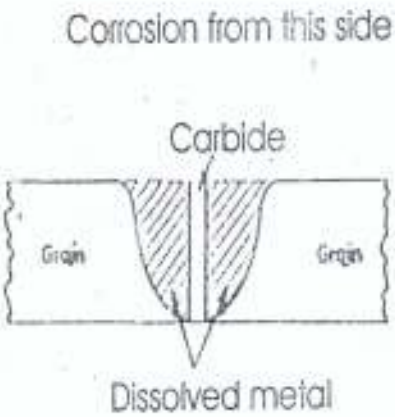


Figure2.5: Cross-section of area shown in Figure 2.4

Table 2: Corrosives reported to induce intergranular corrosion in sensitized austenitic stainless steels (Nelson, 1960)

Acetic acid	Oxalic acid
Acetic acid + salicylic acid	Phenol + naphtenic acid
Ammonium nitrate	Phosphoric acid
Ammonium sulfate	Phthalic acid
Ammonium sulfate+H_2SO_4	Salt spray
Bee juice	Sea water
Calcium nitrate	Silver nitrate+acetic acid
Chromic acid	Sodium bisulfate
Chromium chloride	Sodium hydroxide + sodium sulfide
Copper Sulfate	Sodium hypochlorite
Crude oil	Sulphuric acid
Fatty acid	sulfite cooking liquor
Ferric chloride	sulfite digester acid
Ferric sulfate	(calcium bisulfite + sulfur dioxide)
Formic acid	Sulfite solution
Hydrocyanic acid	Sulfur dioxide (wet)
Hydrocyanic acid + sulfur dioxide	Sulfuric acid
Hydrofluoric acid + ferric sulfate	Sulfuric acid + acetic acid
Lactic acid	Sulfuric acid+ copper sulfate
Lactic acid + nitric acid	Sulfuric acid + ferrous sulfate
Maleic acid	Sulfuric acid + methanol
Nitric acid	Sulfuric acid + nitric acid
Nitric acid + hydrochloric acid	Sulfurous acid
Nitric acid + hydrofluoric acid	Water + starch + sulfur dioxide

2.3.1 Control Of Intergranular Corrosion In Austenitic Stainless Steel

Three methods are used to control or minimize intergranular corrosion of the austenitic stainless steels (Avery, 1963).

(i) Employing high – temperature solution heat treatment, commonly termed quench – annealing or solution – quenching.

(ii) Adding elements that are strong carbide formers (called stabilizers), and

(iii) Lowering the carbon content to below 0.03%.

Commercial solution- quenching treatments consist of heating to 1050 to 1100 °C followed by water quenching. Chromium carbide is dissolved at these temperatures, and a more homogenous alloy is obtained. Quenching, or rapid cooling from the solution temperature, is very important. If cooling is slow, the entire structure will be susceptible to intergranular corrosion.

The strong carbide formers or stabilizing elements, columbium or (columbium plus tantalum) and titanium, are used to produce types 347 and 321 stainless steels, respectively. These elements have a much greater affinity for carbon than does chromium and are added in sufficient quantity to combine with all of the carbon in the steel. The stabilized steels eliminate the economic and other objections of solution – quenching the unstabilized steels after fabrication or weld repair.

The original 18/8 steels contained around 0.2% carbon, but this was quickly reduced to 0.08% because of rapid and serious weld decay failures. Lowering the carbon content much below 0.08% was by blowing oxygen through the melt to burn out carbon and by the addition of low – carbon ferrochrome (Fontana, 1986).

Carbon pick up (surface carburization) during production of austenitic stainless steels has caused premature failures. It occurs when these are cast into molds containing carbonaceous materials, such as organic binders and washes or baked oil sand. The hot metal absorbs carbon from the carbon – containing environment.

Increased carbon content of the stainless steel can degrade corrosion resistance particularly to environments that are aggressive from the stand point of intergranular attack. Resistance to pitting is also decreased (Fontana, 1986).

2.4 PASSIVITY IN STAINLESS STEEL

Stainless steels owe their corrosion resistance to the formation of the so – called passive film (Llewellyn, 1992). This consists of a layer only 20- 30A° thick, of hydrated chromium oxide (Cr_2O_3), which is extremely adherent and resistant to chemical attack. If the passive film is damaged by abrasion or scratching, a healing process or repassivation occurs almost immediately. In general, the corrosion and oxidation resistance of stainless steels increase with chromium content and the materials are used in a wide range of aggressive media in the chemical and process plant industries.

Ostrander (1971), believed that for a coated chromium film when in contact with water the hexavalent chromium portion of the film gradually leaches out thus providing a local supply of dissolved chromate inhibitor to prevent corrosion at any exposed areas of metal surface. The trivalent chromium portion of the film, while essentially insoluble and incapable of providing local corrosion inhibition, does provide important general protection of the mechanical exclusion type. While the films will not withstand continuous rubbing or harsh abrasion, suitably dried coatings can withstand the abrasion incident to the drawing or stamping of metal. Undried, freshly formed films are fragile and easily damaged. Dehydration of chromate coatings will occur on baking at temperatures of 150F (66°C) and higher or from subjecting the films to drying in closed silica gel or calcium chloride desiccators. This changes the structure of the coating to the extent that an x – ray pattern of chromium chromate can then be found. It is not known whether this structure had previously

been present in the normal film in a particle size too small for diffraction. As a consequence of dehydration the film becomes relatively insoluble and its protective value is reduced in water immersion or salt spray tests. It has been noted, however, that resistance to abrasion and to the action of weak acids and alkalis improves.

2.5 HEAT TREATMENT OF STAINLESS STEEL

Heat treating of stainless steel serves to produce changes in physical condition, mechanical properties and residual stress level, and to restore maximum corrosion resistance when that property has been adversely affected by previous fabrication or heating. Frequently, a combination of satisfactory corrosion resistance and optimum mechanical properties is obtained in the same heat treatment (Metals Handbook, 1964). Unstabilized compositions (the series 200 steels and types 301 through 317) cannot be hardened by heat treatment but will harden as a result of cold working. These steels are annealed to insure maximum corrosion resistance and to restore maximum softness and ductility.

Avery (1963), reported that during annealing, carbides which markedly decrease resistance to intergranular corrosion are dissolved. Because carbide precipitation can occur at temperatures between 450 and 860°C, it obviously is desirable that the annealing temperature should be safely above this limit. Moreover, because all carbides should be in solution before cooling begins, and because the chromium carbide dissolves slowly, the highest practical temperature consistent with limited grain growth is selected. This temperature is in the vicinity of 1100°C. It is frequently necessary to homogenize some alloys above 1100°C to promote uniformity of chemical composition and microstructure. Cooling from the annealing temperature must be rapid, but it must also be consistent with limitations of distortion. Whenever considerations of distortion permit, water quenching is used, thus insuring that dissolved carbides remain in solution (because they precipitate carbides more rapidly,

types 309 and 310 invariably require water quenching). Where practical considerations of distortion rule out such a fast cooling rate, cooling in an air blast is used. With some thin – section parts, even this intermediate rate of cooling produces excessive distortion, and parts must be cooled in still air.

The austenitic alloys achieve maximum resistance to intergranular corrosion by the high – temperature heating and quenching procedure know as solution annealing or solution – quenching. Solution annealing procedures for all austenitic alloys are similar, and consist of heating to a temperature of above 950°C, holding for a time sufficient to accomplish complete solution of carbides, and quenching at a rate fast enough to prevent reprecipitation of the carbides – particularly while cooling through the range from 815 to 540°C (Metals Handbook, 1964).

2.5.1 Heat And Corrosion Resistance Of Austenitic Stainless Steels

According to Lakhtin (1979), Dell (1981), and John (1994), scale resistance is increased primarily by the addition of chromium. An addition of 5 to 8 percent chromium raises the scale resistance to 700 – 750°C; increasing the chromium content to 15 to 17 percent prevents scaling up to 950 – 1000°C. Heat resistance depends only on the composition of the steel and not on its structure. Consequently, ferritic and austenitic steels with equal chromium contents have practically the same heat or scale resistance.

Austenitic stainless steels, usually alloyed by chromium and nickel (or manganese), have an austenite structure after being cooled to room temperature. They have good corrosion resistance in oxidizing media. These steels are paramagnetic. Chromium makes its electrochemical potential positive and it becomes resistant to corrosion in the atmosphere, salt (and sweet) water, and many acids, alkalis and salts.

Molybdenum raises the resistance to corrosion in organic acids, and salt water. Chromium – manganese – nickel stabilizes the austenite. Ulick (1982) reported that alloy containing molybdenium possesses practically no salt forming properties. The 18/8 alloys containing 3-4% molybdenum are essentially designed to withstand oxygen – free or reducing acid conditions; they are not well suited to strongly oxidizing acid liquids, where the molybdenum may pass into the liquid in the hexavalent condition. Austenitic stainless steels containing molybdenum are especially useful for resisting sulphurous acid-a reagent important in the paper and textile industry and many other situations where a higher corrosion resistance than that of a 18/8 austenitic alloy is required (Ulick, 1982).

2.5.2 Effects Of Heat Treatment Temperatures On Austenitic Stainless Steel

Sangdall (1964) presented the following as effect of various heat treatment temperature on austenitic stainless steel

(1) Heating in the range from 480 to 815°C will precipitate chromium carbides in the grain boundaries of unstabilized grades.

(2) Heating in the range from 540 to 930°C may result in the formation of hard, brittle sigma phase, which can decrease both corrosion resistance and ductility.

(3) Slow cooling an unstabilized grade (other than an extra – low carbon grade) through either of the above temperatures ranges will allow sufficient time for these detrimental effects to take place.

(4) Heating at 815 to 930°C will cause the coalescence of chromium carbide precipitates or sigma phase to a form less harmful to corrosion resistance or mechanical properties.

(5) Heating at 950 to 1100°C (an annealing treatment) will cause all grain – boundary chromium carbide precipitates to redisolve, will transform sigma back to ferrite, and will fully soften the steel.

Fig. 2.6 by Schneider (1960) illustrated the structures produced in a wide range of compositions after rapid cooling from 1050°C.

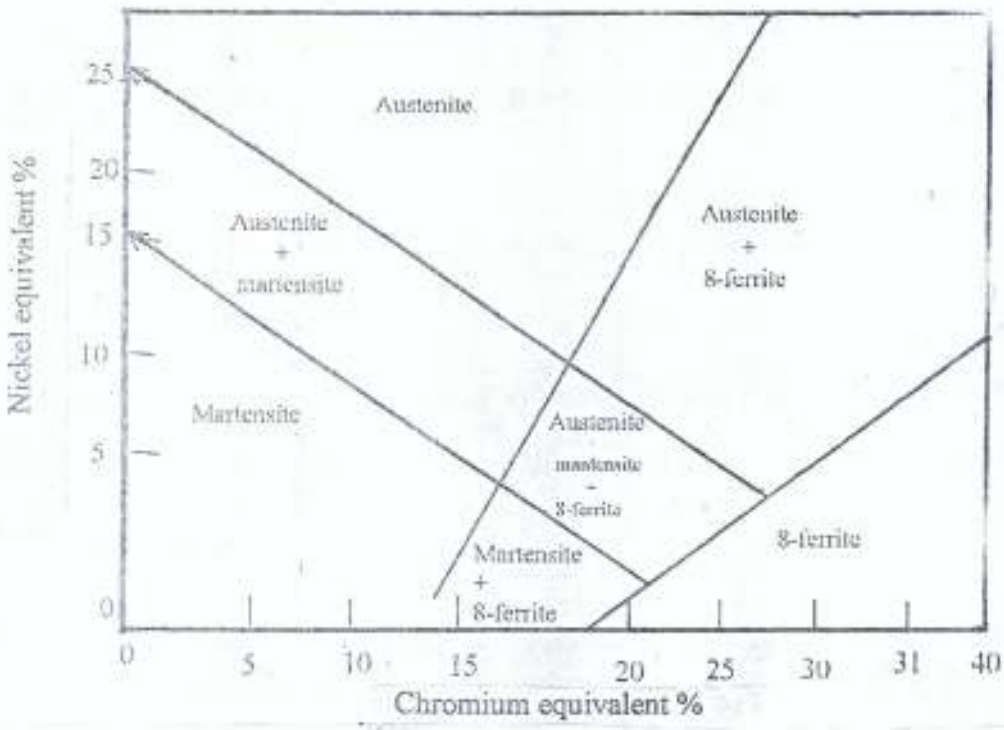


Figure 2.6: Microstructures obtained from rapidly cooling stainless steel From 1050°C (Schneider, 1960)

There are really only two processes used on austenitic stainless steel (Kenneth, 1983) : annealing and stress relieving. Annealing is performed at 1800 to 2000 F (1000 or 1100°C) followed by a water quench. The quench is imperative if sensitisation is to be prevented. Effective stress relieving requires temperatures above 1600 F(870°C), followed by as rapid a cooling rate as the part will permit. Some grades of stainless steel can be used in continuous service in temperatures up to 2000F (1000°C) in oxidizing atmospheres such as in hot – air furnaces.

At room temperatures, types 316 and 317 show low corrosion rates at low concentrations (less than 10%) sulphuric acid and at very high concentrations (greater than 95%). At higher temperatures, they are resistant only to very dilute solutions (less than 1%) sulphuric acid. In order to give the stainless austenitic and ferritic austenitic steels their best corrosion properties, they are heat – treated by accelerated cooling (quenching). The steel is heated to a high temperature, 1320 – 1420 K for austenitic and 1220 – 1270 K for ferritic – austenitic steels. All carbides are then dissolved and a homogenous austenitic structure is obtained after quenching (Kenneth, 1983).

2.6 APPLICATION OF THERMODYNAMIC PRINCIPLES TO CORROSION STUDIES

Iron is a fairly reactive metal; in thermodynamic terms all its compounds are formed with a resulting decrease of free energy, so it may be anticipated that reaction can occur in almost any environment. It must again be stressed however that the fact that a reaction can occur does not necessarily mean that it will continue to do so at such a rate that corrosion become serious.

The following stages of reaction take place during corrosion.

1. Iron + Oxygen

$$2Fe + O_2 \longrightarrow 2FeO \longrightarrow Fe_2O_3 \longrightarrow Fe_3O_4 \quad \text{.....(i)}$$

This reaction is slow at room temperature, but accelerates with increasing temperature. At low temperatures and under carefully controlled conditions of purity a durable tarnish film may be produced, but more usually, and especially at high temperature, the film thickens to a layered and friable scale of varied composition which tends or flake and crumble, so affording little protection (Ross, 1977).

2. Iron + Water

$$Fe + 2H^- \longrightarrow Fe^{2+} + H_2 \quad \text{.....(ii)}$$

This reaction progresses without hindrance on metal surfaces until either the environment becomes locally saturated with iron salts or the discharge of hydrogen is impeded.

3. Iron + Oxygenated water:

$$2Fe + 2H_2O + O_2 \longrightarrow 2Fe(OH)_2 \longrightarrow Fe(OH)_3 \quad \text{.....(iii)}$$

This is perhaps the most common reaction in the rusting of irons and steel. Corrosion in the presence of oxygenated water comprises three steps as follows:

i. Dissolution or corrosion of iron,

$$Fe \longrightarrow Fe^{2+} + 2e^- \quad \text{..... (iv)}$$

ii. Reduction of dissolved oxygen,

$$2H_2O + O_2 + 4e^- \longrightarrow 4(OH)^- \quad \text{.....(v)}$$

iii. Precipitation of rust by admixture of products of (i) and (ii)

$$2Fe^{2+} + 2H_2O + O_2 \longrightarrow 2Fe(OH)_2 \quad \text{.....(vi)}$$

Ferrous hydroxide precipitates from solution. However, this compound is unstable in oxygenated solutions and is oxidized to the ferric salt.

$$2Fe(OH)_2 + H_2O + \tfrac{1}{2} O_2 \longrightarrow 2Fe(OH)_3 \quad \text{.....(vii)}$$

CHAPTER THREE

3.0 MATERIALS AND METHODS

3.1 MATERIALS AND PREPARATION

The material used for this study was a hot rolled flat bar austenitic stainless steel supplied by Galvanising Industries Limited, Lagos. The chemical composition presented in Table 3 was carried out at Nigerian Foundries Limited, Lagos.

Table 3: **Chemical Composition of Austenitic Stainless Steel Sample**

Chem. Comp.	Fe	C	Si	Mn	P	S	Cr	Mo	Ni
Wt%	66.81	0.06	0.531	1.48	0.043	0.019	17.24	2.3	11.94

The as-received material was of dimension 230mm x 60mm x 12mm. It was cut into pieces of dimensions 60mm x 15mm x 12mm with power hack saw. These were further reduced to dimension 18mm x 14mm x 12mm. Thirty one samples each of 18mm x 14mm x 12mm were used for this study.

3.2 EXPERIMENTAL METHOD

Electrical full-muffle type furnace was used for the heat-treatments of the austenitic stainless steel samples.

3.2.1 NORMALIZING AT 1000°C HELD FOR 1 HOUR

A sample was heated from room temperature (27°C) to austenitic temperature of 1000°C and was held at this temperature for one (1) hour, after which it was cooled in air to room temperature. For the sake of this study, this procedure is termed "one – cycle" normalizing. This procedure was repeated once for another sample that had previously undergone one-cycle normalizing. This operation may be termed "two-cycle" normalizing. Using these aforementioned approach "three-cycle", "four-cycle" and "five-cycle" normalizing were carried out.

3.2.2 NORMALIZING AT 1000°C WITHOUT SOAKING

The procedures described under 3.2.1 were used except that no soaking period in the furnace was allowed.

3.2.3 ANNEALING AT 1000°C HELD FOR 1 HOUR

A sample was heated in the furnace from room temperature (27°C) to austenitic temperature of 1000°C and was held at this temperature for one hour after which it was cooled in the furnace to room temperature. This operation is termed "one-cycle" annealing. This operation was repeated once for another sample that had previously undergone one-cycle annealing and this is termed "two-cycle" annealing. Using these aforementioned approach "three-cycle", "four-cycle" and "five-cycle" annealing were carried out.

3.2.4 ANNEALING AT 1000°C WITHOUT SOAKING.

The procedures described under 3.2.3 were used except that no soaking period in the furnace was allowed.

3.2.5 SOLUTION-QUENCHED AT 1000°C HELD FOR 1 HOUR

A sample was heated in the furnace from room temperature (27°C) to austenitic temperature of 1000°C and was held at this temperature for one hour after which it was rapidly quenched in water at ambient temperature. This procedure is termed "one-cycle" solution-quenching.

The above procedure was repeated once for another sample that had previously undergone one-cycle solution quenching. This operation may be termed "two-cycle" solution quenching. This aforementioned approach was used for "three-cycle", "four-cycle" and "five-cycle" solution quenching operations.

3.2.6 SOLUTION-QUENCHED AT 1000°C WITHOUT SOAKING

The procedures described under 3.2.5 were used except that no soaking period in the furnace was allowed.

3.3 SAMPLE PREPARATION AFTER HEAT TREATMENT

The scales formed on the samples after heat-treatment were removed by filling and later polished on electronic polishing machine with silicon carbide paper of grade P220C. The samples were washed with jet of distilled water.

3.4 ELECTRODE POTENTIAL MEASUREMENTS

Scale and grease free samples obtained from various heat-treatment cycles were each completely immersed in 400ml beaker containing 0.5M H_2SO_4 solution. The electrode potentials for each sample were taken five times daily for the period of thirty days. Digital Multimeter model AVD890F ALDA 8900 series was used for measuring the electrode potentials with Zinc rod as the reference electrode. The values obtained from electrode potential measurement were converted to values to be obtained if Saturated Calomel Electrode (SCE) were to be used using the formula:

Electrode potential mV (SCE) = (Ezn – 1030) mV (Hibbert D. et al, 1984).

Where: Ezn - electrode potential values using high purity zinc as reference electrode

1030 – constant

mV – millivolt. (unit of measurement)

3.5 ESTIMATION OF CORROSION RATE FROM WEIGHT LOSS MEASUREMENT

Chemical weighing balance of model Mettler AE 160 was used to measure the weight loss of the samples. The initial weight of the samples were measured before being immersed in the 0.5M H_2SO_4 solution. They were withdrawn at 5 days interval, washed with distilled water and dried cleaned with cotton wool to avoid scratching of surfaces before re-weighing.

The corrosion rate (r) during the exposure time (T) is expressed as

$$r = \frac{W}{A\,(T/365)} \qquad (mg/cm^2/yr) \qquad \text{(Fontana, 1986)}$$

W = weight loss (mg)

A = total surface area (cm^2)

T/365 = Exposure time in days extrapolated to a year

A = 2[BL + BH + LH]

L = length (mm), B = Breadth (mm) and H = Height (mm)

For the used samples

L = 18mm B = 14mm, H = 12mm

Therefore

Area (A) = 1272mm^2 or 12.72cm^2

CHAPTER FOUR

RESULTS AND DISCUSSION

The results of the Electrode potentials versus exposure time and corrosion rates versus exposure time of as received and heat-treated austenitic stainless steel samples immersed in 0.5M H_2SO_4 solution are presented below:

4.1 ELECTRODE POTENTIALS AND CORROSION RATES AT VARIOUS EXPOSURE TIMES FOR NORMALISED SAMPLE AT 1000°C FOR 1 HOUR

Figure 4.1.1 shows the plot of electrode potentials versus exposure time and Figure 4.1.2 shows the plot of corrosion rates versus exposure time of as-received and normalised samples of austenitic stainless steel immersed in 0.5M H_2SO_4 solution at various cycles of normalisation held at 1000°C for 1 hour. It is observed that as-received sample displays highest electrode potentials at periods between 24 and 240 hours, followed by the values of electrode potentials from 4-cycle, 3-cycle, 2-cycle and 1-cycle specimens. The 5-cycle normalisation specimen indicates the least electrode potentials between the same periods. However, the electrode potentials increase sharply for all samples from –230 mV (SCE) to –80 mV (SCE) at periods between 450 and 750 hours with 1-cycle normalisation specimen displaying the highest electrode potentials and as-received specimen the least. A drop in electrode potentials is observed for as-received specimen at periods between 400 and 500 hours.

Whitney (1964), suggested that the initial corrosion attack may be due to the embedded particles of iron on the surfaces of the samples as a result of machining and mechanical polishing operations carried out before and after heat treatment operations

respectively. Shaweesh et al (2001) observed that iron contamination on stainless steel parts, components and systems is almost always confined to the surface. Most surface defects on stainless steel are produced during fabrication and are difficult to remove, thus contributing to corrosion. Surface defects tend to initiate corrosion that would not occur so readily in their absence.

It can be said that $0.5MH_2SO_4$ solution acted as passivating solution to the steel as passivity sets in after 10 days of exposure periods. According to Whitney's (1964) observation, in order to prevent initial corrosion, semi finished parts are given a passivation treatment. This treatment consists of immersing the stainless steel in a solution of inorganic acids (e.g. Nitric acid) to dissolve the embedded or smeared iron and restores the original corrosion-resistant surface by forming a thin, transparent oxide film (passive layer). Natural passivity effects would show up during tests to determine the effect of time and temperature on corrosion as revealed in Figure 4.1.2.

The electrode potentials drop observed for as-received sample between 400 and 500 hours exposure periods may be attributed to film break down; this quickly heals up owing to oxygen diffusion towards the metal surface - a character peculiar to stainless steel. The anodic curves in Figure 4.1.1 indicate that the material electrode potentials after 200 hours exposure periods is in a passive state and in a state of dynamic equilibrium of film partial breakdown and repair. This is also reflected in Figure 4.1.2, corrosion rates above 10 days exposure periods are less than $5mg/cm^2/yr$ for the rest of exposure time. At the exposure period above 240 hours (10 days) film dissolution rate is equal to the rate of film formation, the film stability have been reached and adherence force has increased thereby preventing the migration of metallic ion to the surface.

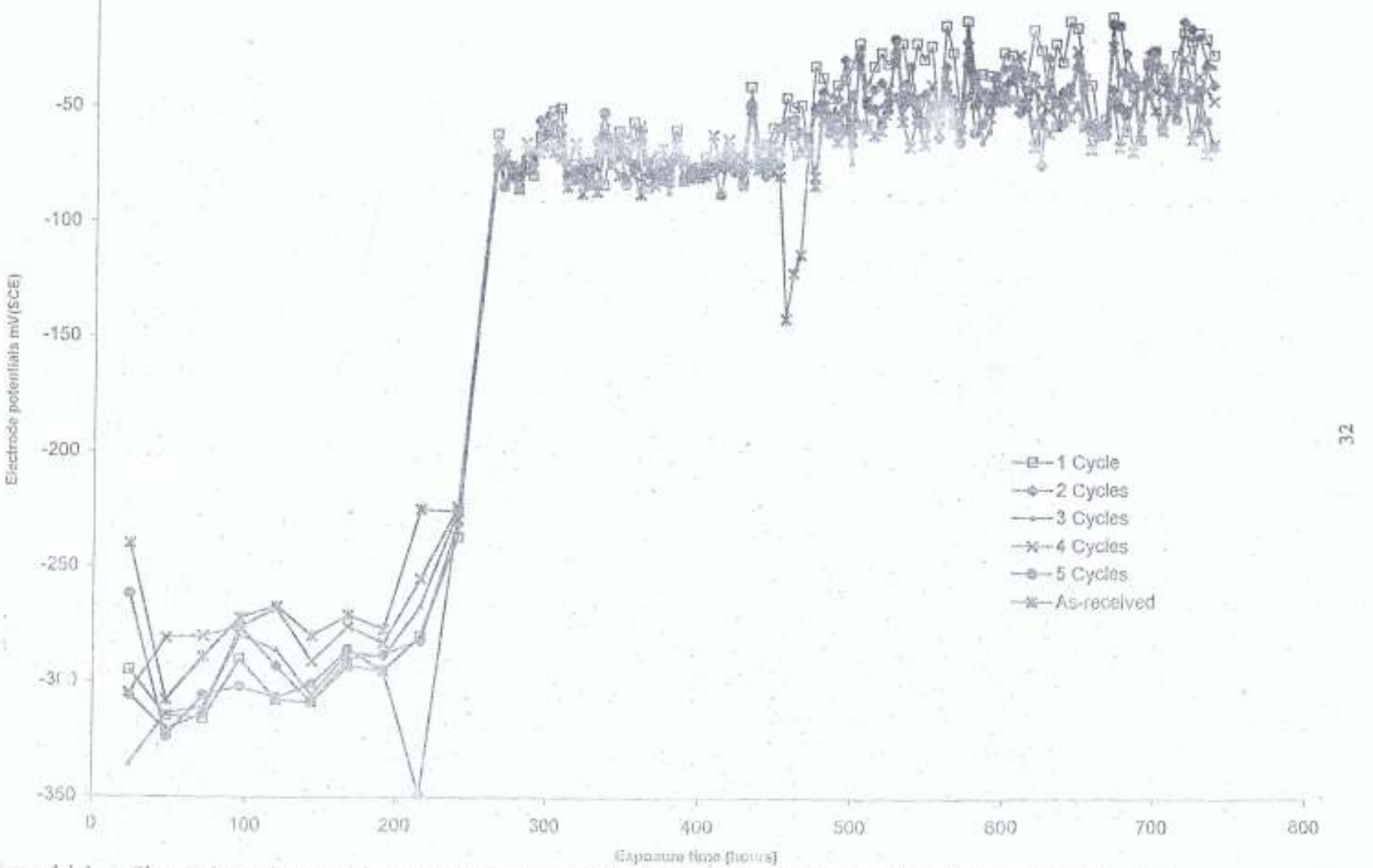
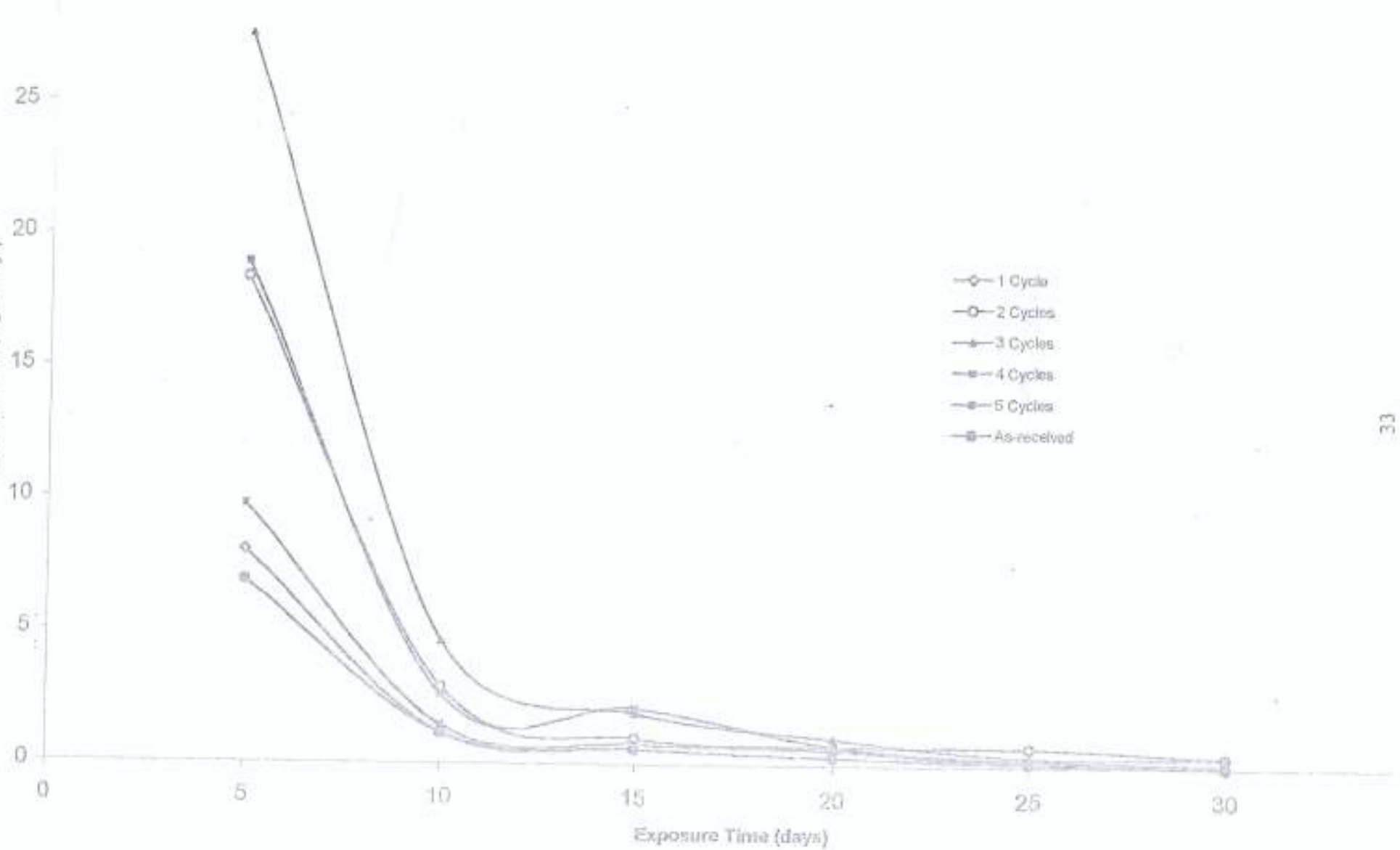


Figure 4.1.1 Plot of electrode potentials versus exposure time of as-received and normalised samples of austenitic stainless steel immersed in 0.5M H_2SO_4 solution at various cycles of heat-treatment held at 1000°C for 1 hour.



Plot of Corrosion Rate versus Exposure Time of as-received and Normalised samples of austenitic stainless steel immersed in 0.5 M H_2SO_4 solutions at various cycles of heat- treatment held at 1000°C for 1 hour.

4.2 ELECTRODE POTENTIALS AND CORROSION RATES AT VARIOUS EXPOSURE TIMES FOR NORMALISED SAMPLES AT 1000°C WITHOUT SOAKING

Figure 4.2.1 shows the plot of electrode potentials versus exposure time and Figure 4.2.2 shows the plot of corrosion rates versus exposure time of as-received and normalised samples of austenitic stainless steel immersed in 0.5M H_2SO_4 solution at various cycles of normalisation at 1000°C without soaking. It is observed that the electrode potentials in the decrease order is as follows: as-received, 1-cycle, 4-cycle, 3-cycle, 2-cycle and 5-cycle normalisation specimens at exposure periods between 24 and 240 hours. A general increase in electrode potential is observed for all the specimens from –235mV (SCE) to –38mV (SCE) at the exposure periods above 240 hours. The electrode potentials for the samples remain within the range of –58mV (SCE) and –8mV (SCE) at exposure period between 250 and 750 hours. Corrosion rates are observed to decrease sharply with the exposure time in this order: three-cycle, five-cycle, one-cycle, two-cycle, four-cycle normalisation specimens and as-received specimen features the least. All the corrosion rates tend towards zero after 10 days of the exposure periods.

The higher electrode potential and the lower corrosion rates of almost zero value observed after 240 hours (10 days) exposure periods is a pointer to the fact that the metal is not susceptible to corrosion at various cycles of heat-treatments. The corrosion experienced in the initial 10 days exposure periods is due to explanation given under section 4.1. Reports of Avery (1963) show that at higher temperatures sufficient to dissolve all the carbides into solution, the steel remains a single phase, and smaller sections can be cooled in air to keep dissolved carbides in solution.

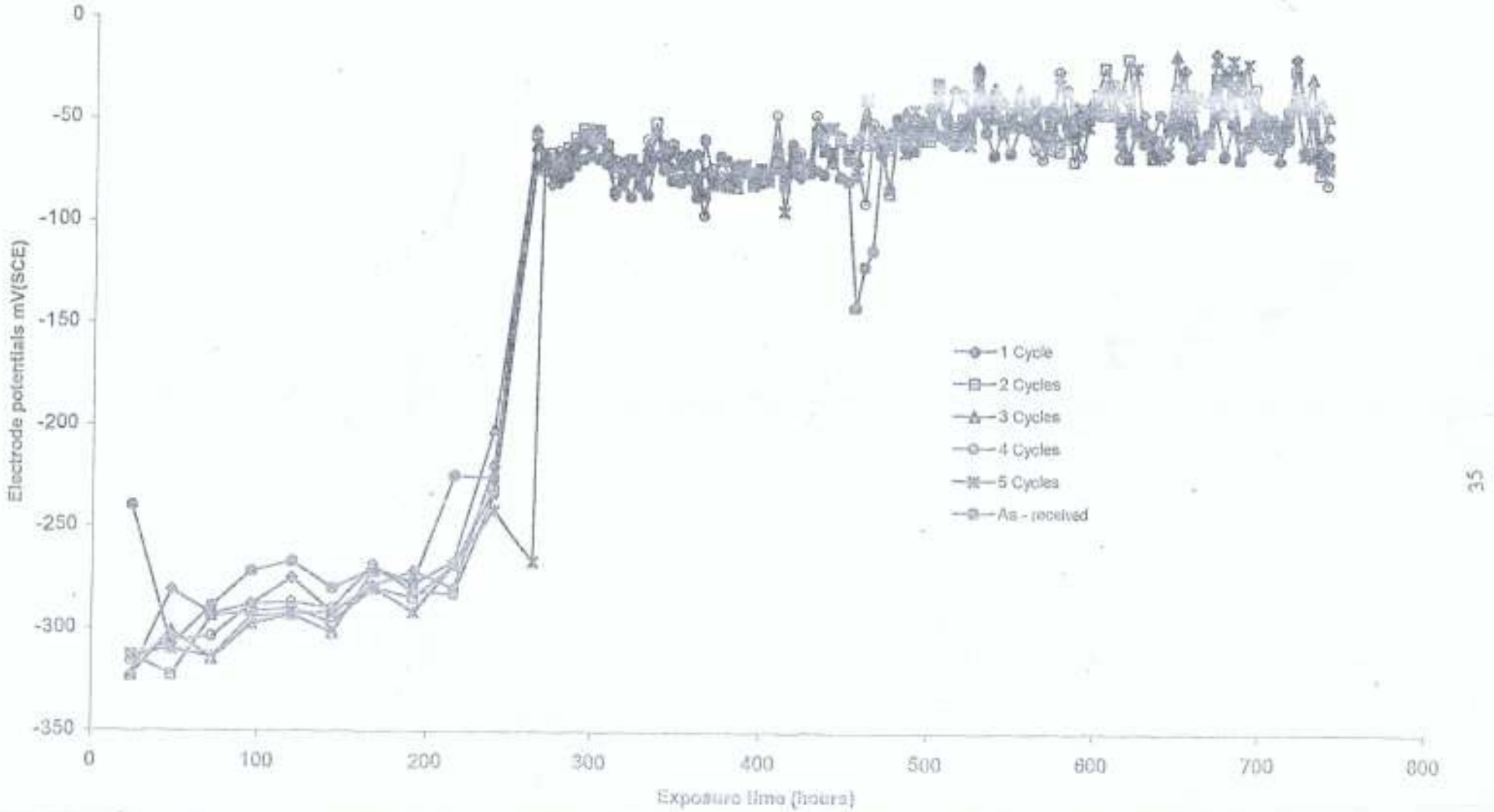
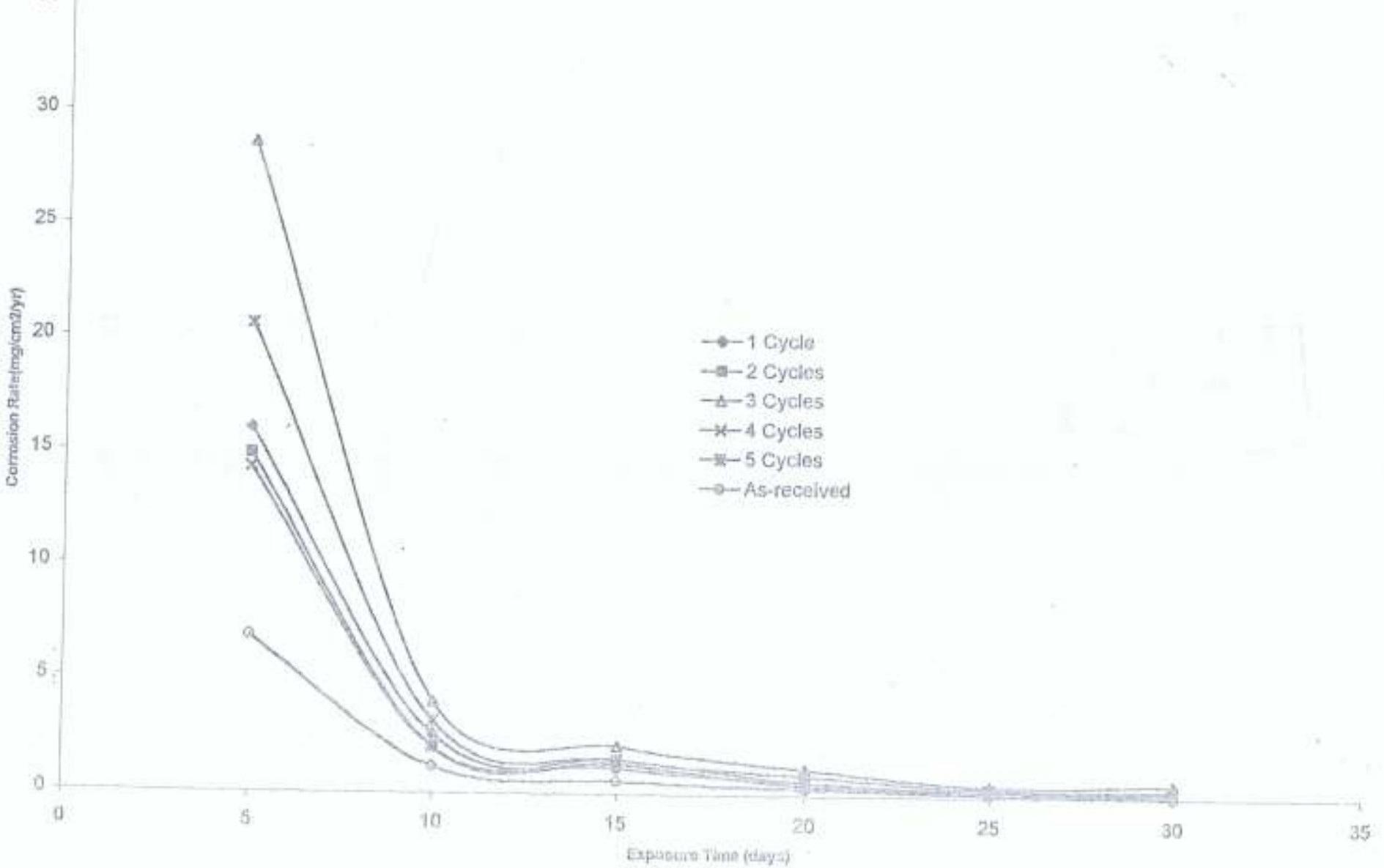


Figure 4.2.1: Plot of electrode potentials versus exposure time of as-received and normalised samples of austenitic stainless steel immersed in $0.5MH_2SO_4$ solution at various cycles of heat-treatment held at 1000°C without soaking.



e 4.2.2: Plot of Corrosion Rate versus Exposure Time of as-received and Normalised samples of austenitic stainless steel immersed in 0.5 M H_2SO_4 Solution at various cycle of heat – treatments at 1000°C (without soaking).

4.3 ELECTRODE POTENTIALS AND CORROSION RATES AT VARIOUS EXPOSURE TIMES FOR ANNEALED SAMPLES HELD AT 1000°C FOR 1 HOUR

Figure 4.3.1 shows the plot of electrode potentials versus exposure time and Figure 4.3.2 presents the plot of corrosion rates versus exposure time of as-received and annealed samples of austenitic stainless steel immersed in 0.5M H_2SO_4 solution at various cycles of annealing held at 1000°C for 1 hour. A progressive increase of electrode potentials from –340mV (SCE) to -220 mV (SCE) is observed for all the samples at the exposure periods between 24 and 240 hours. A sharp increase of electrode potential to –70mV (SCE) is noticed at 240 hours. The electrode potentials between –70mV (SCE) and –50mV (SCE) are maintained from the period of 300 to 450 hours. Further increase in electrode potentials from –50mV (SCE) to –24 mV (SCE) is observed at the exposure period between 450 and 750 hours for all the samples. From Figure 30, the corrosion rates decreases in the following order: three-cycle, two-cycle, five-cycle, four-cycle, one-cycle annealing specimens and as-received specimen displays the least.

Avery (1963) reported that during annealing, carbides, which markedly decrease resistance to intergranular corrosion are dissolved. The operating temperature of 1000°C is above the carbides re-precipitation temperature which is between 450°C and 860°C, also the 1 hour holding time observed is sufficient to keep all the carbides in solution. Therefore, the samples are fully homogenised. Homogenisation of the specimens result to an absence of galvanic cell when in contact with electrolyte. There is absence of galvanic corrosion that might take place due to heterogeneous phases.

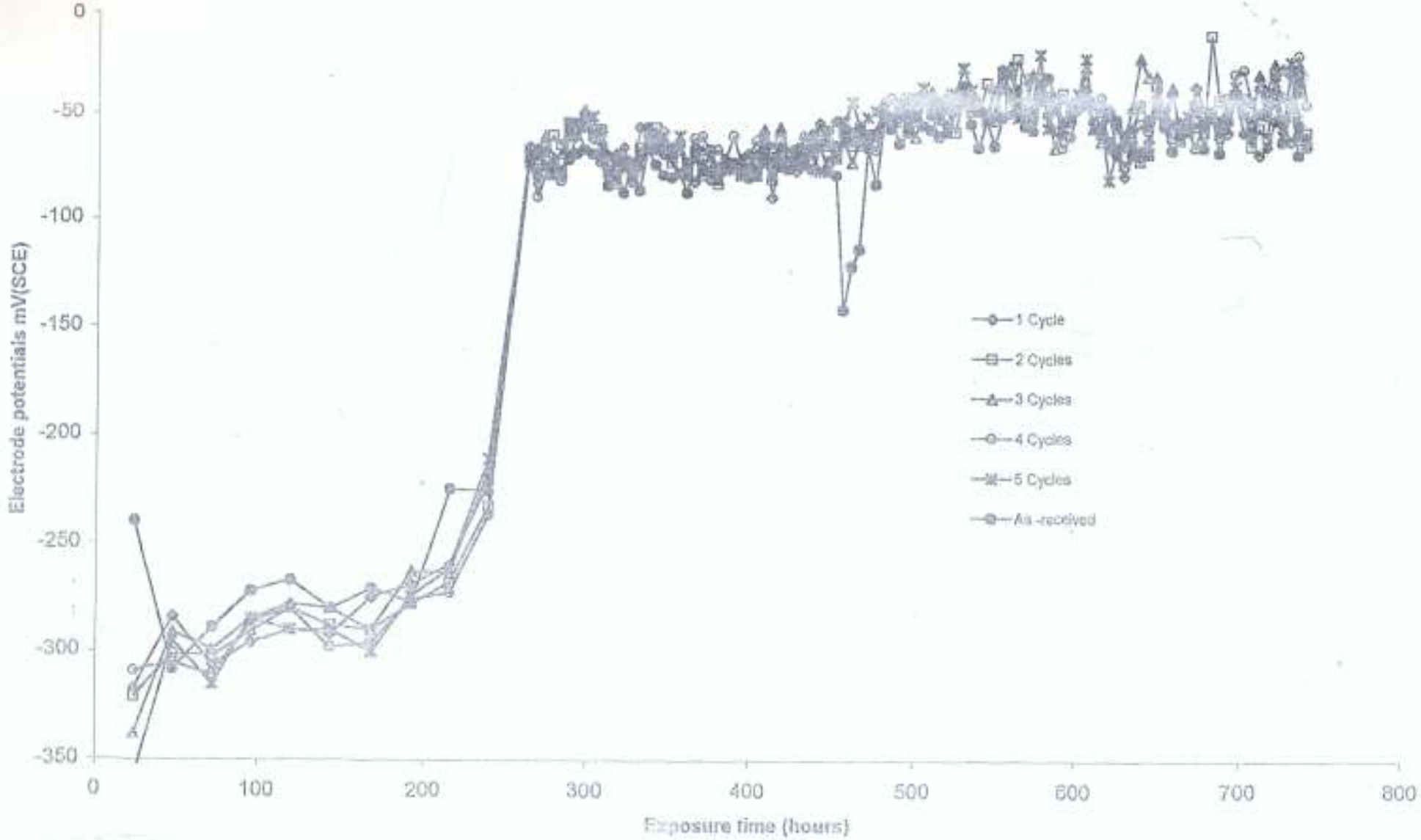
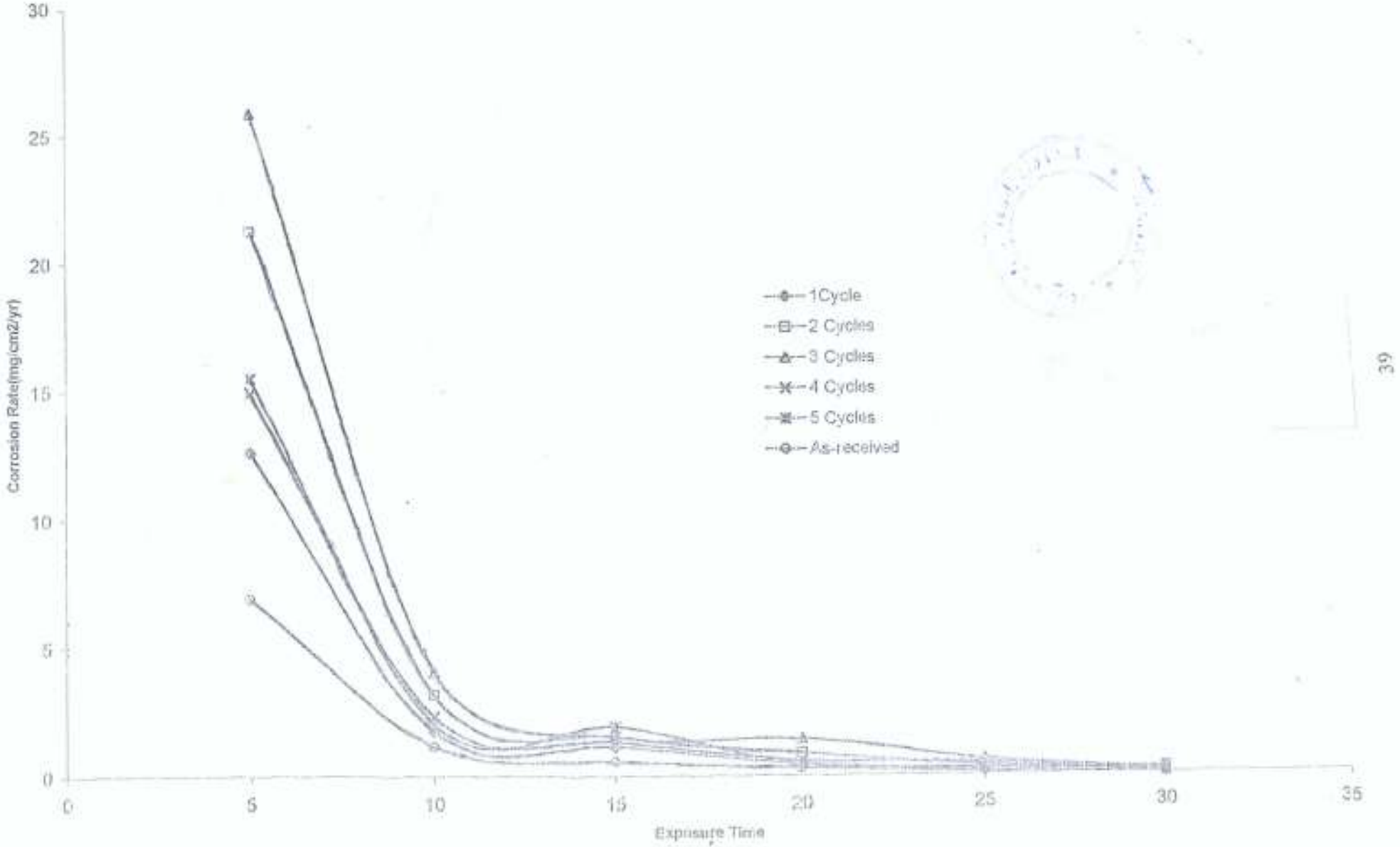


Figure 4.3.1: Plot of electrode potentials versus exposure time of as-received and annealed samples of austenitic stainless steel immersed in


Corrosion Rate(mg/cm2/yr)
Exposure Time
1Cycle
2 Cycles
3 Cycles
4 Cycles
5 Cycles
As-received
0
5
10
15
20
25
30
35

4.4 ELECTRODE POTENTIALS AND CORROSION RATES AT VARIOUS EXPOSURE TIMES FOR ANNEALED SAMPLES AT 1000°C WITHOUT SOAKING

Figure 4.4.1 shows the electrode potentials versus exposure time and Figure 4.4.2 shows the plot of corrosion rates versus exposure time of as-received and annealed samples of austenitic stainless steel immersed in 05.M H_2SO_4 solution at various cycle of annealing at 1000°C without soaking. The highest electrode potential is observed for 2-cycle annealed specimen at the exposure periods between 400 and 750 hours. Corrosion rates are observed to decrease in the order of four-cycle, two-cycle, five-cycle and one-cycle annealing specimens. All the corrosion rates tend towards zero after 10 days of exposure to the end of the exposure periods. These later values of electrode potentials and corrosion rates are related to the existence of a passive layer, that is, all the specimens formed a more stable film on the surface.

Lakhtin (1979), Dell (1981), and John (1994), showed that austenitic stainless steels usually alloyed by chromium and nickel (or manganese), have an austenite structure after being cooled to room temperature. This structure enhances good corrosion resistance in oxidizing media. Furthermore, chromium makes its electrochemical potential positive and it becomes resistant to corrosion in many acids. From the report of Chamberlain et al (1996), the presence of chromium greater than 12 percent in steel significantly improves the corrosion resistance, hence, the term 'stainless' steels. Contrary to the findings of Sangdall (1964), it appears that precipitation of carbides is suppressed at very slow cooling at various cycles of heat treatments. This may be due to the higher percentages of nickel and chromium in the specimens. Also there could be enough chromium in the matrix to resist corrosion in the acidic media should carbides precipitation occurs with 0.06 percent carbon present in the specimens.

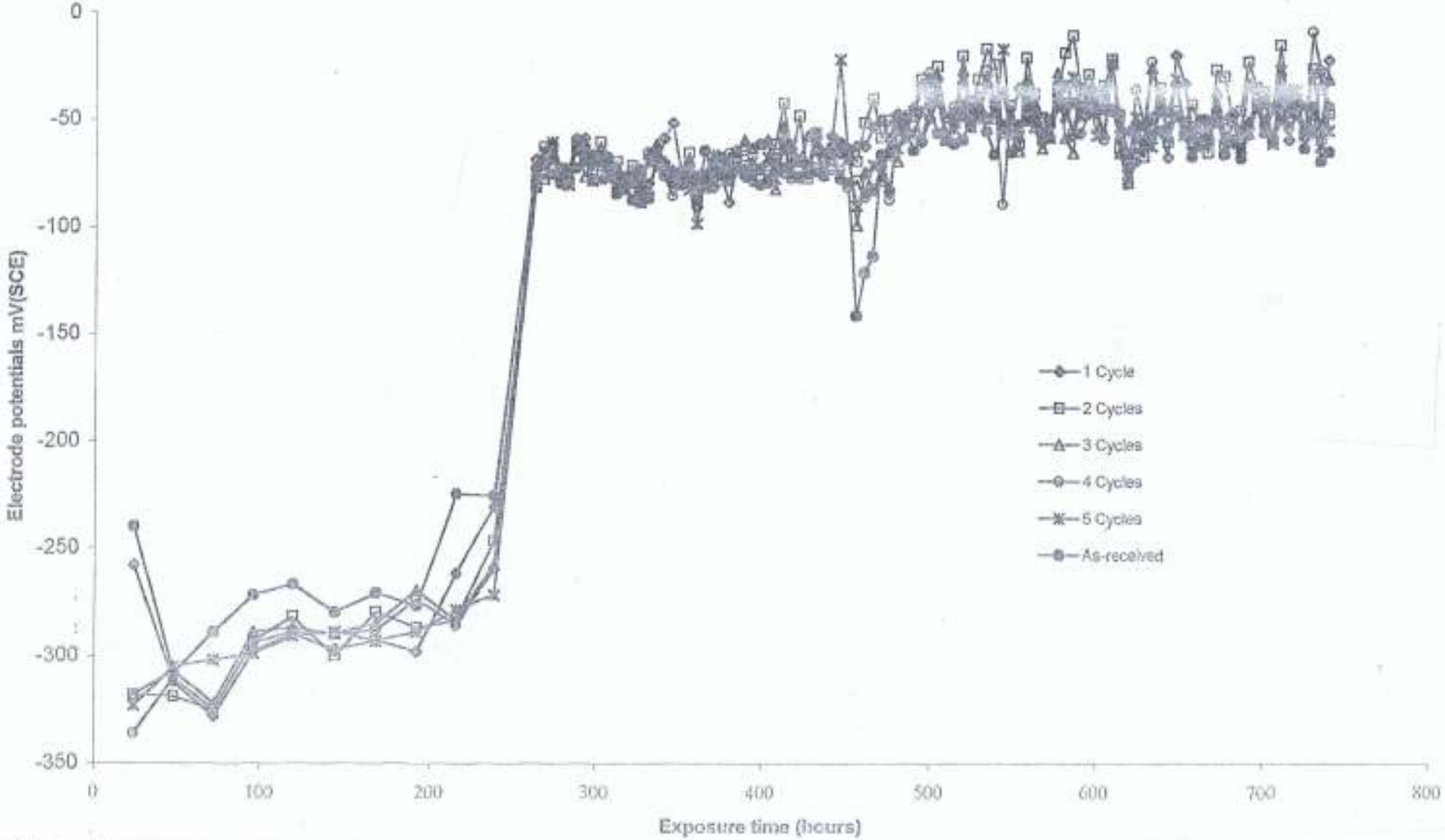
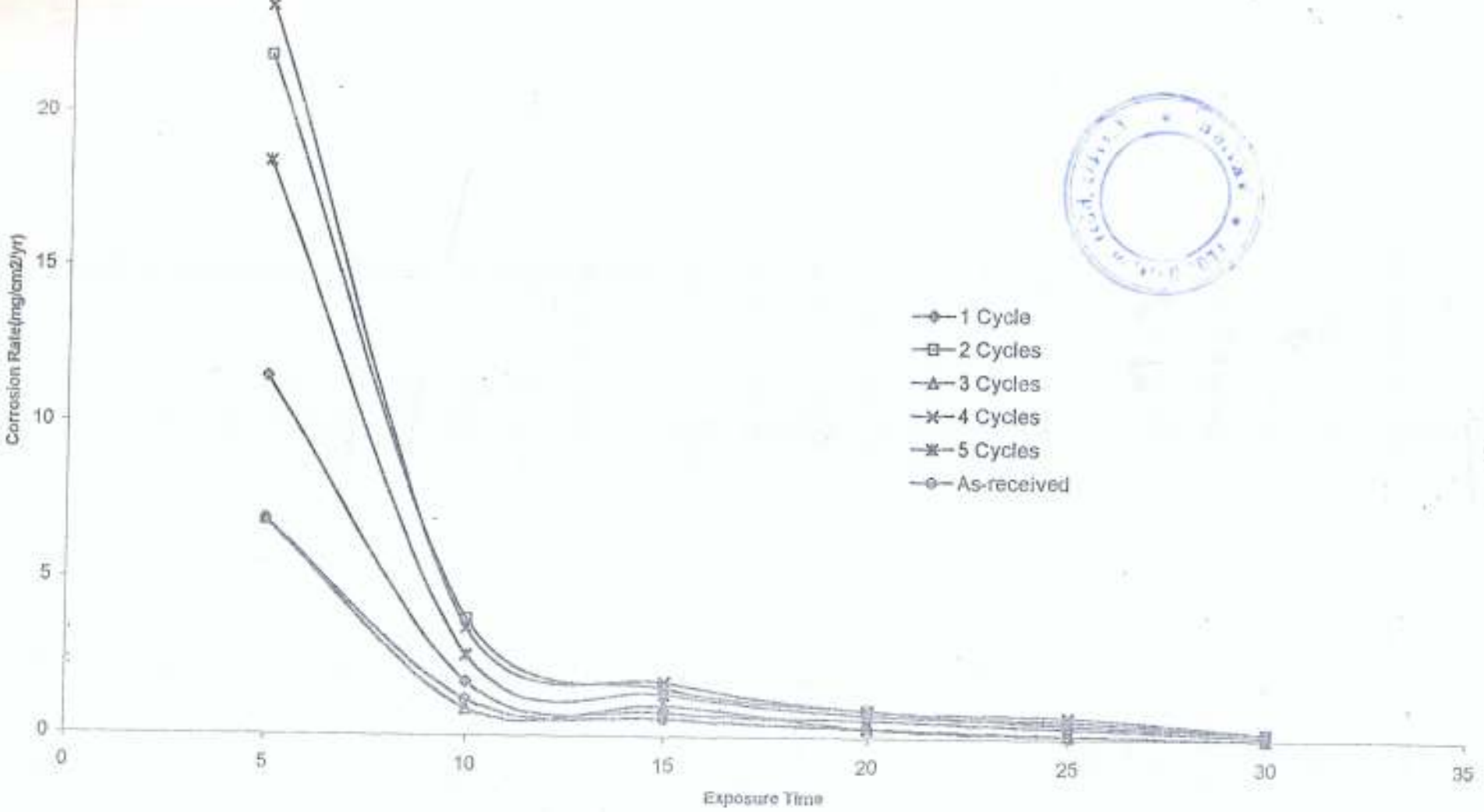


Figure 4.4.1: Plot of electrode potentials versus exposure time of as-received and annealed samples of austenitic stainless steel immersed in 0.5MH$_2$SO$_4$ solution at various cycles of heat treatment



4.4.2: Plot of Corrosion Rate versus Exposure Time of as-received and annealed samples of austenitic stainless steel immersed in 0.5 M H_2SO_4 Solution at various cycle of heat – treatments at 1000°C (without soaking).

4.5 ELECTRODE POTENTIALS AND CORROSION RATES AT VARIOUS EXPOSURE TIMES FOR SOLUTION-QUENCHED SAMPLES HELD AT 1000°C FOR 1 HOUR

Figure 4.5.1 shows the plot of electrode potentials versus exposure time and Figure 4.5.2 presents the plot of corrosion rates versus exposure time of as-received and solution-quenched samples of austenitic stainless steel immersed in $0.5MH_2SO_4$ solution at various cycle of solution-quenching held at 1000°C for 1 hour. A general increase of electrode potential from -340mV (SCE) to –230mV is observed for all the specimens at the exposure time between 24 and 240 hours. Further rise of electrode potential to –80mV (SCE) is observed at period over 240 hours. All the samples maintained electrode potentials ranging from –80mV (SCE) to –10 mV (SCE) at periods between 300 and 750 hours. It is observed in Figure 4.5.2 that three-cycle and four-cycle solution-quenched specimens feature the same corrosion rates, also two-cycle and one-cycle specimens have the same corrosion rates. Corrosion rates decrease in the order of five-cycle, two-cycle, three-cycle specimens and as-received specimen displays the least.

The above observations agreed with the findings of Fontana (1986) and Avery (1963) that austenitic alloys achieve maximum resistance to intergranular corrosion by the high temperature heating and quenching procedure known as solution-annealing or solution-quenching. Holding the temperature at 1000°C for 1 hour is sufficient to accomplish complete solution of carbides. Quenching at a fast rate in water has prevented the re-precipitation of the carbides; hence a higher resistance to corrosion is observed in Figure 4.5.1. This also supports the argument of Chamberlain et al (1996) that fast cooling in water of austenitic stainless steel from above 1000°C suppresses carbide $\{(FeCr)_{23}C_6\}$ formation in samples containing greater than 0.03 percent carbon.

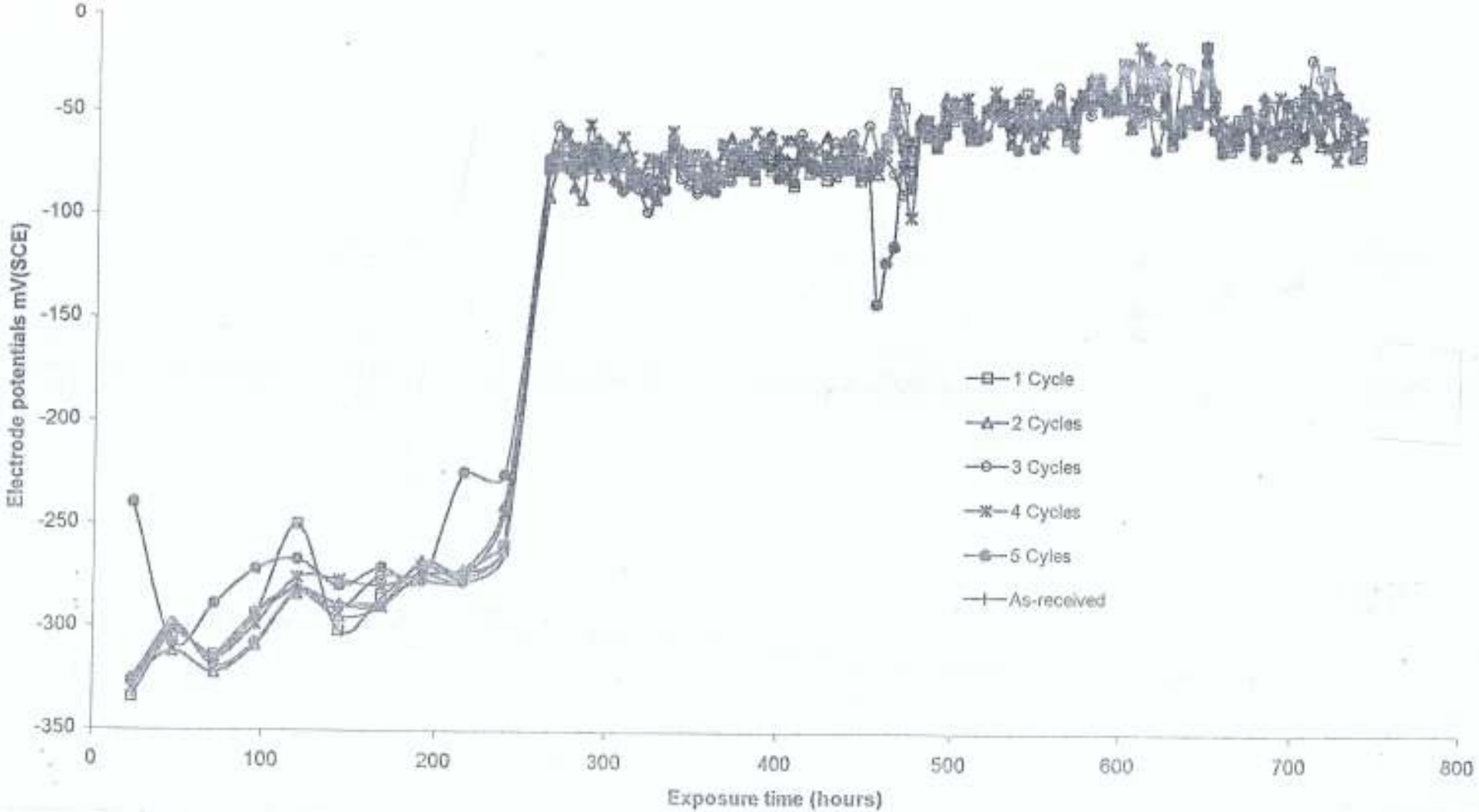
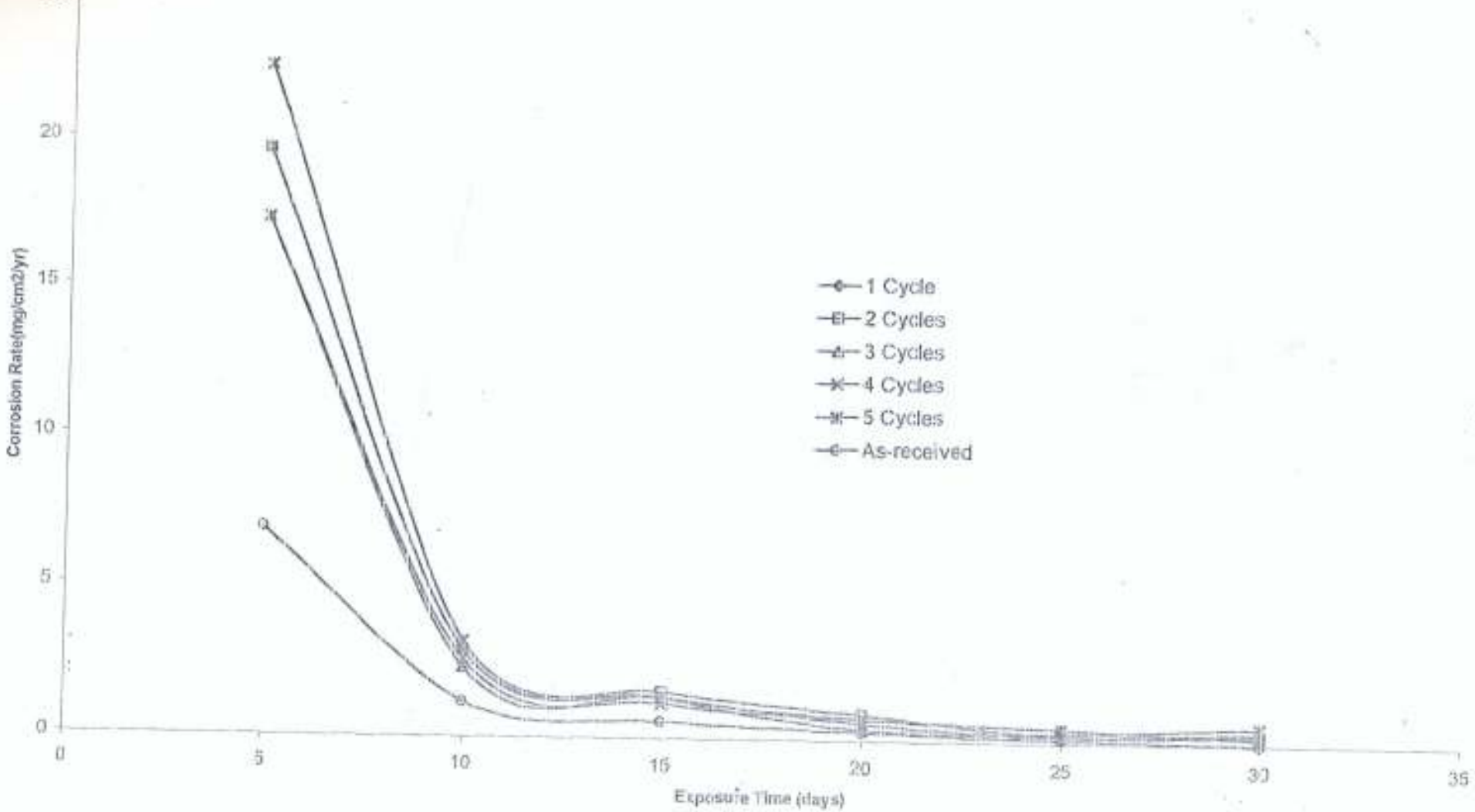


Figure 4.5.1: Plot of electrode potentials versus exposure time of as-received and solution-quenched samples of austenitic stainless steel immersed in $0.5MH_2SO_4$ solution at various cycles of heat-treatment held at 1000°C for 1 hour.



4.5.2: Plot of Corrosion Rate versus Exposure Time of as-received and solution - quenched samples of austenitic stainless steel immersed in 0.5 M H_2SO_4 Solution at various cycle of heat -treatments held at 1000°C for 1 hour.

ELECTRODE POTENTIALS AND CORROSION RATES AT VARIOUS EXPOSURE TIMES FOR SOLUTION-QUENCHED SAMPLES AT 1000°C WITHOUT SOAKING

Figure 4.6.1 displays the plot of electrode potential versus exposure time and e 4.6.2 shows the plot of corrosion rates versus exposure time of as-received and on-quenched samples of austenitic stainless steel immersed in $0.5MH_2SO_4$ solution ious cycle of solution-quenching at 1000°C without soaking. Figure 4.6.1 is similar gure 4.5.1, however, 2-cycle solution-quenched specimen maintain the highest ode potential at the exposure times between 240 and 750 hours. Initial electrode tial drop from –260mV (SCE) to –348mV (SCE) is observed for all the samples at sure periods between 24 and 48 hours. Electrode potential drop is observed for both s-received and 3-cycle specimens at the periods between 450 and 500 hours. The sion rates for three-cycle, one-cycle, four-cycle and five-cycle specimens do not ase sharply as in normalised and annealed specimens. However, the highest sion rate is observed for two-cycle, solution-quenched specimen. In Figure 4.6.2, sion rates decrease with increasing exposure times and tend towards zero from 10 to 30 days.

Corrosion attack is prominent in the initial 10 days exposure periods as electrode ntials are more of negative values. This might be due to explanation given under on 4.1. Although passivity sets in after 10 days exposure periods, in the case of a metal free from all production defects, there can be selective corrosion attack at boundaries. The natural passivity that must have set in after 10 days exposure ds caused the drastic reduction in the corrosion rates as shown in Figure 4.6.2.

The minimum corrosion rate is observed for three-cycle solution-quenched specimen. The corrosion rate remains below that of the as-received specimen. This may be attributed to a maximum homogenisation of the specimen at the end of the third-cycle solution-quenching operations. The fast quenching in water has suppressed re-precipitation of chromium carbides along the grain boundaries. Third-cycle solution-quenching operation markedly enhances the resistance of austenitic stainless steel of the composition in Table 3.1 to intergranular corrosion.

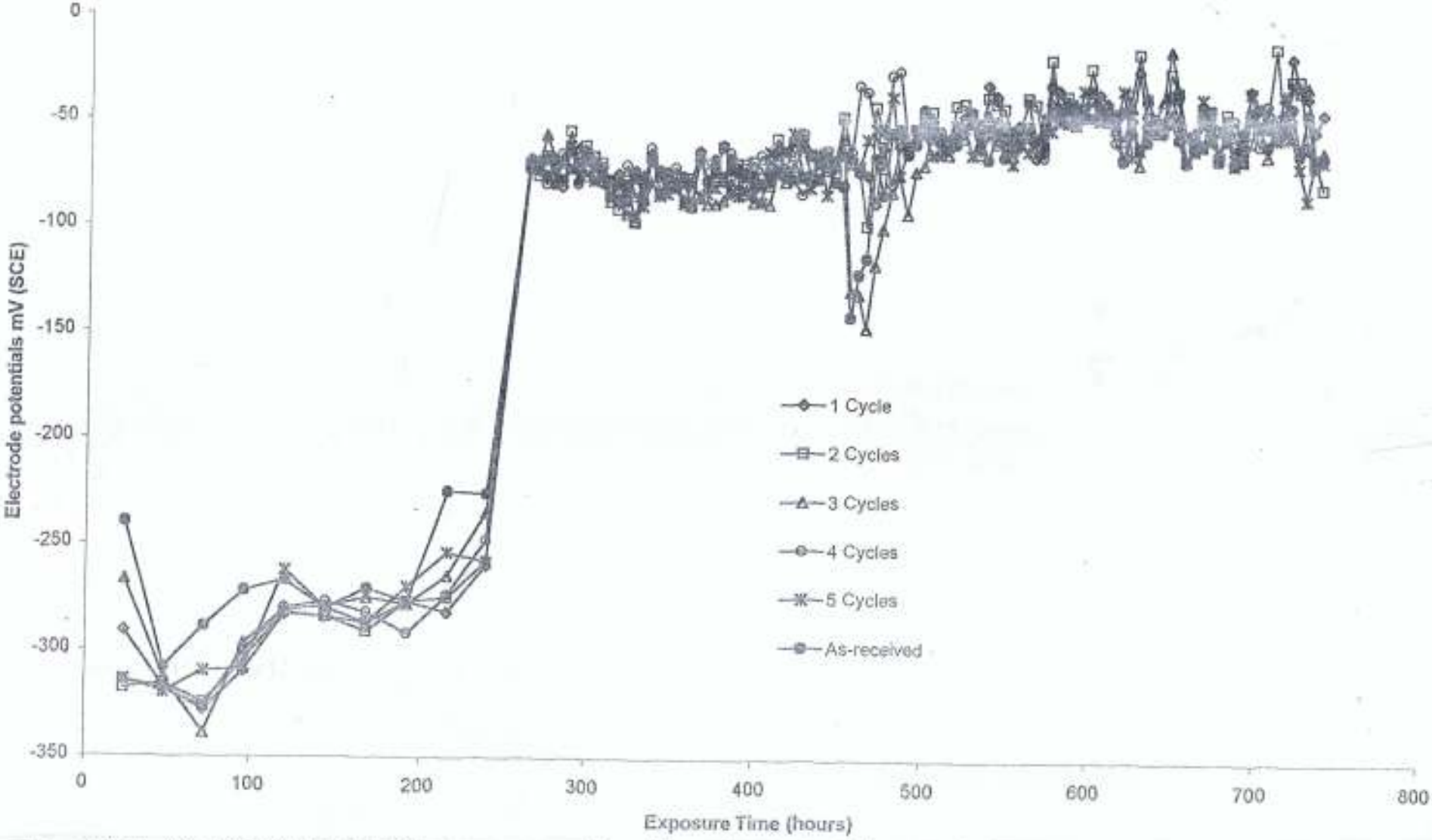
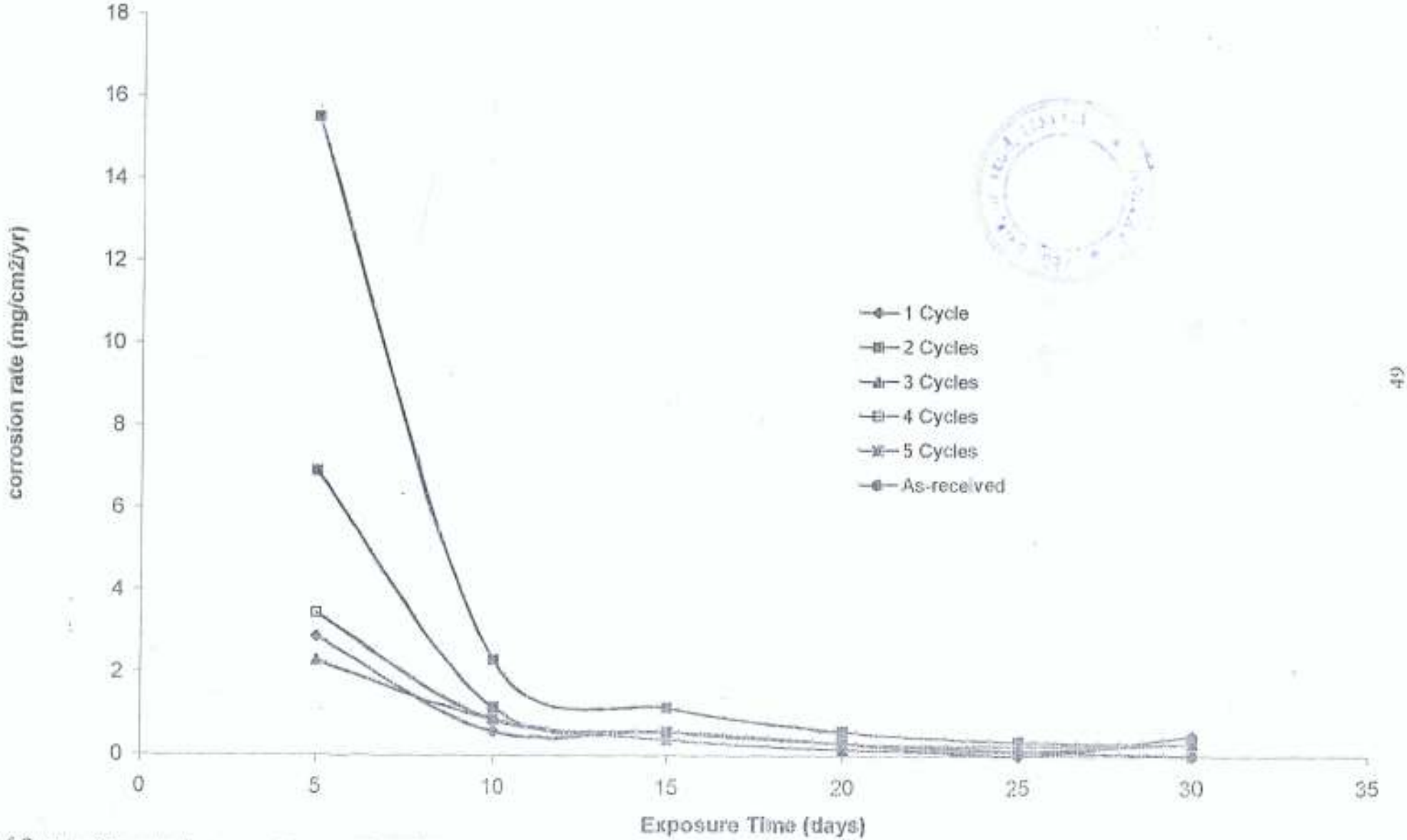


Figure 4.6.1: Plot of electrode potentials versus exposure time of as-received and solution-quenched samples of austenitic stainless steel immersed in $0.5MH_2SO_4$ solution at various cycles of heat-treatment held at 1000°C without soaking.



4.6.2: Plot of Corrosion Rate versus Exposure Time of as-received and Solution-quenched samples of austenitic stainless steel immersed in 0.5 M H_2SO_4 Solution at various cycle of eat -treatments at 1000°C (without soaking).

4.7 COMPARATIVE RESULTS OF ELECTRODE POTENTIALS AND CORROSION RATES AT VARIOUS EXPOSURE TIMES AT DIFFERENT CYCLES OF HEAT-TREATMENT CARRIED OUT AT 1000°C FOR 1 HOUR

Figure 4.7.1 presents the comparative plot of electrode potential versus exposure time of specimens subjected to one cycle of the different heat-treatment. It is observed that the normalised specimen has the least electrode potentials at exposure time between 48 and 240 hours. It maintains the highest electrode potentials ranging from –80mV (SCE) to –11mV (SCE) at the exposure period between 250 and 750 hours. The as-received specimen displays a decrease in electrode potential within the initial 48 hours from –240mV (SCE) to –310mV (SCE) which later increases to –230mV (SCE) at 240 hours. The as-received and annealed samples display the same electrode potentials whose values range between –80mV (SCE) and –50mV (SCE) at the exposure periods between 240 and 750 hours. Solution-quenched samples display electrode potentials whose values lie between those of normalised and annealed samples at the same exposure periods.

Figures 4.7.2, 4.7.3, 4.7.4 and 4.7.5 present the comparative electrode potentials versus exposure time plots for two-cycle, three-cycle, four-cycle and five-cycle of heat-treatments respectively. At the periods between 250 and 450 hours, annealed samples display the highest electrode potentials while the as-received and solution-quenched specimens feature the least. At the exposure periods between 450 and 750 hours, normalised, annealed and solution-quenched specimens maintain peak electrode potentials. The electrode potentials in the range of –340mV (SCE) and –230mV (SCE) are observed to occur between 24 and 240 hours for all the samples.

Figure 4.7.6 presents the comparative plot of corrosion rates versus exposure time of specimens subjected to on-cycle of the different heat-treatments. It is observed that corrosion rates decrease sharply for all the specimens in the initial 10 days of exposure periods and tend towards zero value from 15 days to 30 days exposure periods. Initial highest corrosion rate is observed for solution-quenched specimen followed by annealed specimen while normalised as well as the as-received specimens display the least corrosion rates. As presented in Figure 4.7.7, the annealed specimen displays the initial highest corrosion rates followed by solution-quenched and normalised specimens. The as-received specimen features the least corrosion rates. The plot of the corrosion rates for three-cycle of various heat-treatments is presented in Figure 4.7.8. Normalised specimen displays the highest corrosion rates followed by annealed and solution-quenched specimens. The as-received specimen displays the least corrosion rates. Figures 4.7.9 and 4.7.1.0 present the comparative corrosion rates versus exposure time plots for four-cycle and five-cycle of heat-treatments respectively. Normalised specimen displays the initial highest corrosion rates followed by solution-quenched and annealed specimen as shown in Figure 4.7.9. Solution-quenched specimen displays the initial highest corrosion rates followed by corrosion rate value from annealed and normalised specimens as shown in Figure 4.7.1.0. The as-received specimen features the least corrosion rate. However, all the specimens display the corrosion rates that tend towards zero value at the exposure periods beyond 20 days.

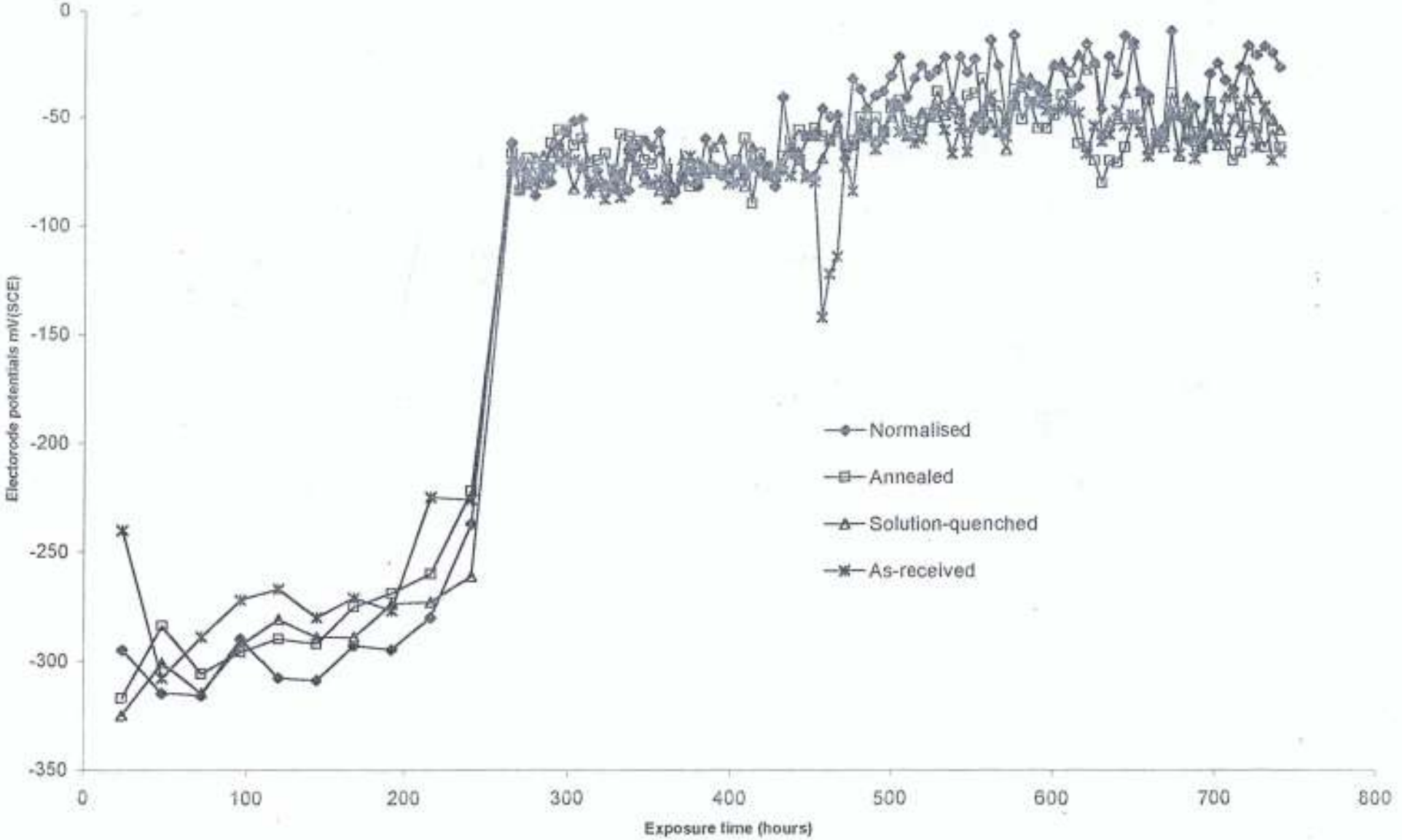


Figure 4.7.1: Plot of Electrode Potentials versus Exposure time of one-cycle heat-treated austenitic stainless steel samples held at 1000°C for 1 hour, immersed in $0.5MH_2SO_4$ solution.

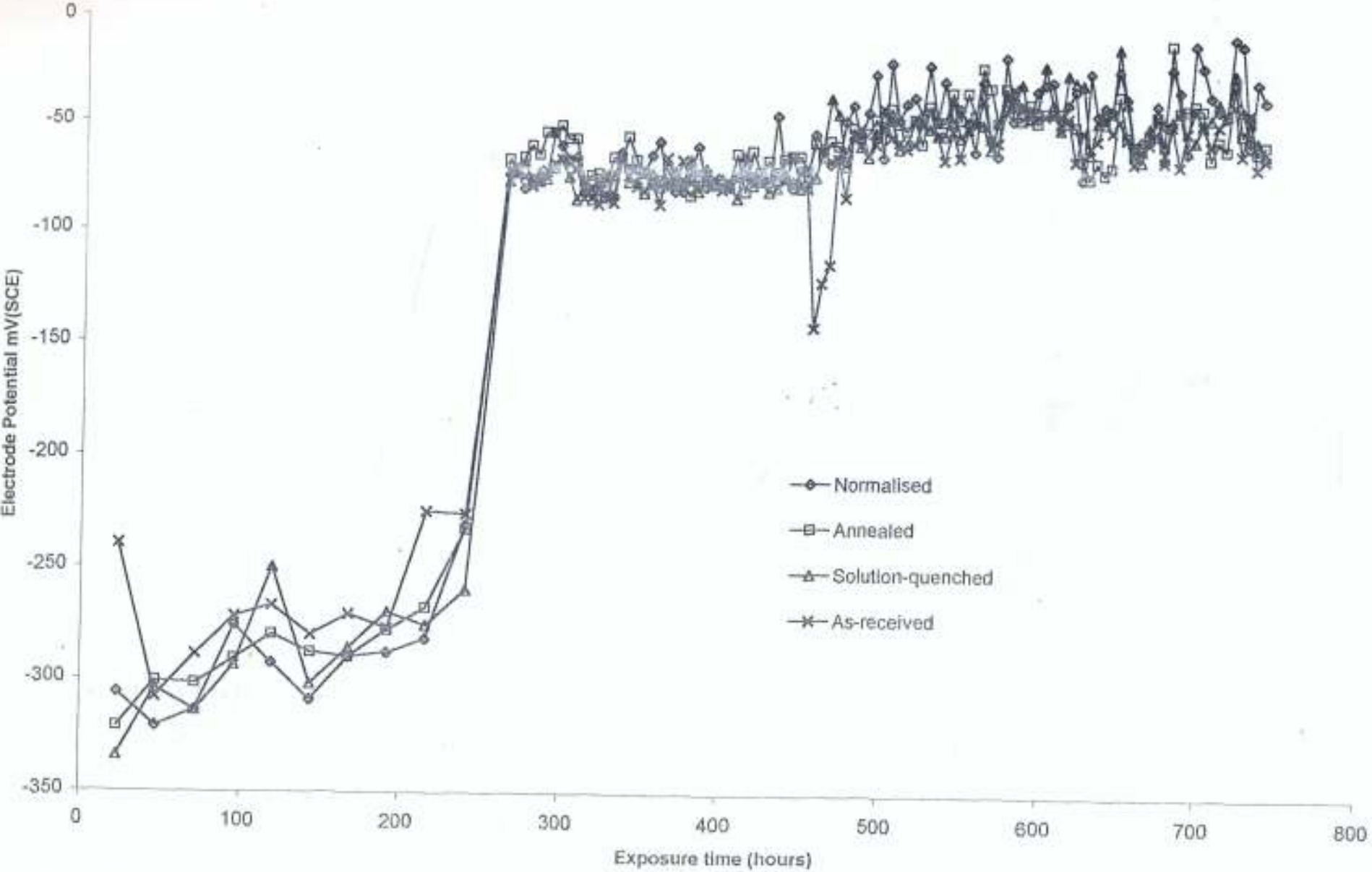


Figure 4.7.2: Plot of Electrode Potentials versus Exposure time of two-cycle heat-treated austenitic stainless steel samples held at 1000°C for 1 hour, immersed in 0.5M H_2SO_4 solution

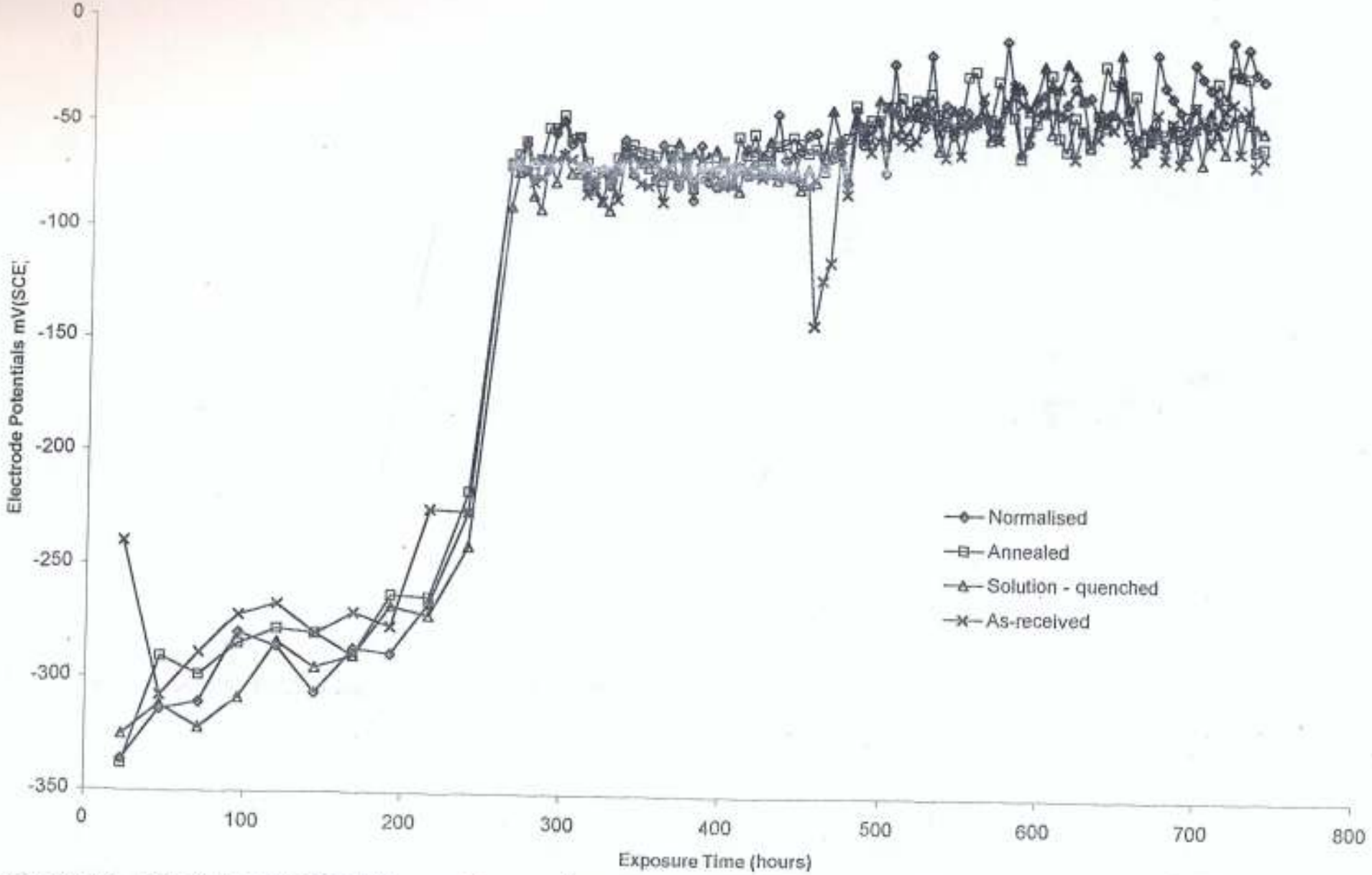


Figure 4.7.3: Plot of Electrode Potentials versus Exposure time of three-cycle heat-treated austenitic stainless steel samples held at 1000°C for 1 hour, immersed in $0.5MH_2SO_4$ solution.

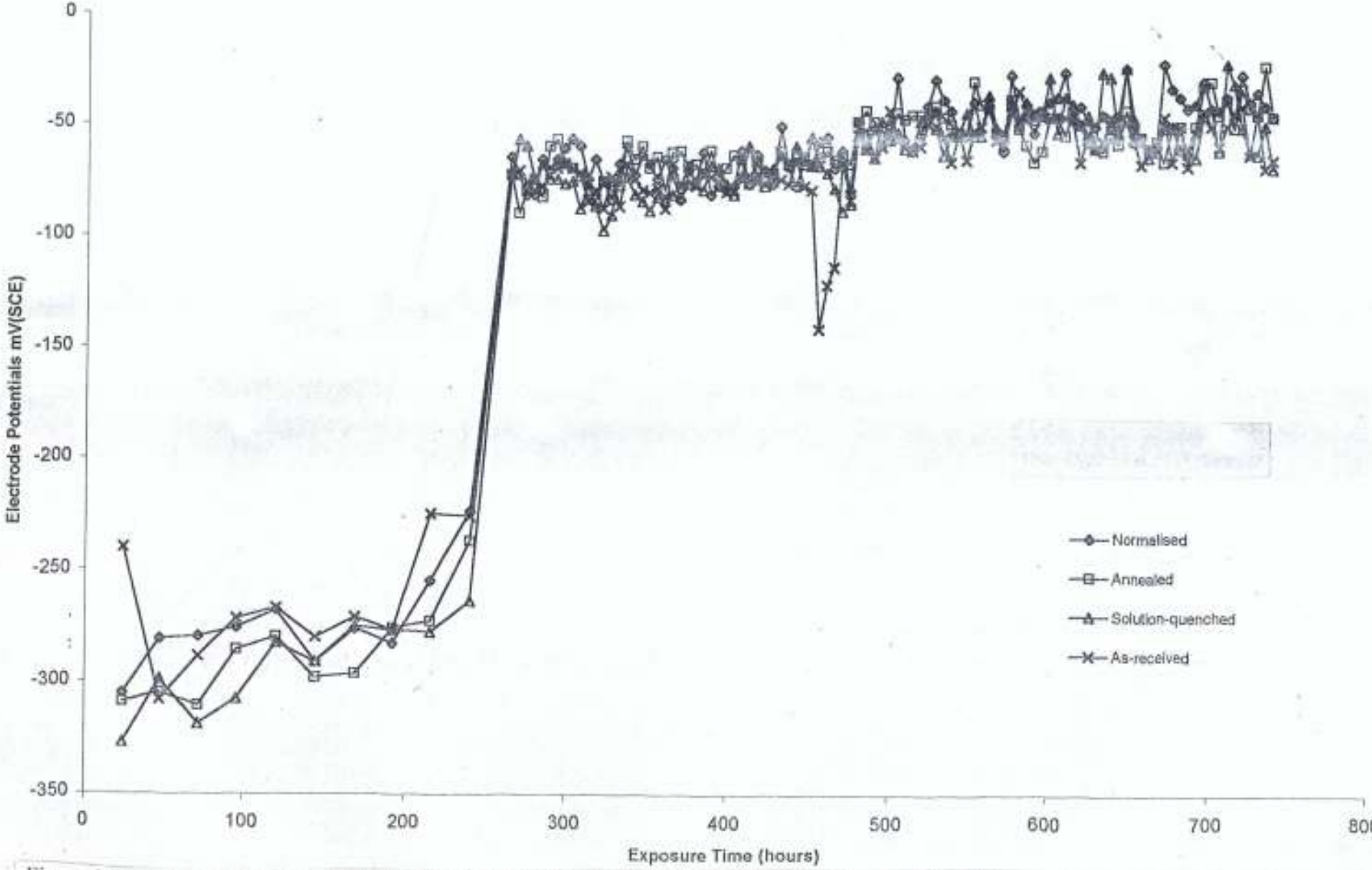


Figure 4.7.4: Plot of Electrode Potentials versus Exposure time of four-cycle heat-treated austenitic stainless steel samples held at 1000°C

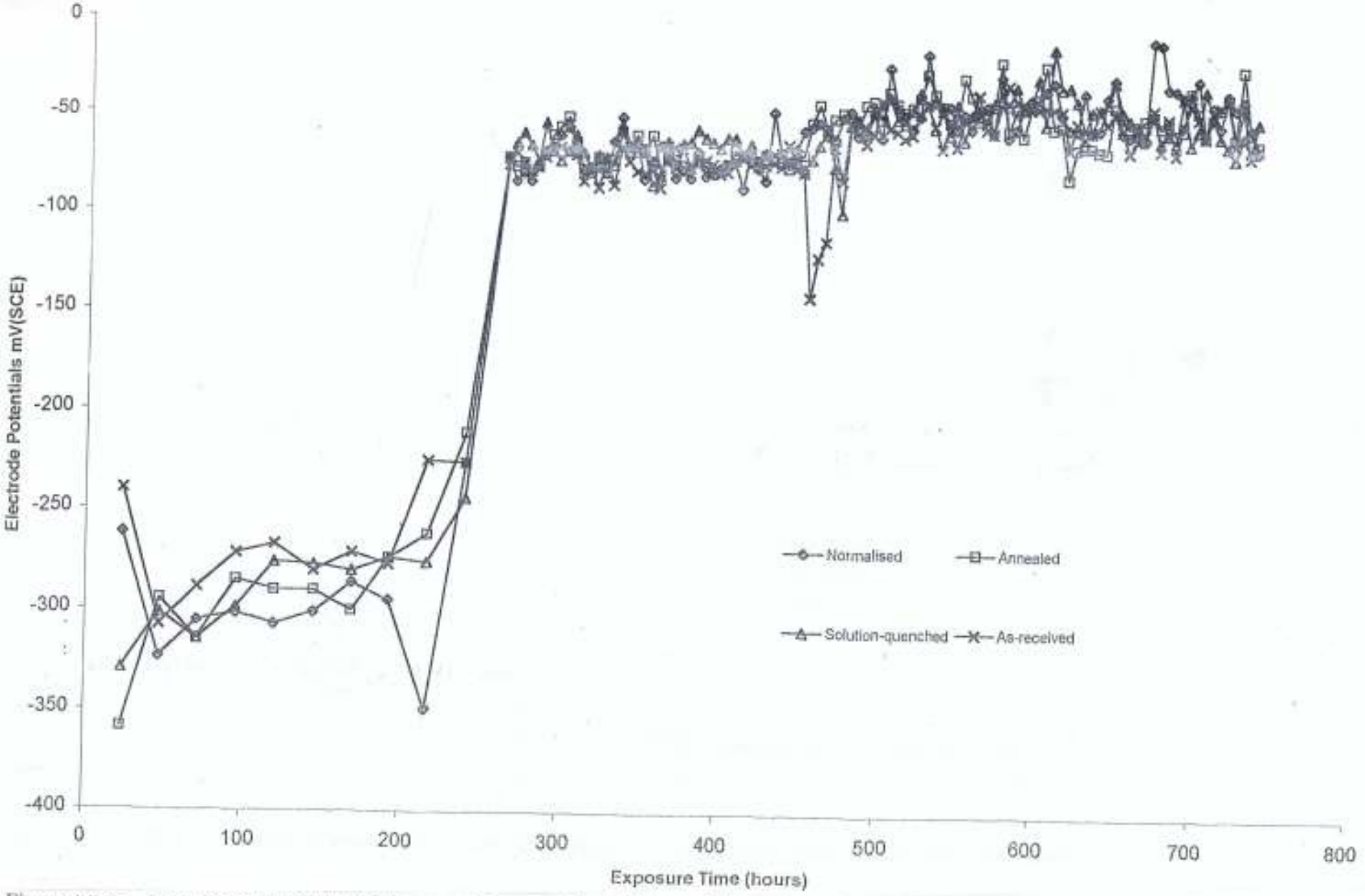


Figure 4.7.5: Plot of Electrode Potentials versus Exposure time of five-cycle heat-treated austenitic stainless steel samples held at 1000°C

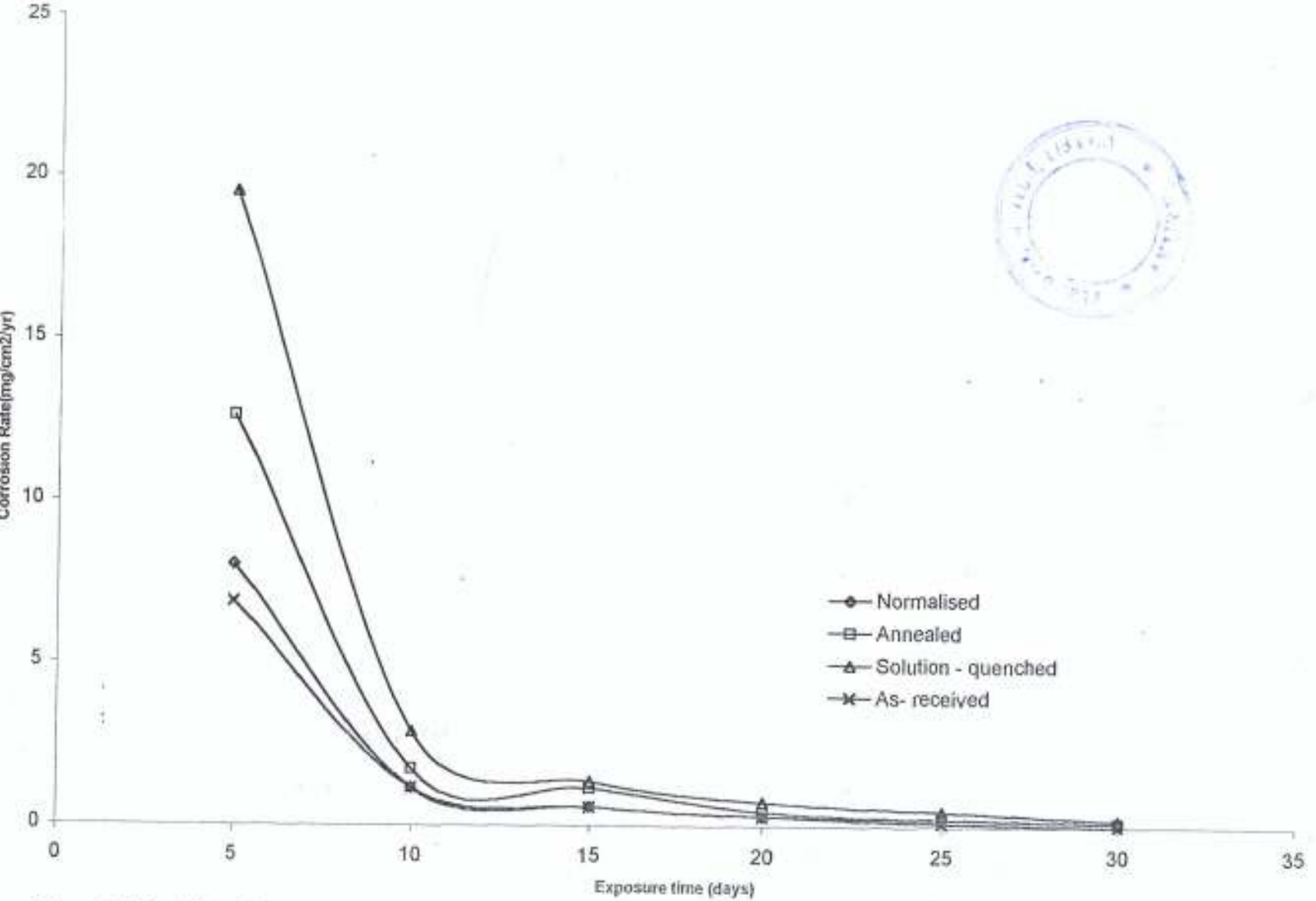


Figure 4.7.6: Plot of corrosion rates versus Exposure time of one-cycle heat-treated austenitic stainless steel samples held at 1000°C for 1 hour, immersed in $0.5MH_2SO_4$ solution.

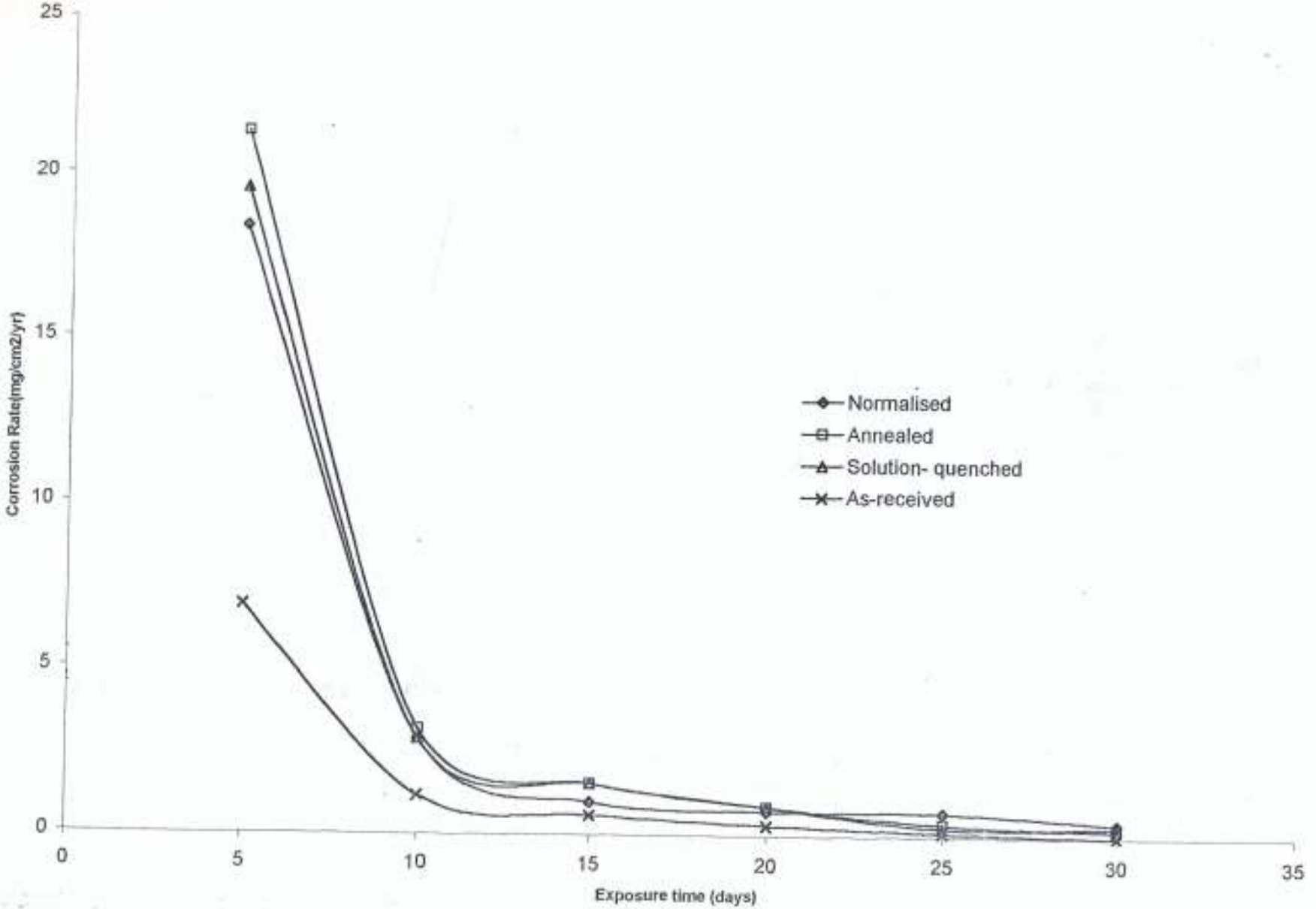


Figure 4.7.7: Plot of Corrosion Rates versus Exposure time of two-cycle heat-treated austenitic stainless steel samples held at 1000°C

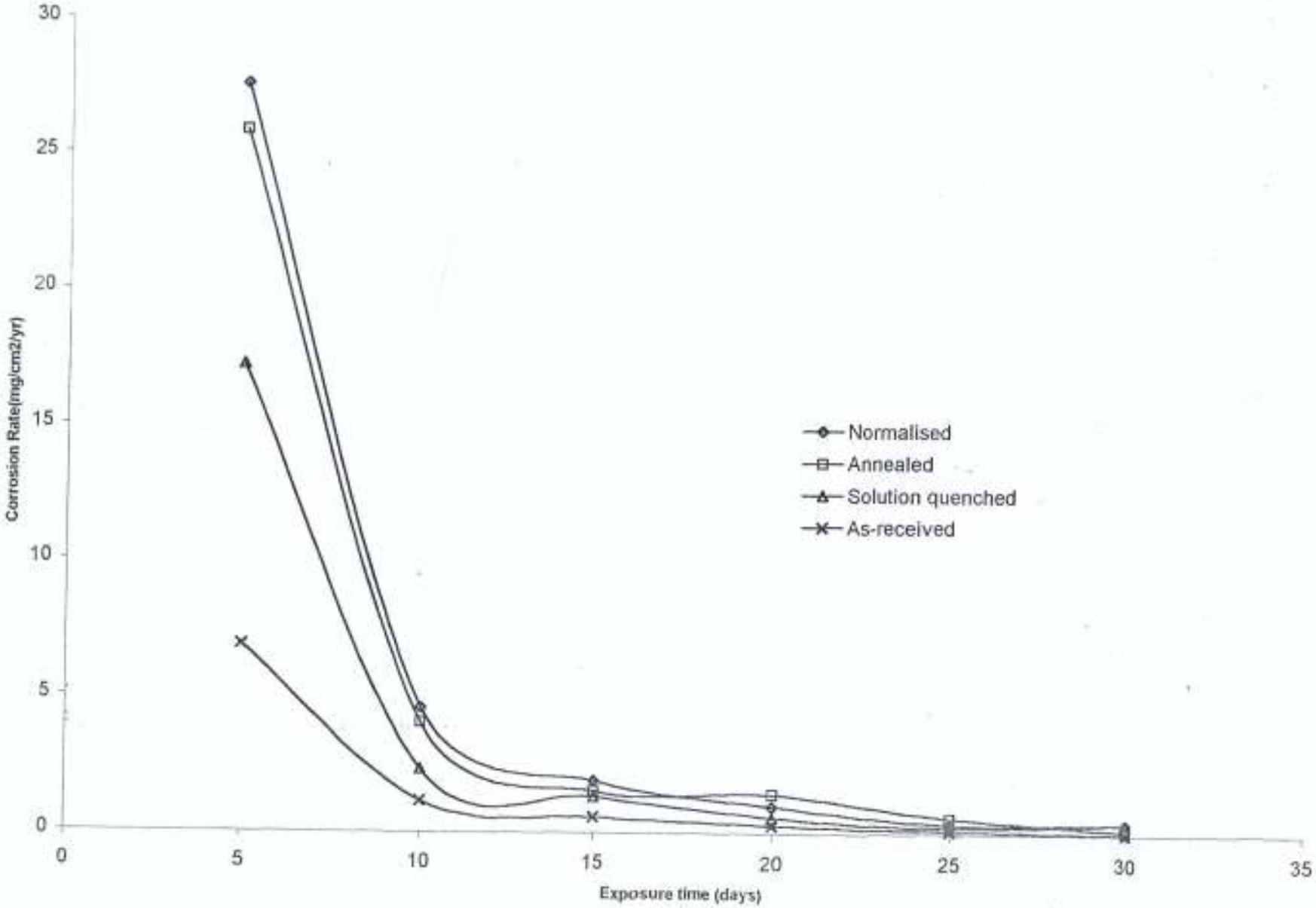


Figure 4.7.8: Plot of Corrosion Rates versus Exposure time of three-cycle heat-treated austenitic stainless steel samples held at 1000°C for 1 hour, immersed in $0.5MH_2SO_4$ solution.

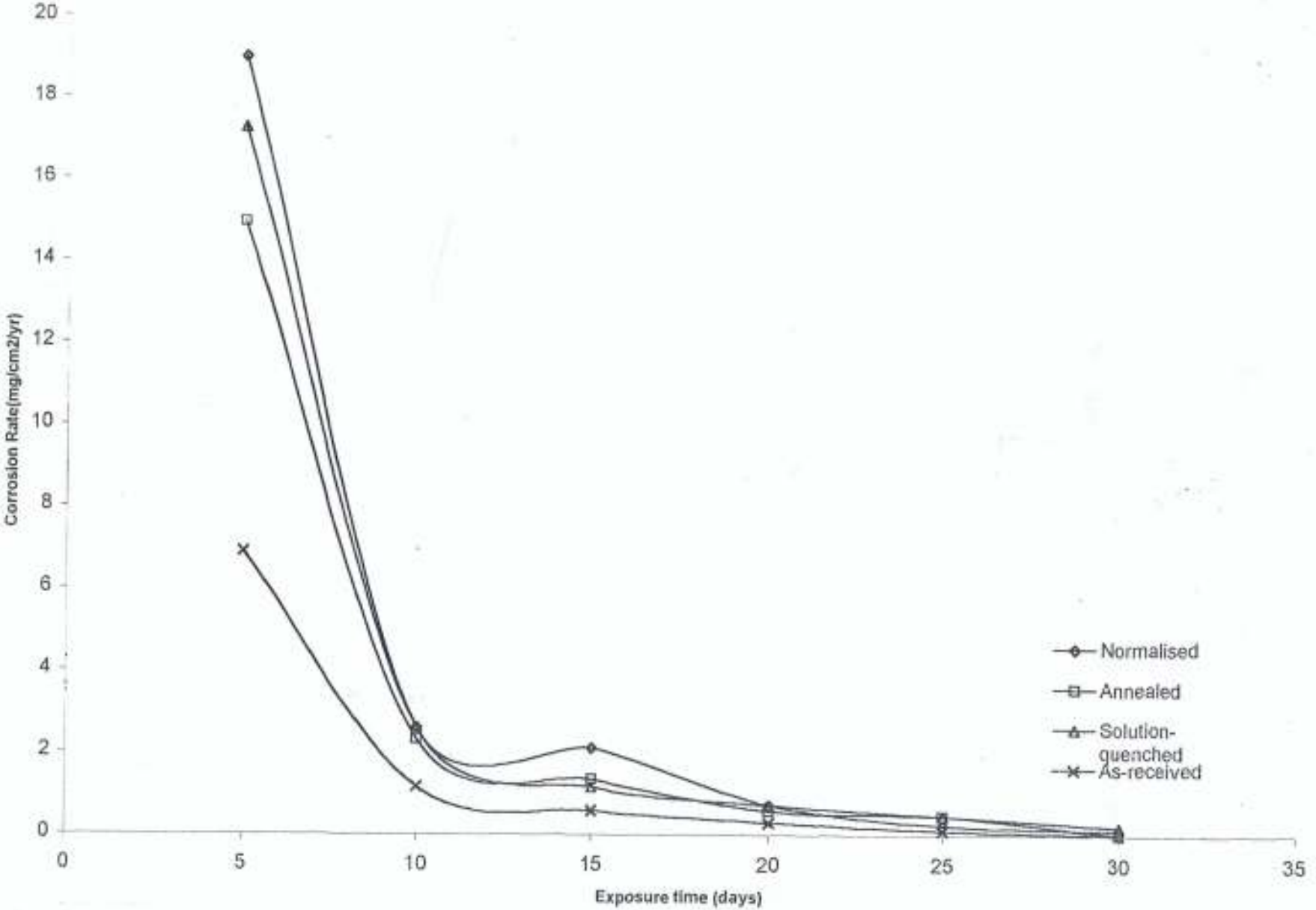


Figure4.7.9: Plot of Corrosion Rates versus Exposure time of four-cycle heat-treated austenitic stainless steel samples held at 1000°C for

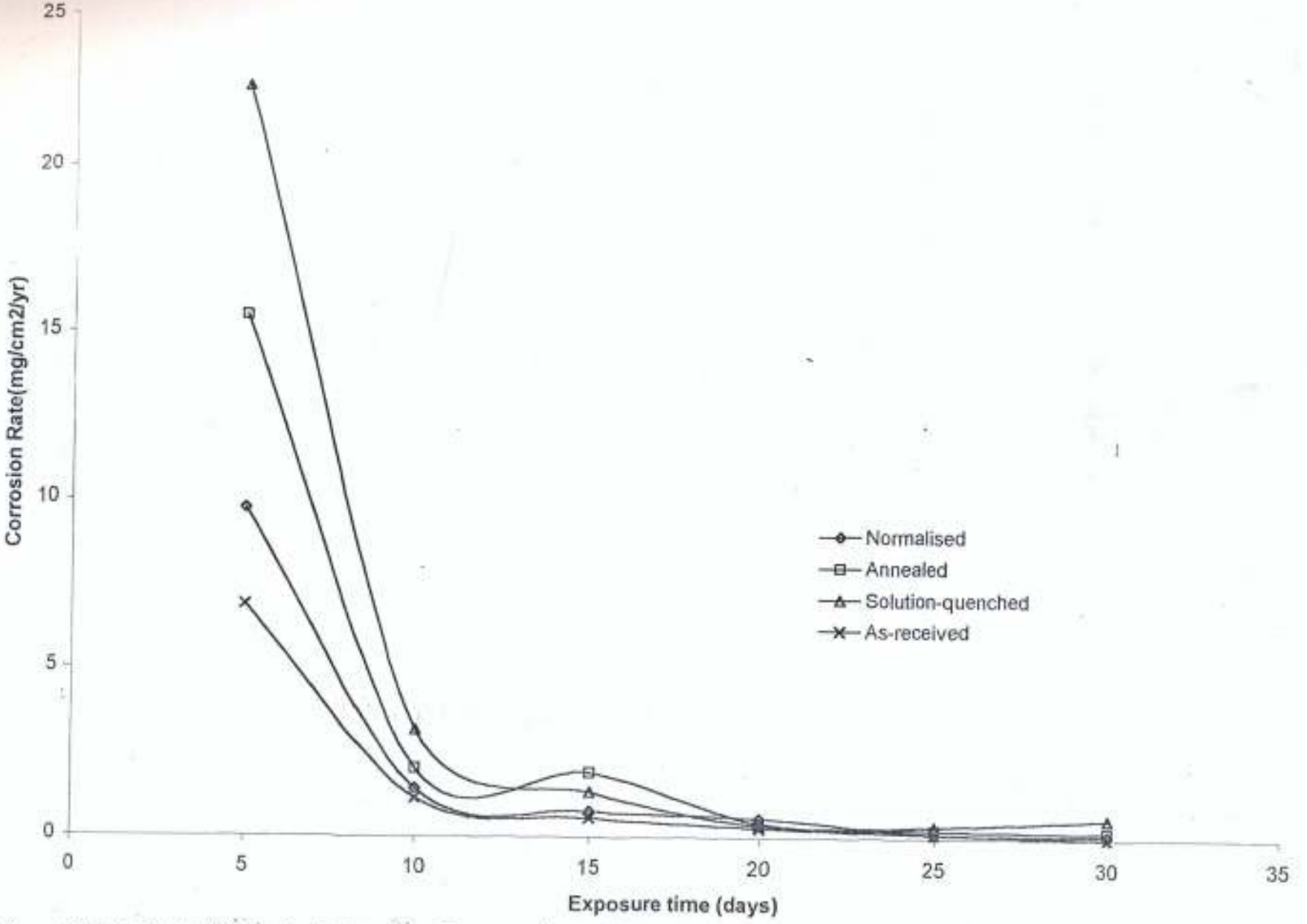


Figure 4.7.1.0: Plot of Corrosion Rates versus Exposure time of five-cycle heat-treated austenitic stainless steel samples held at 1000°C for

4.8 COMPARATIVE RESULTS OF ELECTRODE POTENTIALS AND CORROSION RATES AT VARIOUS EXPOSURE TIMES AT DIFFERENT CYCLES OF HEAT-TREATMENT CARRIED OUT AT 1000°C WITHOUT SOAKING

Figures 18, 19, 20, 21 and 22 present the comparative plots of the electrode potentials versus the exposure time for specimens subjected to various cycles of heat-treatments. It could be observed that the annealed samples display the least electrode potentials at the exposure periods between 24 and 240 hours. The as-received samples feature the highest electrode potentials, followed by normalised and solution-quenched samples at the same exposure periods. A sharp increase in electrode potentials for all the samples is observed at the exposure periods between 240 and 250 hours. In figure 18, normalised samples display the highest electrode potentials ranging from –58mV (SCE) to –17mV (SCE) at the periods between 300 and 750 hours. As shown in figure 19, the annealed samples display highest electrode potentials at the periods between 300 and 750 hours. In figure 20, solution-quenched and normalised samples display highest electrode potentials at the exposure periods between 650 and 700 hours. It is observed in figure 21 that all the samples except the as-received maintain peak electrode potentials at the exposure periods between 250 and 750 hours. The highest electrode potentials are observed for both annealed and normalised specimen as shown in Figure 22 at the periods between 400 and 750 hours.

Normalised specimen features the initial highest corrosion rates followed by the values of corrosion rates from annealed and as-received specimens at one-cycle heat-treatment as shown in Figure 39, solution-quenched specimen displays the least corrosion rates. In figure 40, annealed specimen features the initial highest corrosion

rates followed by the values of corrosion rates from solution-quenched and normalised specimens. The as-received specimen displays the least corrosion rates. It is observed in Figure 41 that normalised specimen displays the highest corrosion rates, followed by annealed and as-received specimens of almost the same value of corrosion rates, while the solution-quenched specimen displays the least corrosion rates. Annealed specimen features the initial highest corrosion rate, followed by the values of corrosion rates from normalised, as-received and solution-quenched specimens as shown in Figure 42. It is observed in Figure 43 that normalised specimen displays the initial highest corrosion rates, followed by the values of corrosion rates from the annealed specimen. The as-received and solution-quenched specimens have almost the same corrosion rates which is the lowest relative to the corrosion rates of the normalised and annealed specimens. All the corrosion rates decrease sharply in the initial 10 days of the exposure periods.

The steel displays a high resistance to corrosion due to the naturally occurring chromium-rich oxide film formed on the surface of the steel. Although extremely thin, this invisible, inert film is tightly adherent to the metal and extremely protective in a wide range of corrosive media. The film is rapidly self-repairing in the presence of oxygen. As evidenced in the as-received specimen, damage by scratching is quickly repaired. Also, under most conditions of exposure, the persistence of the passive film is subject either to continue growth or to the dynamic equilibrium of partial breakdown and repair as depicted by the spontaneous changes of the electrode potentials with exposure periods above 240 hours. During film breakdown the electrode potentials become more negative, and more positive as the film repairs.

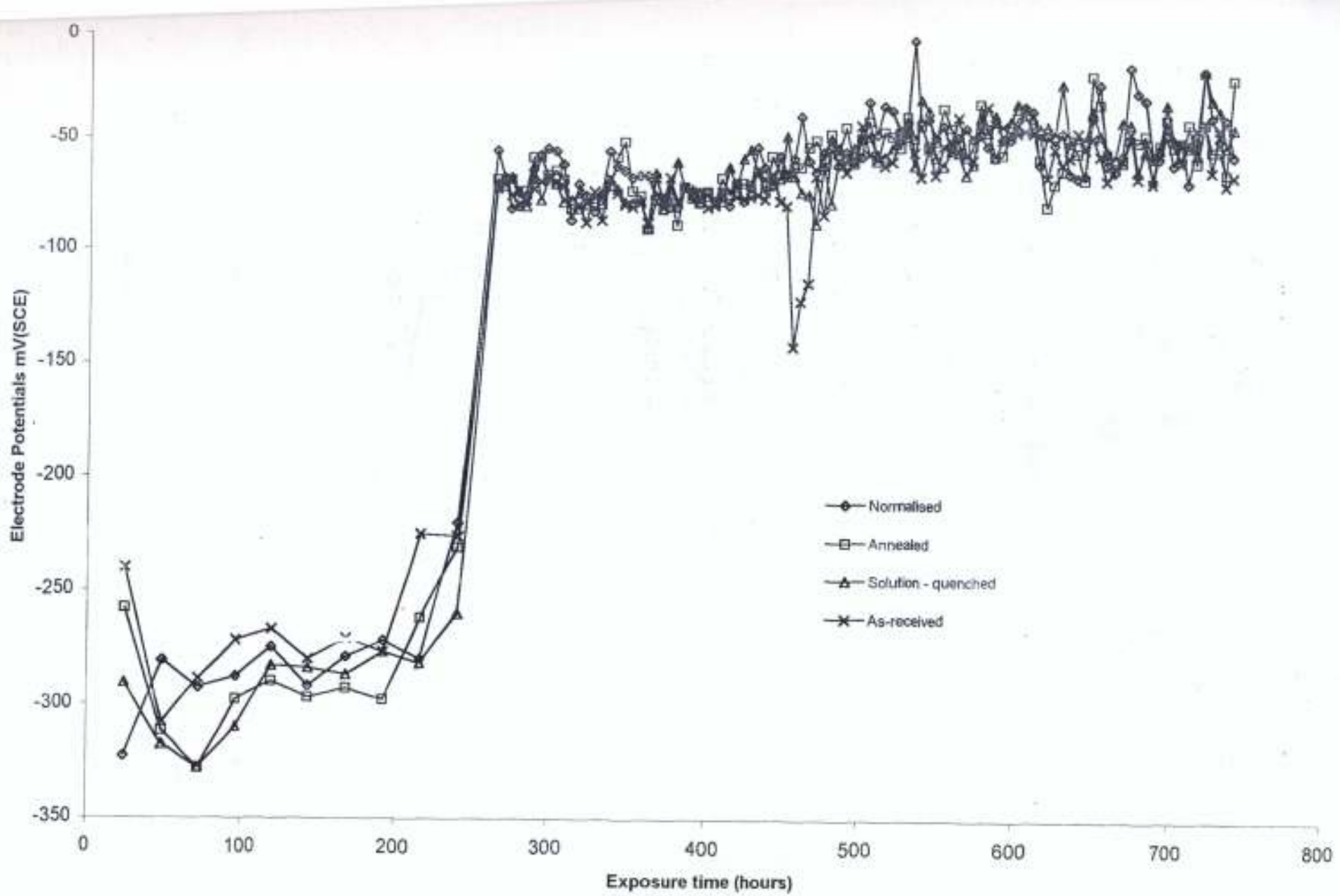


Figure 4.8.1: Plot of Electrode Potentials versus Exposure time of one-cycle heat-treated austenitic stainless steel samples at 1000°C without soaking, immersed in 0.5MH_2SO_4 solution

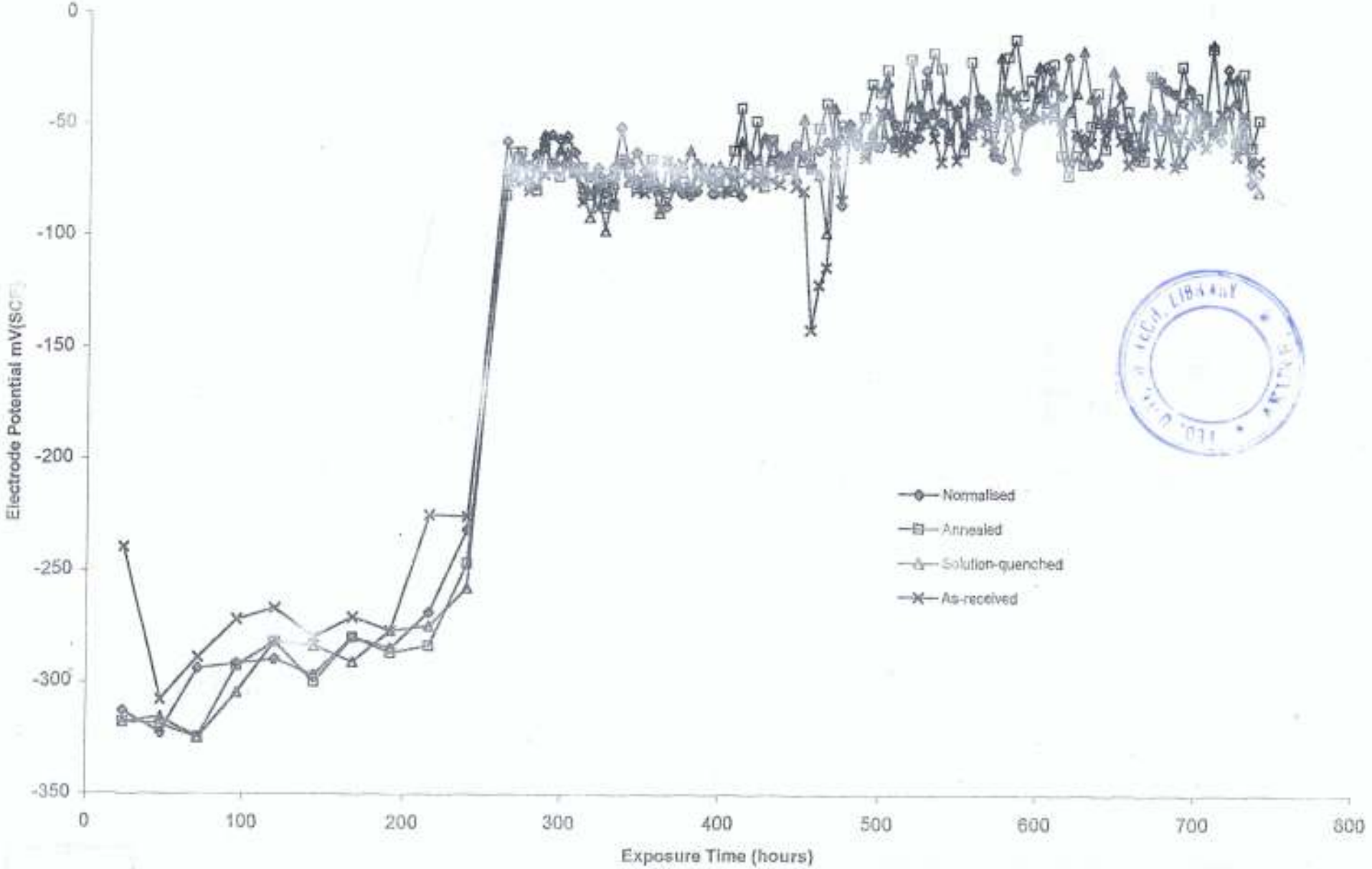


Figure 4.8.2: Plot of Electrode Potentials versus Exposure time of two-cycle heat-treated austenitic stainless steel samples at 1000°C without soaking, immersed in $0.5MH_2SO_4$ solution.

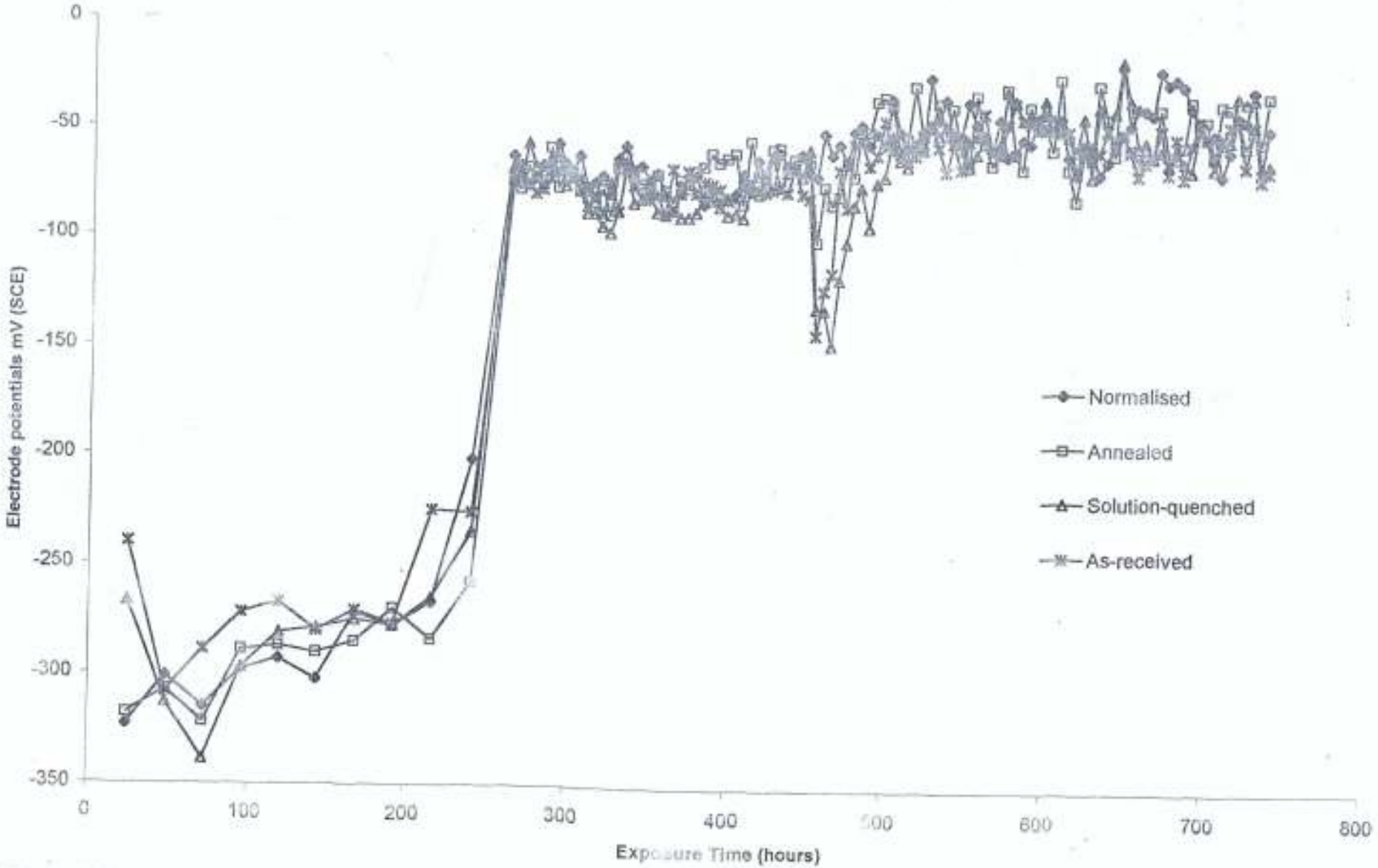


Figure 4.8.3: Plot of Electrode Potentials versus Exposure time of three-cycle heat-treated austenitic stainless steel samples at 1000°C without soaking, immersed in $0.5MH_2SO_4$ solution.

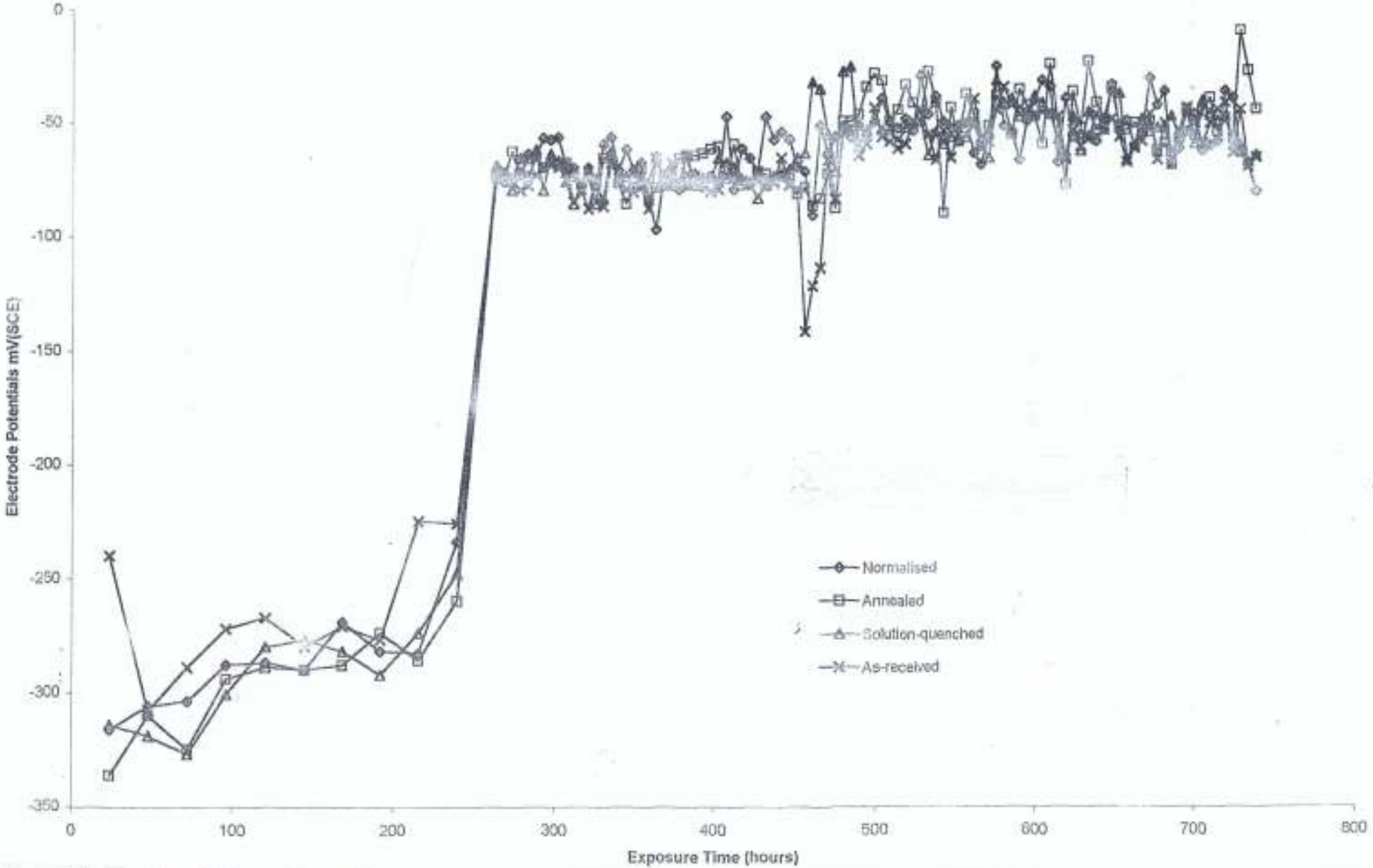


Figure 4.8.4: Plot of Electrode Potentials versus Exposure time of four-cycle heat-treated austenitic stainless steel samples at 1000°C without soaking, immersed in $0.5MH_2SO_4$ solution.

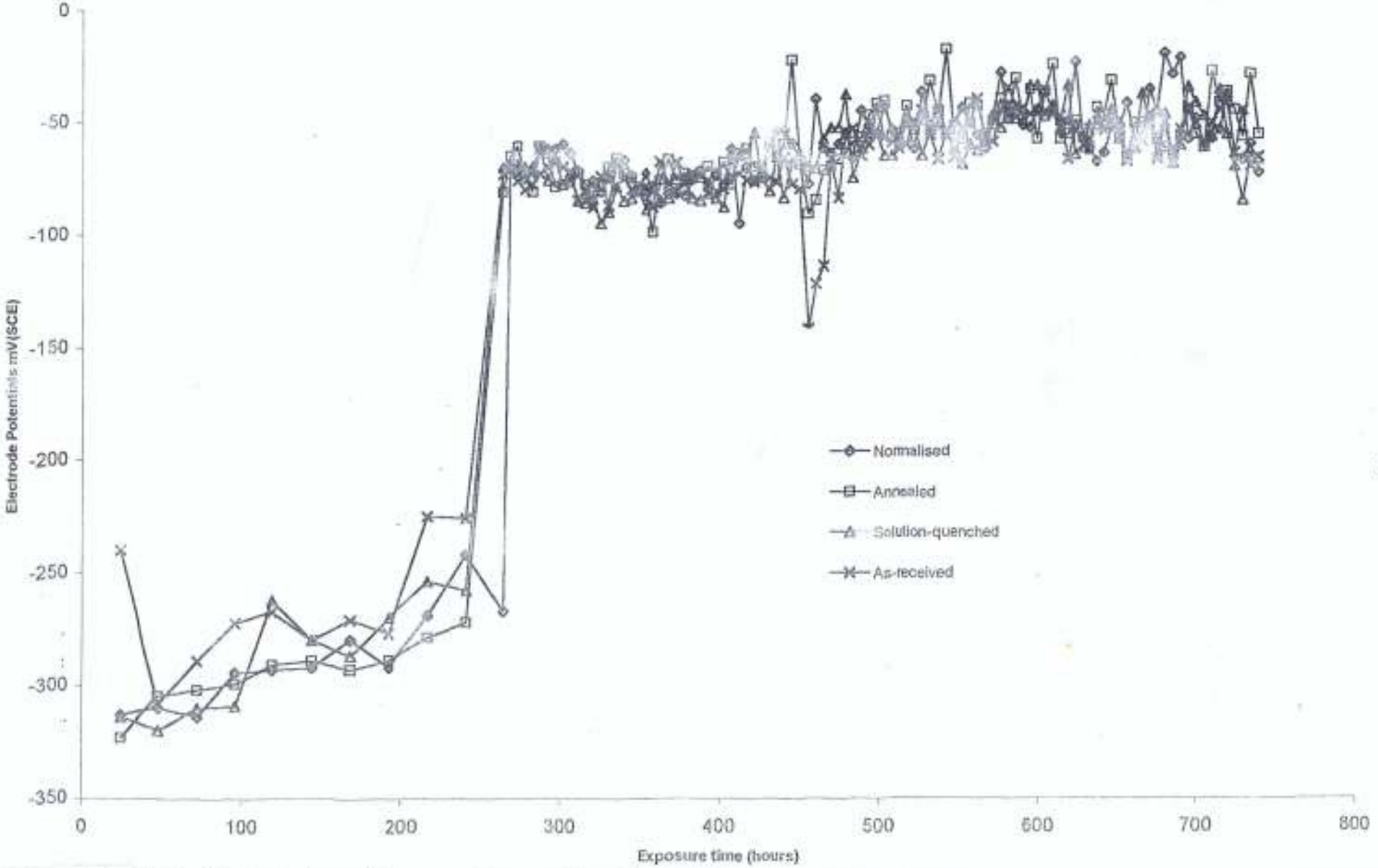


Figure 4.8.5: Plot of Electrode Potentials versus Exposure time of five-cycle heat-treated austenitic stainless steel samples at 1000°C without soaking immersed in $0.5MH_2SO_4$ solution.

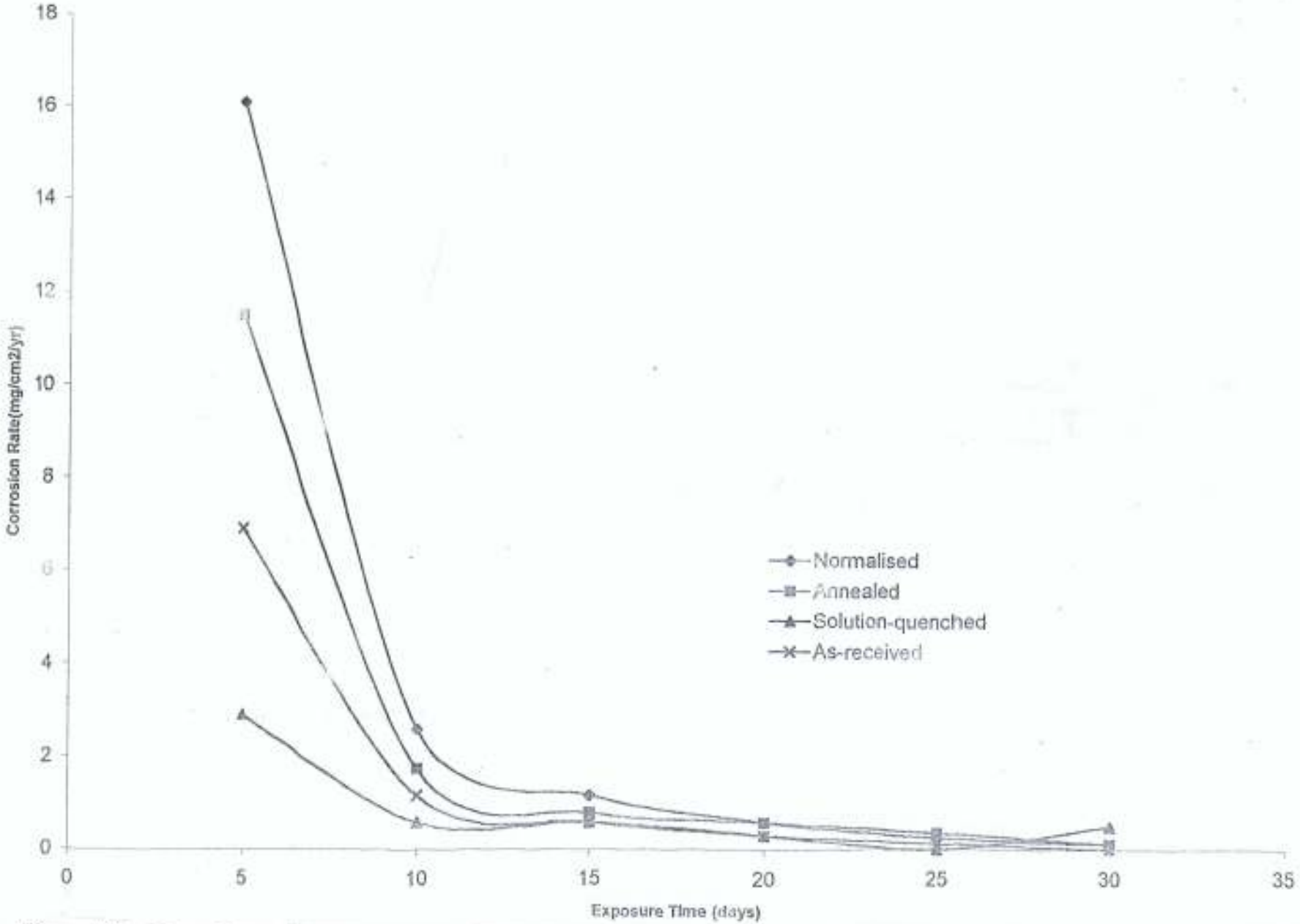


Figure 4.8.6: Plot of Corrosion Rates versus Exposure time of one-cycle heat-treated austenitic stainless steel samples at 1000°C without

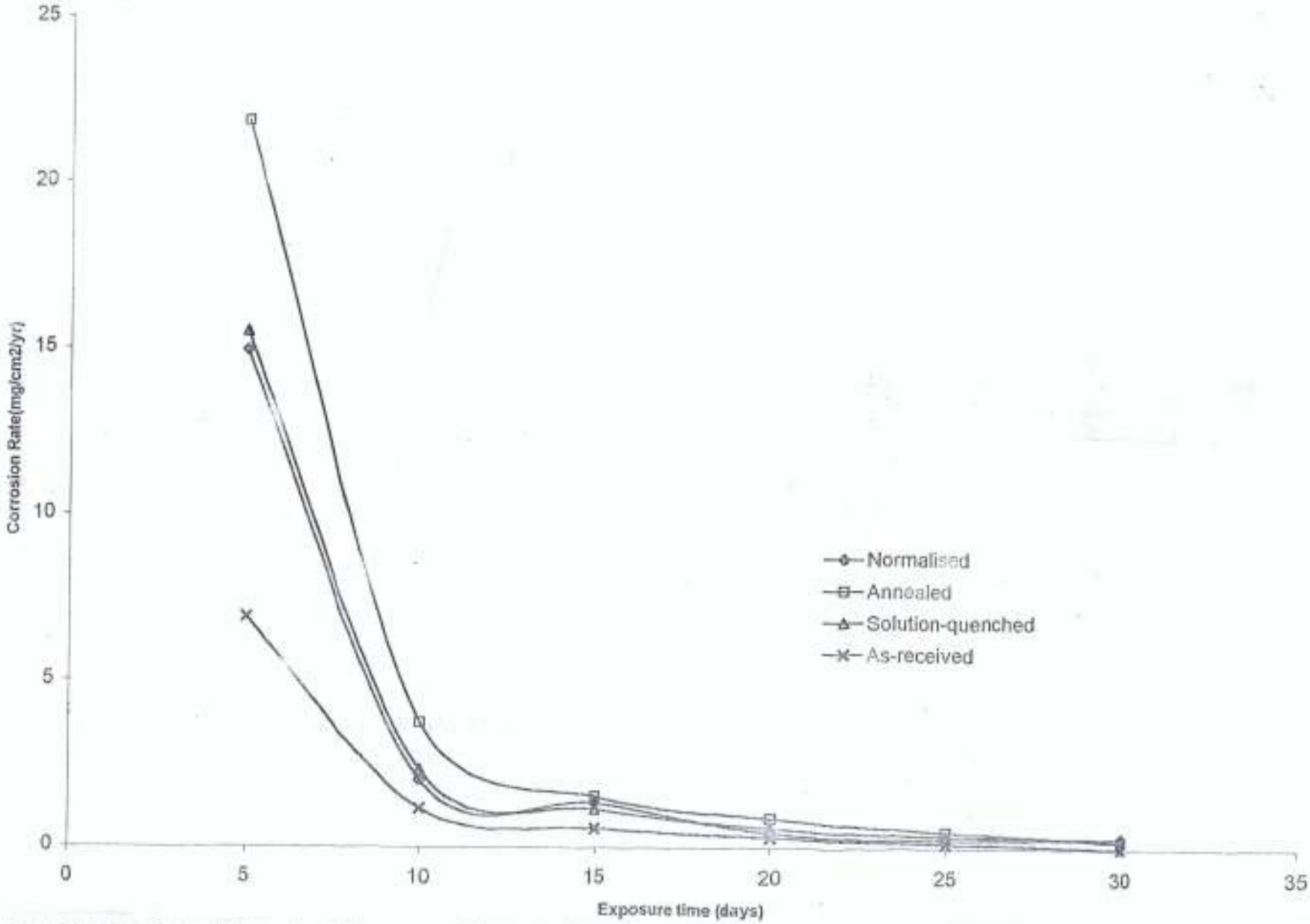


Figure 4.8.7: Plot of Corrosion Rates versus Exposure time of two-cycle heat-treated austenitic stainless steel samples at 1000°C without soaking immersed in $0.5MH_2SO_4$ solution

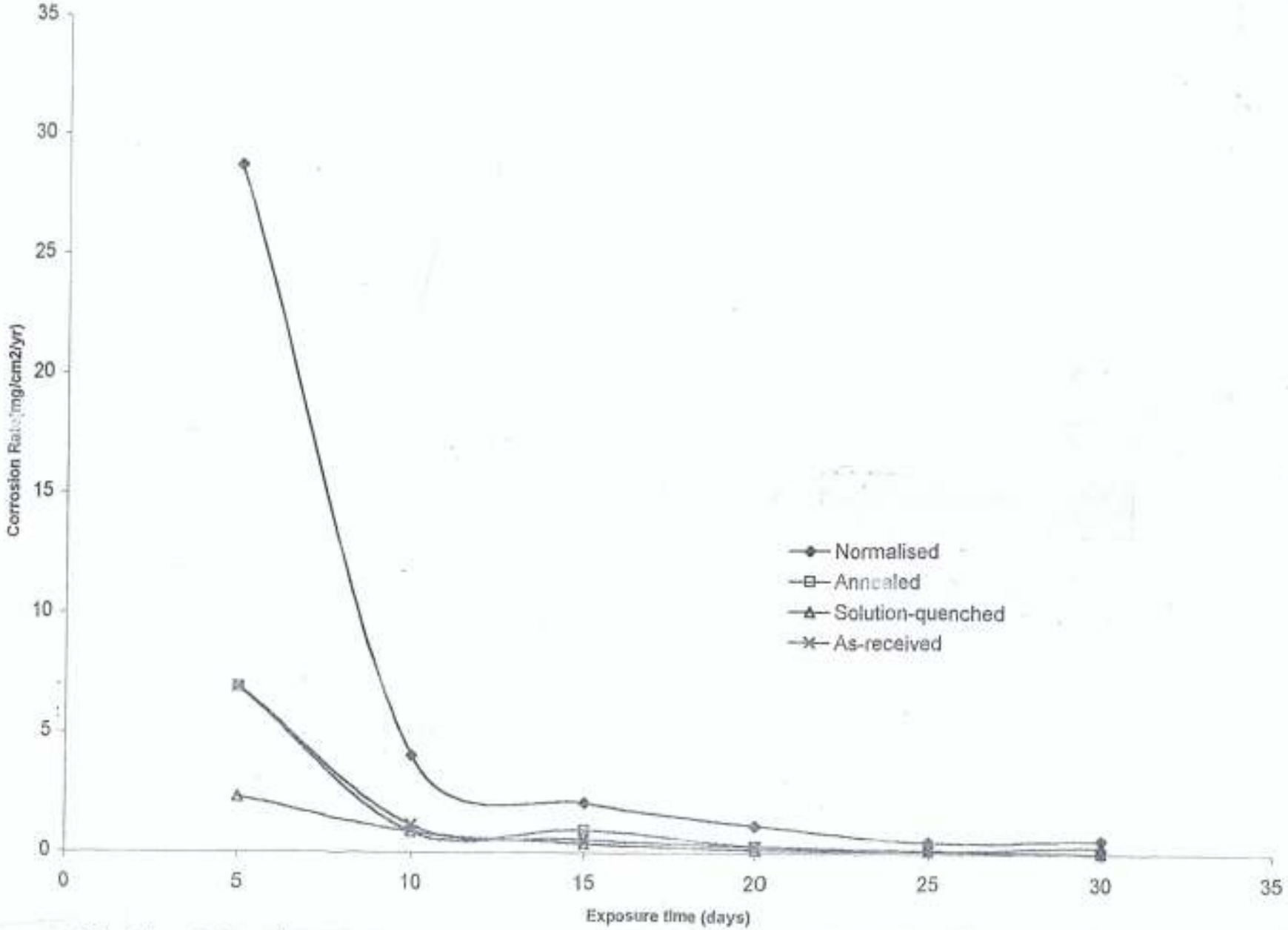


Figure 4.8.8: Plot of Corrosion Rates versus Exposure time of three-cycle heat-treated austenitic stainless steel samples at 1000°C without soaking immersed in 0.5M H_2SO_4 solution

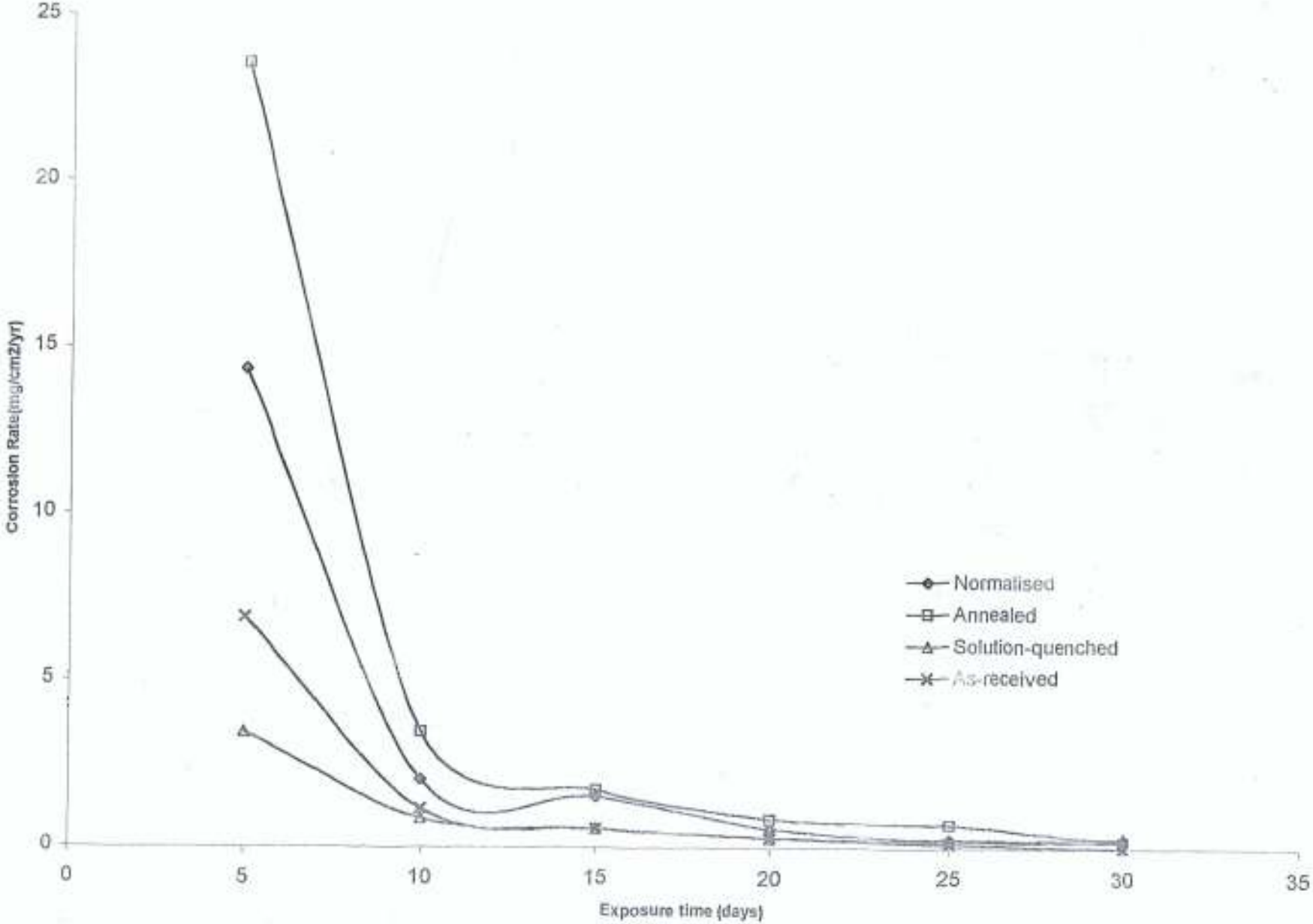


Figure 4.8.9: Plot of Corrosion Rates versus Exposure time of four-cycle heat-treated austenitic stainless steel samples at 1000°C without

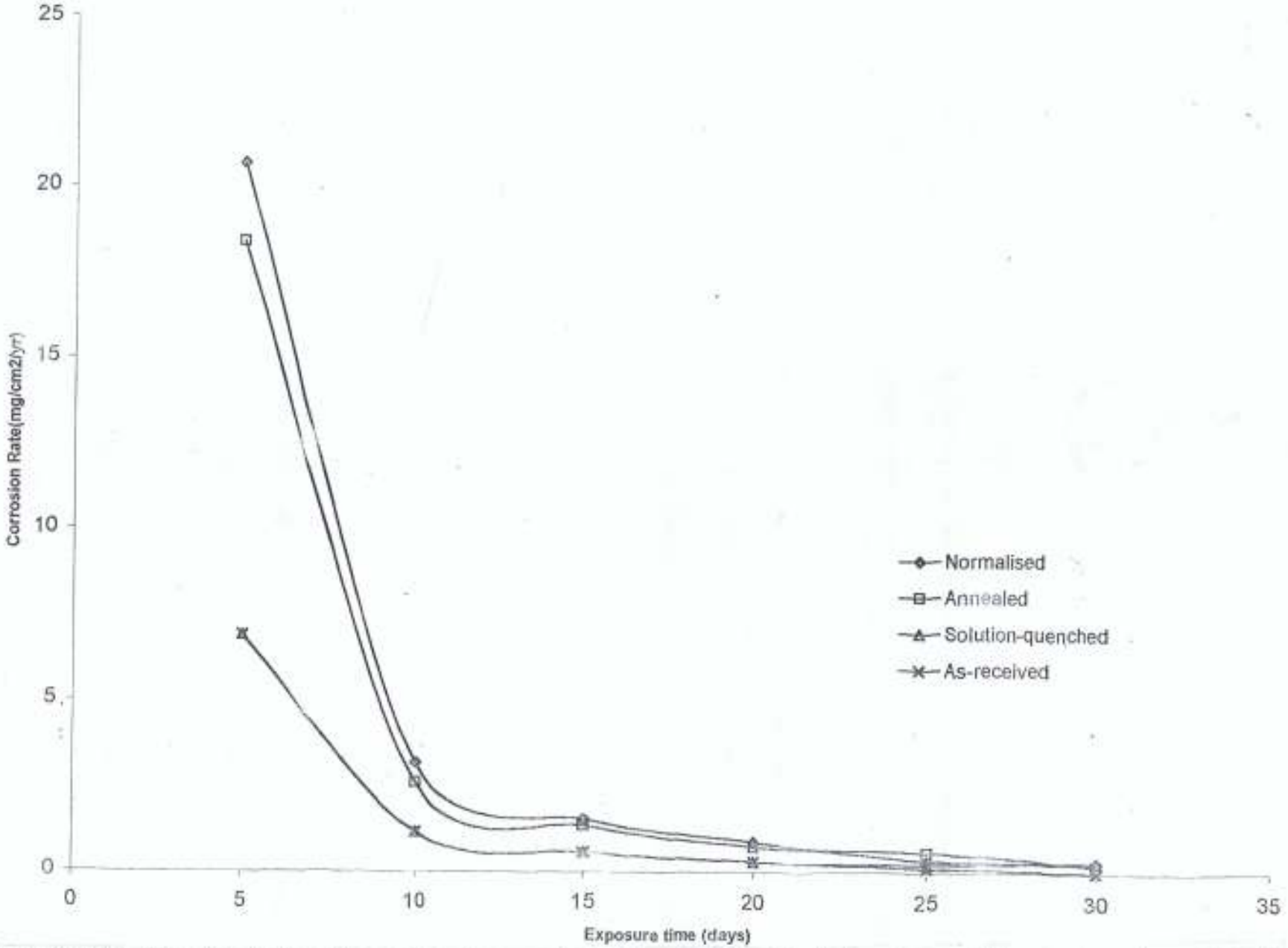


Figure 4.8.1.0: Plot of Corrosion Rates versus Exposure time of five-cycle heat-treated austenitic stainless steel samples at 1000°C without soaking immersed in 0.5MH_2SO_4 solution.

CHAPTER FIVE

5.0 CONCLUSIONS AND RECOMMENDATIONS

5.1 CONCLUSIONS

From the results of the present investigation, the following conclusions can be drawn on the influence of fluctuating heat treatment operations on corrosion of austenitic stainless steel in sulphuric acid solution.

(1) Repeated cycles of normalisation, annealing and solution-quenching at 1000°C enhance corrosion resistance of austenitic stainless steel in sulphuric acid solution.

(2) Enhancement of corrosion resistance of Austenitic Stainless Steel in sulphuric acid solution increases in the following order of thermal cycling operations: Normalisation, Annealing and Solution-quenching.

(3) Corrosion resistance of austenitic stainless steel subjected to Normalisation, Annealing and Solution-quenching at elevated temperature without soaking period at 1000°C is superior to that which was soaked for 1 hour at the temperature.

5.2 RECOMMENDATIONS

The following are the recommendations as a result of this study.

(1) Since austenitic stainless steel resists corrosion at repeated cycles of slow thermal fluctuations, it may be selected in the designing of components for applications in practical engineering systems whose operations may involve repeated thermal fluctuations. Examples are in the production of exhaust pipes of internal combustion engines and construction of furnace body structure.

(2) Austenitic stainless steel may find application in the construction of baths and reactors (vessels) where sulphuric acid solutions are used; as in the textile and paper industries.

REFERENCES

AUDOUARD, J. P., DESESTRET, A., LEMOINE, L. and MORIZOR, Y. (1984): Special Stainless Steels for use in Sea Water. UK Corrosion Conference, Wembley. Papers of 12th, 13th and 14th November.

AVERY, C. H. (1963): Heat Treating of Stainless Steel, Metals Handbook, 8th edition, American Society For Metals, Ohio. Vol. 2 p 243.

BANERJEE, S. N. (1985): An Introduction to the Science of Corrosion and its Inhibition. Oxonian Press PVT Limited, Germany. pp 255-327

BIRK, N. and SMITH, C. A. (1997): 'Materials issues in heat exchangers andboilers', Conference Proceeding ,The Institute of Materials, London. Vol. 51

CHAMBERLAIN, J. and TRETHEWAY, K.R. (1996): Corrosion for Students of Science and Engineering., 2nd Edition, Longman Singapore. pp 139-142

DELL, K. A. (1981): Metallurgy Theory and Practice. American Technical Publishers inc. Illinois. pp 229, 454

FONTANA, M. G. (1986): Corrosion Engineering. McGraw – Hill Inc, New York. pp 78-203

HIBBERT, D. B. and JAMES, A. M. (1984): Dictionary of Electrochemistry. Macmillan Press, London.

JOHN, E. N. (1994): Practical Metallurgy and Materials of Industry. 4th Edition. Prentice Hall, New Jersey. pp 80-261

KENNETH, B. (1983): Engineering Materials Properties and selection. 2nd Edition. Prentice Hall, New Jersey. pp 9-98

LAKHTIN, Y. (1979): Engineering Physical Metallurgy and Heat – Treatment. Mic Publishers Moscow. pp 318-322

LLEWELLYN, D. T. (1992): Steels Metallurgy and Applications. Butterworth – Heinemann. pp 214-231

METALS HANDBOOK (1964): Heat Treating, Cleaning and finishing. American Society for Metals, Ohio. Vol. 2, p 260

NELSON, G.A.(1960): Corrosion Data Survey, Shell Development Company. p11

OLDFIELD, J. W. (1990): Ambient – temperature Stress Corrosion Cracking of Austenitic Stainless Steel in Swimming Pools. Materials Reference, Vol. 29, pp 60 – 62.

OSTRANDER, C. W. (1971): Chromate Conversion Coatings, Electroplating Engineering Handbook. Van Nostrand Reinhold Company, London.

ROSS, T.K. (1977): Metal Corrosion, Engineering Design Guides, Oxford University Press. Vol. 21, p 13.

SANGDAHL, G. S. (1964): Stress Relieving of Austenitic stainless steel, Metals Handbook, 8th edition American Society For Metals, Ohio. Vol. 2 p 253.

SCULLY J. C. (1980): The Fundamental of Corrosion. 3rd Edition. Pergamon,Oxford p107

SHAWESH, F. EL, HOUD, A. EL. and RAGHAI, O. (2001): Role of surface finish and post-fabrication cleaning on localised corrosion of type 316L austenitic stainless steel flash chambers. British Corrosion Journal, IOM Communications, London.Vol.36 No.3 pp 184-189.

SHNEIDER, H. (1960): Foundry Trade Journal. Vol. 108 p 562.

SHRIER, L. C. (1963): Corrosion, Metal and Environment. Newness – Butter works. London. Vol. 1, pp 56-63

SOURCE BOOK ON STAINLESS STEELS, 1976: Section I: Stainless Types and Their Characteristics, American Society for Metals, pp 1-2

STONE, P. G., HUDSON, R. M. and JOHN, D. R. (1984): Stainless Steels. Proceedings, The Institute of Metals, Gotenborg. p 178.

TRETHEWAY, K. R. (1985): Corrosion for Students of Science and Engineering. Willey and Sons Inc, United State.

UHILIG, H. H. (1971): Corrosion and Corrosion Control; An Introduction to Corrosion Science and Engineering, Wiley, New York.

ULICK, R. E. (1982): An Introduction to Metallic Corrosion. 3rd Edition, Edward Arnold, Great Britain. pp 75-90

WHITNEY, S. (1964): Cleaning And Finishing Of Stainless Steel, Metals Handbook, 8th Edition, American Society for Metals, Ohio. Vol. 2, p 605.

APPENDIX A

Table A1 : Electrode Potentials mV (SCE) of normalized (1000oC, 1hr) austenitic stainless steel immersed in 0.5M H2SO4

Exposure Time (Hours)	Number of Cycles					
	1 Cycle	2 Cycle	3 Cycle	4 Cycle	5 Cycle	As-received
24	-295	-306	-336	-305	-262	-240
48	-315	-321	-314	-281	-324	-308
72	-316	-314	-311	-280	-306	-289
96	-290	-276	-280	-276	-302	-272
120	-308	-293	-286	-268	-307	-267
144	-309	-309	-306	-291	-301	-280
168	-293	-290	-287	-276	-286	-271
192	-295	-288	-289	-283	-295	-277
216	-280	-282	-267	-255	-349	-225
240	-237	-231	-226	-224	-226	-226
264	-62	-75	-72	-65	-73	-73
266.8	-84	-74	-71	-73	-85	-71
273.6	-81	-81	-75	-82	-81	-76
278.4	-86	-77	-79	-82	-85	-80
283.2	-73	-78	-70	-66	-78	-78
288	-80	-75	-74	-71	-70	-73
292.8	-63	-56	-58	-66	-68	-70
297.6	-57	-62	-53	-61	-62	-68
302.4	-52	-68	-63	-57	-58	-70
307.2	-51	-67	-60	-60	-66	-72
312	-82	-82	-81	-75	-80	-85
316.8	-75	-82	-80	-66	-78	-80
321.6	-81	-82	-74	-76	-78	-88
326.4	-82	-84	-81	-84	-81	-74
331.2	-76	-84	-74	-68	-65	-87
336	-84	-65	-61	-60	-53	-68
340.8	-64	-72	-76	-73	-67	-75
345.6	-61	-72	-70	-64	-67	-80
350.4	-64	-73	-71	-68	-84	-81
355.2	-57	-66	-78	-75	-75	-79
360	-80	-60	-63	-66	-67	-88
364.8	-85	-79	-76	-73	-74	-6
369.6	-79	-82	-81	-84	-83	-78
374.4	-75	-82	-76	-71	-79	-68
379.2	-82	-73	-87	-76	-83	-78
384	-60	-62	-63	-63	-70	-73
388.8	-73	-80	-79	-82	-82	-74
393.6	-77	-79	-81	-78	-81	-76
398.4	-76	-80	-78	-80	-78	-81
403.2	-72	-80	-80	-78	-76	-80
408	-78	-75	-72	-62	-75	-74
412.8	-72	-74	-76	-77	-88	-73
417.6	-70	-75	-71	-64	-69	-76
422.4	-73	-76	-75	-71	-78	-77
427.2	-82	-78	-72	-77	-84	-76
432	-41	-48	-49	-51	-50	-75
436.8	-67	-74	-69	-69	-70	-77
441.6	-70	-80	-77	-78	-74	-66
446.4	-53	-72	-65	-66	-68	-78
451.2	-59	-75	-58	-68	-59	-80
456	-46	-56	-57	-58	-57	-142
460.8	-50	-62	-72	-56	-55	-122
465.6	-49	-69	-68	-70	-61	-114
470.4	-69	-67	-65	-62	-63	-67
475.2	-32	-50	-79	-80	-52	-84
480	-37	-43	-47	-49	-49	-55
484.4	-46	-58	-62	-60	-61	-53
489.6	-16	-46	-55	-52	-60	-65
494.4	-16	-29	-52	-59	-50	-60

TABLE A1 CONTINUED

Exposure Time	Electrode Potentials mV (SCE) of normalized(1000 o c, 1 hr) austenitic stainless steel immersed in 0.5M H2SO4					
(Hours)	1 Cycle	2 Cycle	3 Cycle	4 Cycle	5 Cycle	As-received
499.2	-16	-66	-74	-57	-62	-44
504	-22	-24	-26	-29	-27	-57
508.8	-41	-49	-51	-57	-47	-59
513.6	-32	-42	-49	-62	-51	-62
518.4	-26	-39	-49	-55	-57	-60
523.2	-31	-47	-54	-42	-51	-50
528	-28	-25	-22	-30	-20	-45
532.8	-22	-49	-53	-39	-44	-56
537.6	-42	-32	-44	-44	-47	-67
542.4	-22	-50	-57	-55	-46	-54
547.2	-29	-44	-46	-55	-57	-66
552	-23	-50	-47	-40	-50	-53
556.8	-56	-63	-52	-53	-57	-51
561.6	-14	-32	-42	-41	-48	-40
566.4	-26	-57	-51	-53	-46	-57
571.2	-52	-65	-58	-62	-59	-59
576	-12	-21	-15	-28	-31	-45
580.8	-36	-49	-39	-48	-61	-35
585.6	-35	-43	-65	-47	-57	-43
590.4	-36	-48	-60	-54	-47	-44
595.2	-20	-36	-42	-44	-42	-47
600	-26	-33	-39	-40	-44	-47
604.8	-27	-32	-35	-38	-38	-46
609.6	-39	-52	-48	-27	-34	-47
614.4	-12	-42	-43	-48	-50	-48
619.2	-16	-36	-36	-42	-57	-67
624	-25	-75	-41	-48	-57	-54
628.8	-46	-28	-40	-51	-39	-61
633.6	-22	-47	-49	-46	-58	-58
638.4	-30	-43	-49	-51	-57	-47
643.2	-12	-44	-47	-46	-41	-54
648	-15	-27	-32	-25	-32	-49
652.8	-37	-39	-45	-58	-58	-57
657.6	-40	-60	-58	-56	-60	-68
662.4	-59	-58	-63	-61	-59	-62
667.2	-55	-58	-57	-60	-62	-59
672	-10	-42	-21	-23	-13	-47
676.8	-50	-50	-35	-34	-14	-67
681.6	-60	-26	-40	-38	-36	-51
686.4	-45	-36	-46	-43	-37	-69
691.2	-64	-63	-58	-41	-40	-54
696	-12	-15	-25	-31	-39	-44
700.8	0	-25	-31	-43	-32	-51
705.6	-11	-38	-36	-44	-59	-60
710.4	-37	-44	-41	-38	-45	-51
715.2	-27	-47	-51	-47	-55	-45
720	-17	-12	-15	-28	-39	-42
724.8	-21	-15	-30	-40	-46	-64
729.6	-17	-60	-18	-36	-42	-45
734.4	-20	-32	-29	-42	-56	-70
739.2	-27	-40	-32	-47	-67	-66

TABLE A2: Electrode Potentials mV (SCE) of normalized(1000 °C without soaking) austenitic stainless steel immersed in 0.5M H2SO4

Exposure Time (Hours)	Number of Cycles					
	1 Cycle	2 Cycle	3 Cycle	4 Cycle	5 Cycle	As-received
24	-303	-313	-323	-316	-313	-240
48	-281	-323	-301	-306	-310	-206
72	-293	-294	-315	-304	-314	-289
96	-288	-292	-298	-288	-294	-272
120	-275	-290	-293	-287	-293	-267
144	-292	-297	-302	-290	-292	-280
168	-279	-280	-272	-269	-20	-271
192	-272	-285	-278	-282	-292	-277
216	-280	-269	-267	-283	-269	-225
240	-220	-232	-202	-234	-242	-226
264	-56	-58	-63	-75	-267	-73
268.8	-68	-70	-67	-76	-69	-71
273.6	-82	-67	-76	-75	-72	-76
278.4	-81	-69	-73	-66	-75	-80
283.2	-77	-64	-67	-64	-72	-78
288	-69	-59	-69	-65	-62	-73
292.8	-58	-55	-57	-57	-62	-70
297.6	-55	-57	-66	-58	-62	-68
302.4	-56	-56	-69	-57	-60	-70
307.2	-62	-63	-62	-69	-64	-72
312	-87	-71	-76	-70	-72	-85
316.8	-71	-74	-74	-79	-83	-80
321.6	-76	-70	-71	-70	-76	-88
326.4	-83	-81	-79	-78	-81	-74
331.2	-75	-70	-63	-60	-75	-87
336	-56	-52	-57	-57	-68	-68
340.8	-63	-68	-68	-71	-67	-75
345.6	-65	-63	-66	-62	-74	-80
350.4	-68	-71	-72	-71	-79	-81
355.2	-67	-72	-69	-68	-73	-79
360	-67	-79	-79	-84	-80	-68
364.8	-76	-87	-86	-97	-86	-6
369.6	-77	-78	-78	-74	-82	-78
374.4	-81	-81	-70	-78	-82	-68
379.2	-81	-82	-70	-80	-83	-78
384	-72	-80	-83	-78	-76	-73
388.8	-76	-74	-76	-73	-72	-74
393.6	-79	-81	-82	-79	-80	-76
398.4	-74	-76	-80	-74	-74	-81
403.2	-79	-80	-78	-76	-78	-80
408	-74	-71	-69	-48	-62	-74
412.8	-80	-82	-77	-80	-95	-73
417.6	-70	-64	-63	-62	-70	-76
422.4	-78	-72	-71	-66	-76	-77
427.2	-72	-72	-76	-72	-74	-76
432	-54	-57	-59	-48	-59	-75
436.8	-63	-64	-65	-58	-54	-77
441.6	-71	-70	-66	-55	-54	-66
446.4	-58	-59	-60	-58	-60	-78
451.2	-66	-64	-68	-70	-66	-80
456	-59	-62	-70	-72	-78	-142
460.8	-40	-62	-50	-91	-40	-122
465.6	-60	-58	-60	-62	-62	-114
470.4	-65	-58	-55	-64	-64	-67
475.2	-56	-86	-64	-57	-60	-84
480	-51	-50	-49	-52	-55	-55
484.4	-62	-59	-46	-57	-66	-53
489.6	-56	-60	-55	-52	-45	-65
494.4	-60	-56	-50	-56	-52	-60
499.2	-58	-60	-55	-50	-55	-44
504	-33	-32	-34	-40	-43	-57
508.8	-48	-50	-49	-51	-54	-59

TABLE A2 CONTINUED

Exposure Time (Hours)	Electrode Potentials mV (SCE) of normalized(1000 o c, without s· oaking) austenitic stainless steel immersed in 0.5M H2SO4					
	1 Cycle	2 Cycle	3 Cycle	4 Cycle	5 Cycle	As-received
518.4	-37	-55	-54	-49	-49	-60
523.2	-46	-56	-56	-54	-62	-50
528	-42	-26	-24	-30	-37	-45
532.8	-40	-45	-43	-47	-52	-56
537.6	-42	-49	-34	-39	-44	-67
542.4	-41	-54	-53	-50	-56	-54
547.2	-49	-46	-60	-53	-51	-66
552	-44	-39	-35	-52	-44	-53
556.8	-54	-52	-45	-51	-51	-51
561.6	-49	-49	-50	-64	-56	-40
566.4	-45	-45	-57	-69	-62	-57
571.2	-50	-64	-43	-57	-48	-59
576	-48	-65	-60	-26	-28	-45
580.8	-53	-52	-58	-52	-49	-35
585.6	-58	-70	-52	-55	-47	-43
590.4	-50	-50	-54	-67	-52	-44
595.2	-50	-48	-42	-50	-53	-47
600	-45	-37	-40	-41	-45	-47
604.8	-35	-24	-47	-32	-37	-46
609.6	-37	-32	-42	-35	-46	-47
614.4	-61	-37	-60	-68	-55	-48
619.2	-48	-20	-68	-40	-50	-67
624	-52	-55	-55	-57	-24	-54
628.8	-47	-56	-55	-56	-58	-61
633.6	-65	-68	-68	-57	-62	-58
638.4	-67	-67	-62	-59	-68	-47
643.2	-66	-49	-54	-54	-64	-54
648	-40	-44	-18	-34	-53	-49
652.8	-25	-37	-45	-52	-52	-57
657.6	-61	-60	-37	-50	-42	-68
662.4	-64	-66	-38	-61	-59	-62
667.2	-57	-60	-40	-56	-57	-59
.672	-17	-29	-20	-31	-36	-47
676.8	-29	-30	-26	-43	-46	-67
681.6	-32	-34	-24	-37	-20	-51
686.4	-57	-36	-27	-69	-29	-69
691.2	-58	-40	-37	-62	-22	-54
696	-48	-34	-43	-55	-58	-44
700.8	-61	-55	-51	-47	-49	-51
705.6	-51	-49	-49	-63	-61	-60
710.4	-69	-54	-68	-61	-58	-51
715.2	-50	-57	-57	-59	-54	-45
720	-19	-25	-41	-37	-38	-42
724.8	-40	-40	-43	-40	-66	-64
729.6	-50	-50	-29	-64	-67	-45
734.4	-41	-76	-65	-69	-60	-70
739.2	-57	-72	-47	-81	-73	-66

TABLE A3: Electrode Potentials mV (SCE) of annealed (1000 o C, 1 hr) austenitic stainless steel immersed in 0.5M H2SO4

Exposure Time (Hours)	Number of Cycles					
	1 Cycle	2 Cycle	3 Cycle	4 Cycle	5 Cycle	As received
24	-317	-321	-338	-309	-359	-240
48	-284	-301	-291	-305	-295	-308
72	-306	-302	-299	-311	-315	-289
96	-296	-291	-285	-286	-285	-272
120	-290	-280	-278	-280	-290	-267
144	-292	-288	-280	-298	-290	-280
168	-275	-290	-290	-296	-300	-271
192	-269	-278	-263	-276	-274	-277
216	-260	-268	-264	-273	-262	-225
240	-222	-233	-217	-237	-211	-226
264	-67	-68	-72	-73	-74	-73
268.8	-84	-73	-68	-90	-78	-71
273.6	-69	-67	-62	-80	-76	-76
278.4	-76	-62	-72	-75	-80	-80
283.2	-81	-66	-70	-83	-78	-78
288	-62	-56	-56	-60	-71	-73
292.8	-56	-56	-56	-57	-62	-70
297.6	-56	-53	-50	-68	-58	-68
302.4	-63	-59	-61	-70	-53	-70
307.2	-60	-59	-60	-74	-67	-72
312	-71	-78	-71	-84	-79	-85
316.8	-70	-75	-75	-84	-81	-80
321.6	-67	-74	-75	-76	-73	-88
326.4	-83	-78	-79	-78	-81	-74
331.2	-58	-67	-69	-78	-74	-67
336	-59	-67	-64	-58	-60	-68
340.8	-61	-58	-63	-66	-68	-75
345.6	-70	-68	-66	-60	-62	-80
350.4	-72	-74	-67	-70	-69	-81
355.2	-66	-79	-68	-65	-62	-79
360	-74	-77	-79	-80	-80	-88
364.8	-83	-72	-65	-63	-71	-6
369.6	-78	-80	-68	-62	-74	-78
374.4	-82	-78	-71	-69	-77	-69
379.2	-76	-84	-82	-68	-78	-76
384	-75	-77	-77	-72	-75	-73
388.8	-74	-76	-75	-62	-77	-74
393.6	-75	-79	-72	-74	-80	-76
398.4	-76	-78	-70	-70	-79	-81
403.2	-70	-79	-80	-64	-76	-80
408	-60	-65	-59	-66	-69	-74
412.8	-90	-82	-67	-77	-72	-73
417.6	-67	-64	-59	-67	-72	-76
422.4	-73	-73	-69	-74	-74	-77
427.2	-76	-68	-67	-72	-71	-76
432	-68	-73	-63	-62	-72	-75
436.8	-61	-67	-62	-68	-70	-77
441.6	-56	-66	-59	-65	-78	-66
446.4	-59	-66	-62	-65	-73	-78
451.2	-55	-72	-66	-69	-71	-80
456	-59	-60	-64	-65	-55	-142
460.8	-60	-66	-74	-62	-46	-122
465.6	-57	-59	-63	-66	-58	-114
470.4	-63	-61	-61	-65	-53	-67
475.2	-61	-64	-59	-68	-50	-84
480	-50	-53	-45	-49	-52	-55
484.4	-56	-57	-58	-44	-57	-53
489.6	-50	-55	-51	-49	-46	-65
494.4	-57	-49	-51	-53	-44	-60
499.2	-46	-52	-62	-55	-53	-44
504	-42	-44	-45	-45	-39	-57
508.8	-52	-51	-41	-48	-45	-59
513.6	-53	-54	-47	-46	-49	-62

TABLE A3 CONTINUED

Exposure Time (Hours)	Electrode Potentials mV (SCE) of annealed (1000 o C, 1 hr) austenitic stainless steel immersed in 0.5M H2SO4					
	1 Cycle	2 Cycle	3 Cycle	4 Cycle	5 Cycle	As-received
518.4	-56	-49	-42	-46	-47	-60
523.2	-48	-60	-49	-48	-42	-50
528	-38	-43	-39	-42	-30	-45
532.8	-45	-50	-48	-47	-40	-56
537.6	-48	-50	-49	-50	-46	-67
542.4	-51	-37	-54	-54	-54	-54
547.2	-40	-49	-52	-55	-52	-66
552	-39	-37	-31	-31	-32	-53
556.8	-32	-47	-29	-41	-41	-51
561.6	-45	-26	-49	-43	-53	-40
566.4	-45	-35	-50	-54	-51	-57
571.2	-53	-50	-33	-55	-46	-59
576	-37	-35	-43	-39	-24	-45
580.8	-51	-43	-49	-52	-58	-35
585.6	-43	-49	-67	-59	-47	-43
590.4	-55	-42	-55	-67	-61	-44
595.2	-55	-50	-52	-62	-46	-47
600	-49	-46	-47	-47	-42	-47
604.8	-40	-46	-30	-47	-26	-46
609.6	-45	-46	-58	-55	-57	-47
614.4	-62	-50	-64	-44	-56	-48
619.2	-64	-52	-49	-50	-62	-67
624	-70	-67	-55	-58	-67	-54
628.8	-80	-75	-62	-61	-66	-61
633.6	-70	-68	-48	-62	-66	-58
638.4	-71	-73	-26	-56	-68	-47
643.2	-64	-70	-34	-59	-69	-54
648	-51	-38	-34	-44	-46	-49
652.8	-53	-56	-52	-52	-51	-57
657.6	-42	-62	-39	-56	-56	-68
662.4	-56	-62	-63	-61	-55	-62
667.2	-57	-55	-59	-58	-52	-59
672	-39	-47	-57	-67	-50	-47
676.8	-48	-60	-56	-52	-56	-67
681.6	-50	-15	-52	-51	-53	-51
686.4	-57	-45	-54	-62	-59	-69
691.2	-60	-44	-48	-51	-57	-54
696	-43	-42	-44	-33	-38	-44
700.8	-60	-44	-50	-31	-48	-51
705.6	-63	-66	-55	-46	-60	-60
710.4	-70	-58	-33	-39	-51	-51
715.2	-66	-62	-40	-52	-46	-45
720	-54	-33	-28	-34	-41	-42
724.8	-55	-55	-30	-43	-61	-64
729.6	-64	-49	-31	-46	-27	-45
734.4	-56	-63	-63	-24	-58	-70
739.2	-64	-60	-62	-47	-64	-66

TABLE A4: Electrode Potentials mV (SCE) of annealed (1000oC without soaking) austenitic stainless steel immersed in 0.5M H2SO4

Exposure Time (Hours)	Number of Cycles					
	1 Cycle	2 Cycle	3 Cycle	4 Cycle	5 Cycle	As-received
24	-258	-318	-318	-336	-323	-240
48	-312	-319	-307	-310	-305	-308
72	-328	-325	-322	-325	-302	-289
96	-298	-293	-289	-294	-299	-272
120	-290	-282	-287	-289	-291	-267
144	-297	-300	-290	-290	-289	-280
168	-293	-280	-285	-288	-293	-271
192	-298	-287	-270	-274	-289	-277
216	-262	-264	-284	-286	-279	-225
240	-231	-247	-258	-260	-272	-226
264	-69	-82	-75	-73	-81	-73
268.8	-70	-63	-78	-74	-65	-71
273.6	-69	-63	-68	-63	-61	-76
278.4	-77	-78	-73	-73	-73	-83
283.2	-74	-80	-76	-74	-81	-78
288	-59	-61	-59	-65	-61	-73
292.8	-59	-68	-77	-70	-64	-70
297.6	-67	-74	-69	-68	-79	-68
302.4	-65	-61	-72	-69	-78	-70
307.2	-69	-72	-77	-68	-77	-72
312	-82	-70	-78	-74	-73	-85
316.8	-82	-82	-83	-77	-78	-80
321.6	-80	-72	-77	-74	-79	-80
326.4	-79	-68	-89	-86	-81	-74
331.2	-80	-86	-67	-66	-70	-87
336	-72	-66	-62	-65	-66	-68
340.8	-59	-72	-70	-77	-73	-75
345.6	-52	-79	-74	-86	-75	-80
350.4	-74	-76	-76	-79	-80	-81
355.2	-76	-66	-70	-71	-83	-79
360	-91	-80	-77	-84	-99	-88
364.8	-78	-81	-82	-79	-76	-6
369.6	-82	-75	-72	-73	-67	-78
374.4	-76	-79	-76	-73	-76	-68
379.2	-89	-79	-68	-66	-73	-78
384	-72	-70	-66	-65	-74	-73
388.8	-77	-72	-60	-65	-75	-74
393.6	-79	-75	-64	-6	-70	-76
398.4	-74	-74	-62	-62	-73	-81
403.2	-76	-72	-60	-61	-68	-80
408	-68	-62	-63	-75	-78	-74
412.8	-75	-43	-54	-60	-64	-73
417.6	-71	-68	-70	-78	-62	-76
422.4	-70	-49	-69	-78	-70	-77
427.2	-71	-78	-58	-72	-71	-76
432	-65	-57	-57	-73	-60	-75
436.8	-74	-68	-61	-76	-66	-77
441.6	-58	-69	-62	-74	-60	-66
446.4	-66	-62	-61	-74	-23	-78
451.2	-67	-67	-64	-82	-69	-80
456	-63	-70	-100	-79	-91	-142
460.8	-63	-52	-75	-87	-85	-122
465.6	-54	-41	-83	-84	-72	-114
470.4	-51	-59	-79	-73	-70	-67
475.2	-59	-51	-63	-88	-67	-84
480	-48	-52	-70	-50	-64	-55
484.4	-57	-56	-49	-50	-59	-53
489.6	-45	-47	-49	-47	-57	-65
494.4	-61	-32	-35	-35	-47	-60
499.2	-50	-36	-33	-29	-42	-44
504	-42	-26	-37	-32	-41	-57
508.8	-60	-60	-58	-50	-57	-59
513.6	-47	-51	-52	-45	-55	-62

TABLE A4 CONTINUED

Exposure Time (Hours)	Electrode Potentials mV (SCE)of annealed (1000oC without soaking) austenitic stainless steel immersed in 0.5M H2SO4					
	1 Cycle	2 Cycle	3 Cycle	4 Cycle	5 Cycle	As-received
523.2	-54	-47	-51	-42	-54	-50
528	-40	-32	-47	-47	-50	-45
532.8	-50	-18	-35	-28	-32	-56
537.6	-44	-25	-50	-42	-47	-67
542.4	-53	-57	-38	-90	-18	-54
547.2	-51	-44	-57	-44	-57	-66
552	-38	-62	-65	-59	-58	-53
556.8	-45	-22	-32	-38	-42	-51
561.6	-62	-39	-54	-47	-43	-40
566.4	-58	-50	-64	-61	-56	-57
571.2	-53	-51	-59	-52	-52	-59
576	-34	-38	-29	-39	-40	-45
580.8	-45	-20	-59	-43	-49	-35
585.6	-58	-12	-66	-56	-31	-43
590.4	-57	-37	-37	-36	-49	-44
595.2	-48	-30	-45	-43	-36	-47
600	-40	-38	-44	-48	-50	-47
604.8	-39	-35	-57	-60	-37	-46
609.6	-46	-23	-24	-25	-25	-47
614.4	-59	-64	-66	-51	-58	-48
619.2	-60	-73	-60	-78	-56	-67
624	-70	-64	-58	-37	-50	-54
628.8	-64	-68	-60	-53	-54	-61
633.6	-50	-51	-27	-24	-63	-58
638.4	-56	-36	-42	-42	-44	-47
643.2	-68	-61	-59	-50	-47	-54
648	-21	-47	-49	-35	-32	-49
652.8	-34	-52	-56	-53	-58	-57
657.6	-56	-44	-61	-54	-65	-68
662.4	-63	-55	-60	-51	-51	-62
667.2	-60	-66	-60	-53	-51	-59
672	-50	-28	-38	-50	-52	-47
676.8	-51	-31	-59	-63	-58	-67
681.6	-48	-52	-50	-60	-62	-51
686.4	-58	-47	-55	-64	-64	-69
691.2	-52	-24	-34	-58	-57	-54
696	-41	-36	-45	-45	-45	-44
700.8	-53	-38	-43	-57	-56	-51
705.6	-59	-46	-60	-46	-62	-60
710.4	-43	-16	-36	-40	-28	-51
715.2	-60	-50	-37	-55	-42	-45
720	-43	-45	-42	-49	-37	-42
724.8	-55	-58	-55	-58	-45	-64
729.6	-55	-27	-48	-10	-57	-45
734.4	-65	-60	-53	-28	-29	-70
739.2	-23	-48	-32	-45	-56	-66

TABLE A5: Electrode Potentials mV (SCE) of 1 solution-quenched (1000oC, 1hr) austenitic stainless steel immersed in 0.5M H2SO4

Exposure Time (Hours)	Number of Cycles					
	1 Cycle	2 Cycle	3 Cycle	4 Cycle	5 Cycle	As-received
24	-325	-334	-325	-327	-330	-240
48	-301	-304	-312	-299	-302	-308
72	-315	-314	-322	-319	-315	-289
96	-293	-294	-309	-308	-299	-272
120	-281	-250	-284	-283	-276	-267
144	-289	-302	-295	-291	-277	-280
168	-289	-286	-290	-275	-280	-271
192	-274	-270	-268	-277	-274	-277
216	-273	-276	-272	-278	-276	-225
240	-261	-260	-241	-264	-244	-226
264	-75	-78	-91	-73	-77	-73
268.8	-75	-74	-76	-57	-66	-71
273.6	-77	-69	-62	-60	-61	-76
278.4	-69	-79	-86	-75	-67	-80
283.2	-68	-74	-92	-68	-75	-78
288	-66	-77	-70	-75	-56	-73
292.8	-63	-72	-80	-75	-70	-70
297.6	-71	-68	-67	-77	-75	-68
302.4	-83	-70	-76	-76	-70	-70
307.2	-73	-86	-76	-88	-62	-72
312	-80	-80	-77	-77	-71	-85
316.8	-82	-86	-83	-87	-79	-80
321.6	-84	-79	-88	-98	-72	-88
326.4	-82	-85	-92	-91	-73	-74
331.2	-79	-75	-74	-71	-77	-87
336	-63	-67	-64	-74	-59	-68
340.8	-73	-78	-68	-82	-70	-75
345.6	-76	-78	-71	-85	-70	-80
350.4	-81	-83	-73	-89	-70	-81
355.2	-84	-74	-71	-83	-87	-79
360	-84	-80	-75	-84	-82	-88
364.8	-83	-78	-70	-82	-65	-6
369.6	-70	-73	-62	-83	-69	-78
374.4	-71	-78	-68	-77	-66	-68
379.2	-71	-68	-66	-77	-65	-78
384	-76	-82	-70	-80	-59	-73
388.8	-64	-72	-68	-69	-63	-74
393.6	-60	-78	-65	-70	-65	-76
398.4	-72	-76	-70	-77	-68	-81
403.2	-72	-78	-73	-82	-63	-80
408	-82	-85	-83	-74	-63	-74
412.8	-64	-66	-64	-60	-71	-73
417.6	-73	-79	-65	-74	-66	-76
422.4	-77	-77	-67	-78	-75	-77
427.2	-77	-82	-61	-70	-70	-76
432	-74	-80	-76	-64	-72	-75
436.8	-63	-75	-76	-68	-72	-77
441.6	-70	-77	-76	-60	-75	-66
446.4	-77	-80	-82	-68	-73	-78
451.2	-77	-79	-73	-56	-78	-80
456	-69	-75	-79	-69	-73	-142
460.8	-61	-62	-66	-72	-66	-122
465.6	-53	-40	-47	-79	-58	-114
470.4	-73	-47	-60	-89	-77	-67
475.2	-63	-70	-77	-86	-100	-84
480	-58	-56	-52	-55	-56	-55
484.4	-59	-61	-59	-61	-53	-53
489.6	-61	-66	-61	-65	-57	-65
494.4	-56	-55	-42	-49	-48	-60
499.2	-50	-53	-45	-50	-45	-44
507	-45	-49	-47	-54	-42	-57
508.8	-59	-62	-56	-61	-53	-59
513.6	-58	-60	-57	-61	-55	-62
518.4	-48	-49	-47	-49	-48	-60

TABLE A5 CONTINUED

Exposure Time (Hours)	Electrode Potentials mV (SCE) of solution-quenched (1000oC, 1hr) austenitic stainless steel immersed in 0.5M H2SO4					
	1 Cycle	2 Cycle	3 Cycle	4 Cycle	5 Cycle	As-received
523.2	-50	-50	-42	-46	-39	-50
528	-50	-53	-50	-52	-45	-45
532.8	-49	-56	-64	-64	-57	-56
537.6	-42	-56	-53	-55	-48	-67
542.4	-47	-40	-46	-54	-60	-54
547.2	-57	-57	-54	-45	-45	-66
552	-50	-53	-51	-55	-63	-53
556.8	-46	-49	-50	-55	-50	-51
561.6	-53	-51	-49	-37	-47	-40
566.4	-57	-62	-59	-52	-54	-57
571.2	-65	-50	-51	-59	-44	-59
576	-42	-40	-44	-40	-40	-45
580.8	-32	-40	-34	-50	-45	-35
585.6	-32	-33	-36	-40	-36	-43
590.4	-41	-48	-46	-48	-47	-44
595.2	-38	-44	-40	-46	-44	-47
600	-20	-25	-26	-29	-32	-47
604.8	-25	-42	-56	-54	-55	-46
609.6	-29	-53	-36	-38	-17	-47
614.4	-21	-29	-25	-41	-37	-48
619.2	-28	-32	-30	-50	-36	-67
624	-25	-33	-41	-46	-43	-54
628.8	-58	-64	-62	-60	-58	-61
633.6	-52	-50	-52	-27	-48	-58
638.4	-51	-50	-53	-29	-47	-47
643.2	-39	-45	-47	-54	-44	-54
648	-16	-17	-21	-25	-33	-49
652.8	-38	-41	-44	-48	-48	-57
657.6	-65	-63	-60	-57	-52	-68
662.4	-60	-67	-55	-65	-56	-62
667.2	-64	-52	-53	-62	-60	-59
672	-49	-55	-54	-52	-50	-47
676.8	-68	-65	-61	-51	-63	-67
681.6	-41	-49	-56	-61	-59	-51
686.4	-61	-45	-57	-63	-63	-69
691.2	-64	-62	-64	-65	-41	-54
696	-58	-58	-53	-46	-64	-44
700.8	-63	-50	-69	-43	-57	-51
705.6	-41	-48	-46	-62	-37	-60
710.4	-39	-41	-55	-23	-50	-51
715.2	-57	-46	-64	-32	-60	-45
720	-28	-28	-49	-52	-65	-42
724.8	-39	-44	-47	-63	-71	-64
729.6	-46	-56	-51	-64	-60	-45
734.4	-51	-58	-53	-51	-67	-70
739.2	-56	-64	-55	-70	-53	-66

TABLE A8: Electrode Potentials mV (SCE (SCE) of solution-quenched (1100C without soaking) austenitic stainless steel immersed in 0.5M H_2SO_4

Exposure Time (Hours)	Number of Cycles					
	1 Cycle	2 Cycle	3 Cycle	4 Cycle	5 Cycle	As-received
24	-291	-318	-267	-314	-314	-240
48	-318	-316	-313	-319	-320	-308
72	-328	-325	-339	-327	-310	-289
96	-310	-305	-297	-301	-309	-272
120	-283	-282	-281	-280	-262	-267
144	-284	-284	-279	-277	-280	-280
168	-287	-291	-275	-282	-287	-271
192	-277	-277	-278	-292	-270	-277
216	-282	-275	-265	-274	-254	-225
240	-260	-258	-235	-248	-258	-226
264	-71	-72	-68	-69	-70	-73
268.8	-72	-76	-72	-74	-69	-71
273.6	-68	-67	-57	-80	-69	-76
278.4	-74	-77	-68	-72	-70	-80
283.2	-81	-70	-70	-66	-70	-78
288	-63	-55	-64	-62	-62	-73
292.8	-78	-70	-63	-80	-76	-70
297.6	-69	-62	-76	-64	-66	-68
302.4	-71	-67	-72	-67	-78	-70
307.2	-79	-70	-78	-76	-74	-72
312	-78	-79	-88	-86	-85	-85
316.8	-75	-92	-88	-80	-86	-80
321.6	-80	-78	-94	-71	-84	-88
326.4	-77	-98	-97	-86	-95	-74
331.2	-76	-77	-87	-84	-90	-87
336	-69	-67	-65	-63	-79	-68
340.8	-73	-76	-83	-75	-85	-75
345.6	-79	-75	-81	-74	-84	-80
350.4	-79	-77	-78	-72	-81	-81
355.2	-78	-77	-87	-76	-89	-79
360	-90	-90	-88	-77	-79	-88
364.8	-64	-66	-86	-65	-85	-6
369.6	-82	-70	-89	-78	-84	-78
374.4	-73	-72	-89	-76	-81	-68
379.2	-61	-62	-87	-77	-76	-78
384	-72	-68	-79	-65	-84	-73
388.8	-73	-70	-81	-78	-85	-74
393.6	-74	-70	-84	-75	-82	-76
398.4	-76	-69	-88	-75	-84	-81
403.2	-79	-72	-87	-66	-88	-80
408	-78	-80	-89	-68	-64	-74
412.8	-63	-58	-74	-70	-65	-73
417.6	-72	-64	-78	-75	-64	-76
422.4	-59	-66	-71	-77	-55	-77
427.2	-55	-56	-61	-84	-75	-76
432	-65	-66	-75	-78	-81	-75
436.8	-69	-69	-68	-74	-65	-77
441.6	-70	-64	-66	-73	-84	-66
446.4	-68	-74	-70	-71	-68	-78
451.2	-49	-48	-58	-66	-65	-80
456	-67	-69	-130	-64	-70	-142
460.8	-74	-73	-131	-33	-71	-122
465.6	-75	-99	-147	-36	-58	-114
470.4	-88	-43	-117	-56	-53	-67
475.2	-62	-57	-100	-72	-53	-84
480	-79	-55	-83	-28	-38	-55
484.4	-61	-57	-75	-26	-75	-53
489.6	-61	-63	-93	-61	-62	-65
494.4	-54	-53	-79	-54	-55	-60
499.2	-50	-48	-70	-49	-55	-44
504	-49	-45	-51	-54	-65	-57
508.8	-57	-56	-62	-54	-65	-59
513.6	-58	-60	-65	-59	-57	-62
518.4	-54	-42	-55	-53	-51	-60

TABLE [illegible] CONTINUED

Exposure Time (Hours)	Electrode Potentials mV (SCE) of solution-quenched 00oC without soaking austenitic stainless steel immersed in 0.5M H2SO4					
	1 Cycle	2 Cycle	3 Cycle	4 Cycle	5 Cycle	As-received
523.2	-47	-41	-57	-51	-52	-50
528	-49	-49	-46	-50	-65	-45
532.8	-62	-52	-50	-65	-54	-56
537.6	-32	-38	-46	-55	-52	-67
542.4	-37	-41	-49	-60	-56	-54
547.2	-58	-43	-51	-57	-63	-66
552	-62	-57	-65	-58	-69	-53
556.8	-55	-54	-59	-57	-60	-51
561.6	-56	-38	-51	-51	-63	-40
566.4	-66	-41	-57	-61	-58	-57
571.2	-61	-62	-54	-66	-47	-59
576	-40	-20	-30	-32	-53	-45
580.8	-47	-48	-34	-42	-43	-35
585.6	-39	-37	-43	-40	-49	-43
590.4	-50	-48	-40	-48	-50	-44
595.2	-42	-46	-44	-46	-34	-47
600	-34	-24	-34	-39	-34	-47
604.8	-36	-40	-42	-42	-48	-46
609.6	-40	-42	-48	-47	-43	-47
614.4	-45	-52	-50	-58	-50	-48
619.2	-44	-44	-57	-66	-34	-67
624	-48	-36	-42	-48	-65	-54
628.8	-25	-17	-69	-63	-62	-61
633.6	-51	-38	-37	-46	-52	-58
638.4	-49	-49	-48	-54	-52	-47
643.2	-50	-51	-39	-49	-50	-54
648	-37	-26	-16	-36	-45	-49
652.8	-48	-35	-34	-38	-51	-57
657.6	-55	-55	-55	-67	-67	-68
662.4	-58	-62	-53	-61	-62	-62
667.2	-42	-46	-60	-49	-38	-59
672	-43	-43	-57	-55	-51	-47
676.8	-64	-52	-63	-53	-65	-67
681.6	-55	-45	-57	-53	-47	-51
686.4	-67	-55	-62	-48	-69	-69
691.2	-53	-67	-66	-57	-61	-54
696	-34	-58	-47	-46	-35	-44
700.8	-50	-42	-50	-60	-42	-51
705.6	-51	-46	-65	-41	-48	-60
710.4	-53	-14	-53	-47	-57	-51
715.2	-56	-51	-52	-50	-36	-45
720	-19	-29	-32	-48	-56	-42
724.8	-32	-29	-34	-61	-70	-64
729.6	-37	-60	-32	-61	-65	-45
734.4	-53	-66	-67	-68	-66	-70
739.2	-45	-80	-63	-65	-67	-66

APPENDIX B

Table B1: Weight loss (mg) of normalized (1000°C,1hr) immersed in 0.5M H_2SO_4

Exposure time days	1 Cycle	2 Cycle	3 Cycle	4 Cycle	5 Cycle	As Received
5	1.4	3.2	4.8	3.3	1.7	1.2
10	0.9	1.0	1.7	0.9	0.5	0.9
15	0.3	0.5	1.0	1.1	0.4	0.3
20	0.2	0.5	0.7	0.5	0.4	0.2
25	0.2	0.6	0.3	0.2	0.1	0.1
30	0.1	0.4	0.4	0.1	0.1	0.0
Total	2.6	6.2	8.9	6.1	3.2	2.2

Table B2: Weight loss (mg) of normalized (1000°C without soaking) immersed in 0.5M H_2SO_4

Exposure time days	1 Cycle	2 Cycle	3 Cycle	4 Cycle	5 Cycle	As Received
5	2.8	2.6	5.0	2.5	3.6	1.2
10	0.9	0.7	1.4	0.7	1.1	0.9
15	0.6	0.7	1.1	0.8	0.8	0.3
20	0.4	0.3	0.8	0.4	0.6	0.2
25	0.2	0.2	0.4	0.2	0.3	0.1
30	0.1	0.3	0.6	0.2	0.3	0.0
Total	5.0	4.8	9.3	4.8	6.7	2.2

Table B3: **Weight loss (mg) of annealed (1000^{0}C, 1hr) immersed in 0.5M H_2SO_4**

Exposure time days	1 Cycle	2 Cycle	3 Cycle	4 Cycle	5 Cycle	As Received
5	2.2	3.7	4.5	2.6	2.7	1.2
10	0.6	1.1	1.9	0.8	0.7	0.9
15	0.6	0.8	0.8	0.7	1.0	0.3
20	0.3	0.6	1.0	0.4	0.3	0.2
25	0.2	0.3	0.5	0.4	0.2	0.1
30	0.1	0.2	0.1	0.0	0.2	0.0
Total	4.0	6.2	8.3	4.9	5.1	2.2

Table B4: **Weight loss (mg) of annealed (1000^{0}C without soaking) immersed in 0.5M H_2SO_4**

Exposure time days	1 Cycle	2 Cycle	3 Cycle	4 Cycle	5 Cycle	As Received
5	2.0	3.8	1.2	4.1	3.2	1.2
10	0.6	1.3	0.3	1.2	0.9	0.9
15	0.4	0.8	0.5	0.9	0.7	0.3
20	0.4	0.6	0.2	0.6	0.5	0.2
25	0.3	0.4	0.1	0.6	0.5	0.1
30	0.1	0.2	0.0	0.2	0.2	0.0
Total	3.8	7.1	2.3	7.6	6.0	2.2

Table B5: **Weight loss (mg) of Solution-quenched (1000^{0}C, 1hr) immersed in 0.5M H_2SO_4**

Exposure time days	1 Cycle	2 Cycle	3 Cycle	4 Cycle	5 Cycle	As Received
5	3.4	3.4	3.0	3.0	3.9	1.2
10	1.0	1.0	0.8	0.9	1.1	0.9
15	0.7	0.8	0.7	0.6	0.7	0.3
20	0.5	0.6	0.4	0.5	0.6	0.2
25	0.9	0.2	0.2	0.4	0.3	0.1
30	0.2	0.3	0.4	0.2	0.6	0.0
Total	6.2	6.3	5.5	5.6	7.2	2.2

Table B6: **Wight loss (mg) of Solution-quenched (1000^{0}C without soaking) immersed in 0.5M H_2SO_4**

Exposure time days	1 Cycle	2 Cycle	3 Cycle	4 Cycle	5 Cycle	As received
5	0.5	2.7	0.4	0.6	1.2	1.2
10	0.2	0.8	0.3	0.3	0.4	0.9
15	0.3	0.6	0.2	0.3	0.3	0.3
20	0.2	0.4	0.1	0.2	0.2	0.2
25	0.0	0.3	0.1	0.2	0.2	0.1
30	0.5	0.3	0.3	0.3	0.3	0.0
Total	1.7	5.1	1.4	1.9	2.6	2.2

APPENDIX C

Table C1: Corrosion Rate ($mg/cm^2/yr$) of normalized (1000^0C, 1hr) immersed in 0.5M H_2SO_4

Exposure time days	1 Cycle	2 Cycle	3 Cycle	4 Cycle	5 Cycle	As Received
5	8.03	18.36	27.55	18.94	9.76	6.89
10	1.15	2.87	4.88	2.58	1.43	1.15
15	0.57	0.97	1.91	2.10	0.77	0.57
20	0.29	0.72	1.00	0.72	0.57	0.29
25	0.23	0.69	0.34	0.23	0.11	0.11
30	0.10	0.38	0.38	0.10	0.10	0.00

Table C2: Corrosion Rate ($mg/cm^2/yr$) of normalized (1000^0C, without soaking) immersed in 0.5M H_2SO_4

Exposure time days	1 Cycle	2 Cycle	3 Cycle	4 Cycle	5 Cycle	As received
5	16.07	14.92	28.69	14.35	20.00	6.89
10	2.58	2.01	4.02	2.01	3.16	1.15
15	1.15	1.39	2.10	1.53	1.53	0.57
20	0.57	0.43	1.15	0.52	0.86	0.29
25	0.23	0.23	0.46	0.23	0.34	0.11
30	0.10	0.29	0.57	0.19	0.29	0.00

Table C3: Corrosion Rate (mg/cm^2/yr) of anncaled (1000^{0}C, 1hr) immersed in 0.5M H_2SO_4

Exposure time days	1 Cycle	2 Cycle	3 Cycle	4 Cycle	5 Cycle	As Received
5	12.63	21.23	25.83	14.92	15.50	6.89
10	1.72	3.16	4.02	2.30	2.01	1.15
15	1.15	1.53	1.53	1.39	1.91	0.57
20	0.43	0.86	1.43	0.52	0.43	0.29
25	0.23	0.39	0.57	0.46	0.23	0.11
30	0.10	0.19	0.10	0.09	0.19	0.00

Table C4: Corrosion Rate (mg/cm^2/yr) of annealed (1000^{0}C, without soaking) immersed in 0.5M H_2SO_4

Exposure time days	1 Cycle	2 Cycle	3 Cycle	4 Cycle	5 Cycle	As Received
5	11.48	21.81	6.89	23.53	18.36	6.89
10	1.72	3.73	0.86	3.99	2.58	1.15
15	0.77	1.53	0.97	1.72	1.34	0.57
20	0.57	0.86	0.29	0.86	0.72	0.29
25	0.34	0.46	0.11	0.69	0.57	0.11
30	0.10	0.19	0.00	0.09	0.19	0.00

Table C5: Corrosion Rate ($mg/cm^2/yr$) of solution-quenched (1000^0C, 1hr) immersed in 0.5M H_2SO_4

Exposure time days	1 Cycle	2 Cycle	3 Cycle	4 Cycle	5 Cycle	As Received
5	19.51	19.51	17.22	17.22	22.38	6.89
10	2.87	2.87	2.30	2.58	3.16	1.15
15	1.39	1.53	1.39	1.15	1.34	0.57
20	0.72	0.86	0.57	0.72	0.86	0.29
25	0.96	0.23	0.23	0.46	0.34	0.11
30	0.19	0.29	0.38	0.19	0.57	0.00

Table C6: Corrosion Rate ($mg/cm^2/yr$) of solution-quenched (1000^0C, without soaking) immersed in 0.5M H_2SO_4

Exposure time days	1 Cycle	2 Cycle	3 Cycle	4 Cycle	5 Cycle	As Received
5	2.87	15.50	2.30	3.44	6.89	6.89
10	0.57	2.30	0.86	0.86	1.15	1.15
15	0.57	1.15	0.38	0.57	0.57	0.57
20	0.29	0.57	0.19	0.29	0.29	0.29
25	0.00	0.38	0.11	0.23	0.23	0.11
30	0.48	0.29	0.29	0.29	0.29	0.00