CORROSION STUDIES ON SHAPE WELDED STEELS

A DISSERTATION

submitted in partial fulfilment of the
requirements for the award of the degree
of
MASTER OF ENGINEERING
in
MECHANICAL ENGINEERING
(With Specialization in Welding Engineering)

By

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CANDIDATE'S DECLARATION

I hereby certify that the works which is being presented in this thesis entitled **"CORROSION STUDIES ON SHAPE WELDED STEELS"** in partial fulfillment of the requirements for the award of the Degree of Master of Engineering in Mechanical Engineering (with Specialization in Welding Engineering) submitted in the Department of Mechanical & Industrial Engineering, University of Roorkee, Roorkee, is an authentic record of my own work carried out during the period from July 1995 to February 1996, under the supervision of **Dr. Satya Prakash,** Professor, Department of Metallurgical Engineering & **Shri Ajay Agarwal,** Lecturer, Department of Mechanical & Industrial Engineering.

The matter embodied in this thesis has not been submitted by me for award of any other degree.

Date : 6, March, 1996

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ABSTRACT

The present study has been done to analyze the aqueous corrosion behaviours of low carbon steel (C - 0.097%, Mn - 1.59%, Si - 0.24%, S - 0.024%, Cu - 0.3%, P - 0.04%) processed by the new method of shape welding and rolled steel (C - 0.12%, Mn - 0.4%, S - 0.01%) in the 92% and 96% concentrated sulphuric acid solutions. This shape welding technique is being developed to manufacture the components like rotor of shafts, reactor pressure vessels and valves etc. by welding method alone. Shape welding method has specific advantages for the manufacturing of thick walled components of any dimensions and to a wide range of high strength, creep and corrosion resistant properties.

The corrosive behaviour was studied under total immersion test (92% and 96% H_2SO_4 solution). The exposure time was around 250 hours. The corrosion rate was calculated by weight loss method and was calculated in terms of mdd.

The shape welded steel was found to have better corrosion resistance than the rolled steel of similar composition under similar conditions of temperature and concentration of H_2SO_4 solutions. Increase in temperature from 15^oC to 50^oC led to substantial increase in corrosion resistance for both the steels. While the concentration of H_2SO_4 from 92% to 96% led to a marginal increase in corrosion. The corrosion rates decreased with increase in time of exposure.

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CHAPTER - I

INTRODUCTION :

The emergent techniques of welding are fast replacing the conventional joining methods of rivetting fasteners etc. in engineering practices. These welded structures are subjected to a variety of corrosive environments; sound technical design makes it imperative to consider these environmental influences responsible for corrosion besides the conventional parameters like force and strength, in order to reduce cost and enhance service life and safety of these parts.

The influence of these service conditions is in no way straight forward, but is complicated by a host of different parameters. The problem of corrosion of welded structures has thrown up a challenge before the welding engineer. Extensive research is being carried out on various aspects of corrosion phenomenon to evolve corrosion resistant alloy and materials to suit specific service conditions.

The fact development of technology in heavy industries, power sector, transportation etc. requires development of large size components. Similarly in case of chemical industry, to increase the capacity, large size of reactors are required. Conventional methods of making such vessels leads to lot of inhomogeneity and defects, which may effect the performance of these equipment in service. Keeping the above requirements of large size components and reactors vessels, some works has been done in the area of shape welding i.e. forming the components by welding process. Here, mostly submerged arc welding is used and near shape components are formed by welding process. These shape welded products show isotropy as far as far as the mechanical properties are concerned. Thereby the shortcomings of rolling or forging or casting, where the mechanical properties are different in different directions is overcome in shape welding which provides near uniform property in all directions.

Corrosion becomes an important factor when we talk of chemical reactors or thermal plants etc. and so it is important to test the corrosive behaviour of shape welded steel under corrosive environment. Laboratory corrosion tests may give suitability, life expectancy of a given material in a particular environment. Inference regarding its actual behaviour in service can be drawn from the corrosion tests.

CHAPTER - II

SHAPE WELDING

2.1 SHAPE WELDING

The cost of construction for any production units like chemical and process engineering or units in energy engineering are reduced by increasing the unit output(1). This results in ever increasing requirements for the size of the individual components. This increase is limited, however, not only by the maximum production size of the individual component but also by the availability of the large component, and by the long delivery times in the case of failure of the unit.

Large components of such systems as steel vessels for chemical reactors, regenerators, separators and containers; rotors for rollers, centrifuges, and generator or turbine shafts; press dies, forging dies, and valve assemblies; and valve pump or turbine casings have been scaled up to such a size that it is generally not possible to manufacture the necessary semi-finished products from one piece. Basically, it is possible to manufacture one large piece composed of smaller parts by welding; this has been carried out during recent years when welding cast, forged or rolled parts to a component, inhomogeneities may occur, affecting the material performance in the weld joint region. In the worst cases, incomplete joint penetration, heat-affected zone (HAZ) embrittlement, or hot, cold or stress relief cracking occurs.

Alternative large component manufacturing methods, such as pre-stressed cast pressure vessels and pre-stressed concrete vessels, have been tested. They again presented special difficulties as a result of the high weight and volume and the poor in-service inspectability, so they are not considered for geometrically more complex parts.

The above problems prompted Thyssen Company on plans for constructing components of any dimension and shape, made only of weld metal. Instead of casting and forging or rolling a multilayer weldment is produced by the submerged arc welding (SAW) process. This method called "Shape Welding" for brevity has specific advantages for the manufacturing of thick-walled components of any dimensions and to a wide range of high strength, creep and corrosion resistant properties. Thyssen feels that this method will meet the most stringent safety requirements and be more economical than the conventional manufacturing methods(2).

2.1.1. ADVANTAGES OF SHAPE WELDING (1)

The advantages of shape welding technique are :

1. For build-up welding there is no limitations as to component shape, size and weight.

2. The investments for a shape welding unit are low in comparison to the costs for expansion of conventional process stages.

3. No extensive manufacturing units for filler materials are necessary, as the welding electrodes are produced in the available modern continuous casting and rolling plants.

4. There is less risk of rejects in employing the shape welding method, because possible defects occurring during manufacturing can easily be detected by means of non destructive examination and repaired if required.

5. The weight of the machined shape welded component is usually over 80 percent that of the continuous casting for the welding electrodes.

6. The shape welding units can be dismantled and assembled on-site in case of transportation problems.

7. It is possible to compose the body layer by layer from material with various chemical composition or strengths for low or high temperature applications and severe environmental conditions.

8. Homogeneity and purity of the alloy composition of the final product are obtained because the continuous casting guarantees a uniform alloy distribution in the welding electrode.

9. For a given alloy content, there is an increase in both strength and toughness of shape welded structures compared to those manufactured by conventional methods.

10. Cost-benefit analysis(2) of the production of parts by shape welding shows that this process is a viable alternative to forging, particularly for the parts of intricate geometry. The economy of this method is further confirmed by other studies also (3), (4).

2.1.2 SCOPE OF SHAPE WELDING (1)

It is anticipated that the potential of this new technology will include :

1. The possibility exists of fully hardening and tempering low alloy steel vessels of any thickness by means of weld layer heat treatment. A substantial transformation in the lower bainite and, thus, thus an optimum upper shelf toughness at very low nil ductility transition(NDT) temperature is obtained by cooling rapidly behind the weld spot.

2. No macro-segregation develops because there are only small solidification areas.

3. The shape welded parts are homogeneous and isotropic with respect to all material characteristics. They do not have any forging or rolling texture which may cause a strength and toughness decrease in the transverse direction in rolled plates or forged courses due to the banded structure, and they do not have course dentritic primary solidification zones as occur in the course of casting or electroslag welding.

4. Optimized weldability exists in the shape welded semi-finished products, since the weld metal has a lower carbon content than the base metal with increased strength and enhanced toughness; thus, a lower maximum hardness can be obtained. The overheated region of the HAZ is less susceptible to course grain formation than in forged base metal.

5. Weldments exhibit a very small grain size which increases the strength and the toughness as a result of multiple annealing and rapid cooling after the welding process.

6. Excellent ultrasonic inspectability results from the small grain size.

2.2 LABORATORY CORROSION TESTS

Laboratory corrosion tests are used for a wide variety of purposes, and the type of corrosion test that will be found useful depends upon the end results desired. These tests are widely used because of its simplicity, low cost, short duration, adaptability to multiple tests and ease of operation (5). It was hoped that some form of an accelerated corrosion tests could be made available which in a short time would give a rating of the behaviour of the various materials that could be correlated with service exposures. The first laboratory tests on the selected materials should be as simple as possible, the nature of the environment in which the material is to be used being kept in mind and at least the more important controlling factors being simulated. It is often advisable to make use of existing equipment and environments for the purpose of exposing small samples of the materials that are being considered (6).

2.2.1 TOTAL IMMERSION TEST

This test usually consists of the immersion of one or more metal test specimens in a small volume of solution for some period of time. The total immersion test performed with proper precautions can be made a reliable type of corrosion test. Adequate control of the important variables is in general obtainable. Complete immersion of the specimen in the corrosive provides relatively simple conditions, but a number of factors must be controlled to attain adequate reproducibility of the results. Bengough and Wormwell (7) have pointed out that surface, size, shape and method of suspension the specimen, depth of immersion, volume of the solution are the physical factors that may affect the corrosion rate.

The main difficulty in total immersion test is that there must be contact of the specimen with its support in the corrosive environment, and that corrosion may be locally accelerated by this contact. The effect may be minimised by using point contacts and the minimum number of contacts. Horizontal specimens are sometimes advantageous, since they can be viewed without removal. But where both sides of a horizontal specimen are exposed to corrosive environment, it is possible that the different conditions prevailing at the upper and lower surface may complicate the corrosion test. Another way of avoiding this possibility is to arrange the specimen vertically, for example suspending the specimen from a glass or nylon rod or thread, although this requires the provision of at least one hole in the specimen (8). In general, a depth of not less than 2.0 cm is recommended for applied corrosion tests. The volume of solution recommended per sq. cm of metal surface varied from 6 ml to 40 ml.

A constant rate of corrosion is encountered much more often in total immersion test than in other (6). Nevertheless it is not safe to assume that this relationship holds without the supporting evidence of a corrosion time curve. If the test is of too short duration, some materials build up protective corrosion product films slowly may be ruled out as unsatisfactory. If a test is too long, the effects of exhaustion of ingredients or accumulation of corrosion products may be pronounced.

CHAPTER - III

CORROSION

3.1 Definition and Importance

Corrosion is a destructive attack of a metal by chemical or electrochemical reaction with its environment. Deterioration by physical causes is not corrosion, but is described as erosion, wear etc. Eg. Rusting applies to corrosion of iron-base alloys(9).

Many authorities have give 'transformation' definition of corrosion in which metallic corrosion embraces all interactions of a metal or an alloy (solid or liquid) with its environment, irrespective of whether this is deliberate and beneficial or unintended and deleterious.

The scope of the term 'corrosion' is continually being extended by the recent research scholars. Fontana and staehle have stated that corrosion will include the reaction of metals, glasses, ionic solids, polymeric solids and composites with environments that embrace liquid metals, gases, non-aqueous electrolytes and other non-aqueous solutions. Vermilyea has defined corrosion as a process in which atoms or molecules are removed one at a time, and considers that evaporation of metal into vacuum should come within the scope of the term, since atomically, it is similar to other corrosion processes[10]. Evans regards corrosion as a branch of chemical thermodynamic or kinetics, as the outcome of electron affinities of metals and non-metals, as short circuited electrochemical cells, or as the demolition of the crystal structure of a metal(11).

Corrosion studies have enormous importance in the present context. The first area of significance is economic, including the objective of reducing material losses resulting from the corrosion of piping, tanks, metal components of various machinery etc. The second area is improved safety of operating equipment, which through corrosion, may fail with catastrophic consequences, as in pressure vessels, boilers, nuclear reactors etc. Third is conservation, applied primarily to metal resources, the world's supply of which is limited. Equally important is the accompanying conservation of human effort entering into the design and rebuilding of corroded metal equipments.

Economic losses are divided into :

1. Direct losses and
2. Indirect losses

Direct losses include the cost of replacing corroded structure and machinery or their components. The indirect losses are more difficult to access. Typical examples of this kind are as follows.

a. Shut-down

b. Loss of product

c. Loss of efficiency

d. Contamination of a product

e. Overdesign.

3.2 Types of Corrosion

Corrosion has been variously classified by different authorities. A broad classification of corrosion into five major types is as follows.

1. Uniform attack

In this form of attack, all areas of the metal corrode at the same (or similar) rate. Eq. Oxidation and tarnishing; active dissolution in acids; anodic oxidation and passivity, chemical and electrochemical polishing; atmospheric and immersed corrosion in certain cases.

Rate of uniform attack is measured in various units like inches penetration per year(ipy), **milligrams** per square decimeter per day (mdd) etc, Metals are generally classified according to corrosion rates and intended application as.

a) <0.005 ipy (<0.015 cm/year)

Metals in this category have good corrosion resistance to the extent that they are suitable for critical parts eg. value seats, impellers etc.

b) **0.005 to 0.05 ipy (0.015-0.15 cm/year)**

Metals in this group are satisfactory if a higher rate of corrosion can be tolerated. Eg tanks, Bolts heads etc.

c) **>0.005 ipy (>0.015 cm/year)**

Usually not satisfactory

2. **Localised attack**

Here certain areas of the metal surface corrode at higher rate than others due to heterogeneities in the metal, the environment, or in the geometry of the structure as a whole. Attack ranges from slightly localised to pitting. The various heterogeneities in metals are as follows :

i) **Atomic heterogeneities.**

a) Sites within a given surface layer : which vary according to the particular crystal plane.

b) Sites at edges of partially completed layers.

c) Point defects in the surface layer : vacancies (molecules missing in the surface layer), kink sites (molecules missing at edge of layer), molecules adsorbed on top of complete layer.

d) Disordered molecules at point of emergence of dislocations (screw or edge) in metal surface.

ii) **Microscopic heterogeneities**

a) Grain boundaries-usually, but not invariably, more reactive than grain interior.

b) Phase-metallic (single metals, solid solutions, intermetallic compounds), non-metallic metal compounds, impurities, etc. - heterogeneous due to thermal or mechanical causes.

iii) Macroscopic heterogeneities

a) Grain boundaries

b) Discontinuities on metal surface-out edges, scratches, discontinuities in oxide films (or other chemical films) or in applied metallic or non-metallic coatings.
or in applied metallic or non-metallic coatings.

c) Bimetallic couples of dissimilar metals.

d) Geometrical factors-general design, crevices, contact with non-metallic materials etc.

3. Pitting

Highly localised attack at specific areas resulting in small pits that penetrate into the metal and may lead to perforation. Refer fig. (3.2.a) Eg. Iron burried in soil, stainless steel immersed in sea water etc.

4. Selective Dissolution

One component of the alloy (usually the most active) is selectively removed from alloy. Eg. Dezincification, dealuminification, graphitisation etc. The alloy so corroded often retains its original shape, and may appear undamaged except for surface tarnish, but its tensile strength and especially ductility are seriously reduced.

5. Conjoint action of corrosion and a mechanical factor

Localised attack or fracture due to the synergistic action of a mechanical factor and corrosion. Eg. erosion-corrosion, impingement attack wherein metals corrode when subjected to high velocity liquids, fretting corrosion which results from slight relative motion of two substances in contact, cavitation damage, stress corrosion cracking, hydrogen cracking, corrosion fatigue etc.

3.3. Mechanical of Corrosion

Corrosion takes place by either direct chemical action or due to setting of electromechanical cells i.e. by galvanic action. The two mechanism, however can not always be separated. Corrosion by direct chemical attack occurs when a metal enters into a chemical reaction with other elements such as O_2, Cl_2 and SO_2 etc. thus forming non-metallic compounds eg. pickling of steel.

The three main types of cells that take part in corrosion reactions are as follows :

1. Dissimilar electrode cells

A metal containing electrically conducting impurities on surface as a seperate phase, a copper pipe connected to an iron pipe, illustrate this type of cell. It also includes cold worked metal in contact with the same metal annealed, grain boundary metal in contact with grains, and a single metal crystal of definite orientation in contact with another crystal of differing orientation.

2. Concentration Cells

These cells have two identical electrodes each in contact with a solution of differing composition. It is further divided into two types .

a) Salt concentration cell

Eg. one copper electrode is exposed to a concentrated copper sulphate solution and another to a dilute copper sulphate solution. Refer Fig. (3.3.a). On short circuiting, copper dissolves at anode (electrode in contact with dilute $CuSO_4$) and plates out on cathode. The reaction tends to bring both solutions to same concentration.

b) Differential aeration cell

Eg. two iron electrodes in dilute sodium chloride solution, the electrolyte around one electrode being thoroughly aerated (cathode) and the other deaerated (anode). Refer Fig. (3.3.b).

The difference in oxygen concentration produces a potential difference and causes current to flow .

It also accounts for crevice corrosion at the interface of two coupled pipes or at threaded connections or pitting damage under rust. ref. Fig. (3.3.c).

c) Differential temperature cells

Electrodes of the same metal, each of which is at different temperature immersed in an electrolyte of the same initial composition sets up this cell. Electrode at higher temperature is cathode and that at lower temperature is generally anode. However, the polarity may change for different materials. This is especially important in heat exchangers, boilers etc.

CHAPTER -IV

CORROSION STUDIES :

Corrosion resistance of some ferrous metals in 92% sulphuric acid was studies by E.V. Lyalin (12). The effect of temperature, the motion of the electrolyte and the pressure on the corrosion of the metal was studied. Corrosion was measured by the loss of weight and the change in dimension of the sample. The samples were measured with a micrometer and weighed on an analytical balance before and after the corrosion tests. The ferrous metals studied were Sch 15-32 cast iron (GOST 1412-54); Sch 21-40 Cast iron; MSch 28-48 and MSCh 38-60 cast iron.

It was found that the corrosion decreases with time, apparently due to the formation of protective film on the surface of the metal.

The corrosion rate, like the rate of many chemical reactions, was found to increase with temperature. In unagitated sulphuric acid at 20°C and 50°C the Kh-28 alloy and sormite are almost completely corrosion resistant while at 100°C there is considerable corrosion (tests were made at 20°C, 50°C and 100°C.

It was shown that agitation of the medium increases the corrosion rate by approximately two times with respect to unagitated solution. The motion of the solution increases the supply of oxygen to cathodic areas and fresh aggressive medium to the surface of the metal, inducing acceleration of the corrosion.

For all metals the variation of the pressure had no effect on the corrosion rate. Among all metals the high chromium Kh-28 cast iron is the most resistant under the conditions tested. Corrosion of mild steel (both hot and cold rolled) and type 304 stainless steels in welded and unwelded form was studied in the industrial environment of a steel works by K.G. Mishra and C.R. Das (13). Corrosion rates were determined by weight loss and the nature of the corrosion damage and its propagation were studied by optical and scanning electron microscopy. The welded mild steel samples corroded faster than the unwelded specimens.

Experimental study of pitting corrosion in mild steel and welded mild steel and welded mild steel was carried out by G.R. Wallwork (13a). The experimental technique chosen was a scanning microprobe potentionmetric method. The electrolyte was NaCl. The experiments were designed to evaluate the relationship between sulphide inclusions and pitting corrosion in steels and weld metal of various Mn and sulphur concentration. The sulphur concentration range was from 0.002-0.32%. The literature suggests that pitting corrosion in mild steel is strongly related to the morphology, distribution and composition of sulphide inclusion. The higher sulphur specimens had the highest potential for pitting corrosion.

T.P.S. gill etc. (14) studied the effect of heat input and microstructure on pitting corrosion in AISI 316L submerged arc welds. The pitting resistance of AISI 316L stainless steel weld metal in the as welded and stress relieved conditions was evaluated as the function of heat input. The results obtained indicated a decrease in the value of critical pitting potential in acidic chloride medium as the heat input was increased. However, on stress relieve heat treatment, the critical pitting potential remained unchanged with varying heat input, though its magnitude was much lower than that of the as welded alloy. The deterioration in the pitting resistance on increasing heat input is discussed on the basis of enhanced segregation of solute elements like chromium and molybdenum to delta ferrite and the presence of $M_{23}C_6$ carbides at the austenite/Ferrite interface.

Stress corrosion studies were conducted on weldments of cold rolled 316 stainless steels in boiling solution 5M NaCl + 0.15M Na_2SO_4 using the constant load method at a nominal stress level of 390 mPa (15). As welded and post weld heat treated samples were employed in these studies. It was found that time to fracture decreased with increasing cold work. Failure always occurred in the heat affected zone.

Murthy and Sharma (16) carried out a detailed study of a arc welded 0.23% carbon steel blade. It showed that probably from damp or cellulosic coated electrodes, the gas diffused into parent metal and reacted with maganese sulphide inclusions producing excessive gas pressure. The fracture and observations are described.

The corrosion of mild steel and stainless steels of the type AISI 304 and 316 with and without welding has been studied in marine atmosphere together with some samples of mild steels with magnetic coating (17). The corrosion rate has been determined in each case by loss in weight of the specimens and the nature of corrosion has been examined by the X-ray diffraction and microscopy. The corrosion rate of mild steel is much more than that of stainless steels. Gas welded specimens were found to corrode more quickly than arc welded samples. Magnetite coating properly bonded to the metal surface reduced the rate of corrosion considerably.

The corrosion fatigue behaviour of mild steel & in aqueous 3.5% NaCl solution has been investigated by Tong etc. (18). The corrosion fatigue crack growth rate and the variation of transient polarization currents have been studied simultaneously under anodic and cathodic polarization conditions and at open circuit potential. Under cyclic loading in a corrosive environment, the initiation and growth of fatigue cracks are controlled by both the mechanical parameters and the electrochemical characteristics of the system.

L. Bertini (22) studied the influence of sea-water and residual stresses on fatigue crack growth in C-Mn steel and weld joints. Tests were conducted to identify the behaviour at different locations of the joints as the frequency was varied. Residual stresses in specimens with welds are evaluated to analyse the fatigue crack growth behaviour. Satisfactory predictions were obtained by accounting for residual stresses.

The effect of aerated stagnant solution of mono, di, and trichloroacetic acids on the corrosion behaviour of low carbon steel at different pH values from 1 to 4 and at concentrations of 2.0 to 10 M has been studied by G.K. Gomma, El-Baradie, R.M. Isa and H.E. Shokry. The corrosion rate decreased with increasing pH value from 1 to 4.

EFFECT OF INHIBITORS :

Different investigations have been carried out to study the inhibition of corrosion of mild steels on different organic and inorganic inhibitors. Sodium nitrite has been found to be an effective inhibitor for corrosion of mild steels in aqueous solution of sodium chloride (19).

The corrosion of mild steel is reduced and the potential of steel is ennobled as the concentration of nitrite is increased in aqueous solution of sodium chloride. Sodium nitrite remained effective at higher temperatures up to 353K. The effect of temperature on the corrosion inhibition of low carbon steel in 3% NaCl solution has been investigated by J.C. Lin and H.C. Shih (20). A mixture of aluminium sulphate and thiourea was found to be an effective corrosion inhibitor at temperatures less than 50°C, but both the mixture and aluminium sulphate alone accelerate corrosion at temperature above 60°C.

Sodium benzoate was found to be an effective inhibitor for corrosion of mild steel in aqueous NaCl solution under various experimental conditions (21). The minimum concentrations of benzoate required for complete protection of mild steel in aqueous solution of NaCl for one month at room temperature were found.

CHAPTER - VI

FORMULATION OF PROBLEM :

A critical review of the available literature shows that most of the steels and its alloys are readily corrodable in corrosive environment. It also shows that weld metals are more sensitive to corrosion problems due to the inhomogenities in microstructure and composition, defects and residual stresses which may occur in certain unavoidable conditions. Even though laboratory corrosion tests does not reproduce all the factors involved in the actual field of application, they can be used as an arbitrary performance test for materials before commercial exploitation.

Large components of many system like vessels for chemical reactors, rotors for rollers, generators or turbine shafts and pump and valves, etc. have been scaled up to such a size that it is generally not possible to manufacture the necessary semi finished products from one single piece. Basically, it is possible to manufacture one large piece composed of smaller parts by welding process. Which welding of cast, forged or rolled products, inhomogenities may occur, affecting the material performance in the weld joint design. Keeping in view these problems, plans for components of any dimension and shape, made only of weld metal, have been embarked. Instead of casting and forging or rolling and then welding, a single multilayer weldment is produced by the submerged arc welding process. This method called shape welding has specific advantages for the manufacturing of thick walled components of any compositions, size and shape.

The shape welding technique is still in the developmental stage and has not been yet extensively put into commercial applications, and a very limited studies had been carried out on the corrosion behaviour of these shape welded steels. In the present work, an attempt had been made to study the corrosion behaviour of shape welded steel in sulphuric acid environment and to compare it with the corrosion behaviour of rolled steel of similar composition in similar environment.

In the chemical industry equipment, or parts of equipment are often exposed to sulphuric acid of high concentration. Among such pieces of equipment are the pumps used for sulphuric acid during its production or subsequent use, sulphuric acid is used as a sealer and lubricant in rotational chlorine vaccum compressors used in the rectification of chlorine, and also as a lubricant in the cylinders of chlorine compressors.

CHAPTER - VI

EXPERIMENTAL TECHNIQUES AND PROCEDURES

Low carbon steel specimens manufactured by the method of shape welding and rolled carbon-manganese steel were used for corrosion studies in sulphuric acid solution. Shape welding samples used in present investigation (processed by the submerged arc welding) were supplied by the Welding Research Laboratory, University of Roorkee. The composition of steels used are given in tables 1.1 and 1.2. The parameters used in shape welding are mentioned in table 1.3.

6.1 PREPARATION OF SPECIMEN

The given steel blocks were made into number of rectangular specimens of sizes mentioned in table 2.1. Holes were also drilled through them for hanging them into the corrosive environment. The specimens were first machined on a surface grinder to remove the irregularities on the surface as well as the thick layer of rust. Then the specimens were polished on silicon carbide emergy paper of grade 120, 180, 320, 500, 600, 800, 1000 and 1200 in that order. Fine scratches were removed by polishing on the polishing wheel. The polished specimens were rinsed in distilled water and cleaned with acetone and dried. The exact dimensions of the prepared specimens had been measured with the help of vernier calliper. The surface area of all the specimens were calculated from the dimensions, they are mentioned in table 2.1.The specimens were weighed on an electronic balance.

H_2SO_4 solutions of concentration 92% and 96% were prepared using distilled water. The solution was poured into different beakers for immersion test. The prepared specimens of shape welded steel were suspended in solution with the help of plastic threads which were tied to a rod supported at the mouth of the beaker. The testing time choosen was around 250 hours for each sample. The bath temperatures were kept at $15^{o}C$, $35^{o}C$ and $50^{o}C$ for both the 92% and 96% concentration solutions. One sample from shape welding and the other from rolled plate were dipped in a particular solution simultaneously. The weights of the samples were taken after every 24 hours and weight loss was recorded for all the samples.

6.2 METHODS OF INSPECTION

Apart from the visual inspection the samples were polished and photographed for macro and microstructures. For macro-structure the magnification was 12X and for microstructures it was kept at 200X.

6.3 SCANNING ELECTRON MICROSCOPY

Surface morphologies of the specimens were examined by SEM. The scanning electron microscopy was carried out on PHILIPS SEM-501, which was provided by the University Scientific Instrumentation Centre, University of Roorkee. The photographs taken are attached in the figure

6.4. CLEANING OF THE SPECIMEN AND CALCULATION OF CORROSION RATE

This is one of the most important steps in corrosion testing.

Cleaning procedure consists of holding the specimen under a stream of tap water and vigourously scrubbing the surface with rubber stopper. If appreciable quantities of corrosion product adheres to the specimen, we can go for chemical or electrochemical means.

The specimens were weighed again after cleaning. The weight loss can be calculated by subtracting it from the initial weight. The rate of corrosion can be calculated from the following formulae

$$r = \frac{W_1 - W_2}{2(lb + bw + lw)} \times \frac{1}{t} \text{ mdd}$$

where,

r = rate of corrosion in milligrams per square decimeter per day (mdd)

W_1 and W_2 = initial and final weights of the specimens respectively in milligrams.

l, b, w = length, breadth and width of the specimen respectively in dm.

t = time of exposure in days.

CHAPTER - VII

RESULTS AND DISCUSSION

The results of present study on the corrosion behaviour of shape welded steel and rolled steel in conc. H_2SO_4 solution under total immersion test for various temperatures and concentrations are given below.

7.1 VISUAL INSPECTION

The exposed samples were examined visually after their removal from corrosive environment. During the experiment some amount of corrosive product fell to the bottom of the beaker. The precipitated product has the colour of rusty yellowish brown. A thin greenish layer of ferrous sulphate also got deposited on the samples.

7.2 CORROSION RATE

The weight loss was found out for all samples after every 24 hours of immersion in H_2SO_4 solution. The samples were washed in tap water and dried and then weighed. Corrosion rate in mdd (milligrams per square decimeter per day) was calculated from weight loss, surface area and time of exposure for each specimen. These values are given in tables 3.1 to 8.2.

The graphs, depicting a comparison between the corrosion rates of shape welded steel and rolled steel vs time at constant concentration and temperature of H_2SO_4 solution, have been drawn nin figures 1.1 to 1.6

The graphs shown in figures 1.7, 1.8, 1.9 depict the effect of variation of concentration of H_2SO_4 solution (at constant temperatures) on the corrosion rates of shape welded and rolled steel. The graphs in figure 1.10, 1.11 show the effect of variation of temperature on the corrosion rates of shape welded and rolled steel (at constant concentration of H_2SO_4 solution). Two concentrations of 92% and 96% H_2SO_4 and three temperatures 15°C, 35°C and 50°C were used for total immersion tests.

In the immersion test the corrosion rate was found to be decreasing with increasing time of exposure. The decrease in corrosion rate with time in immersion tests indicates that corrosion reaction is perhaps a diffusion controlled process. The reactions are controlled by the diffusion of oxidant to the metal oxide interface and the diffusion of metal ions to the oxide solution interface. With increase in period of exposure the thickness of oxide layer increases, which makes the diffusion in both the directions difficult.

Among the shape welded and rolled steel, the shape welded one proved to be better corrosion resistant under similar conditions of temperature and concentration of H_2SO_4 solution. From the graphs shown in figures 1.1 to 1.6 it is clear that the ratio of the initial corrosion rates of rolled and shaped welded steel is around 2.5.

From the graphs shown in figures 1.7, 1.8, 1.9 it is clear that corrosion rates of both shape welded and rolled steel increased marginally with increase in concentration of H_2SO_4 solution from 92% to 96% at constant temperature.

The graphs shown in figures1.10, 1.11 depict clearly that the corrosion rates of both shape welded and rolled steels increased substantially with increase in temperature (the concentration of H_2SO_4 solution being held constant). The corrosion rates of both shape welded and rolled steel almost doubled when temperature was increased from 15°C to 50°C.

The rolled steel has 0.12% carbon content. At this carbon content the major phase is ferrite and the minor one is pearlite. In the figures 6.1(b) pearlite is the darker portion deposited mainly along the grain boundaries. The microstructure of shape welded steel (figure 6·1(a))contains accicular structure consisting of a mixture of pearlite and accicular ferrite (depicted by the darker region in the figure 6·1a). The white region depitcted in the microstructure is the accicular ferrite content.

The better corrosion resistance of shape welded steel in comparison to rolled steel is perhaps due to the presence of accicular ferrite which has a needle like structure. This accicular ferrite is fairly hard in character. In rolled steel the local cells tend to form easily due to the difference in phases (pearlite and ferrite) which in turn leads to dissolution of one phase in sulphuric acid. In the case of shape welded steels the cell formation is comparatively difficult due to less difference in phases and thus dissolution due to corrosion becomes less.

CHAPTER - VIII

CONCLUSION

Conclusions of the present investigation on the corrosion behaviour of shape welded and rolled steels in concentrated. H_2SO_4 solution under total immersion tests are summarized as below -

1. Shape welded steels have been found to have better corrosion resistance in comparison to rolled steel of almost similar composition under similar conditions of temperature and concentration of H_2SO_4 solution that were used in the experiment. The corrosion rate of rolled steel was found to be around 2.5 times of that of the shape welded steel.

2. The increase in H_2SO_4 concentration from 92% to 96% and keeping the temperature constant led to a marginal increase in corrosion rates of both shape welded and rolled steel.

3. The increase in temperature from 15^oC to 50^oC led to a substantial increase in corrosion rates for both the shape welded and rolled steels. The corrosion rates for shape welded and rolled steel at 50^oC were found to be almost double from the corresponding ones at 15^oC.

4. The corrosion rates for both the shape welded and rolled steel decreased with increase in time of exposure.

CHAPTER - IX

SCOPE FOR FUTURE WORK

It is suggested that further work can be carried out in the following areas -

1. Studies can be carried out at higher temperatures with the identical specimens of the actual components to get results nearer to service conditions.
2. The effects of different organic and inorganic inhibitors and other protective methods can be studied.
3. Studies can be carried out with a longer duration of time and with various corrosive environments like chloride and ammonium sulphate mediums.

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TABLE 1.1

CHEMICAL COMPOSITION OF THE SHAPE WELDED STEEL

C	Mn	Si	Cu	S	P	Fe
0.09%	1.5%	0.3%	0.3%	.023%	.04%	97.737%

TABLE 1.2

CHEMICAL COMPOSITION OF THE ROLLED STEEL

C	Mn	S
0.12%	0.4%	0.01%

TABLE 1.3

WELDING PARAMETERS USED IN SHAPE WELDING

VOLTAGE (VOLTS)	CURRENT (AMPERES)	WLEDING SPEED (cm/minute)	HEAT INPUT (kJ/cm)
27	600	50	19.4

TABLE 2.1

DIMENSIONS OF SPECIMENS

SPECIMEN NO.	DIMENSIONS IN MM LENGTH l	BREADTH b	WIDTH w	TOTAL SURFACE AREA IN mm^2 $2(l.b+b.w+l.w)$
1.	19.40	19.90	9.45	1514.89
2.	19.50	19.90	9.52	1526.28
3.	19.24	19.96	9.46	1509.72
4.	19.52	19.90	9.45	1524.71
5.	19.32	19.74	9.48	1503.33
6.	19.30	19.88	9.49	1511.08
7.	19.32	19.85	9.42	1504.97
8.	19.80	19.30	9.60	1515.02
9.	19.46	19.70	9.45	1506.72
10.	19.60	19.52	9.55	1512.9
11.	19.32	19.87	9.45	1508.46
12.	19.42	20.04	9.61	1536.78

TABLE 3.1

CORROSION OF SHAPE WELDED SPECIMEN (1) IN 92% H_2SO_4 SOLUTION AT 15^0C TEMPERATURE UNDER TOTAL IMMERSION TEST

TIME IN HRS	WEIGHT IN GMS	CORROSION RATE IN MDD
0	27.200	
24	27.170	198.0
48	27.140	198.0
72	27.120	176.0
96	27.100	165.1
120	27.080	158.4
164	27.060	135.2
210	27.050	113.2
250	27.040	102.4

TABLE 3.2

CORROSION OF ROLLED STEEL SPECIMEN (2) IN 92% H_2SO_4 SOLUTION AT 15^0C TEMPERATURE UNDER TOTAL IMMERSION TEST

TIME IN HRS	WEIGHT IN GMS	CORROSION RATE IN MDD
0	29.380	
24	29.300	524.2
48	29.220	524.2
72	29.160	480.5
96	29.110	442.2
120	29.080	393.0
164	29.050	316.4
210	29.030	262.1
250	29.020	226.4

TABLE 4.1

CORROSION OF SHAPE WELDED SPECIMEN (3) IN 92% H_2SO_4 SOLUTION AT 35^0C TEMPERATURE UNDER TOTAL IMMERSION TEST

TIME IN HRS	WEIGHT IN GMS	CORROSION RATE IN MDD
0	26.990	
24	26.940	331.8
48	26.890	331.8
72	26.850	309.1
96	26.810	298.1
120	26.770	291.4
144	26.740	276.0
168	26.720	255.5
192	26.700	240.1
216	26.680	228.2
240	26.670	211.9

TABLE 4.2

CORROSION OF ROLLED STEEL SPECIMEN (4) IN 92% H_2SO_4 SOLUTION AT 35^0C TEMPERATURE UNDER TOTAL IMMERSION TEST

TIME IN HRS	WEIGHT IN GMS	CORROSION RATE IN MDD
0	29.340	
24	29.190	983.8
48	29.040	983.8
72	28.900	961.9
96	28.760	951.0
120	28.640	918.2
144	28.560	852.6
168	28.470	815.1
192	28.430	746.1
216	28.410	677.7
240	28.400	616.5

TABLE 5.1

CORROSION OF SHAPE WELDED SPECIMEN (5) IN 92% H_2SO_4 SOLUTION AT 50^{0}C TEMPERATURE UNDER TOTAL IMMERSION TEST

TIME IN HRS	WEIGHT IN GMS	CORROSION RATE IN MDD
0	26.460	
24	26.390	465.6
48	26.320	465.6
72	26.260	443.5
96	26.200	432.4
120	26.150	412.4
144	26.110	388.0
168	26.080	361.1
202	26.060	316.1
226	26.040	296.6
250	26.020	280.9

TABLE 5.2

CORROSION OF ROLLED STEEL SPECIMEN (6) IN 92% H_2SO_4 SOLUTION AT 50^{0}C TEMPERATURE UNDER TOTAL IMMERSION TEST

TIME IN HRS	WEIGHT IN GMS	CORROSION RATE IN MDD
0	29.030	
24	28.860	1125.8
48	28.690	1125.8
72	28.530	1098.2
106	28.300	1035.5
130	28.170	1025.4
154	28.050	1011.4
178	27.950	964.3
206	27.870	895.0
230	27.800	850.0
250	27.740	820.1

TABLE 6.1

CORROSION OF SHAPE WELDED SPECIMEN (7) IN 96% H_2SO_4 SOLUTION AT 15^{0}C TEMPERATURE UNDER TOTAL IMMERSION TEST

TIME IN HRS	WEIGHT IN GMS	CORROSION RATE IN MDD
0	26.580	
24	26.540	265.8
48	26.500	265.8
72	26.470	243.7
96	26.450	215.9
120	26.430	199.2
154	26.410	175.9
192	26.400	149.5
234	26.390	129.4
260	26.380	127.6

TABLE 6.2

CORROSION OF ROLLED STEEL SPECIMEN (8) IN 96% H_2SO_4 SOLUTION AT 15^{0}C TEMPERATURE UNDER TOTAL IMMERSION TEST

TIME IN HRS	WEIGHT IN GMS	CORROSION RATE IN MDD
0	29.120	
24	29.030	594.0
48	28.940	594.0
72	28.880	528.1
96	28.830	478.5
120	28.780	448.8
154	28.740	380.6
192	28.710	338.2
234	28.690	291.1
260	28.680	268.8

TABLE 7.1

CORROSION OF SHAPE WELDED SPECIMEN (9) IN 96% H_2SO_4 SOLUTION AT 35^0C TEMPERATURE UNDER TOTAL IMMERSION TEST

TIME IN HRS	WEIGHT IN GMS	CORROSION RATE IN MDD
0	26.760	
24	26.700	401.7
48	26.640	401.7
72	26.590	379.4
96	26.540	368.3
120	26.500	348.2
160	26.470	291.2
212	26.450	234.9
242	26.440	212.5

TABLE 7.2

CORROSION OF ROLLED STEEL SPECIMEN (10) IN 96% H_2SO_4 SOLUTION AT 35^0C TEMPERATURE UNDER TOTAL IMMERSION TEST

TIME IN HRS	WEIGHT IN GMS	CORROSION RATE IN MDD
0	29.080	
24	28.920	1058.2
48	28.760	1058.2
72	28.620	1014.5
96	28.480	992.1
120	28.360	952.4
160	28.220	852.5
212	28.090	741.3
242	28.060	669.0

TABLE 8.1

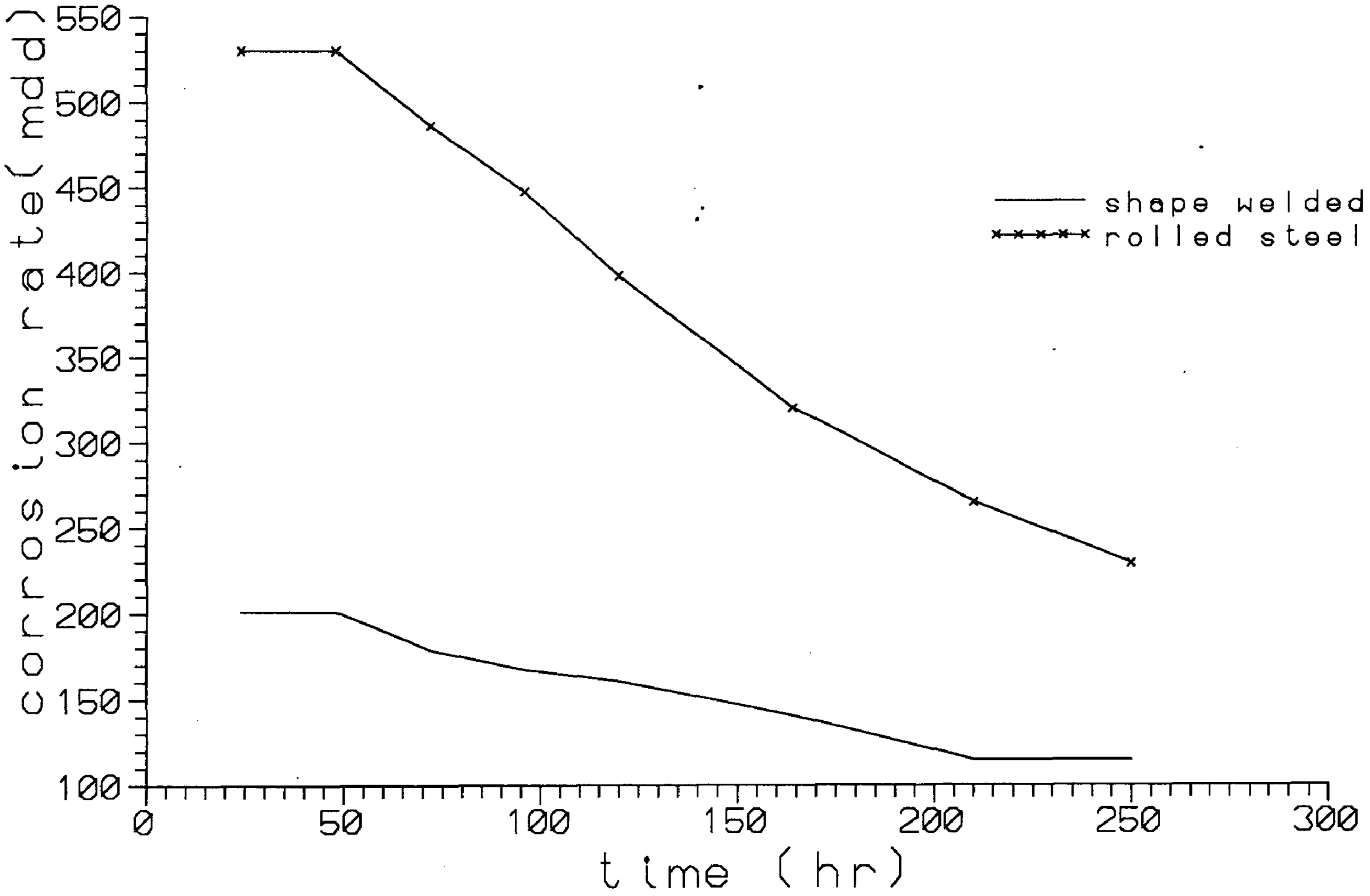
CORROSION OF SHAPE WELDED SPECIMEN (11) IN 96% H_2SO_4 SOLUTION AT 50^0C TEMPERATURE UNDER TOTAL IMMERSION TEST

TIME IN HRS	WEIGHT IN GMS	CORROSION RATE IN MDD
0	26.870	
24	26.780	596.7
48	26.690	596.7
72	26.620	552.3
96	26.550	530.3
120	26.490	503.1
162	26.440	422.3
196	26.400	381.5
220	26.370	361.6
252	26.340	334.5

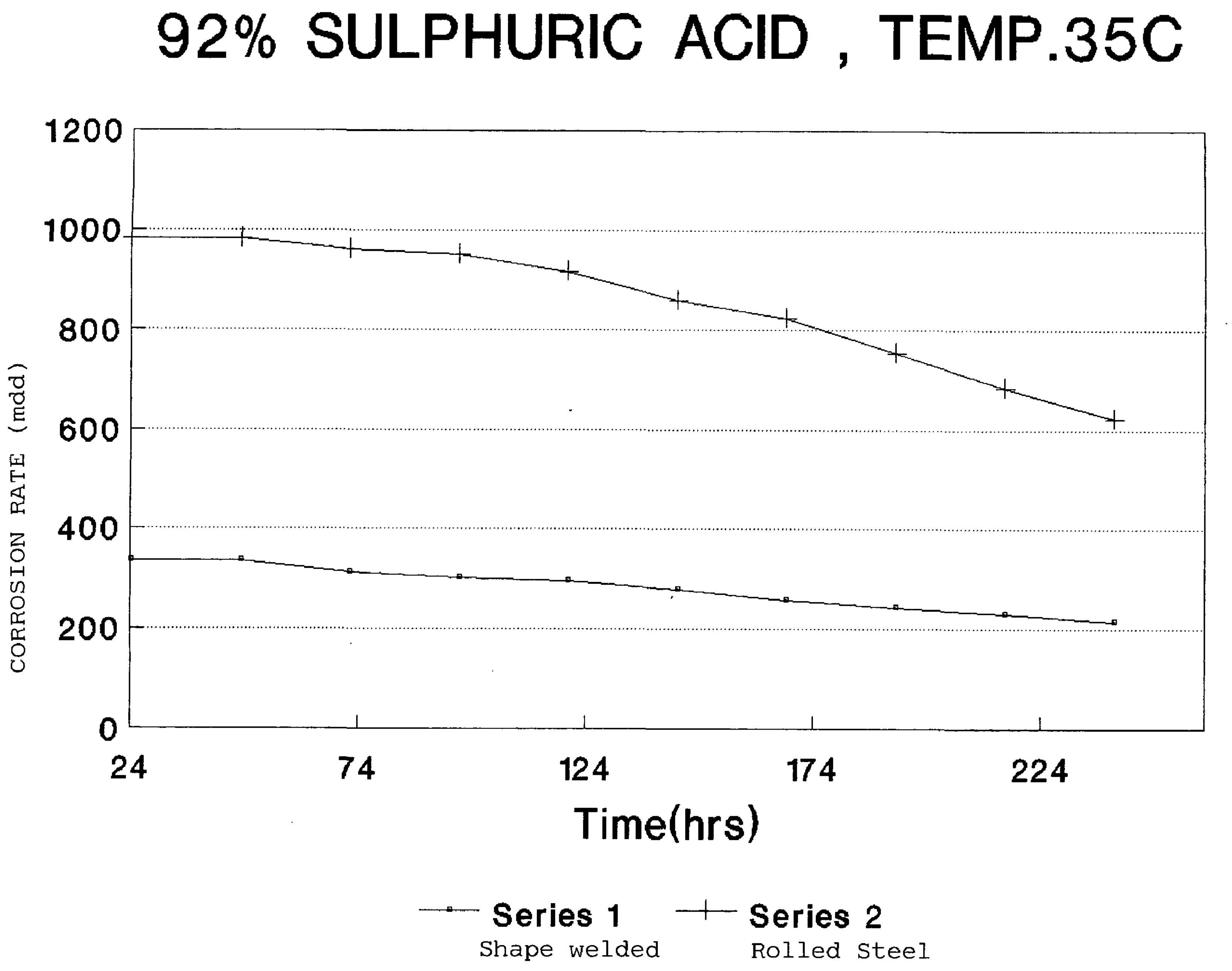
TABLE 8.2

CORROSION OF ROLLED STEEL SPECIMEN (12) IN 96% H_2SO_4 SOLUTION AT 50^0C TEMPERATURE UNDER TOTAL IMMERSION TEST

TIME IN HRS	WEIGHT IN GMS	CORROSION RATE IN MDD
0	29.690	
24	29.510	1171.3
48	29.340	1171.3
72	29.180	1106.2
96	29.030	1073.6
120	28.890	1041.2
162	28.630	1021.9
196	28.480	964.2
220	28.400	915.7
252	28.330	842.8



Temp 15C ;Sulphuric acid 92%



CORROSION RATE VS TIME

Fig.1.2

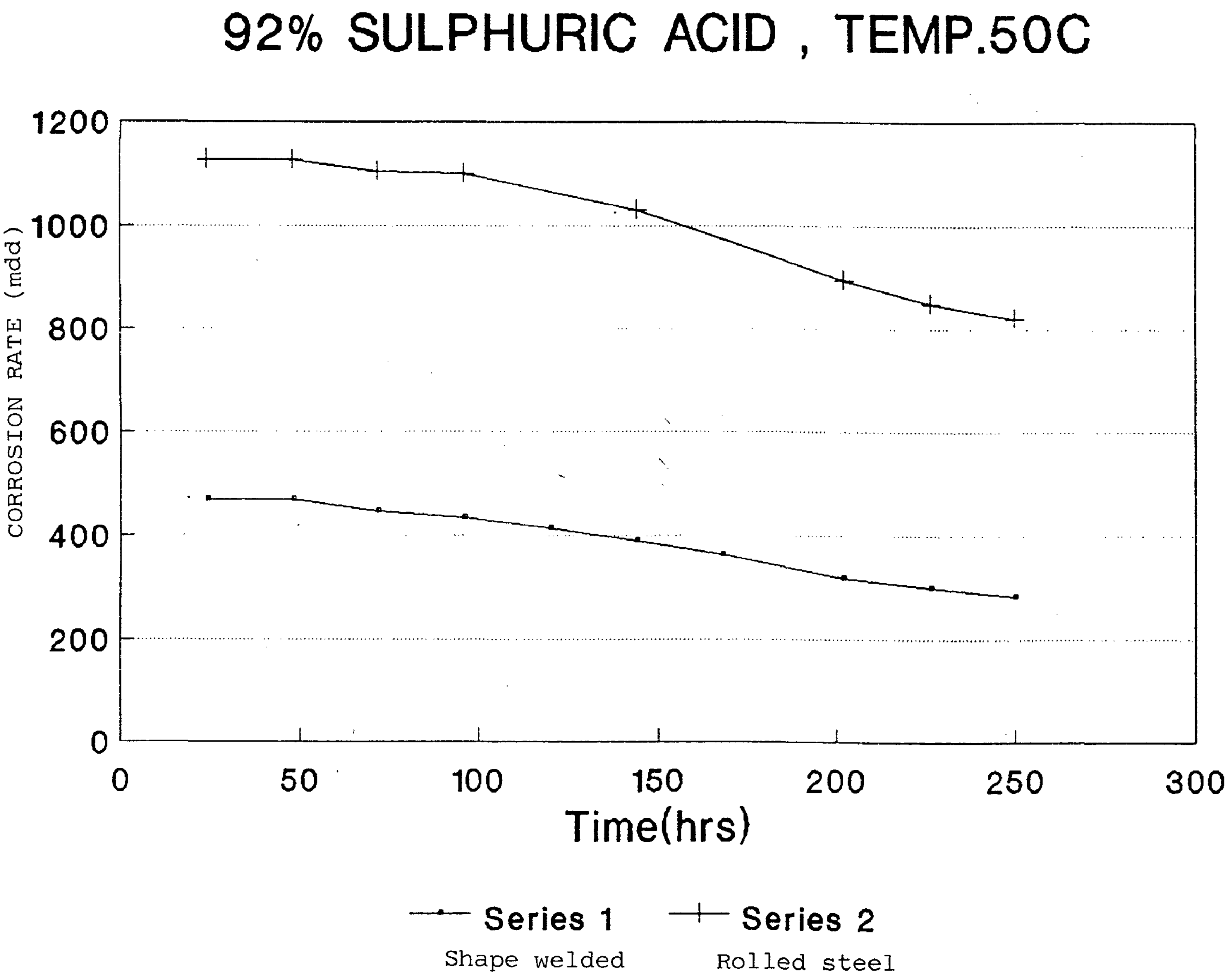


Fig.1.3

CORROSION RATE VS TIME

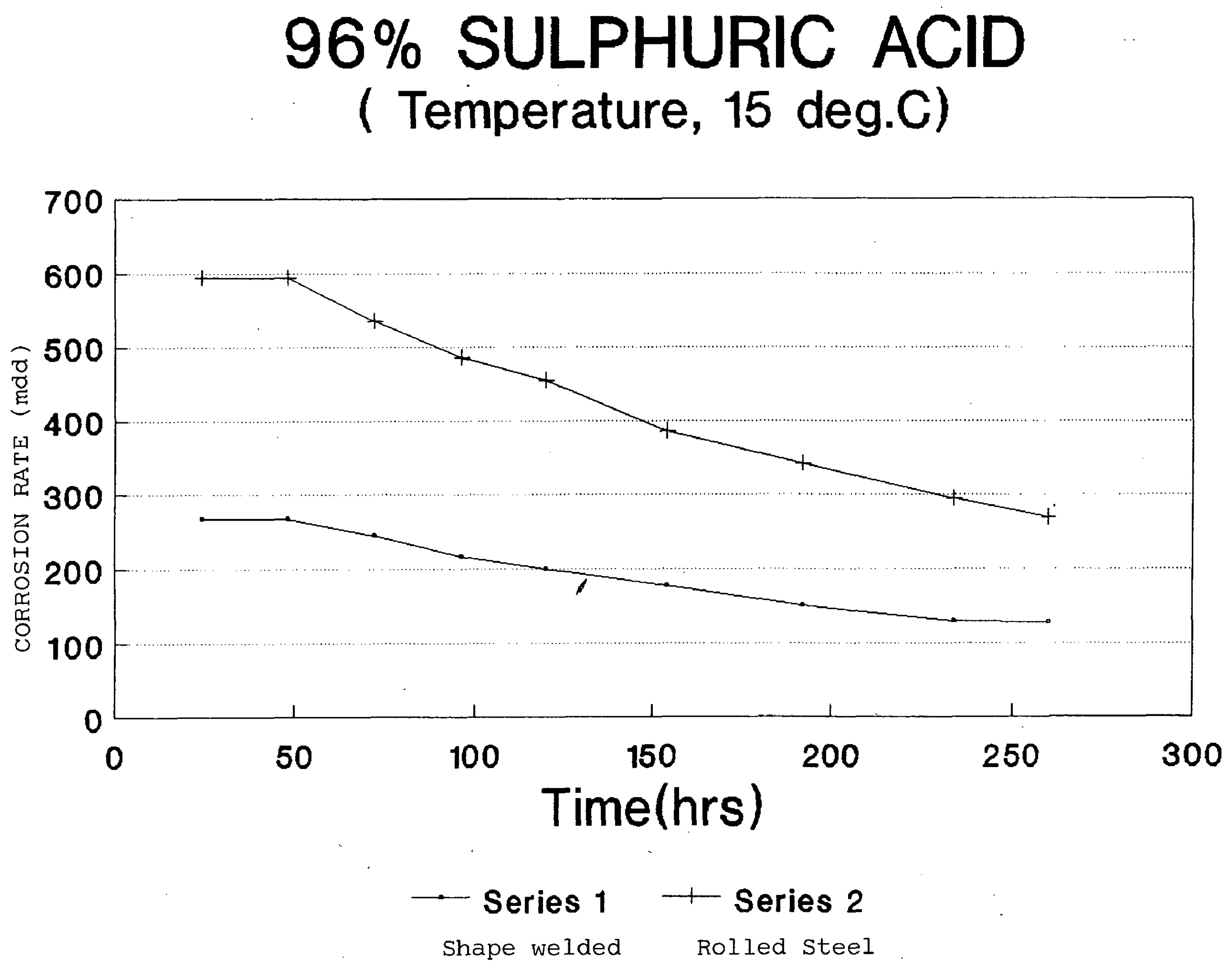


Fig.1.4 CORROSION RATE VS TIME

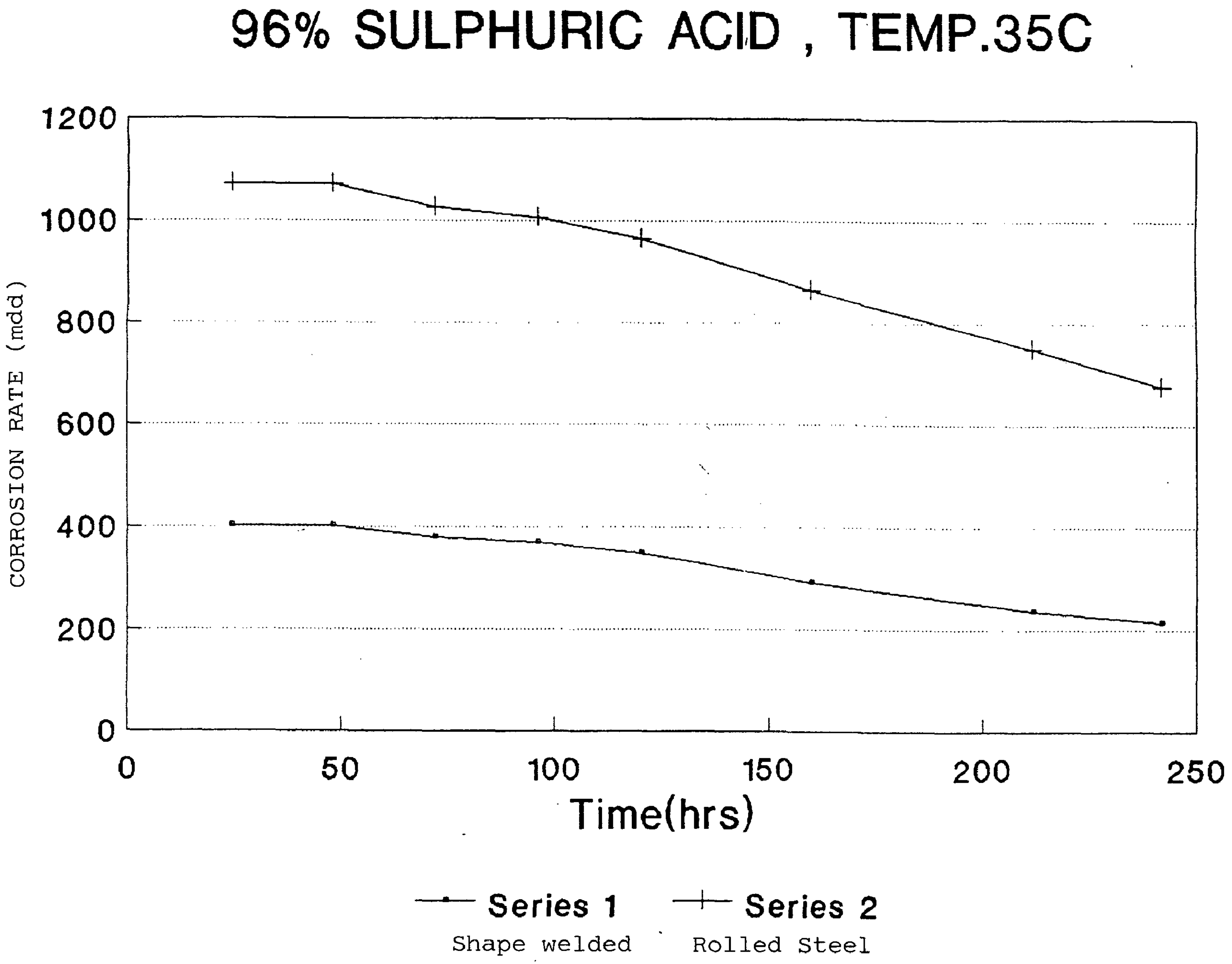


Fig.1.5 CORROSION RATE VS TIME

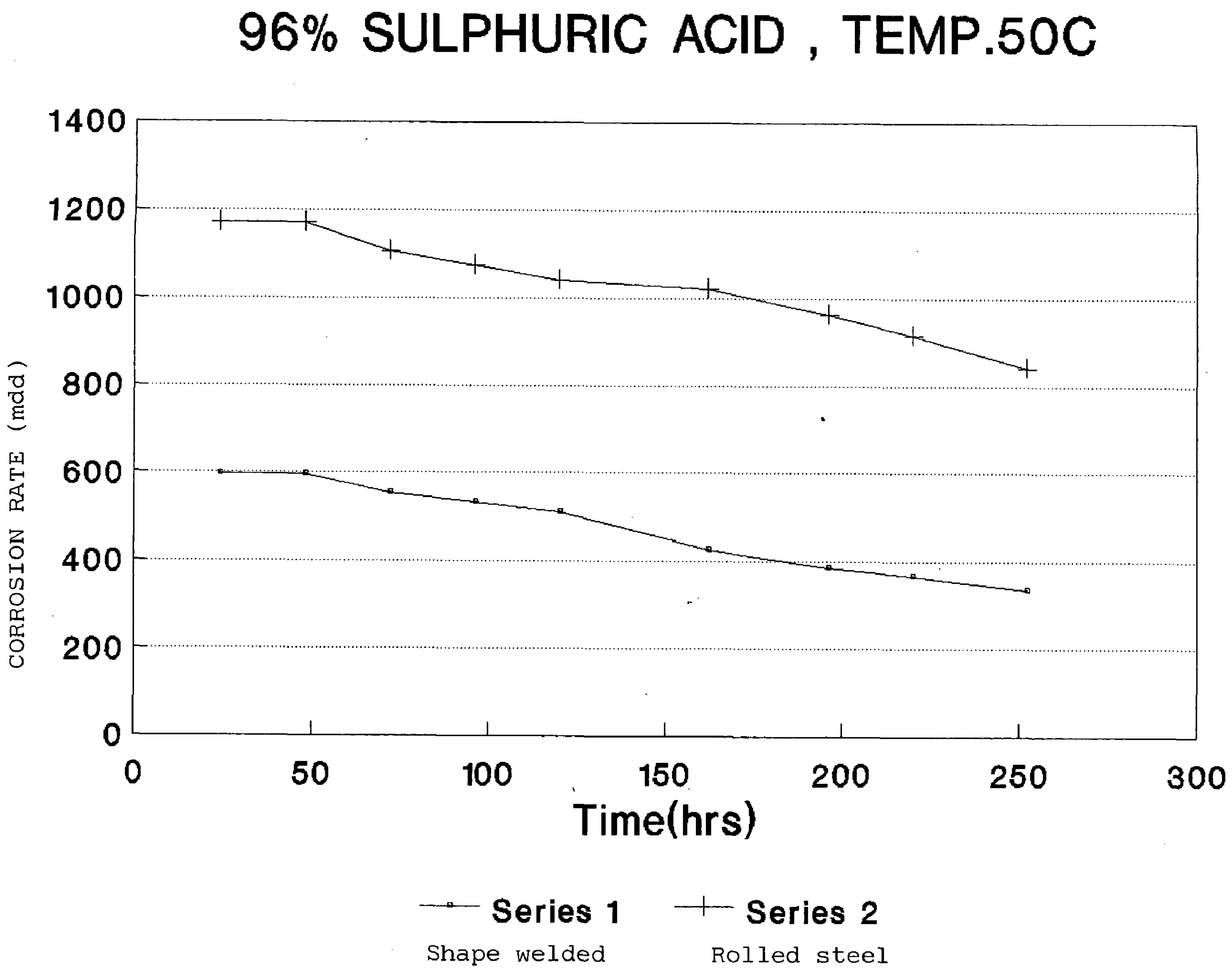


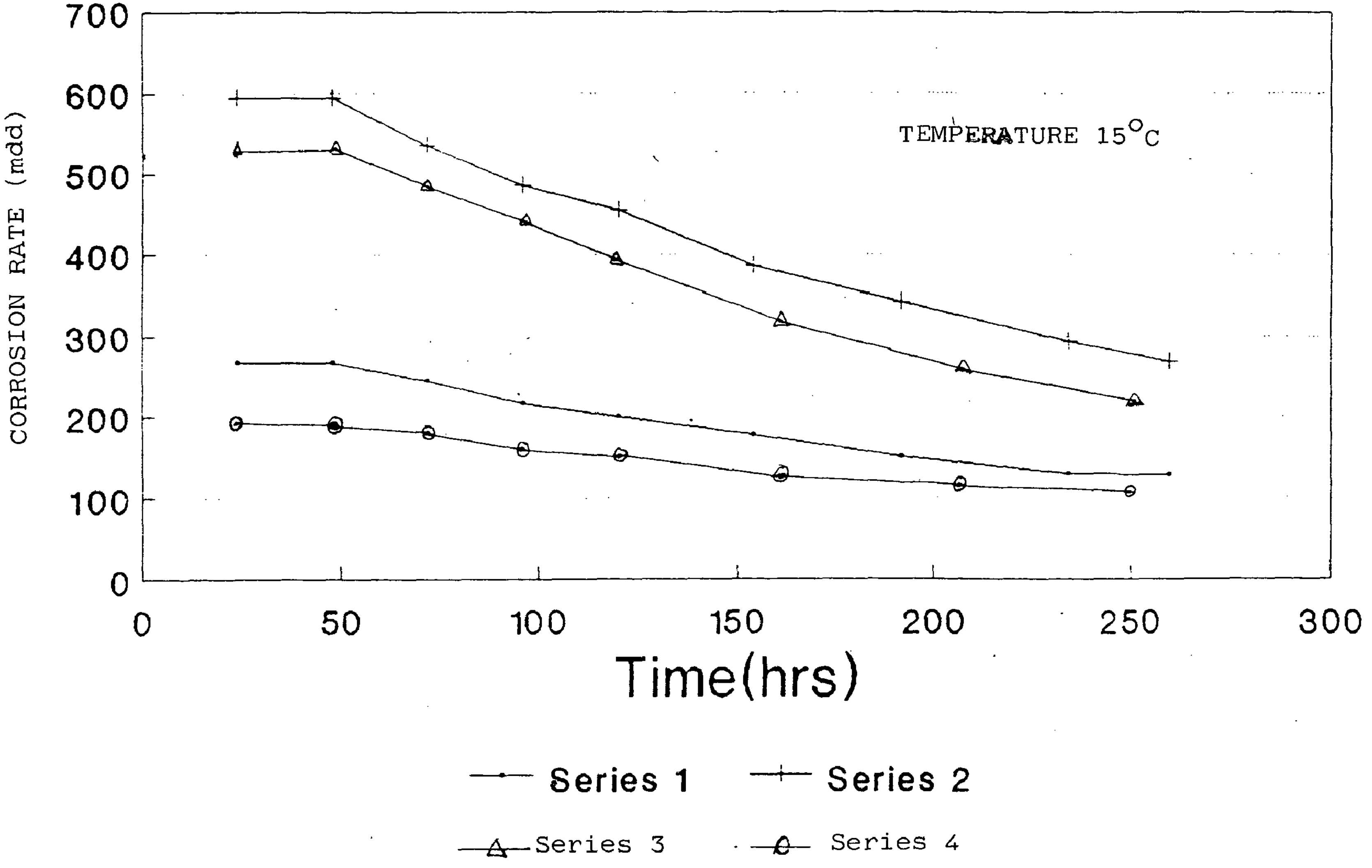
Fig.1.6 CORROSION RATE VS TIME

Series 2: Rolled Steel; 96% H_2SO_4

Series 3: Rolled Steel; 92% H_2SO_4

Series 4: Shape welded steel; 92% H_2SO_4

VARIATION OF CORROSION RATES WITH TIME AT CONSTANT TEMPERATURE OF 15°C AND VARYING CONCENTRATION



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Series 1 : Shape welded; 96% H_2SO_4 Series 2 : Rolled Steel; 96% H_2SO_4

Series 3 : Shape welded; 92% H_2SO_4 Series 4 : Rolled Steel; 92% H_2SO_4

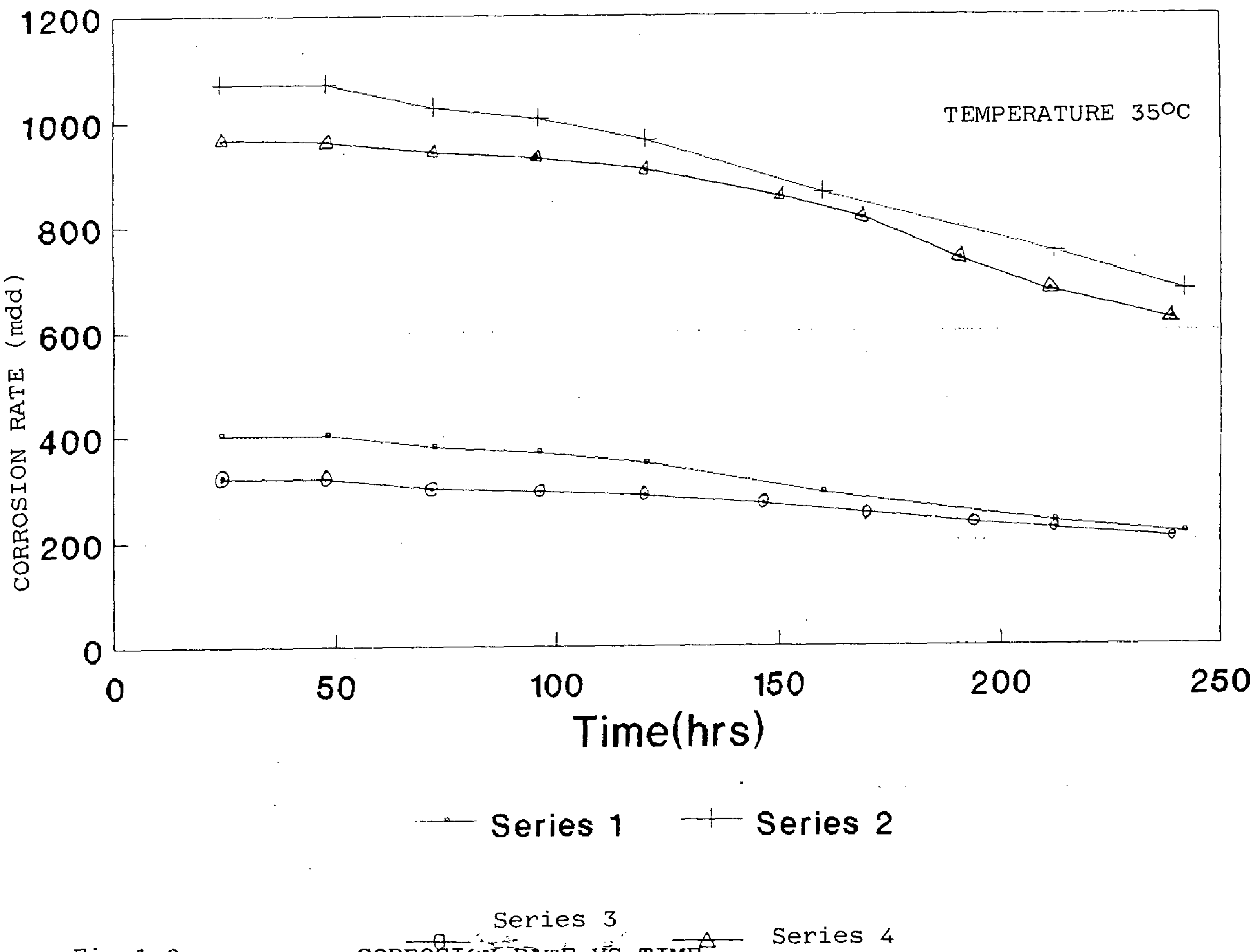


Fig.1.8 CORROSION RATE VS TIME

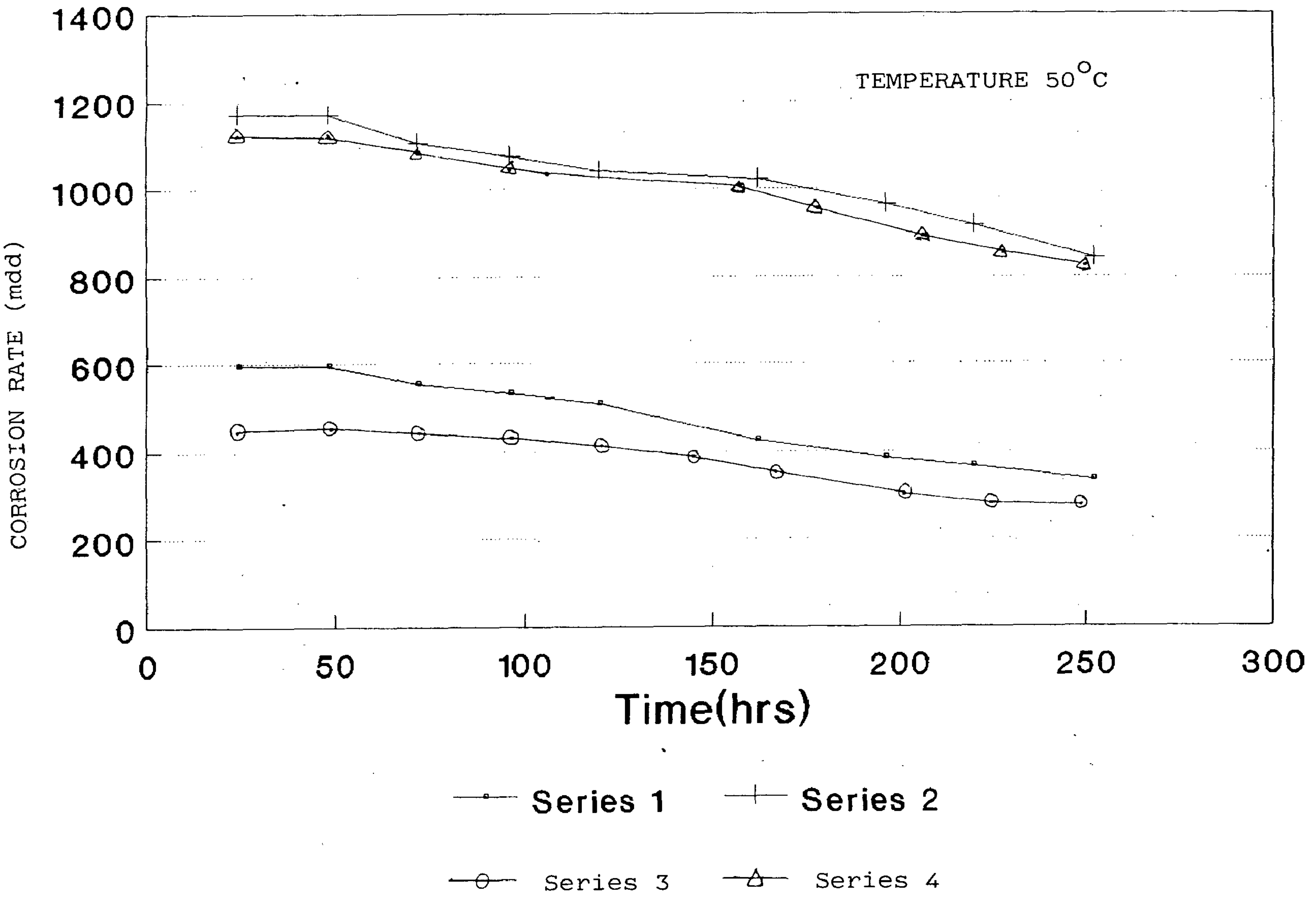
(Variation of corrosion rate with time at

Series 1 : Shape welded; 96% H_2SO_4

Series 2 : Rolled Steel; 96% H_2SO_4

Series 3 : Shape welded; 92% H_2SO_4

Series 4 : Rolled steel; 92% H_2SO_4



VARIATION OF CORROSION RATE WITH TIME AT CONSTANT TEMPERATURE OF 50°C AND VAYING CONCENTRATION

Fig.1.9

Series 1 : Shape welded; 50°C

Series 3 : Rolled Steel; 35°C

Series 5 : Rolled Steel; 15°C

Series 2 : Rolled Steel; 50

Series 4 : Shape welded; 35°C

Series 6 : Shape welded; 15°C

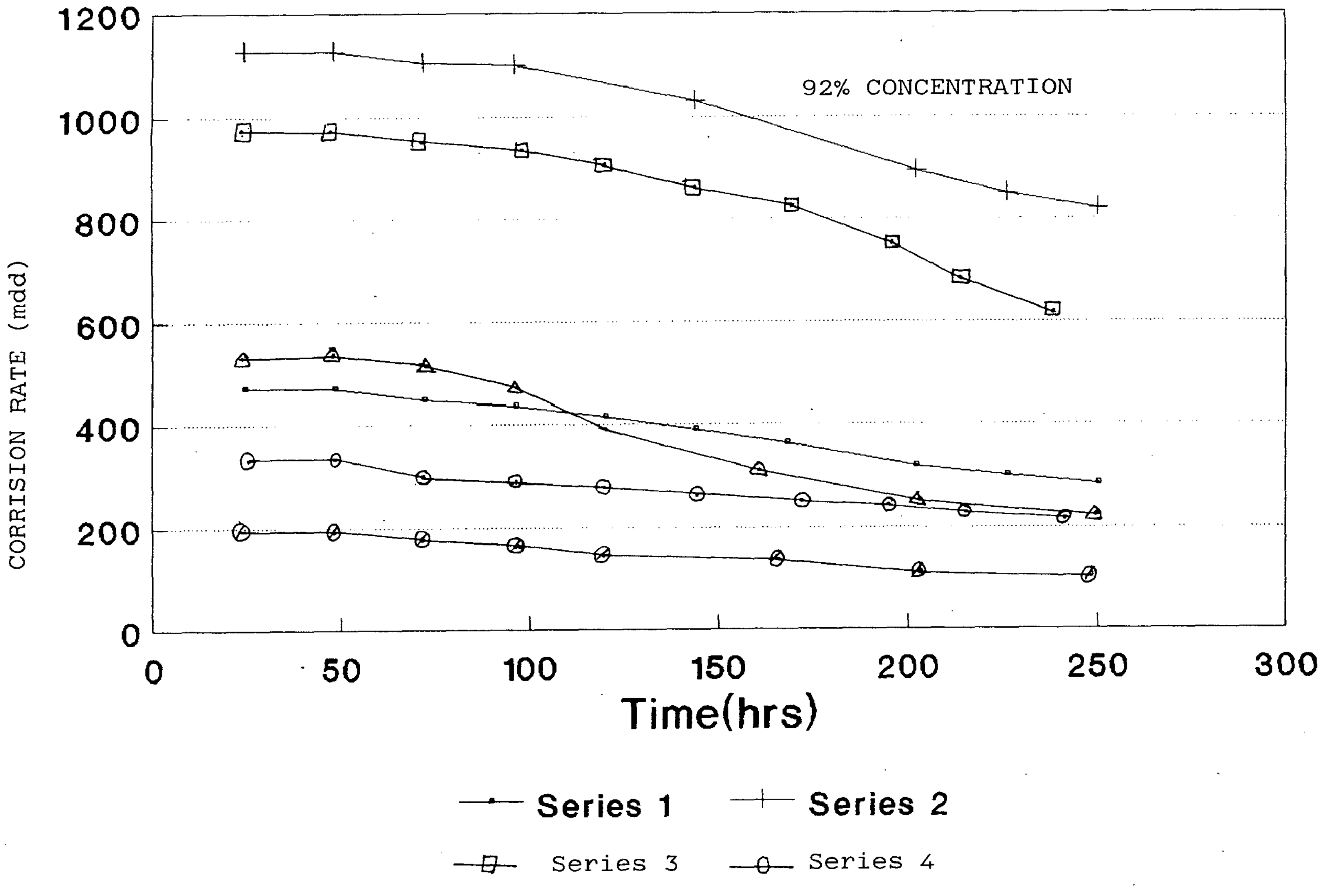


Fig.1.10

VARIATION OF CORROSION RATE WITH TIME AT CONSTANT CONCENTRATION

Series 1 : Shape welded; 50°C
Series 2 : Rolled steel; 50°C
Series 3 : Shape Welded; 35°C
Series 4 : Rolled Steel; 35°C
Series 5 : Shape welded; 15°C
Series 6 : Rolled Steel; 15°C

VARIATION OF CORROSION RATE WITH TIME AT CONSTANT CONCENTRATION OF 96% H_2SO_4 AND VARYING TEMPERATURE

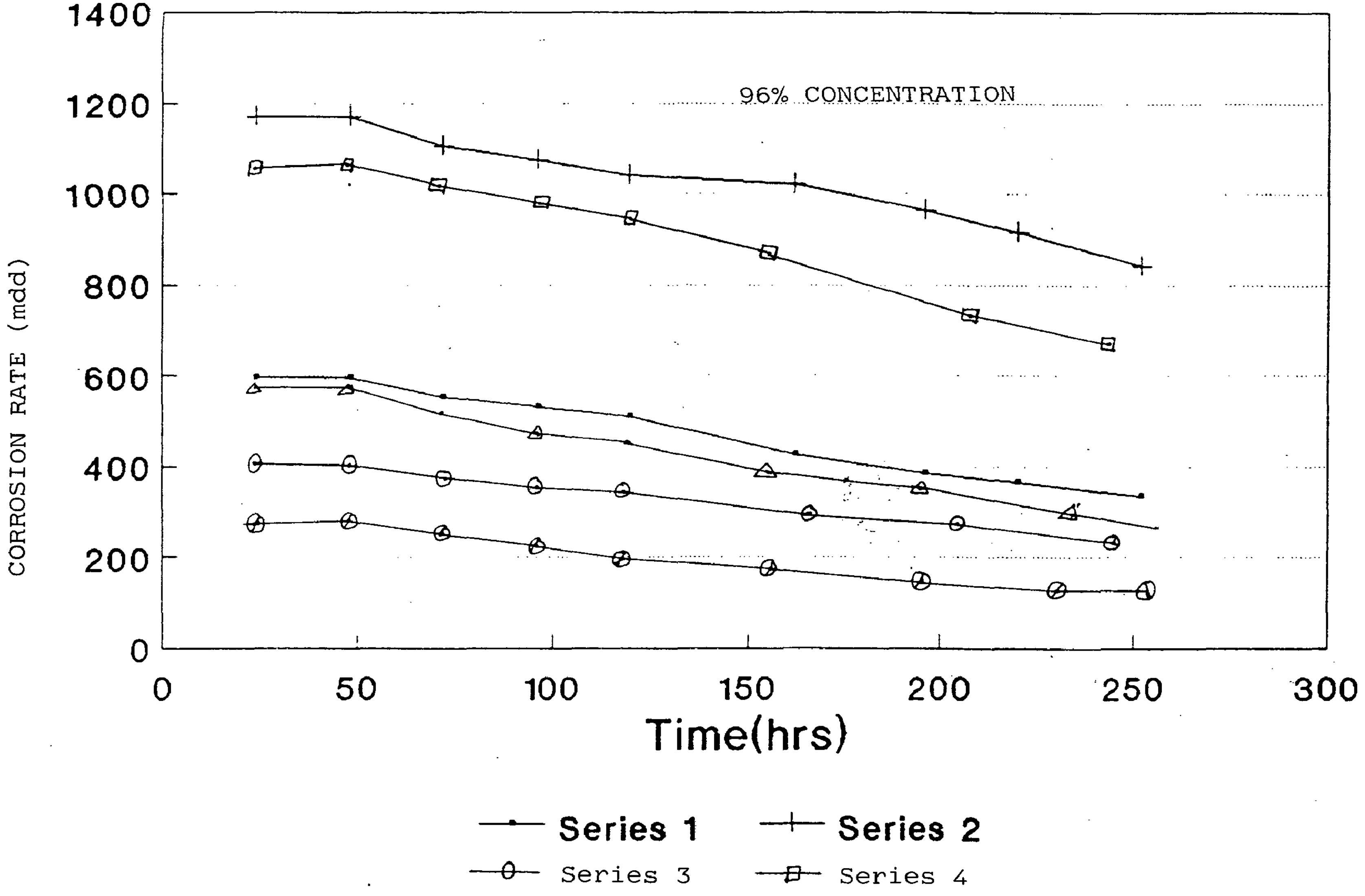
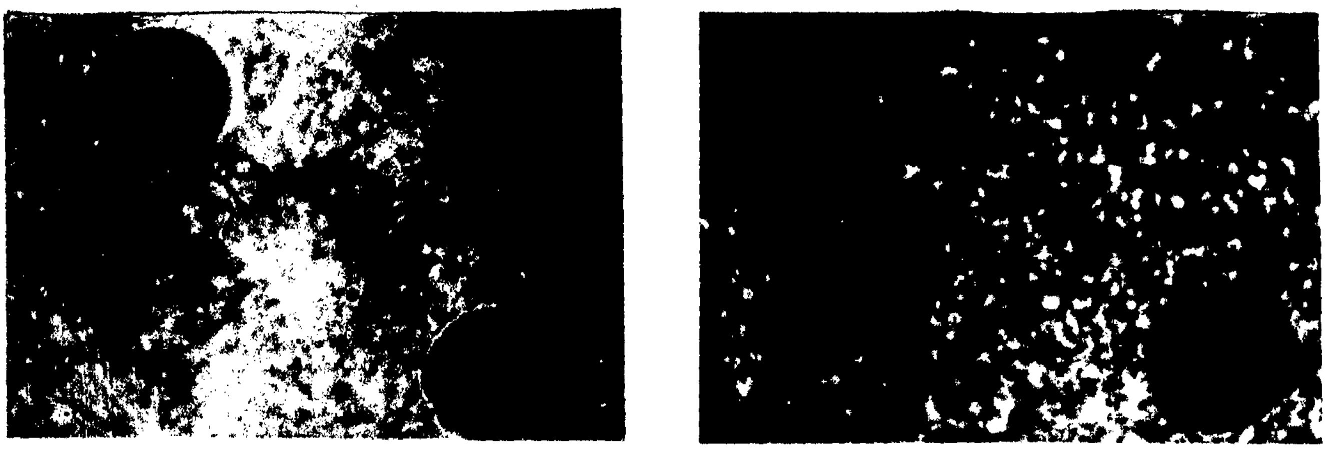


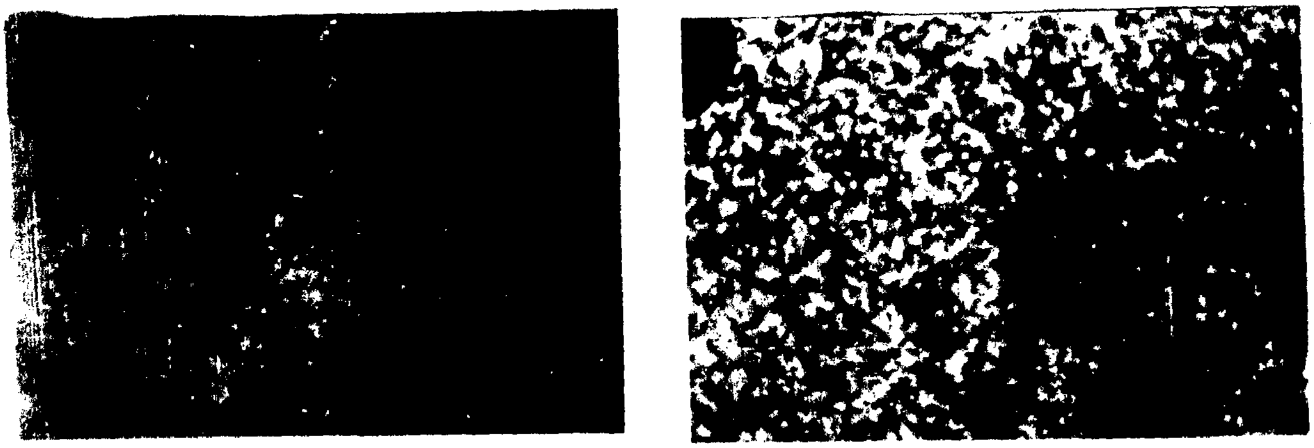
Fig.2.1 Macro structures of samples before corrosion



Shape Welded
12X

Rolled Steel
12X

Fig.2.2 Macro structures of samples after corrosion
in 92% H_2SO_4 a temprature of 15°C



12X

92% H_2SO_4
Temp 15°C
Shape Welded

12X

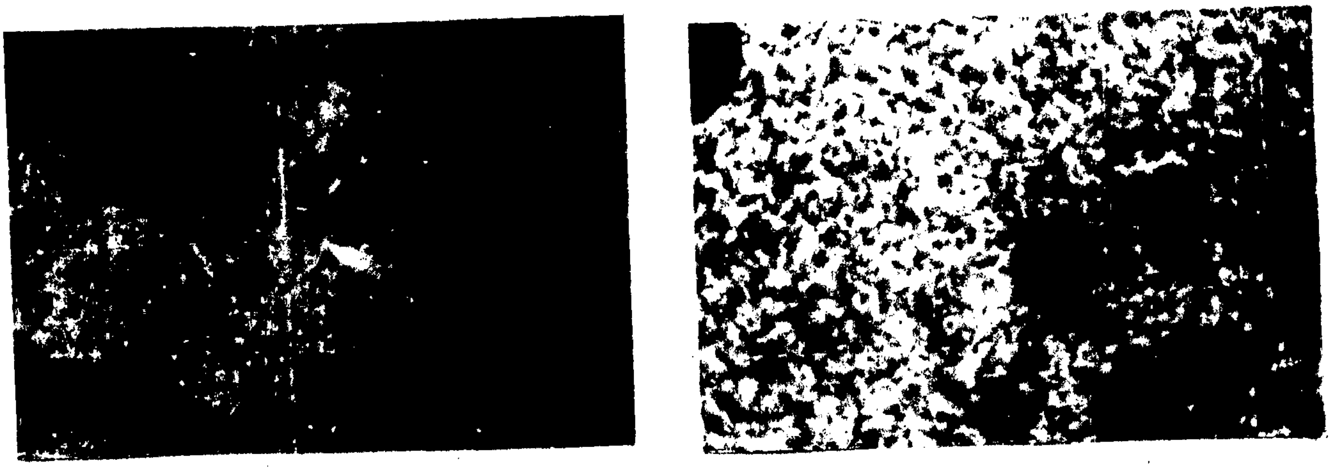
92 % H_2SO_4
Temp 15°C
Rolled Steel

Fig.3.1 Macro structures of samples after corrosion in 92% H_2SO_4 a 35°C temperature.

Shape welded
12X

Rolled Steel
12X

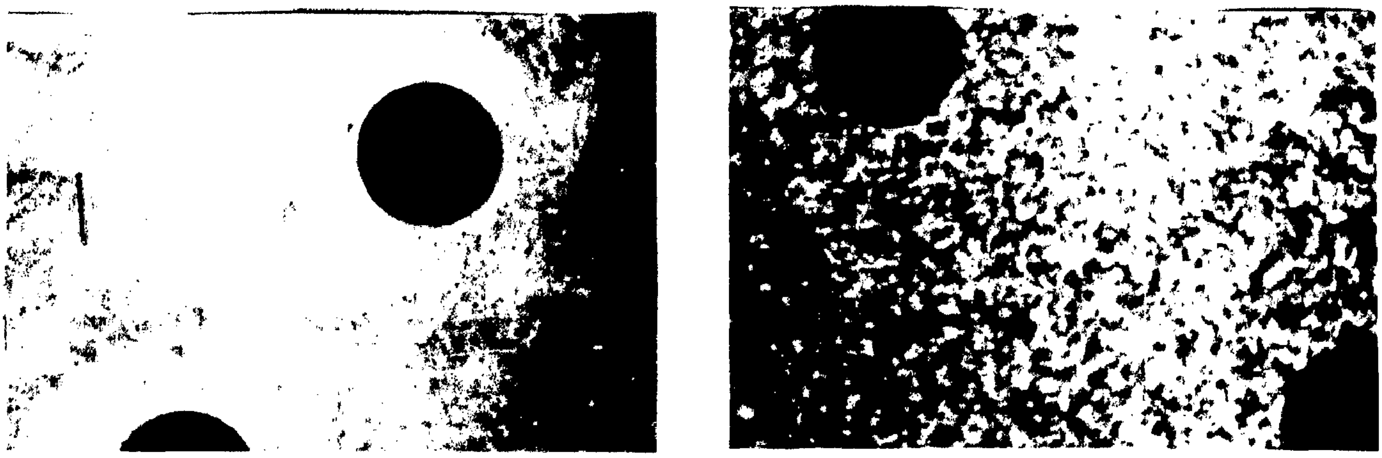
Fig.3.2 Macro structures of samples after corrosion in 92% H_2SO_4 50°C temperature



Shape welded
12X

Rolled steel
12X

Fig.4.1 Macro structures of samples after corrosion in 96% H_2SO_4 a
15°C temperature.



Shape welded
12X

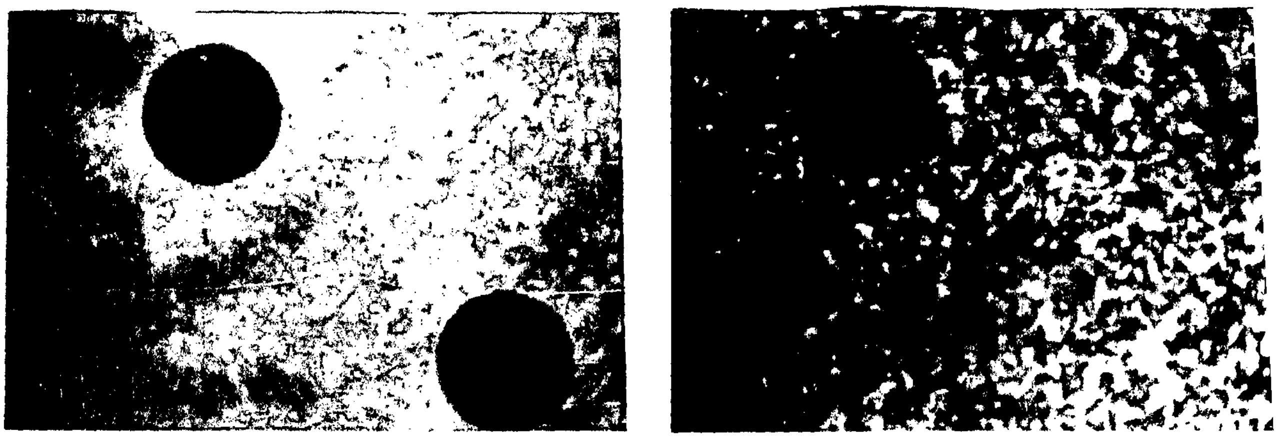
Rolled Steel
12X

Fig.4.2 Macro structure of samples after corrosion in 96% H_2SO_4 a
35°C temperature.

Shape Welded
12X

Rolled Steel
12X

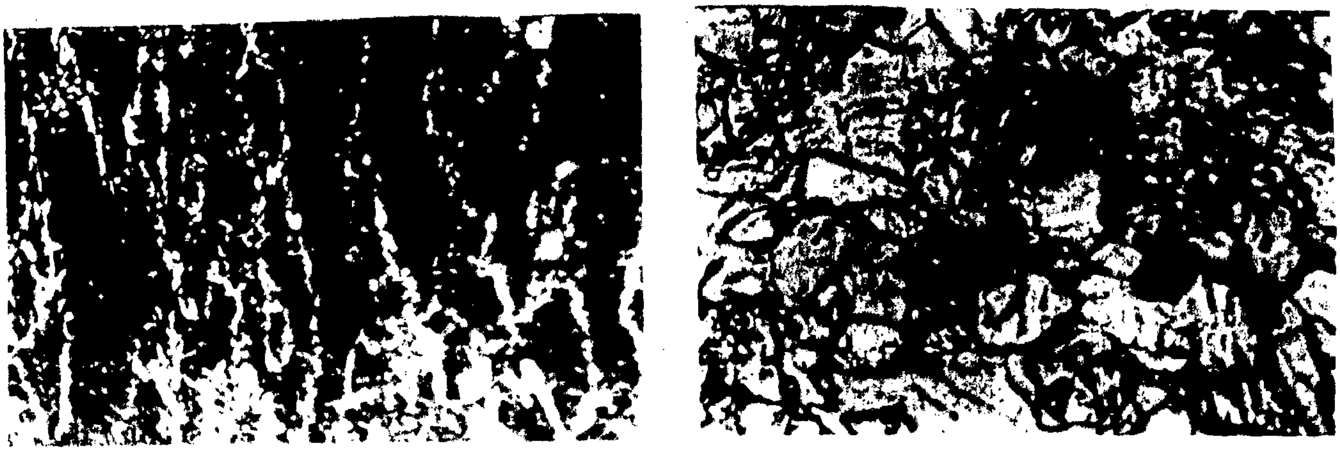
Fig.5.1 Macro structures of samples after corrosion in 96% H_2SO_4 at 50°C temperature.



Shape welded
12X

Rolled Steel
12X

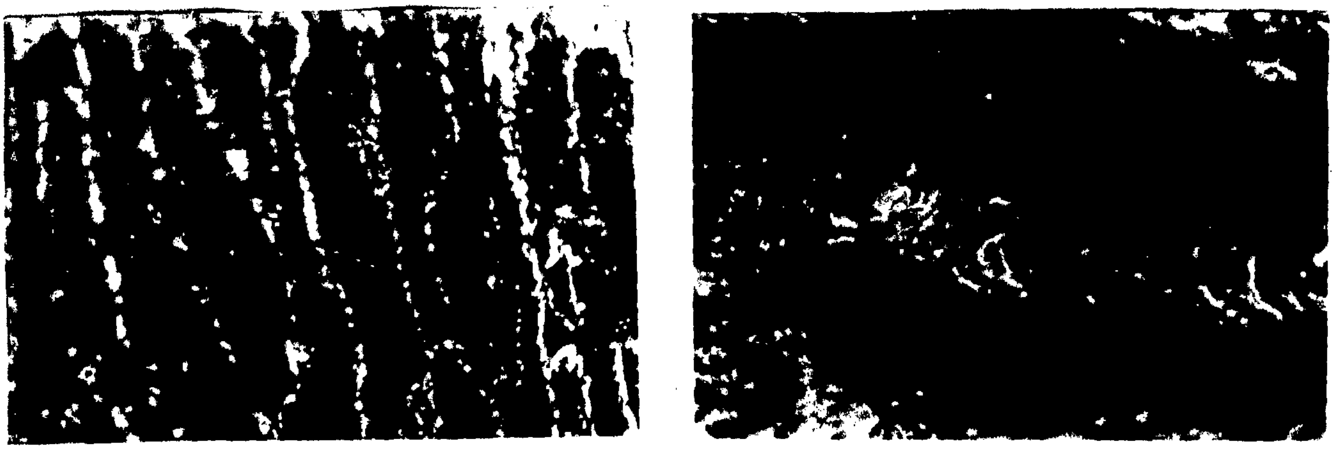
Fig.6.1 Micro structures of samples before corrosion



Shape welded
200X

Rolled Steel
200X

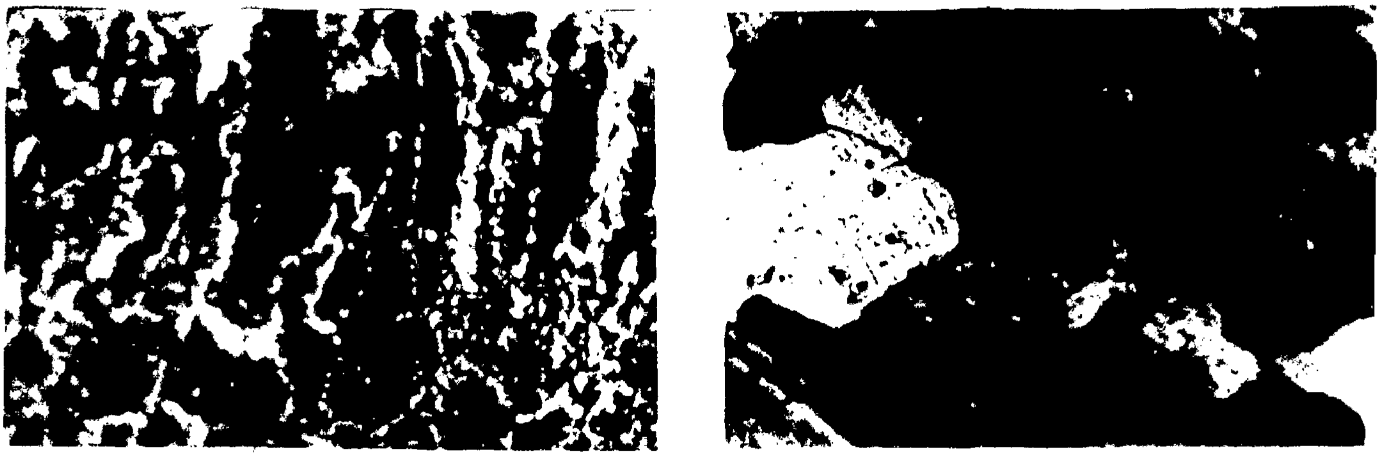
Fig.6.2 Micro structure of samples after corrosion in 92% H_2SO_4 a $15^{\circ}C$ temperature



Shape welded
200X

Rolled steel
200X

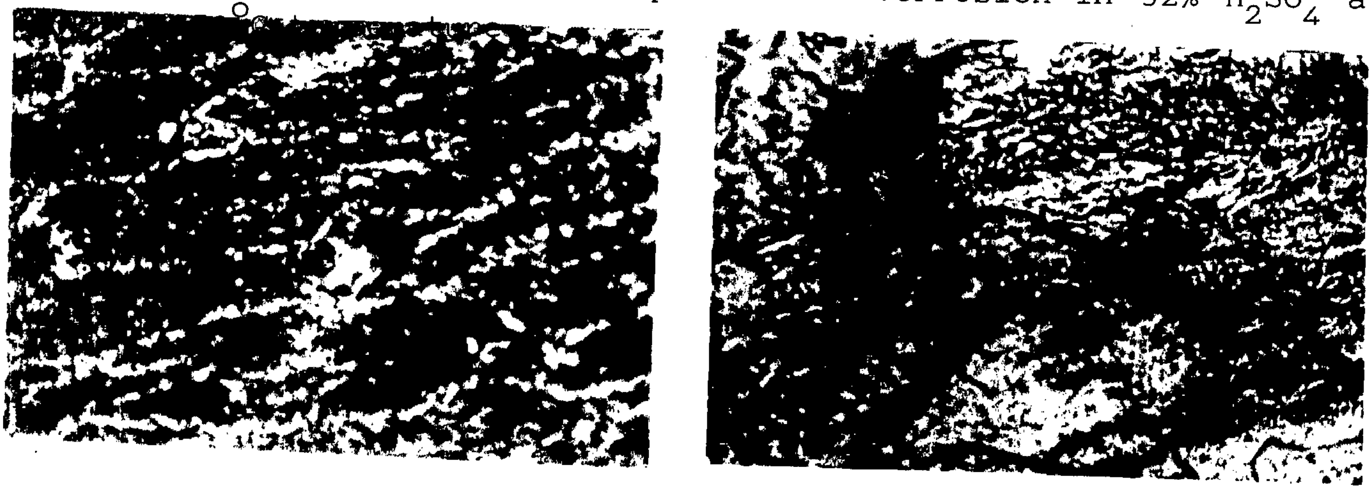
Fig.7.1 Micro structure the samples after corrosion in 92% H_2SO_4 at $35^{\circ}C$ temperature



Shape Welded
200X

Rolled steel
500X

Fig.7.2 Micro Structures of samples after corrosion in 92% H_2SO_4 at



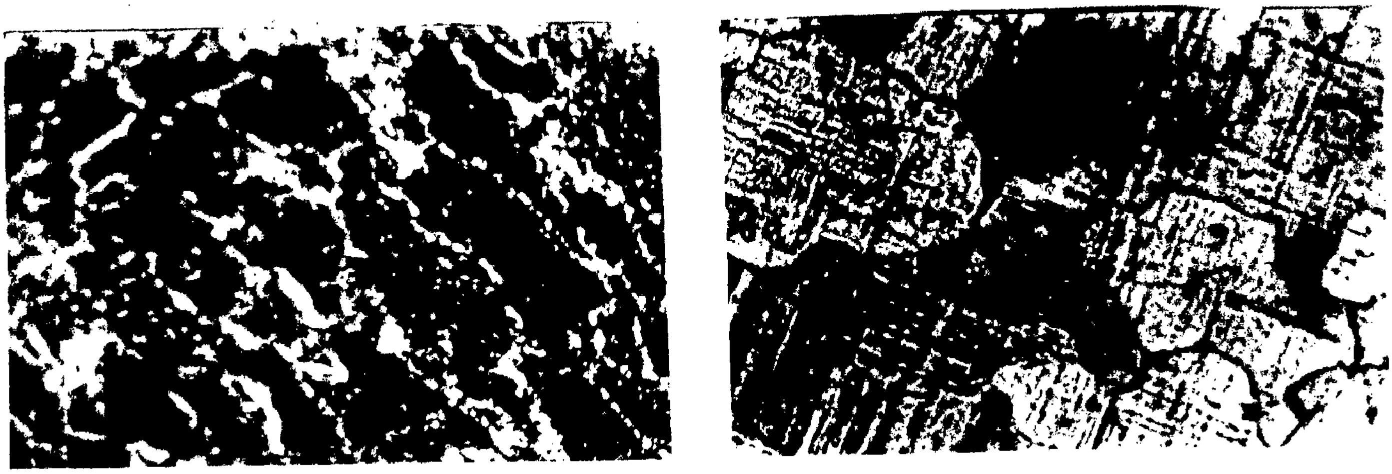
Shape welded

200X

Rolled steel

500X

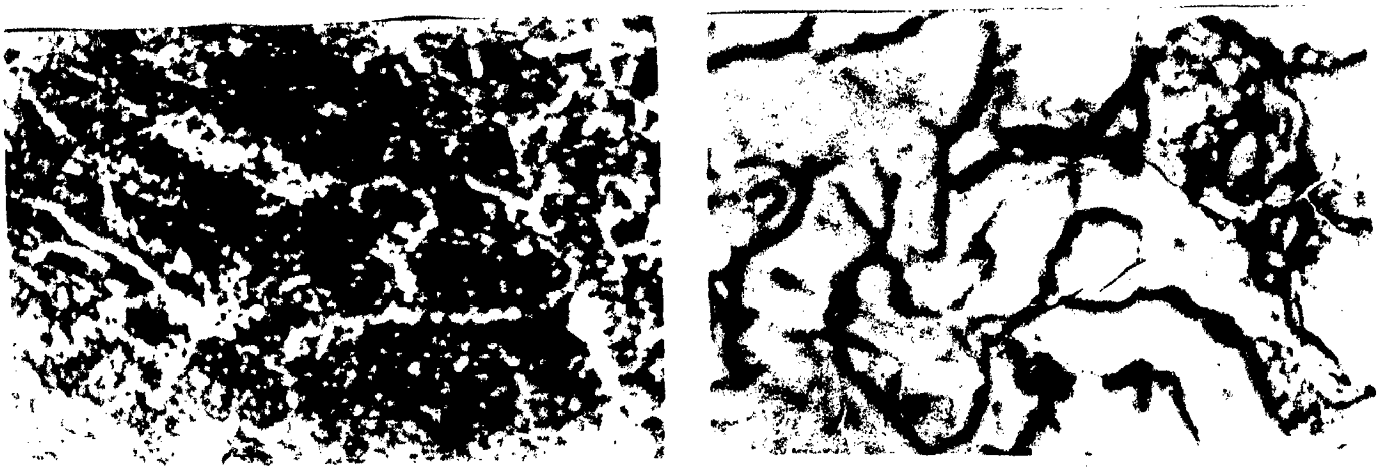
Fig.8.1 Micro structures of samples after corrosion in 96% H_2SO_4 at 15^oC temperature.



Shape welded 200X

Rolled Steel 500X

Fig.8.2 Micro structures of samples after corrosion in 96% H_2SO_4 a 35^oC temperature.



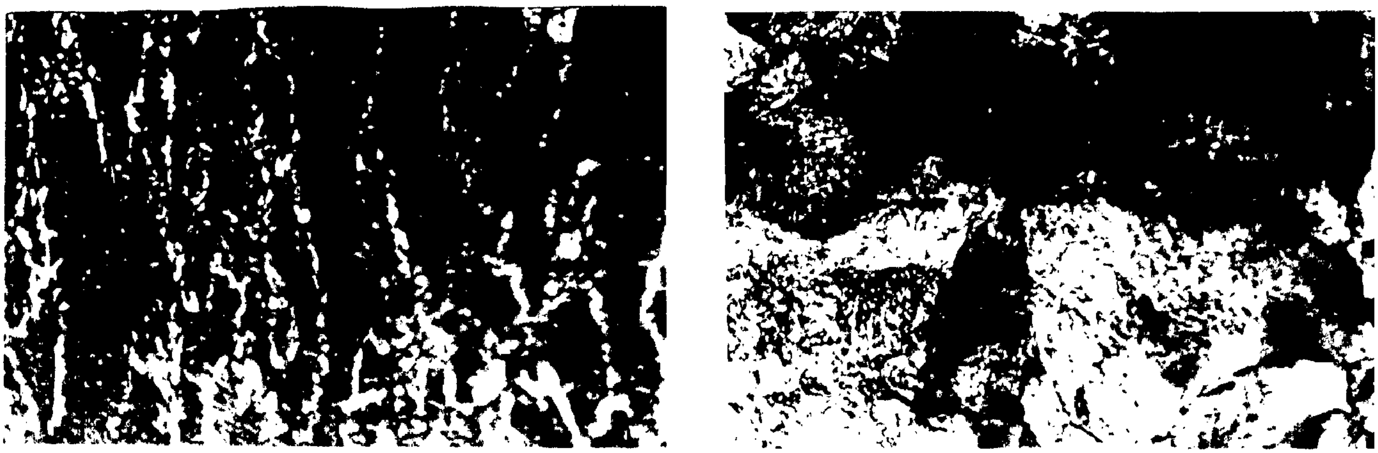
Shape welded

200X

Rolled steel

500X

Fig.9.1 Micro structures of samples after corrosion in 96% H_2SO_4 at 50°C temperature.



Shape welded 200^{X} Rolled steel 500X

Fig.9.2 Micro structure of shape welded steel before corrosion at 1000X.magnification.

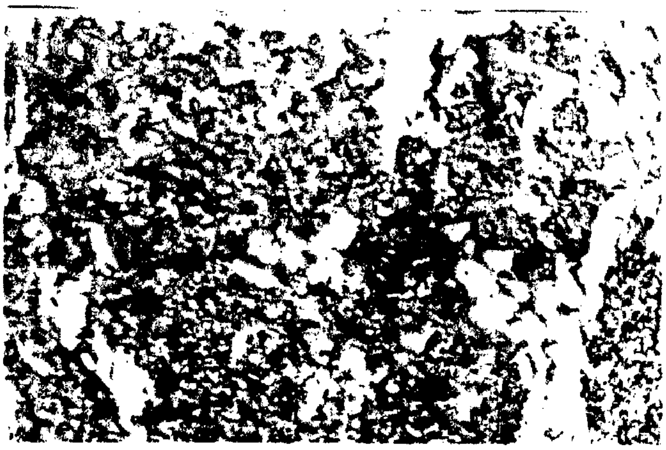
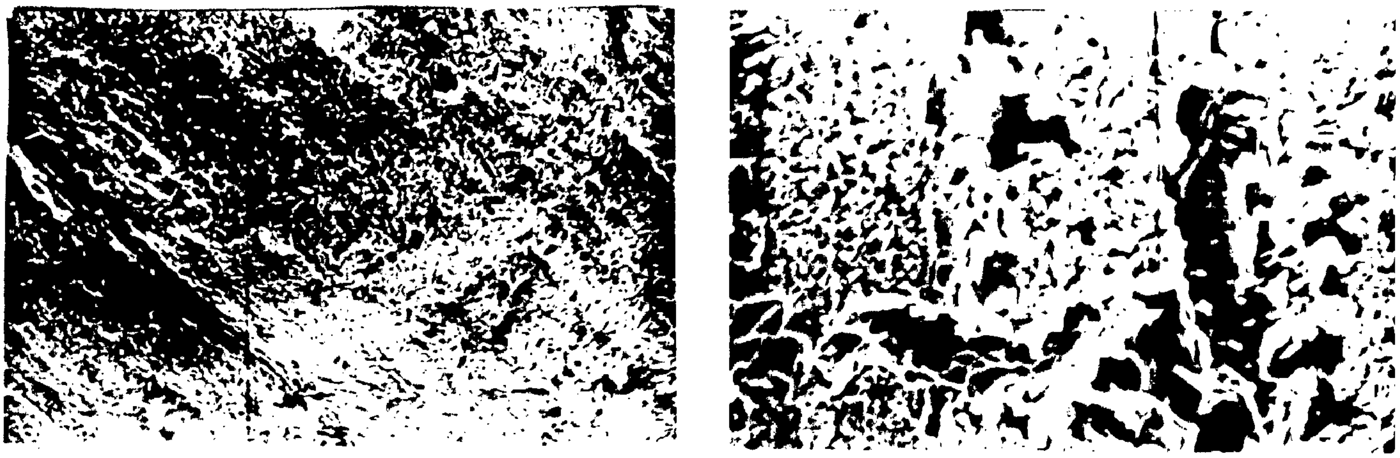


Fig.10.1 SEM micrographs of samples before corrosion

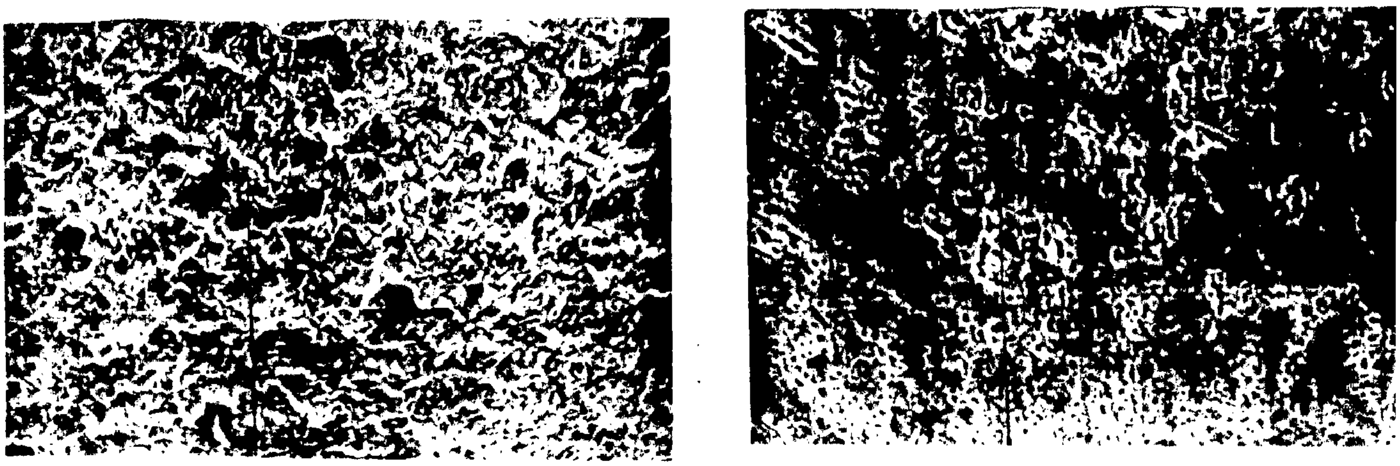
(Correction factor 1.1)



Shape welded 640X

Rolled Steel 640X

Fig.10.2 SEM micrographs of samples after corrosion in 92% H_2SO_4 at 15°C temperature.



Shape welded

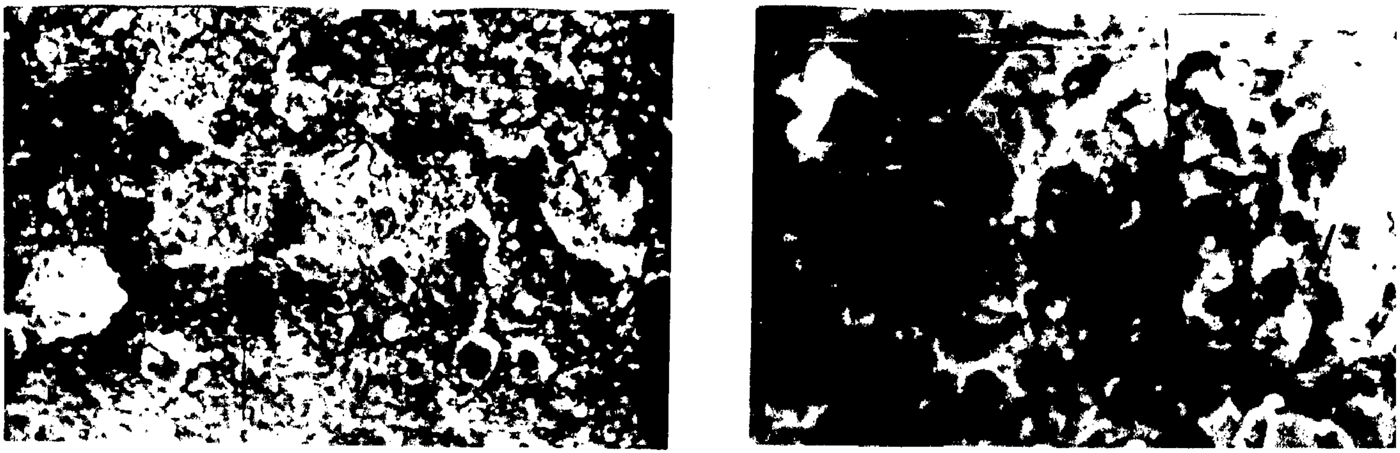
640X

Rolled steel

640X

Fig.11.1 SEM micrographs of samples after corrosion in 92% H_2SO_4 a
50°C temperature.

(Correction factor 1.1)



Shape welded 640X

Rolled Steel 640X

Fig.11.2 SEM micrographs of samples after corrosion in 96% H_2SO_4 a
15°C temperature.

Shape welded

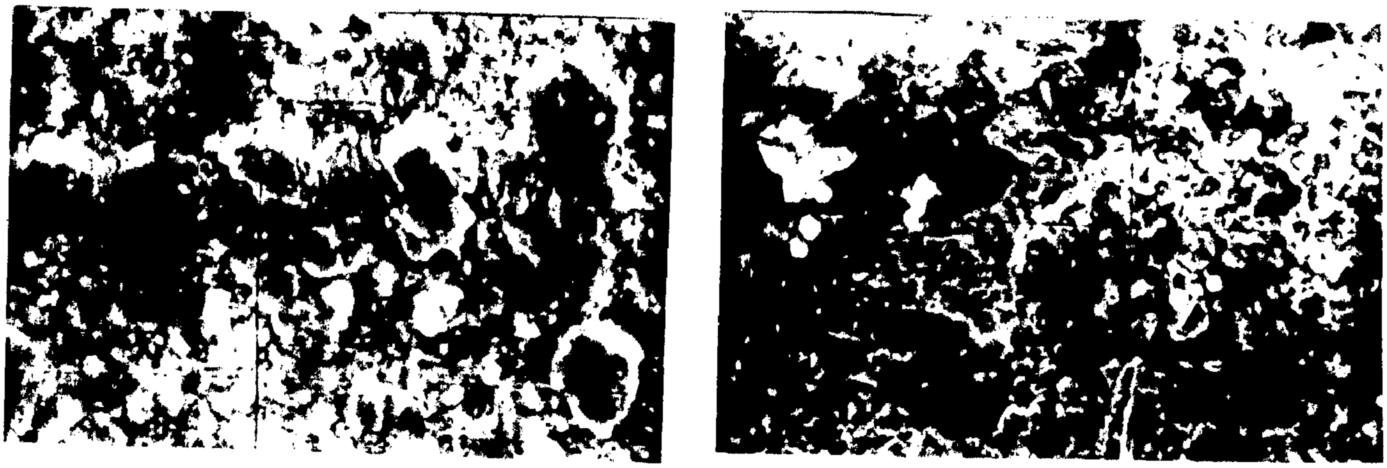
640X

Rolled Steel

320X

Fig.12.1 SEM micrographs of samples after corrosion in 96% H_2SO_4 at 50°C temperature.

(Correction factor 1.1)



Shape welded

320X

Rolled steel

640X

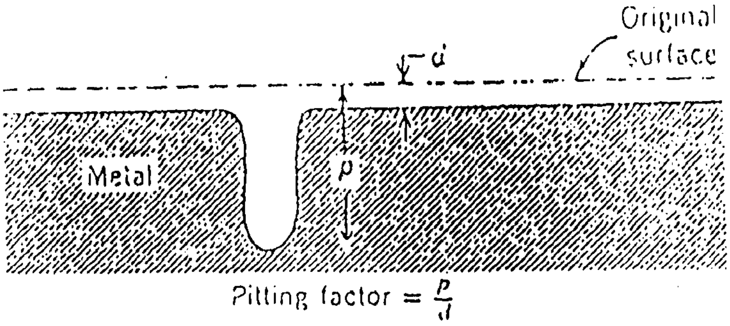


Fig-3.2.a: Schematic Diagram Showing Pitting.

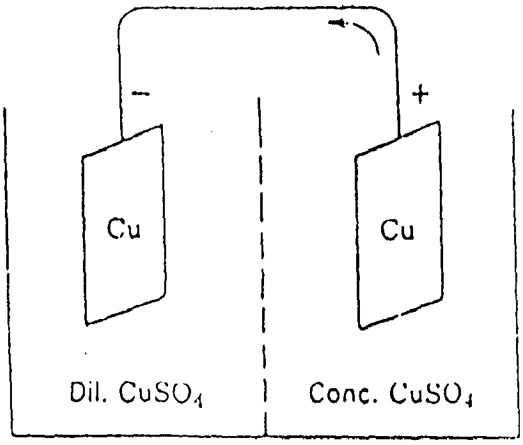


Fig-3.3.a: Salt Concentration Cell.

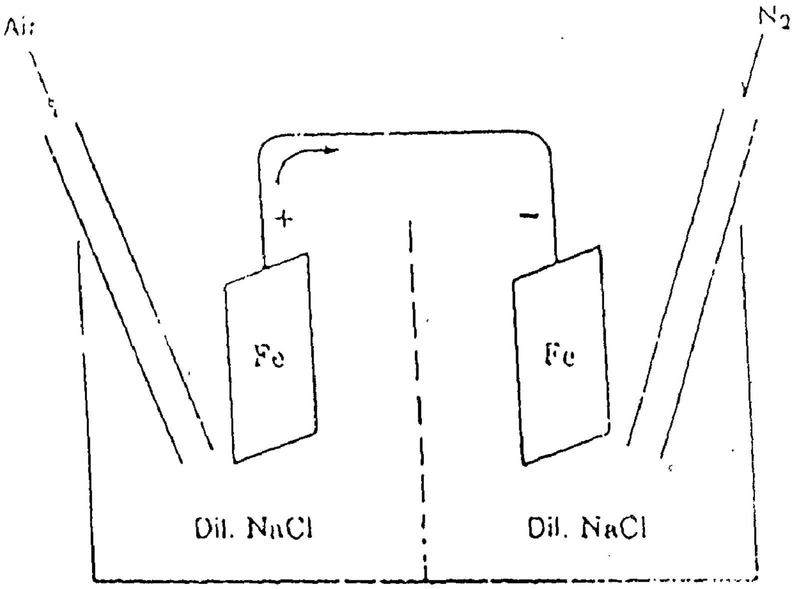


Fig-3.3.b: Differential Aeration Cell.

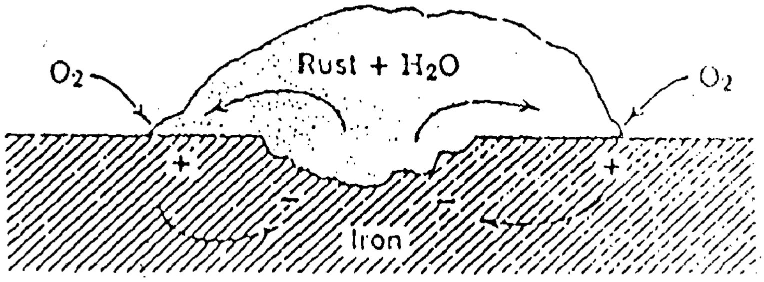


Fig-3.3.c: Pitting Damage under Rust.