EFFECT OF LONG DURATION PWHT ON THE MECHANICAL AND MICROSTRUCTURAL PROPERTIES OF SMA WELDED Cr-Mo ALLOY STEEL AND HOT CORROSION BEHAVIOUR OF Cr-Mo BOILER STEEL IN AIR AND SALT Na_2SO_4-60%V_2O_5 ENVIRONMENTS

A DISSERTATION

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(With Specialization in Welding Engineering)

By

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CANDIDATE'S DECELERATION

I hereby decelerate that the work being presented in the dissertation entitled "Effect of Long Duration PWHT on the Mechanical and Microstructural Properties of SMAW welded Cr-Mo alloy steel and Hot Corrosion behavior of Cr-Mo boiler steel in Air and Salt Na_2So_4-60%V_2O_5 environment" in the partial fulfillment of the requirements of award of the degree of Masters of technology in Mechanical Engineering with specialization in Welding engineering, submitted to the mechanical and industrial engineering department, Indian institute of technology, Roorkee (India) is an authentic record of my own work carried out during July 2009-June 2010, under the supervision of Dr. Ajai Agarwal ,Assistant Professor Department of Mechanical and Industrial Engineering and Dr.B.S.S.Daniel, Associate professor, Department of Metallurgical and Material Science Engineering.

I have not submitted the matter embodied in this dissertation for the award of any other degree of this or any other institute.

Date: 30/6/2010
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This is to certify that the above statement made by the candidate is correct to the best of my knowledge and belief.

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ABSTRACT

Welding process involves melting and subsequent cooling and the result of this thermal cycle causes residual stress and degradation of mechanical properties in weldments, When welding thicker sections. In thicker sections like pressure vessels, boilers which are used in petrochemical, chemical, oil & gas industries the existence of residual stress and undesirable mechanical properties are unacceptable as per criticality of these structures.

In order to impart high mechanical properties to thicker sections for sophisticated applications PWHT is carried out. Extensive research has been done by many authors on PWHT to investigate the effect of PWHT on mechanical properties and residual stresses and the problems occurred during PWHT like reheat cracking.

In the present work the effect of LONG-TIME POST WELD HEAT TREATMENT on the Mechanical and microstructural properties of Chromium-Molybdenum alloy steel of ASTM A 387 Gr-22, 2.25 % Cr-1%Mo alloy steel is determined,which is commonly used in boilers, pressure vessels, heat exchangers, turbine parts etc. The regions in the weldment like base metal, HAZ and weld metal were studied, through the results obtained from the Tensile test, Hardness measurement, Charpy-V-Notch impact test. Microstructural characterization using SEM and Optical microscope and chemical analysis using E-DAX, Spectroscopy. It is concluded that the degradation of mechanical properties of steel is due to the changes that occurred in the microstructure.

Material degradation at high temperature is a serious problem in power plants, which encounter severe corrosion problems resulting in the substantial loss. The problem becomes more prominent as the power plants are getting older. The boiler tubes used for super heaters and re-heaters in the steam generating systems are subjected to corrosion, resulting in tube wall thinning and premature failure. Hot corrosion has been identified as a serious problem in high temperature applications such as boilers, gas turbines, diesel engines, coal gasification plants and chemical plants.

The present study has been performed to evaluate the hot corrosion behaviour of chromium-molybdenum alloy steel when exposed to high temperature oxidation in air and molten salt (Na_2SO_4-60%V_2O_5) environment, under isothermal conditions at a temperature of 900°C, in actual degrading conditions prevailing in a coal fired boiler of a thermal plant. The work is focused on Un-welded material, weld metal and base metal welded by SMAW process. The specimens were characterized by SEM, E-DAX, X-Ray mapping, EPMA, XRD analysis. Isothermal treatments resulted in parabolic oxidation kinetics and thus resulted in weight gain.

CONTENTS

Chapter

LIST OF FIGURES

LIST OF TABLES

ABBREVIATIONS

Word	**Description**
PWHT	Post weld heat treatment
HSLA	High strength low alloy steels
SMAW	Shielded metal arc welding
Cr-Mo	Chromium-Molybdenum
BM	Base metal
WM	Weld metal
HAZ	Heat affected zone
CGHAZ	Coarse grain Heat affected zone
FGHAZ	Fine grain Heat affected zone
ROA	Reduction of area
EAF	Elongation at Fracture
SEM	Scanning Electron Microscopy
E-DAX	ELECTRON DISPERSIVE ANALYSIS
VHN	Vickers hardness number
Temp	Temperature
H or h	hours
V	Arc voltage
I	Welding current
D.C	Direct current
E	Young's modulus
MPa	Mega Pascal's

CHAPTER- 1

INTRODUCTION

Ferritic steels, containing chromium and molybdenum are well known for their excellent mechanical properties combining high temperature strength, and creep resistance with high thermal fatigue life, as well as with good thermal conductivity, weldability, and resistance to corrosion and graphitisation. Because of these characteristics this type of steels have attracted special interest for application in industrial processes related to carbochemistry, oil refining, carbon gasification and energy generation in thermal power plants, and nuclear power plants. Where components like, heat exchangers, boilers, pressure vessels and pipes lines operate at high temperatures and pressures for long periods of time.

Arc Welding is the most commonly used fabrication process employed for joining of HSLA steel in normal varied section size in normal as well as in critical applications. The welding of high strength low alloy (HSLA) steels largely dominates their use in fabrication of various components of modern applications starting from manufacturing of automobile vehicles dealing with sheet metal to heavy engineering employs thick sections.

According to their chemical composition these steels maintain good weldability due to low carbon and alloying contents. However, because of its comparatively complex weld thermal behaviour the multi-pass welding of thick sections of HSLA steels become critical in order to satisfy desired joint properties. Due to the Thermal cycle occurred during welding residual stress can occur in weldment due to the restraint by the parent metal during weld solidification and degradation of mechanical properties will also take place. The stress may be as high as the yield strength of the material itself. When combined with normal load stress these may exceed the design stress.

The development of residual stresses approaching or even exceeding the yield stress is possible when welding thick sections. For certain industry sectors, e.g. Petrochemical, Chemical, Oil and Gas, etc.The existence of residual stress of this magnitude is completely unacceptable. Unfavourable mechanical properties and residual stress resulting from welding are reduced by post weld heat treatment.

All boilers and pressure vessels, regardless of their plate thickness, require mandatory post weld heat treatment (PWHT) as specified in the Australian Standard for Pressure Vessel Manufacture (1). The main role of PWHT is to relieve vessels of elastic residual stresses by allowing plastic flow at the stress relieving temperature as a result of a decrease in yield strength .In addition; stored strain energy is dissipated by thermally activated re-arrangement and annihilation of dislocations. PWHT is mandatory after weld fabrication and it is also mandatory after weld repair.

In the present work "Effect of Long Duration PWHT on the Mechanical and Microstructural Properties of SMAW welded Cr-Mo alloy steel and HOT Corrosion behaviour of Cr-Mo boiler steel in Air and salt Na_2SO_4-60%V_2O_5 environments" the work is attempted on chromium-molybdenum alloy steel of ASTM A 387 Gr-22, 2.25 % Cr-1%Mo alloy steel with an objective to impart high mechanical properties and corrosion resistance to thicker sections for sophisticated applications by using PWHT process.

The principal aim of the research was to establish a need for PWHT to achieve desirable weldment properties. PWHT has been reported to have complementary benefits such as tempering of WM and HAZ regions, and allowing the effusion/diffusion of hydrogen away from the weld region.

The regions in the weldment like base metal, HAZ and weld metal were quantified before and after PWHT times (aswelded,0.5,2,10,50 Hours) through the results obtained from the Tensile test, Hardness measurement, Charpy-V-Notch impact test. Microstructural characterization using SEM and Optical Microscope and chemical analysis using E-DAX, Spectroscopy. It is concluded that the degradation of mechanical properties of steel is due to the changes that occurred in the microstructure.

At high temperature exposure the interaction between a metal or an alloy and the surrounding gases and combustion products leads to corrosion, thus leading to failure for materials and structures [2]. It is commonly reported that as a result of the oxidation process under isothermal conditions a protective Cr-containing oxide and Fe-containing oxide is developed on the surface of the steel causing a decrease of the oxidation rate with time. Oxide scale is constituted by a layered structure with compositional and microstructural variations from the substrate to the outer interface [3]. On the other hand, depending on the oxidation temperature and the chemical composition of the steel, both, the mechanisms of formation and the microstructural characteristics of the oxide scale, along with the degree of protection it provides, are different.

Metals and alloys sometimes experience accelerated oxidation when their surfaces are covered with a thin film of fused salt in an oxidising gas atmosphere at elevated temperatures. This is known as hot corrosion where a porous non-protective oxide scale is formed at the surfaces and sulphides in the substrate [4]. Alloys that are developed for heat and oxidation resistance typically form a protective layer of chromia or alumina. The more rapidly this layer is established, the better protection is offered.

The aim of our research is to study the oxidation behaviour of Cr-Mo boiler steel in Air and salt Na_2SO_4-60%V_2O_5 atmosphere under isothermal conditions in cyclic manner. In this experimental study emphasis is also given to oxide scales which were separated and fell down in boat while oxidation process was going on. The work is focused on Un-welded material, weld metal and base metal welded by SMAW process. The specimens were characterized by SEM, E-DAX, X-Ray mapping, EPMA, XRD analysis.

CHAPTER- 2

LITERATURE REVIEW

2.1. Problem Formulation Statement

To improve mechanical properties in pressure vessels and boilers of thicker sections, considerable research efforts have been directed and many papers have described the methods of improving mechanical properties by conducting tests and results. The literature survey has been concentrated on PWHT, and the effect of PWHT on certain mechanical properties and the problems arising during PWHT.

All Boilers and Pressure vessels, regardless of their plate thickness, require mandatory post weld heat treatment (PWHT) as specified in the Australian Standard for Pressure Vessel Manufacture. The main role of PWHT is to relieve vessels of elastic residual stresses by allowing plastic flow at the stress relieving temperature as a result of a decrease in yield strength .In addition, stored strain energy is dissipated by thermally activated re-arrangement and annihilation of dislocations. PWHT is mandatory after weld fabrication and it is also mandatory after weld repair.

J.G. Nawrocki *et al* [5] has worked on the tempering behavior of simulated coarse-grained heat-affected zone in alloy steel of 2.25Cr-1Mo steel which are used in power generation industry because of their elevated-temperature strength and good creep resistance. The tempering behavior of CGHAZ is done because this is typically the region of a weldment most susceptible to failure like reheat cracking, creep and hydrogen induced cracking.
He concluded that:-

- The hardness decreased as the time increased particularly at temperature of 625^0C and above.
- Microstructural study shows that steel experienced secondary hardening after tempering at 575^0 C for 5 hours; the secondary hardening is due to the Fe-rich M_3C Carbides.

Jinzhu Tan *et al* [6] studied the degradation of 2.25Cr–1Mo pressure vessel steel in a hot-wall hydro fining reactor. A test block containing a weld is exposed to the chemical service environment present within a reactor. After removal from the reactor, chemical composition, tensile behavior, impact performance is quantified and the microstructure characterized using scanning electron microscopy (SEM). Results from the tensile tests indicate that the irradiated weld and base material had similar stress–strain response. Results from the Charpy V-Notch (CVN) impact tests demonstrated that the toughness of both the base material and weld were degraded with the degradation and embrittlement of the weld zone being significantly higher than the base material..

He concluded that:-

- Tensile tests on the base and weld metals conducted at room temperature (24^0 C) and elevated temperature (427^0C) show that the tensile properties have not been degraded.
- CVN impact tests on the base and weld metals reveal that toughness is degraded and the degree of embrittlement for the weld metal is more serious than that of the base metal.
- Based on the test results from specimens that have undergone de-hydrogen, de-brittleness and hydrogen charging treatments, it is concluded that the toughness degradation for the material is mainly caused by high temperature tempering during service exposure and not by hydrogen embrittlement.

J.R. Choa *et al* [7] investigated the residual stress distribution after welding and after a post weld heat treatment by a finite element transient heat flow analysis in conjunction with a coupled thermal–mechanical analysis. To verify the numerical results, the surface residual stresses of a multi-pass butt weld were measured by the hole-drilling technique.

The results are compared favorably with the finite element analysis predictions. The numerical analysis was applied to two multi-pass welds of thick plates used in ship construction a 12-pass K-groove weld joint with 56mm plate, and a nine-pass V-groove weld joint with 32mm plate. A maximum residual stress of 316MPa was found in the 56mm plate, but was reduced to 39MPa after the post weld heat treatment.

C.Sudha *et al* [8] investigated the microstructural stability and elemental redistribution in dissimilar weldments between 9Cr–1Mo and 2.25Cr–1Mo ferritic steels during various post weld heat treatments using microscopy techniques ranging from optical to transmission electron microscopy and electron probe microanalyser. Application of PWHT at 1023 K for various times resulted in the formation of a soft zone in the low Cr side and a carbide rich hard zone adjoining the soft zone in the high Cr side of the weldment.

The width of these zones and their hardness are influenced by the time of exposure at elevated temperature. A measurable increase in the width and a decrease in the hardness of the soft and hard zones with aging times are observed Micro mechanisms responsible for the formation of these zones are proposed. Migration of carbon from low Cr side to high Cr side driven by the gradient in the carbon activity has been found to be responsible for the formation of these zones.

Conclusions were:-

- Exposure of dissimilar weldments of 9Cr–1Mo and 2.25Cr–1Mo steels to elevated temperatures results in the formation of a carbide rich zone in the high chromium side and carbon depleted zone on the low chromium side of the weldment. Width and hardness of these zones are influenced by the duration of heat treatment.
- Carbon which diffuses to the high Cr side gets precipitated in the form of chromium rich carbides leading to the 'precipitate rich' or hard zone on the weld metal side, characterized by a high hardness value.
- A ferrite matrix characterized by a low hardness value called as the soft zone is formed on the HAZ region of 2.25Cr–1Mo base metal.
- There is found to be no redistribution of molybdenum across the 9Cr–1Mo weld metal and 2.25Cr–1Mo base metal interface, whereas the qualitative as well as the quantitative analysis clearly shows the redistribution of carbon.
- The micro mechanisms operative at various stages of the growth of soft and hard zones are identified.

B.C. Muddle *et al* [9] investigated the combined roles of the nature of the oxidizing environment and of secondary precipitation on the extent of void formation in the weldments of chromium molybdenum steel. Samples of microstructurally different regions, viz., weld metal, heat affected zone (HAZ) and base metal, were separated from the weldments of 2.25Cr±1Mo steel and oxidized in environments of steam and air. The resulting oxide scales and the region immediately beneath were characterized systematically using surface analytical techniques. Extensive internal oxidation and oxidation-induced void formation were found to occur during steam oxidation, with a much greater intensity in the case of the HAZ sample.

Results of air- and steam-oxidation of the weld metal, HAZ and base metal regions of a 2.25Cr-1Mo steel weldment, and post-oxidation characterization have led to the following conclusions:-

- Physico-chemical characteristics of the scales (both external and inner scales) formed over the three weldment regions (viz. weld metal, HAZ and base metal) during air-oxidation are similar. The outer layers of the external scales formed during air-oxidation are highly porous and the inner layers have low chromium contents.
- No internal oxidation occurs during air-oxidation of the weld metal, HAZ or the base metal region at 873 K. Cross-sections of the air-oxidized specimens have not suggested any features which could facilitate crack initiation/propagation
- Steam-oxidation of all the three weldment regions results in extensive internal oxide precipitation and formation of distinct subscale zones.
- The inner layers of Fe/Cr oxide layers in the steam oxidized specimens of all the three weldment regions have signicantly high chromium contents and hence can provide protective action. However, a less protective inner scale is developed in the HAZ region which causes a formation of a thicker scale over HAZ than the weld metal and base metal regions.
- The less protective scale formation during steam-oxidation of HAZ surface also results in a subscale zone with extensive internal oxidation, void formation in the subscale and grain boundary capitation in the adjacent alloy matrix.

Victor H.C *et al* [10] studied the effect of banding and nonmetallic inclusions in AISI 4140 steel on the HAZ toughness using the two-layer temper bead technique. The results showed that the amount and the length of the nonmetallic inclusions can negatively influence the toughness. There was a significant difference between the toughness of the materials with banding in the as-welded state and the heat treated.

He concluded that:-

- Banding, besides causing heterogeneousness in terms of hardness, causes a significant reduction in toughness, although the values obtained were still above those found in the quenched and tempered base metal.

- The two-layer temper bead welding technique was able to minimize the effect of banding, proportioning toughness levels above 40 J.
- The presence of a large quantity of nonmetallic elongated inclusions with expressive lengths presented a critical factor for the toughness of these steels.
- The two-layer temper bead welding technique was efficient from the point of view of improving the welded joint properties, especially toughness, presenting results similar to those obtained with PWHT.

L. W. TSAY *et al* [11] Worked on Constant extension rate tensile (CERT) tests were to investigate the fracture characteristics of the A387 steel weldments in an H$S charging environment. The heterogeneous (HG) welds using a stainless steel ER309-16 as a filler metal displayed inferior mechanical properties. In the as-welded condition, the heat-affected zone (HAZ) with coarse-grained structures initiated the crack. The precipitation of Cr-carbides along the fusion boundary was found after tempering at 690°C for 1 h, creating a more favorable path for the crack growth.

He concluded that:-

- The A387 steel plate tested in H2S solution revealed a slight decline in strength and a Drastic decrease in ductility as compared to that tested in air.

- Tensile properties of HM weld in H2S environment could be improved by tempering at 690^0C/l h. The coarse-grained HAZ played as the crack initiation sites for H2S SCC and was more prone to hydrogen embrittlement than other regions in the weld
- The HG welds, with or without PWHT, exhibited inferior tensile properties. In the as-welded condition, both the coarse-grained HAZ and the diluted region in the WM contained a hardened structure were susceptible to hydrogen embrittlement

A.G.Olabi et al [12] worked on low carbon steel AISI 1020 butt-welded components to determine the effect of PWHT on the microstructure and mechanical properties. In order to assess the effect of PWHT, different type of heat treatments have been applied using different soaking temperature, heating rate, time durations and cooling rates. Mechanical testing and microstructural study is done before and after heat treatment.

He had concluded that

- That an improvement in the mechanical properties can be obtained after the application different heat treatments.
- Maximum improvement is obtained by the application of heat treatment with soaking temperature of 650^0 C.
- It is also concluded that the longer soaking time duration and the slower cooling rate improves the mechanical properties.

2.2. Introduction to High Temperature Corrosion

Many very important engineering systems operating at high temperatures (650-1100°C) involve contact of metallic or ceramic materials with combustion product gases or other oxidizing gases containing inorganic impurities, e.g. gas turbines, steam generators, incinerators, and numerous petrochemical process vessels. As the gases are cooled, fused salt films may condense on the hardware to generate a highly corrosive condition analogous in some aspects to aqueous atmospheric corrosion. [13]

Degradation by high-temperature oxidation, hot corrosion and erosion are the main failure modes of components in the hot sections of gas turbines, boilers, industrial waste incinerators, metallurgical furnaces, petrochemical installations, etc. Super alloys have been developed for high temperature applications, but they are not able to meet the requirements of both the high-temperature strength and the high-temperature erosion–corrosion resistance simultaneously. One possible way to overcome these problems is the use of thin high-temperature erosion–corrosion- and wear-resistant coatings.

Stringer *et al* [14] reported that Worldwide ,the majority of electricity is generated in coal-fired thermal power plants, in which the coal is burned to boil water coal is a complex and relatively dirty fuel that contains varying amount of sulphur and a substantial fractional of non combustible mineral constituents, commonly called ash. The coal used in Indian power stations has large amounts of ash (about 50%), which contains abrasive mineral species such as hard quartz, which increase the erosion propensity of coal. The vast technical literature is evidence that corrosion and deposits on the fireside of boiler tube surfaces represents severe problems.

Eliza *et al* [15] reported that Hot corrosion is accelerated is accelerated corrosion of materials at high temperatures, resulting from the presence of salt contaminants such as na2so4,Nacl and V2o5 that combine to form molten deposits, which damage the protective surface oxides. It is also called deposit corrosion ,as the salts first deposit on the surface and then change to liquid melt, either of their own or by forming complex mixture of salts (khanna) According to (Eliza) hot corrosion is divided in to two forms of attack:-type-1(high temperature hot corrosion) and type-2 (low temperature hot corrosion)various parameters may affect the development of these two forms, including alloy composition and thermo-mechanical condition, contaminant composition and flux rate, temperature and temperature cycles, gas composition and velocity and erosion process.

Khanna *et al* [16] reported hot corrosion is observed in boilers, diesel engines, mufflers of internal combustion engines and gas turbines .during the combustion stage in heat engines ,particularly in gas turbines, sodium and sulphur impurities present either in or in combustion air, react to form sodium sulphate(Na_2SO_4).if the concentration of the sulphate exceeds the saturation vapour pressure at the operating metal temperature for turbine blades and vanes(700-1000 0 c),then the deposition of the na2so4 will occur on the surface of these components .at higher temperatures the deposits of na2so4 are molten(melting point-884° C)and can cause accelerated attack of the Ni,Fe and co-base super alloys.

U.K. Chatterjee *et al* [17] has proposed that the cracking of the scale results from the enrichment by Mo at the substrate scale interface, where MoO_2 is formed. This oxide converts into MoO_3 on further oxidation, which is in liquid form at the given temperature of exposure and penetrates along the alloy scale interface. MoO_3 further reacts with Na_2SO_4 to form low melting point Na_2MoO_4 and SO_3 gas contribute to further generation of cracks. The concentration of Mo at the interface will increase as the other alloying elements are oxidized and this MoO_3 gets evaporated at the melt gas interface [18].

The better corrosion resistance of T-91 steels may also be attributed to the absence of a NiO layer in the scale. During investigation, the NiO layer was observed in the oxide scale of T-22 steel. This layer has been suggested to be loose- structured by X. Wu [19], which may not be able to provide effective protection. P. Niranatlumpong [20] have also suggested increase in the pore size of Ni and Cr scale with increase in exposure time, which allows the degrading species to penetrate through the coating thereby resulting in the oxidation of substrate steels.

Ravindra Kumar, *et al* [21] studied the effect of microstructurally different regions on the hot corrosion of tungsten inert gas weldment in 2.25Cr-1Mo (T22) boiler tube steel. Various regions of weldment were oxidized in molten salt Na_2SO_4-60% V_2O_5 environment at 900C.

The conclusions made were:

- The base metal was found to oxidize at much higher rates than the weld metal and the heat-affected zone. Oxide scales formed in the three regions were compared by scanning electron microscopy with energy dispersive of X-ray analysis, X-ray diffraction pattern, and electron probe microanalysis. The research investigates the formation of inner scales with free Cr over the HAZ.
- The weld metal shows a combination of widmanstatten type ferrite, pearlite, and bainite. HAZ region showed coarse grain bainite.
- When the base metal, weld metal, and the HAZ are oxidized in the molten salt environment; all of them follow parabolic kinetics. The weight gain of the base metal and HAZ were greater than that of weld metal. Even weld metal shows a little tendency for spallation and cracks during hot corrosion test. Fe_2O_3 was identified as the major phase by XRD, EDAX, and EPMA analysis in the scale of base metal, weld metal, and HAZ.
- The thicker oxide scale over HAZ was due to the formation of inner scales with free Cr than that of weld metal.
- The corrosion rate (in terms of weight gain) of base metal, weld metal, and HAZ regions has been found in the following order base metal>HAZ>weld metal.

Hazoor Singh Sidhu *et al* [22] worked on Cr_2C_3–NiCr, NiCr, WC–Co and stellite-6 metallic coatings were sprayed on ASTM SA-210 grade A1 steel by the HVOF process. LPG was used as the fuel gas. Hot corrosion studies were conducted on the uncoated as well as HVOF sprayed specimens after exposure to molten salt at 900°-C under cyclic conditions. The thermo-gravimetric technique was used to establish kinetics of corrosion. XRD, SEM/EDAX and EPMA techniques were used to analyse the corrosion products. All these overlay coatings showed better resistance to hot corrosion as compared to that of uncoated steel. NiCr Coating was found to be most protective followed by Cr_2C_3–NiCr coating.WC–Co coating was least effective to protect the substrate steel. It is concluded that the formation of Cr_2O_3, NiO, $NiCr_2O_4$, and CoO may contribute to the development of hot corrosion resistance in the coatings. The uncoated steel suffered corrosion in the form of intense spalling and peeling of the scale, which may be due to the formation of unprotective Fe_2O_3 oxide scale.

Conclusion made were

- The HVOF spray process provides the possibility of developing NiCr, Cr_2C_3–NiCr, WC–Co and stellite-6 coatings on boiler tube steel (GrA1). The uncoated substrate steel showed intense spalling, peeling off scale and weight gain was enormous during hot corrosion study in the aggressive environment of Na_2SO_4–60% V_2O_5 at 900°-C. Fe_2O_3 was identified as the major phase by XRD, EDAX and EPMA analysis in the scale of uncoated steel
- NiCr coating shows the lowest value of porosity and provided highest resistance to hot corrosion. Formation of protective oxides like Cr_2O_3, NiO and $NiCr_2O_4$ in the scale may have contributed to the better corrosion resistance of coatings. Spalling of scale for
- NiCr and Cr_2C_3–NiCr coatings were comparatively less. Suspected evaporation of tungsten from the scale may have attributed to the least corrosion resistance as indicated by WC–Co coating.

CHAPTER-3

EFFECT OF PWHT ON MECHANICAL PROPERTIES AND EFFECT OF HOT CORROSION IN BOILER STEELS

Welding is the most commonly used method of joining the parts together. Due to the thermal cycle occurred during welding residual stress can occur in weldment due to the restraint by the parent metal during weld solidification and degradation of mechanical properties will also take place. The stress may be as high as the yield strength of the material itself. When combined with normal load stress these may exceed the design stress.

The development of residual stresses approaching or even exceeding the yield stress is possible when welding thick sections. Arc Welding is the most commonly used fabrication process employed for joining of HSLA steel in normal varied section size in normal as well as in critical applications. The welding of high strength low alloy (HSLA) steels largely dominates their use in fabrication of various components of modern applications starting from manufacturing of automobile vehicles dealing with sheet metal to heavy engineering employs thick sections.

According to their chemical composition these steels maintain good weldability due to low carbon and alloying contents. However, because of its comparatively complex weld thermal behaviour the multi-pass welding of thick sections of HSLA steels become critical in order to satisfy desired joint properties. For certain industry sectors, eg. Petrochemical, chemical, oil and gas, etc. The existence of residual stress of this magnitude is completely unacceptable. Unfavourable mechanical properties and residual stress resulting from welding are reduced by post weld heat treatment.

PWHT is a process of reheating a weld to below the lower transformation temperature at a controlled rate, holding for a specific time and cooling at a controlled rate. It is the most widely used form of stress relieving on completion of fabrication of welded structures. The principle is that as the temperature is raised, the yield stress and the elastic modulus of the material fall. A point is reached when the yield stress no longer supports the residual stresses and some localized plastic deformation occurs.

All boilers and pressure vessels, regardless of their plate thickness, require mandatory post weld heat treatment (PWHT) as specified in the Australian Standard for Pressure Vessel Manufacture. The main role of PWHT is to relieve vessels of elastic residual stresses by allowing plastic flow at the stress relieving temperature as a result of a decrease in yield strength. In addition; stored strain energy is dissipated by thermally activated re-arrangement and annihilation of dislocations. PWHT is mandatory after weld fabrication and it is also mandatory after weld repair.

3.1. Introduction to Post Weld Heat Treatment

PWHT is a process of reheating a weld to above the upper transformation temperature (A_3) at a controlled rate, holding for a specific time and cooling in a controlled atmosphere. It is the most widely used form of stress relieving on completion of fabrication of welded structures. The principle is that as the temperature is raised, the yield strength of the material falls. A point is reached when the yield strength no longer supports the residual stresses and some localized plastic deformation occurs which reduces the residual stresses.

PWHT is considered to be very important tool of the metallurgy by the metallurgist in which we can alter the properties of steel easily. Now a day's science and technology are advancing very rapidly in purist of higher and higher properties in materials so PWHT plays a very important role.

3.1.1. Why Post weld Heat Treatment is necessary

The process of post weld heat treatment consists of uniform heating of a vessel or part of a vessel to a suitable temperature for the material below the critical range of the base metal, followed by uniform cooling. This process is used to release the locked-up stresses in a structure or weld in order to "stress-relieve" it. Because of the great ductility of steel at high temperatures, usually above 593°C, heating the material to such a temperature permits the stresses caused by deformation or straining of the metal to be released.

Thus post weld heat treatment provides more ductility in the weld metal and a lowering of hardness in the heat-affected zone (HAZ). It also improves the resistance to corrosion and caustic Embrittlement. The proper heat treatment before and after fabrication is one that renews the material as near as possible to its original state. The requirement for post weld heat treatment is largely a function of the material and the thickness. The material (in terms of alloy content) and the thickness (in relation to the quench effect) control the microstructure that will be formed.

Large section thicknesses in alloy steels can result in Martensitic, Pearlitic or Bainitic structures, depending on the cooling rate, the thicker the material that is welded, the greater the amount of residual stress that will be developed on cooling[23].

Heat treatment procedures require careful planning and will depend on a number of factors:

- Temperatures required and time control for material
- Thickness of material and weldment sizes
- Weld contour, or shape
- Heating facilities

To be effective, heat treatment must be carefully watched and recorded. Furnace charts should be reviewed to determine if the rate of heating, holding time, and cooling time are correct. If a vessel is to be fully post weld heat-treated, the furnace should be checked to make sure that the heat will be applied uniformly and without flame impingement on the vessel.

3.1.2. Effects of Welding Heat

Welding metallurgy is the study of the properties of welded metals and the thermal and mechanical effects occurring during the welding process, including the movements of the atoms in the liquid state and the formation of the grain structure of the metal in changing from the liquid to the solid state. It is known that heat treatment can change grain structure, which in turn may affect the physical properties. A metal can be hardened or softened, toughened or made more ductile.

During the cooling and solidification process, the behaviour of the molten metal in the weld joint is similar to that of molten metal poured into a mould to form a steel casting. The mould in the welded joint is the solid parent metal, the melt consists of the molten metal of the electrode plus the liquefied part of the parent metal, the two combining to form a casting within the solid parent metal[23].

When the metal is in the liquid state, its atoms can move about at random. When it cools to the temperature at which it starts to solidify, the atoms are arranged into a regular pattern. This atomic arrangement and the corresponding effect on grain size are influenced by the rate of cooling from the liquid to the solid state and also by the composition. Grain size is influenced by the maximum temperature reached by the metal, the length of time the metal is held at a specific temperature, and the rate of cooling.

For example, annealing, which involves heating to temperatures above the critical range followed by slow cooling, is used to refine the grain size to provide ductility and softness. Stresses can be relieved by heating beneath the critical range, but unless temperatures exceed that range, full grain refinement does not take place.

Normalizing is the process by which the steel is heated to approximately 37 °C above the critical range (as in annealing) and then cooled in still air. This heat treatment also refines the grain size and improves certain mechanical properties. Refinement of ferritic steel can be easily achieved by heat treatment because of the movement of the atoms in the solid metal. Note that the austenitic steels do not have a critical range and that consequently their grain size cannot be changed by heat treatment alone.

The production of a fusion weld naturally results in heating of some of the solid metal adjacent to the weld. The temperature reached by the metal in this zone varies from just below the melting point at the line of fusion to just above room temperature at a distance of a few inches from the weld. The effect of the welding heat on the parent metal depends on the temperature reached, the time this temperature is held, and the rate of cooling after welding.

It is therefore necessary to understand the changes that take place in the zone where the metal is heated to very high temperatures without ever reaching the molten condition. Usually, the weld metal zone is stronger than the parent metal but has less ductility. The heat-affected zone is the region where most cracking defects are likely to occur because the grain structure becomes coarse just beyond the fusion line and the ductility is lowest at this point[24]. This reduction of ductility must be taken into consideration and measures taken to minimize the effects of total welding heat like:

- The correct choice of parent material and welding electrode
- Preheating before welding
- Use of the proper welding procedure
- Post weld heat treatment after welding

3.2. Importance of PWHT

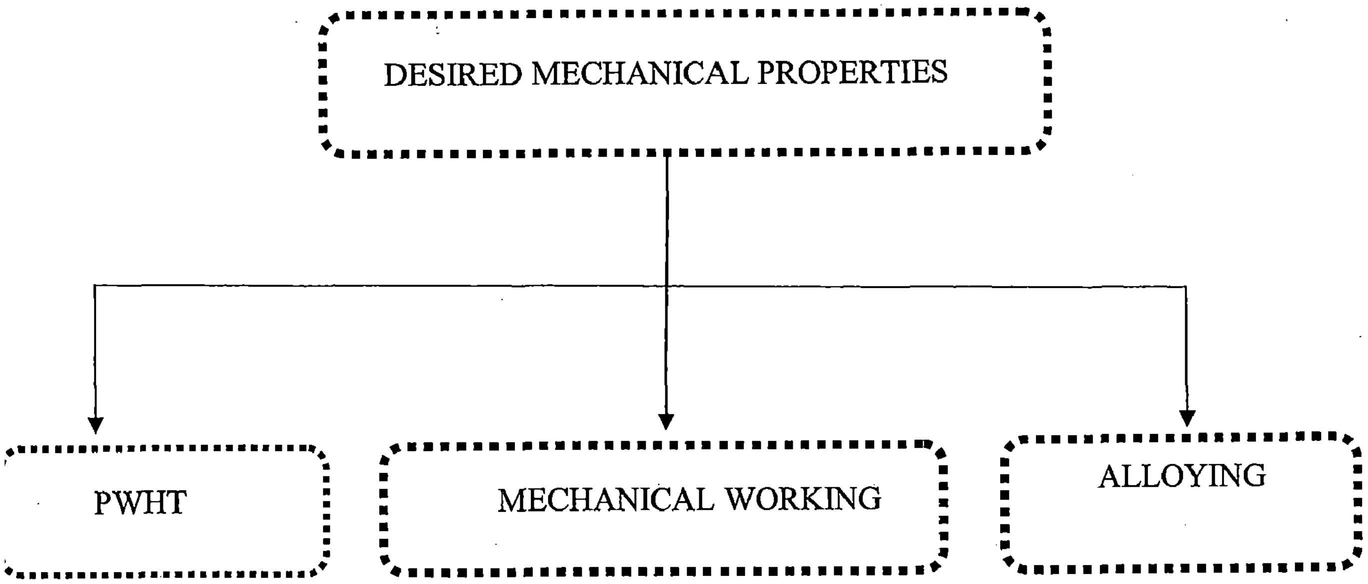


Fig.3.1: Importance of PWHT

Desired mechanical properties can be achieved by PWHT, Mechanical Working, Alloying, but mechanical working methods like peening, overstressing have some disadvantages and alloying can also affect the microstructure and mechanical properties.

- With a high Carbon Equivalent, even slower cooling rates will produce high amounts of Martensite or Bainite.
- PWHT will restore ductility to alloy steels before placing those welded assemblies into service

3.2.1. Advantages of PWHT

- Improve the ductility
- Relieves residual stresses
- Reduces the effect of cold work
- Improving dimensional stability during machine
- Improves the resistance to stress corrosion cracking
- Diffusion of entrapped hydrogen from the weldment
- Softens the heat affected zone and thus improving toughness

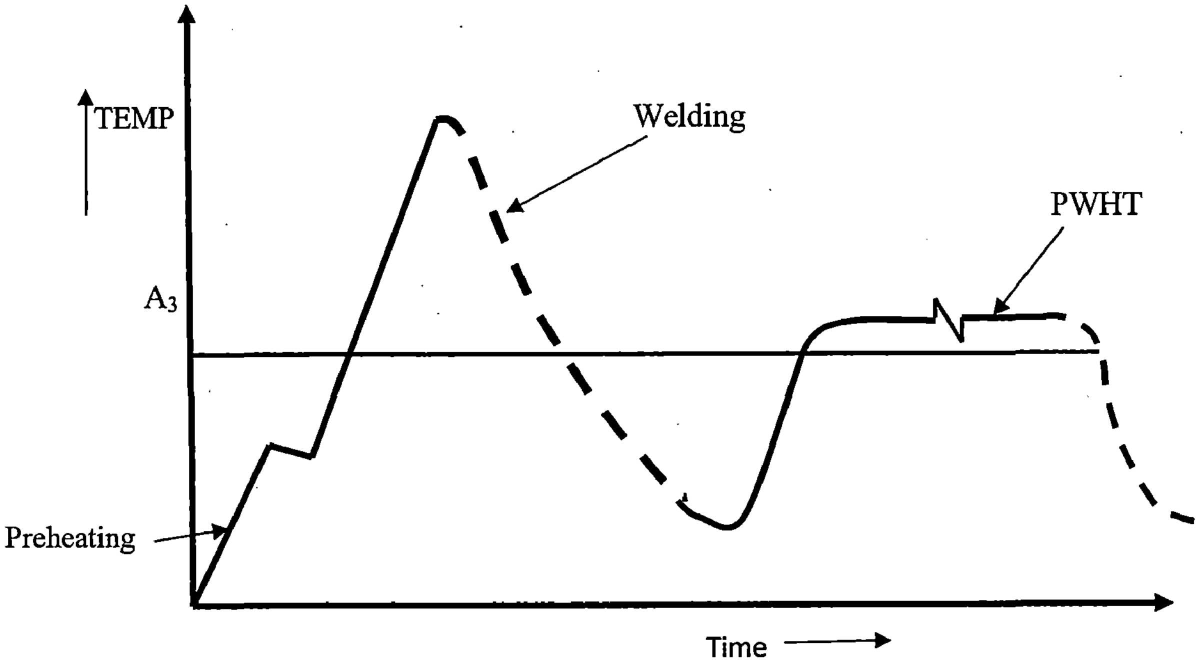


Fig.3.2: Schematic representation of Preheating, Welding, PWHT

3.2.2 Conditions Requiring Post Weld Heat Treatment

Post weld heat treatment requirements (according to materials, thicknesses, service, etc.) include the following

- All carbon-steel weld joints must be post weld heat-treated if their thickness is more and if the material has been preheated to a minimum temperature of 93°C during welding.
- Vessels containing lethal substances must be post weld heat treated.
- Carbon and low alloy steel vessels must be post weld heat treated when the minimum design metal temperatures are below –45 °C, unless exempted from impact tests.
- Ferritic steel flanges must be given a normalizing or fully annealing heat treatment if the thickness of the flange section exceeds 76 mm.
- Unfired steam boilers with a design pressure exceeding 345 KPa and fabricated with carbon or low-alloy steels must be post weld heat –treated.
- Castings repaired by welding to obtain a 90 or 100 percent casting factor must be post weld heat-treated.
- Carbon and low alloy steel plates formed by blows at a forging temperature must be post weld heat-treated.
- Some vessels of integrally clad or applied corrosion-resistance lining material must be post weld heat-treated when the base plate is required to be Post weld heat treated.

- Some high-alloy chromium steel vessels must be post weld heat treated.
- Some welded repairs on forgings require post weld heat treatment.
- When the thickness of welded joints for vessel parts or pressure vessels constructed with carbon steels exceeds 15 mm in and for all thicknesses of low-alloy steels which are subject to direct firing, Post weld heat treatment must be performed [25].

3.2.3. Heating and Cooling Rates

- These are specified in most of the construction Standards, and are reasonably similar. For example, AS 4458 for carbon manganese steel vessels limits heating and cooling rates to 200 ^{0}C/hour for thicknesses up to 25 mm, and 500 ^{0}C/hour divided by the thickness for vessels over 25 mm.
- There is also a minimum limit of 500 ^{0}C/hour for very thick vessels. However, for ½Cr½Mo¼V and 2¼Cr1Mo alloys, the heating rate is limited to half that of carbon manganese steels. For comparison, EN 13445 allows 220 ^{0}C/hour up to 25 mm thick, 550 ^{0}C/thickness for 25 to 100 mm and 550 ^{0}C/hour above 100 mm thick.
- There are no restrictions on the alloy steels in EN 13445. By the time the holding temperature has been reached, the variation in temperature between any two thermocouples should not exceed the range specified for the material.
- In AS 4458, the range is typically 400 ^{0}C (eg. C-Mn steel range is 580 – 620 ^{0}C), and in EN 13445, it is 50 – 70 ^{0}C. In most practical situations, the variation can be limited to much less than this. The issues described here are vital to avoid Post weld heat treatment going wrong and the result being a scrap vessel [25].

3.3. Mechanical Properties Responding To PWHT

3.3.1. Strength

The strength of a material is usually taken as the force required fracturing a test-piece in tension. When we talk about the general strength of a particular material it is usually referring to tensile strength. The strength of weld metal increases as the contents of alloying element is increased .the most important strengthening element is carbon but too high a carbon level is often undesirable in a weld metal. The use of carbon equivalent formula is a useful way of estimating weld metal strength [26].

It is useful for the strength of a weld metal to overmatch that of a parent metal. Overmatching ensures that if a structure is overloaded .The parent metal deforms before the welds. Therefore it is more important for the weld .therefore it is more important for the yield strength to over match them for the tensile strength. Some degree of over matching is advantageous because in most cases the parent metal is less likely to contain defects sufficient to cause premature failure than weld metal.

Overmatching of weld metal strength has some disadvantages like:

- The highest tensile residual stress in an as-welded joint is approximately equal to the yield strength of the weld metal itself. This means the stronger the weld metal the higher will be the maximum residual stress.
- If strength of weld metal is increased the risk of its suffering weld metal hydrogen cracking is also increased.
- Too hard a weld metal may result either in stress corrosion cracking or in hydrogen cracking in service.

Weld metal strength generally increases as temperature falls and decreases as the temperature is increased until it is vanishingly small as the weld metal starts to melt. In as welded condition, the smooth fall in strength with increasing temperature from ambient is interrupted by the effect of strain aging between 100 ^{0}C and 200 ^{0}C. The effect of Post Weld Heat Treatment is it generally reduces Weld Metal, HAZ and often parent metal strength so that repeated PWHT can reduce strength levels below specification values.

Typical values of modulus of elasticity at different temperature

Material	Modulus of elasticity GPa (10^6 psi), at:				
	Room temperature	250 °C (400 °F)	425 °C (800 °F)	540 °C (1000 °F)	650 °C (120 °F)
Carbon steel	207 (30.0)	186 (27.0)	155 (22.5)	134 (19.5)	124 (18.0)
Austenitic stainless steel	193 (28.0)	176 (25.5)	159 (23.0)	155 (22.5)	145 (21.0)
Titanium alloys	114 (16.5)	96.5 (14.0)	74 (10.7)	70 (10.0)	...
Aluminum alloys	72 (10.5)	65.5 (9.5)	54 (7.8)	...	...

Table.3.1.Values of Modulus of Elasticity [27].

3.3.2. Ductility

Ductility is more commonly defined as the ability of a material to deform easily upon the application of a tensile force, or as the ability of a material to withstand plastic deformation without rupture. Ductility may also be thought of in terms of bendability and crushability. Ductile materials show large deformation before fracture. The lack of ductility is often termed brittleness. Usually, if two materials have the same strength and hardness, the one that has the higher ductility is more desirable. The ductility of many metals can change if conditions are altered [26].

An increase in temperature will increase ductility. A decrease in temperature will cause a decrease in ductility and a change from ductile to brittle behaviour. Cold-working also tends to make metals less ductile. The heating of a cold-worked metal to or above the temperature at which metal atoms return to their equilibrium positions will increase the ductility of that metal. This process is called annealing. Ductility is desirable in the high temperature pressure applications in reactor plants because of the added stresses on the metals. High ductility in these applications helps prevent brittle fracture.

At much higher temperature when the weld metal is austenitc, there is a so-called ductility dip range, which can sometimes gives problems. Close to the melting temperature ductility falls to zero. If the ductility is lost at a temperature appreciably lower than that at which strength is lost, the weld metal undergoes liquation cracking [28].

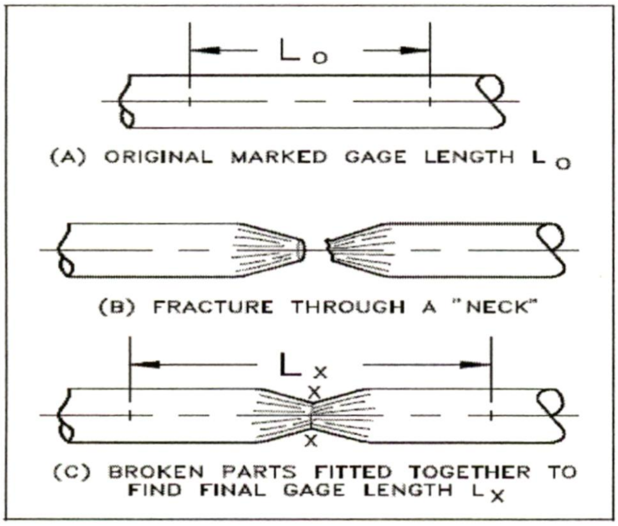


Fig.3.3: Measuring Elongation after Fracture [26].

3.3.3. Toughness

The toughness describes the way a material reacts under sudden impacts. It is defined as the work required to deform one cubic inch of metal until it fractures. Good toughness refers to a good resistance to brittle fracture i.e. a low transition temperature between ductile and brittle fracture. Brittle fracture in welds is particularly dangerous because it can lead to failure apparently below the yield stress of the parent metal and without prior deformation. Even measured toughness is reduced due to PWHT the overall fracture resistance of the joint will be better because of lower residual stresses.

PWHT improves toughness by softening the HAZ it lowers residual stresses so that less toughness is required for a given acceptable or tolerable defect size PWHT temper the HAZ and thus remove the harmful effect of martensitic local brittle zone. PWHT at 600 ° C should be used with carbon and carbon manganese steels. PWHT at 600 °C is likely to improve the measured toughness of HAZ unless they contain elements such as Cr,Mo,Nb,and V which are likely to give secondary hardening .for such steels higher temperature generally at 650 °C or high are recommended.

PWHT does not always improve measured toughness. Deterioration in toughness may be a result of residual austenite in the weld metal transforming on PWHT to Martensite or carbides or to secondary hardening by alloying or micro alloying elements [26].

3.3.4. Toughness testing

Toughness is measured by the Charpy test or the Izod test. Both of these tests use a notched sample. The location and shape of the notch are standard. The points of support of the sample, as well as the impact of the hammer, must bear a constant relationship to the location of the notch.

The tests are conducted by mounting the samples as and allowing a pendulum of a known weight to fall from a set height. The maximum energy developed by the hammer is 120 ft-lb in the Izod test and 240 ft-lb in the Charpy test. By properly calibrating the machine, the energy absorbed by the specimen may be measured from the upward swing of the pendulum after it has fractured the material specimen. The greater the amount of energy absorbed by the specimen, the smaller the upward swing of the pendulum will be and the tougher the material is. Material Toughness Test Indication of toughness is relative and applicable only to cases involving exactly this type of sample and method of loading [26].

Sample of a different shape will yield an entirely different result. Notches confine the deformation to a small volume of metal that reduces toughness. In effect, it is the shape of the metal in addition to the material composition that determines the toughness of the material.

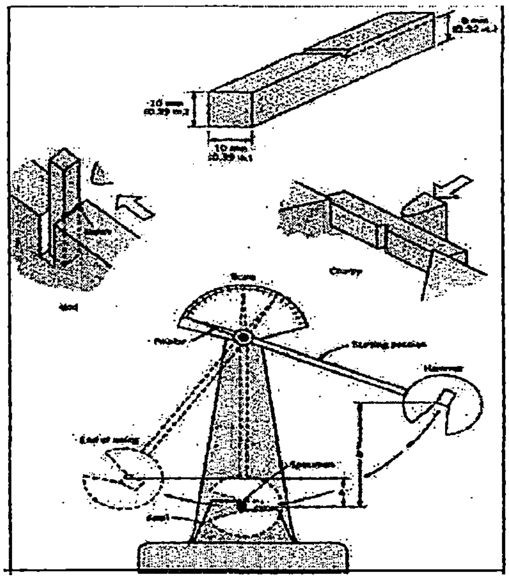


Fig.3.4: Impact testing machine.[26].

Hardness is the property of a material that enables it to resist plastic deformation, penetration, indentation, and scratching. Hardness tests serve an important need in industry. The tests are empirical, based on experiments and observation, rather than fundamental theory. Its chief value is as an inspection device, able to detect certain differences in material when they arise even though these differences may be indefinable. Multiple PWHT cycles shows influence on weld metal hardness.

The hardness at the plate mid thickness is slightly higher than at the surfaces because of the presence of a segregated band. It was reported by Steve lampman (weld integrity and performance, ASM International) that weld metal hardness shows a more noticeable effect when exposed to multiple stress-relief cycles. He conducted experiments on two welds in which weld 1&2 had a fairly uniform hardness of approximately 200 HV, but the third PWHT cycle weldment hardness dropped to 160-200 HV. With multiple PWHT cycles a reduction in Haz hardness occurs, without affecting plate values [26].

3.4. Effect of Alloying Elements on Boiler and Pressure Vessel Steels

Carbon

Carbon increases the tensile properties of steel, hardness and the hardenability. Carbon content in pressure vessel steel is in the range between 0.10-0.22% for enhanced weldability and toughness properties. Carbon plays a vital role in the formation of carbides, which impede dislocation motion and increase the strength of the steel.

Manganese

Manganese is an austenite stabilizer and is added to heat treated, steels as a hardenbility promoter. In carbon steels the manganese content is relatively high 1-1.7 wt%.manganese generally reacts with sulfur to form manganese sulphides and hence minimize the problem of hot cracking in weld metal and base metal.

Silicon

Silicon increases the fluidity and generally improves oxidation resistance. In carbon steels silicon is limited to 0.6% because it can adversely affect the toughness of welds. Silicon is normally regarded as a ferrite former, but can act like an austenite stabilizer by retarding cementite formation and hence suppressing decomposition of austenite to cementite and ferrite.

Chromium

Chromium is added to steel to increase corrosion resistance and oxidation resistance, to increase hardenability or to improve high temperature strength. At high temperature chromium contributes increase strength; chromium is stronger carbide former. The percentage of chromium in carbon steels is 0.3 wt%.

Molybdenum

It increases the hardenability of steel. It is added to suppress temper embrittlement.

Titanium

Titanium is used to retard grain growth and thus improve toughness. Titanium is also used to achieve improvements in inclusion characteristics. Titanium causes sulphide inclusions to be globular rather than elongated this improving toughness and ductility. The wt% of titanium is 0.03 in carbon steels.

Nickel

It increases the hardanability and impact strength of steels at low temperatures. Nickel is added to copper containing steels mainly to provide protection against hot shortness. Hot shortness is embrittlement of a metal at elevated temperature caused by a low melting constituents segregated at grain boundaries during solidification.

Copper

Copper is added for solid solution strengthening and improved corrosion resistance. The wt% of copper in carbon steels is 0.3%. it has been reported(irani et al,1968,pp 75-89)that copper contents above approximately 0.6% cause precipitation hardening.

Sulphur &Phosphorus

These are kept to a minimum and are treated as impurities. The carbon steels contains sulphur &phosphorus as 0.03% increases strength and decreases ductility and notch impact toughness.

Aluminium

It is widely used as a deoxidizer. Aluminium can control grain growth in reheated steels and is therefore added to control grain size. Aluminium is the most effective alloy in controlling grain growth.

Niobium

Increases the yield strength and retards the recrystallization of austenite. Thus promoting a fine grain microstructure having improved strength and toughness.

Vanadium

Increase the yield strength and the tensile strength the impact transition temperature also increases when vanadium is added.

3.5. Residual Stresses in structures

Residual stresses are stresses that remain in a component after any external loading is removed residual stress in metal structures occur for many reasons during manufacturing processes such as rolling, forging, casting and welding. In welding residual stresses results from thermal strains during heating and cooling cycles of the weld metal and the adjacent HAZ.

Residual stress can be classified into two groups according to the mechanism that produce them:-

- Residual stresses produced by structural mismatch
- Residual stresses produced by an uneven distribution of non-elastic strains.

Both of the above mechanisms are possible with all welding process. Residual stresses can produce when materials of different lengths are forcibly connected.

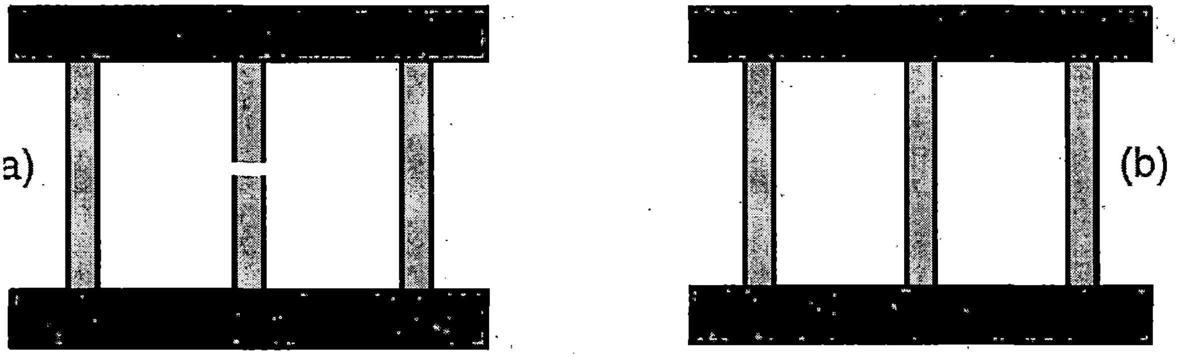


Fig.3.5: (a) Stress Free State & (b) stressed state [30].

Tensile residual stresses are produced in the shorter middle bar and compressive residual stresses are produced in the longer bar. Upon heating the middle bar the residual stresses in this bar decrease and may eventually become compressive due to thermal expansion or phase change.

Residual stresses can be produced by an uneven distribution of non-elastic strains. A residual stress produced by welding tends to occur by this mechanism. If a material is heated uniformly in a welding or alternative process then thermal expansion would occur uniformly and thermal stresses would not develop.

3.5.1. Residual Stresses Related To Welding

Welding is the most common cause of significant residual stresses. The cooler parent metal restrains contraction of the weld metal upon cooling, leading inevitably large residual stresses. Phase and volumetric changes at the microscopic level also contribute to the residual stress phenomenon during welding[30].

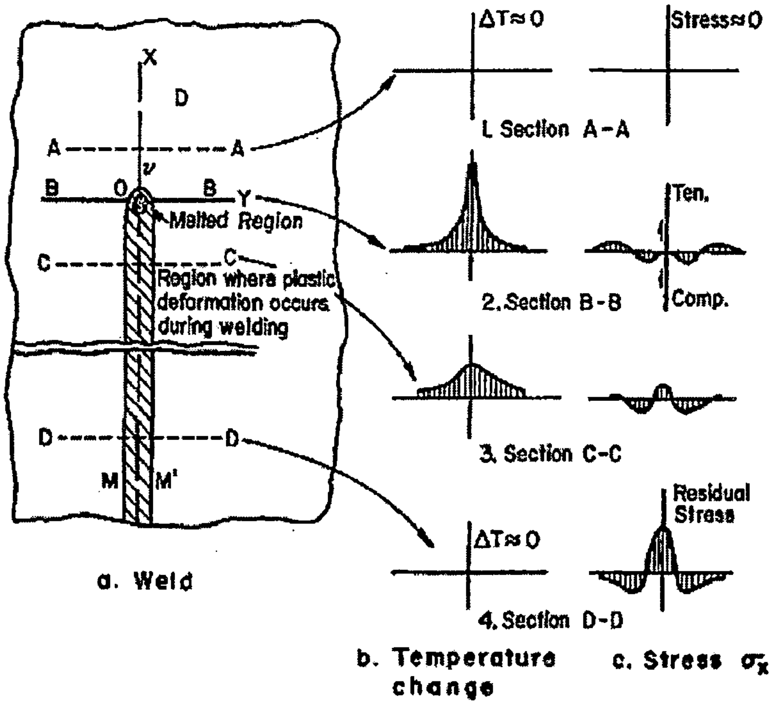


Figure.3.6: Changes in temperature and stresses during welding [30].

In Figure 3.5(a) the weld is shown by the shaded area, the molten weld pool region is shown by the origin O. **Fig** 3.6 shows the temperature profiles along different sections with section B-B bisecting the melted region and section C-C being at a close distance from the weld pool in the solid weld metal. The greater temperature gradient is at the weld pool as shown in section B-B.

Figure 3.6 shows the residual stresses as a result of welding. Section A-A ahead of the weld bead on the parent metal shows no residual stresses. In the melted region (section B-B) there are thermal stresses present but they are close to zero because the molten metal cannot support any loads. In region away from the arc where cooling is occurring the structure s are larger due to the lower temperature and restrained contraction. The maximum magnitude of compressive stresses and tensile stresses occur at section D-D where the tensile stresses are peak in the cooled weld metal and compressive peak in the surrounding parent metal [30].

3.5.2. Factors Affecting Residual Stresses in Welding are

- Time
- Heat input
- Temperature
- Welding process
- Weld geometry
- Deposition sequence
- Thickness joint restraint

3.5.3. Effect of PWHT on Residual Stresses

The main aim of PWHT is to relive the residual stresses induced by welding process .the residual stresses are reduced in the range of 600-650° C .In PWHT it is important to achieve the correct temperature and temperature control within specified limits uniform heating and cooling rate must be obtained through the heaviest section to be treated, especially where the geometry is complex and the thickness is variable. Stress relief is depends on the quantity of heat involved in the PWHT process.

The degree of improvement in properties after PWHT is dependent on the original residual stress distribution in welded joint. It is suggested that if preventative measures were taken to minimize residual stresses then the benefits of PWHT would be more beneficial. In C-Mn steels stress relief heat treatment is beneficial in improving fracture toughness of the haz, allowing service at lower temperature [31].

3.5.4. Hydrogen Embrittlement

Hydrogen Embrittlement reduces the ductility of most ferritic steels, in HAZ and weld metal to some degree after welding. Hydrogen content in the thicker sections takes many years for diffusion of all the hydrogen from the centre of a section. The hydrogen content particularly near free surface is considerably reduced by PWHT [24].

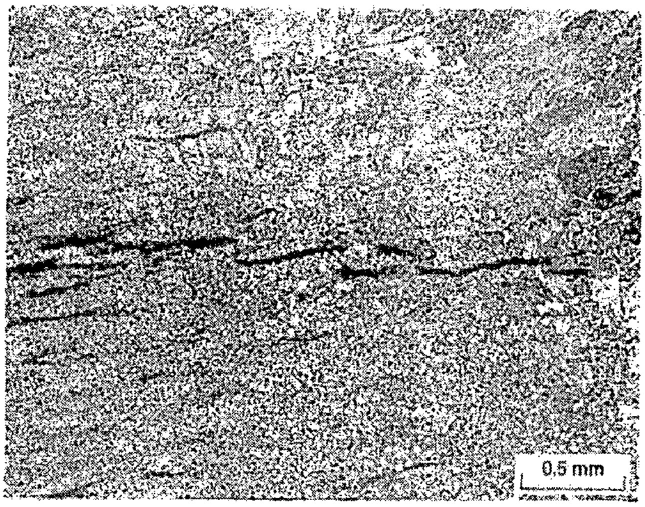


Fig.3.7: HAZ Hydrogen Cracking [24].

3.6. Problems associated during PWHT

3.6.1. Reheat Cracking

Cracking that occurs during PWHT is called reheat cracking, this is commonly found in thicker sections containing Cr, Mo, V or weldments containing at least two of the elements. Which are added to enhance corrosion resistance and elevated temperature strength. When these alloy steels are PWHT at 550-650^0 C to relive stress to reduce susceptibility to hydrogen cracking or stress corrosion cracking, mostly cracking can occur in the HAZ during reheating [26].

Mechanism of Reheat Cracking

During welding the HAZ near the fusion line is heated to temperatures very high in the austenite phase field, where pre-existing carbides of Cr, Mo, and V dissolve and the austenite grains grow. Subsequent rapid cooling does not allow enough time for carbides to reprecipitate, resulting in super saturation of these alloying elements as the austenite transforms to martensite when the coarse-grained HAZ is reheated to elevated temperature for stress relieving, fine carbides precipitate at dislocations in the prior austenite grain interiors and strengthen them before stresses are relieved. Since the grain interiors are strengthened more than the grain boundaries and since this occurs before stresses are relieved, cracking can occur along the grain boundaries.

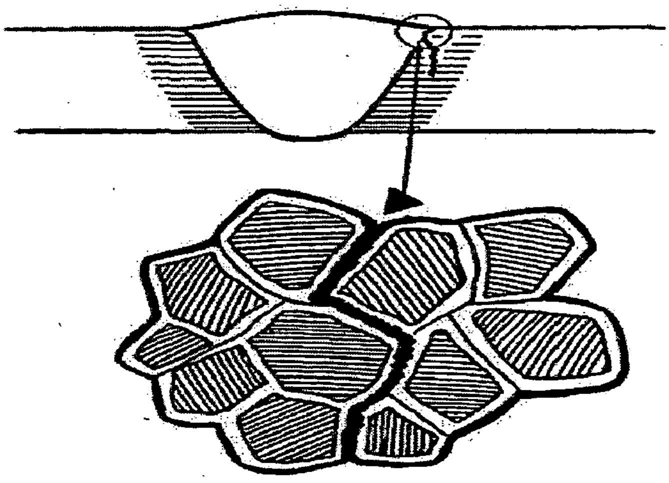


Fig.3.8: Reheat cracking [24].

Fig.3.9: Micro section showing cracking [24].

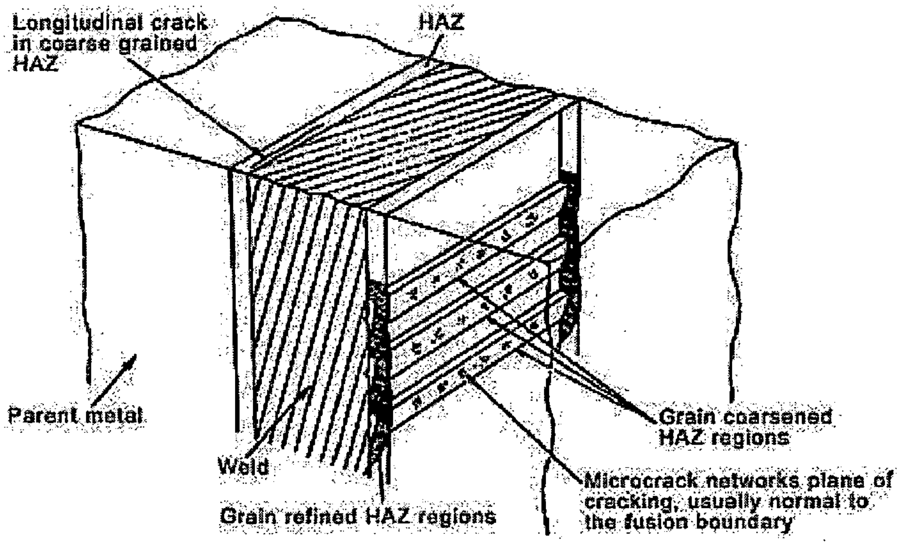


Fig.3.10: Location of reheat cracks in a nuclear pressure vessel steel[54].

3.6.2. VISUAL APPEARANCE

Reheat cracking may occur in low alloy steels containing alloying additions of chromium and molybdenum or chromium, molybdenum and vanadium when the welded component is being subjected to post weld heat treatment, such as stress relief heat treatment, or has been subjected to high temperature service (typically in the range 350 to 550°C).Cracking is almost exclusively found in the coarse grained regions of the heat affected zone (HAZ) beneath the weld, or cladding, and in the coarse grained regions within the weld metal. The cracks can often be seen visually, usually associated with areas of stress concentration such as the weld toe.

Cracking may be in the form of coarse macro-cracks or colonies of micro-cracks. A macro-crack will appear as a 'rough' crack, often with branching, following the coarse grain region, Cracking is always intergranular along the prior austenite grain boundaries. Macro-cracks in the weld metal can be oriented either longitudinal or transverse to the direction of welding. Cracks in the HAZ, however, are always parallel to the direction of welding.

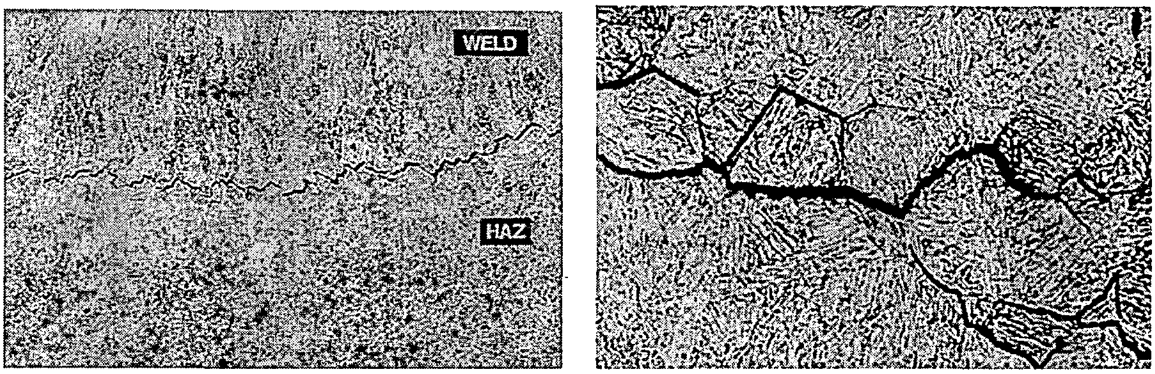


Fig.3.11: a) Cracking in the coarse grained HAZ and b) Intergranular morphology of reheat cracks. [41].

3.6.3. Causes of Reheat Cracking

Austenite Grain Size

The coarser the grain size, the fewer are the grain boundary region available both for deformation during stress relief and also to which any embrittling impurities can segregate. Hence cracking is most likely to occur at region with a coarser prior austenite grain size.

The principal cause is that when heat treating susceptible steels, the grain interior becomes strengthened by carbide precipitation, forcing the relaxation of residual stresses by creep deformation at the grain boundaries. The presence of impurities which segregate to the grain boundaries and promote temper embrittlement, e.g. antimony, arsenic, tin, sulphur and phosphorus, will increase the susceptibility to reheat cracking.

Presence of Notches

Presence of notches at weld toes can in marginal circumstances promote cracking. The joint design can increase the risk of cracking. For example, joints likely to contain stress concentration, such as partial penetration welds, are more liable to initiate cracks. The welding procedure also has an influence. Large weld beads are undesirable, as they produce coarse columnar grains within the weld metal and a coarse grained HAZ which is less likely to be refined by the subsequent pass, and therefore will be more susceptible to reheat cracking.

Heating rate

Heating rate is one of the cause of reheat cracking it has less influence on cracking slower heating rate can cause reheat cracking. To reduce the risk of reheat cracking the heating rate should be as fast as strictly controlled in order to avoid unequal heating of different parts, particularly if they are of different thickness.

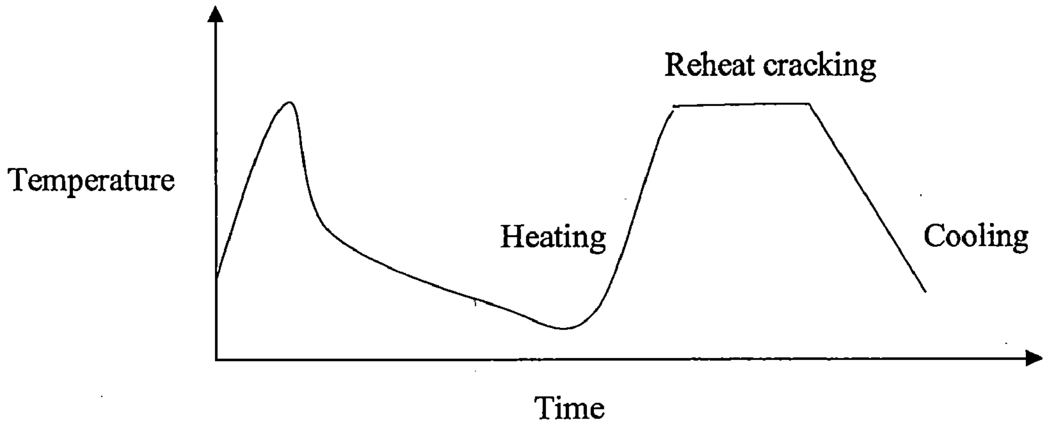


Fig.3.12: Schematic representation of reheat cracking.

3.6.4. Preventive Technique

The welding procedure can be used to minimize the risk of reheat cracking by

- Producing the maximum refinement of the coarse grain HAZ
- Limiting the degree of austenite grain growth
- Eliminating stress concentrations

The procedure should aim to refine the coarse grained HAZ by subsequent passes. In butt welds, maximum refinement can be achieved by using a steep-sided joint preparation with a low angle of attack to minimize penetration into the side-wall. In comparison, a larger angle V preparation produces a wider HAZ, limiting the amount of refinement achieved by subsequent passes. Narrow joint preparations, however, are more difficult to weld, due to the increased risk of lack of side-wall fusion [54].

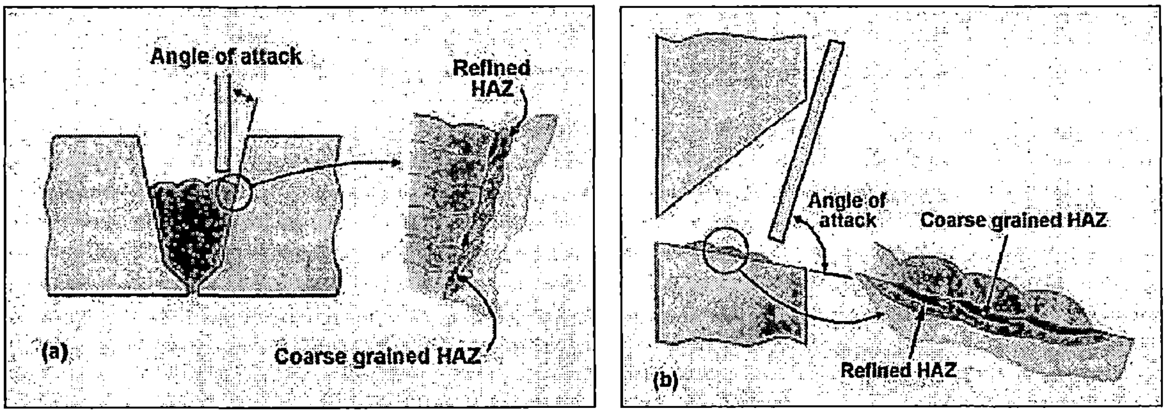


Fig.3.13: a) Welding in the flat position - high degree of HAZ refinement and b) Welding in the horizontal/vertical position - low degree of HAZ refinement [54].

Refinement of the HAZ can be promoted by first buttering the surface of the susceptible plate with a thin weld metal layer using a small diameter (3.2mm) electrode. The joint is then completed using a larger diameter (4-4.8mm) electrode, which is intended to generate sufficient heat to refine any remaining coarse grained HAZ under the buttered layer.

The degree of austenite grain growth can be restricted by using a low heat input. However, precautionary measures may be necessary to avoid the risk of hydrogen-assisted cracking and lack-of-fusion defects. For example, reducing the heat input will almost certainly require a higher preheat temperature to avoid hydrogen-assisted cracking.

The joint design and welding technique adopted should ensure that the weld is free from localized stress concentrations which can arise from the presence of notches. Stress concentrations may be produced in the following situations:

- welding with a backing bar
- a partial penetration weld leaving a root imperfection
- internal weld imperfections such as lack of sidewall fusion
- the weld has a poor surface profile, especially sharp weld toes

The weld toes of the capping pass are particularly vulnerable, as the coarse grained HAZ may not have been refined by subsequent passes. In susceptible steel, the last pass should never be deposited on the parent material, but always on the weld metal, so that it will refine the HAZ. Grinding the weld toes with the preheat maintained has been successfully used to reduce the risk of cracking in 0.5Cr 0.5Mo 0.25V steels [24].

3.7. Introduction to Hot Corrosion

3.7.1. Definition of Corrosion:

Corrosion is defined as the destruction or deterioration of a material because of reaction with its environment [32].Corrosion can be fast or slow depending upon its environment.

Corrosion can be classified in eight forms

1) Uniform corrosion
2) Galvanic corrosion
3) Crevice corrosion
4) Pitting corrosion
5) Intergranular corrosion
6) Erosion corrosion
7) Stress corrosion
8) High temperature corrosion (Hot corrosion)

3.7.2. Hot corrosion

Hot corrosion has been defined by Rapp and Zhang [33] as the accelerated oxidation of materials, at elevated temperatures, induced by a thin film of fused salt deposit. Hot corrosion may be defined as an accelerated corrosion, resulting from the presence of salt contaminants such as Na_2SO_4, NaCl, and V_2O_5 that combine to form molten deposits, which damage the protective surface oxides.

The operation of high-temperature engineering systems, despite their associated materials problems, is inherent to advanced technologies that strive to gain an advantage in thermodynamic driving force or in reaction kinetics or both. Certain high-temperature systems involve the contact of materials with a large quantity, or significant depth, of a salt solution above its liquidus temperature. Such systems include the molten carbonate fuel cell (MCFC), molten chloride baths to melt used aluminum beverage cans, the Hall-Heroult aluminum electrowinning cell with a fluoride (cryolite)-base electrolyte, nitrate/nitrite heat-exchange salts, and fused salt descaling or heat treatment baths. In other important engineering systems, the accelerated corrosion of materials results from the contact of materials with thin films of deposited fused salts. This important corrosion mode, analogous in certain aspects to aqueous atmospheric corrosion near room temperature, is called hot corrosion.

Applications of Cr-Mo Boiler steel are

- Pressure vessels
- Boilers
- Heat exchanger tubes
- Super heater
- Nozzle guide vanes
- Automotive brake discs
- Piston rings
- Gas turbine blades and Combustion cans

3.7.3. Effect of High Temperature Corrosion

Many very important engineering systems operating at high temperatures (650-1100°C) involve contact of metallic or ceramic materials with combustion product gases or other oxidizing gases containing inorganic impurities, e.g. gas turbines, steam generators, incinerators, and numerous petrochemical process vessels. As the gases are cooled, fused salt films may condense on the hardware to generate a highly corrosive condition analogous in some aspects to aqueous atmospheric corrosion.

Degradation by high-temperature oxidation, hot corrosion and erosion are the main failure modes of components in the hot sections of gas turbines, boilers, industrial waste incinerators, metallurgical furnaces, petrochemical installations, etc. Super alloys have been developed for high temperature applications, but they are not able to meet the requirements of both the high-temperature strength and the high-temperature erosion–corrosion resistance simultaneously.

Khanna *et al* [34] reported hot corrosion is observed in boilers, diesel engines, mufflers of internal combustion engines and gas turbines .during the combustion stage in heat engines ,particularly in gas turbines, sodium and sulphur impurities present either in or in combustion air, react to form sodium sulphate(Na_2SO_4).if the concentration of the sulphate exceeds the saturation vapour pressure at the operating metal temperature for turbine blades and vanes(700-1000^{0} c),then the deposition of the Na_2SO_4 will occur on the surface of these components .at higher temperatures the deposits of Na_2OS_4 are molten(melting point-884^0C)and can cause accelerated attack of the Ni, Fe and Co-base super alloys.

According to Ranjab, eliaz *et al* [35] Hot Corrosion is divided in to two forms of attack:-type-1(high temperature hot corrosion) and type-2 (low temperature hot corrosion)various parameters may affect the development of these two forms, including alloy composition and thermo-mechanical condition, contaminant composition and flux rate, temperature and temperature cycles, gas composition and velocity and erosion process.

3.7.4. High Temperature (type-1) hot corrosion (HTHC)

Type -1 hot corrosion os observed mainly with the temperature range 850-950^0C when the salt deposit on the metal or alloy is in liquid state. Type-1 hot corrosion involves the transport of the sulphur from a sulphatic deposit (generally Na_2SO_4) across a preformed oxide in to the metallic material with the formation of the most suitable sulphides. The dominant salt in type-1 hot corrosion is $na_2 so_4$ due to its high thermodynamic stability. Other impurities present either in the fuel or in the air, such as vanadium, phosphorous, lead and chlorides, can combine with $na_2 so_4$ to form compounds with lower melting points. For example Na_2SO_4 (m.p.884 ^{0}C) when combined with V_2O_5 (m.p.670 ^{0}C), a eutectic is formed with a melting point of 550 ^{0}C, thus providing a aggressing environment. The macroscopic appearance of HTHC is characterised in many cases by severe peeling of the metal and by significant colour changes. For instance, greenish tone appears on the surface of the metals and alloys due to the formation of NiO in the area of accelerated attack. Microscopically the morphology of type-1 is characterized by sulphidised and depleted region beneath the porous, on-protective scale.

3.7.5. Low Temperature (type-11) hot corrosion (LTHC)

Type-II Hot corrosion usually occurs below melting point of Na_2SO_4.the reaction product morphology is characterized by a non-uniform attack in the form of pits, with only little sulphide formation close to the scale interface and little depletion of Cr or al in the alloy substrate this form of hot corrosion is observed mainly within the temperature range 650-800 ^{0}C the localized nature of attack is related to localized failure of the scale as result of thermal cycling, erosion or chemical reactions. Microscopic sulphidation or chromium depletion is not generally observed in type-II hot corrosion.

Several metals and alloys react with air at elevated temperature, oxidation resistance must be considered in most of metallurgical engineering application. As the temperature is increased, the importance of metal oxidation also increases as in application for gas turbine, rocket engines, furnaces and high temperature petrochemical system [32]. Given below is the cross section of boiler where it is clear that boiler tubes are exposed to very high temperature.

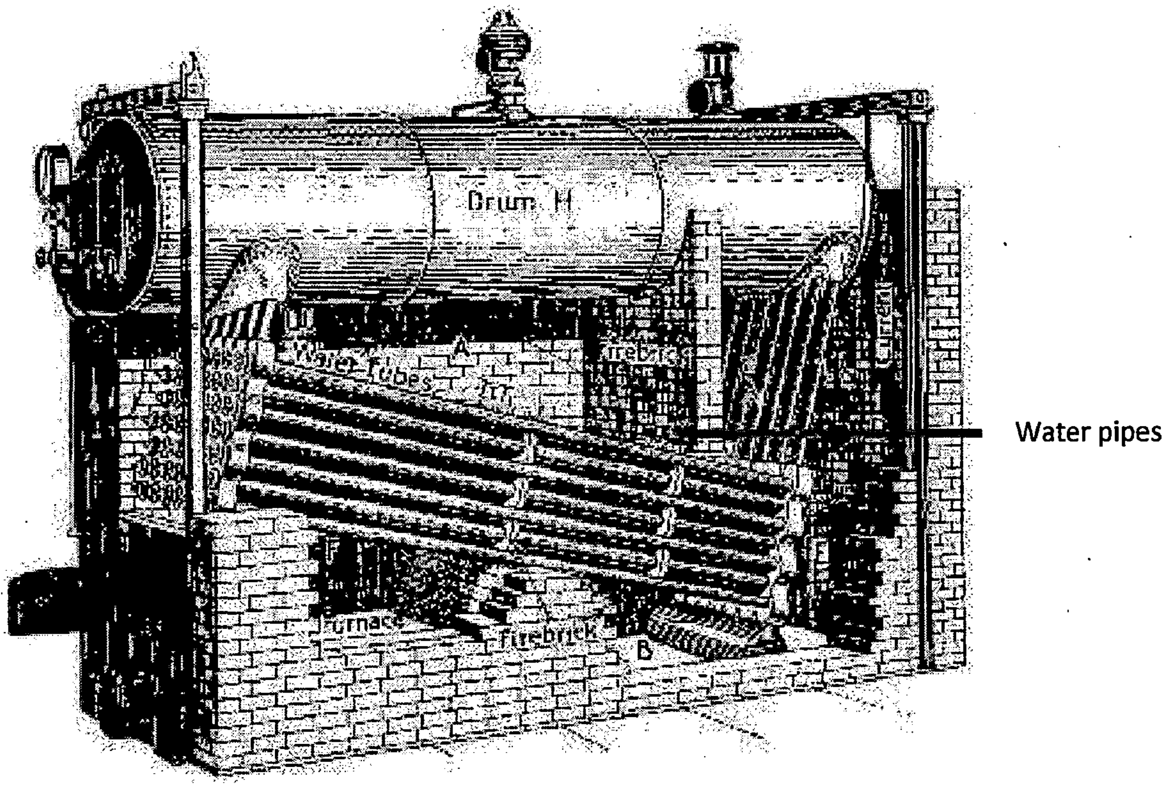


Fig 3.14: Babcock & Wilcox boiler [36].

In water tube boiler, boiler feed water flows through the tubes and enters the boiler drum. The circulated water is heated by the combustion gases and converted into steam at the vapour space in the drum. These boilers are selected when the steam demand as well as steam pressure requirements are high as in the case of process cum power boiler / power boilers. Forced, induced and balanced draft provisions help to improve combustion efficiency.

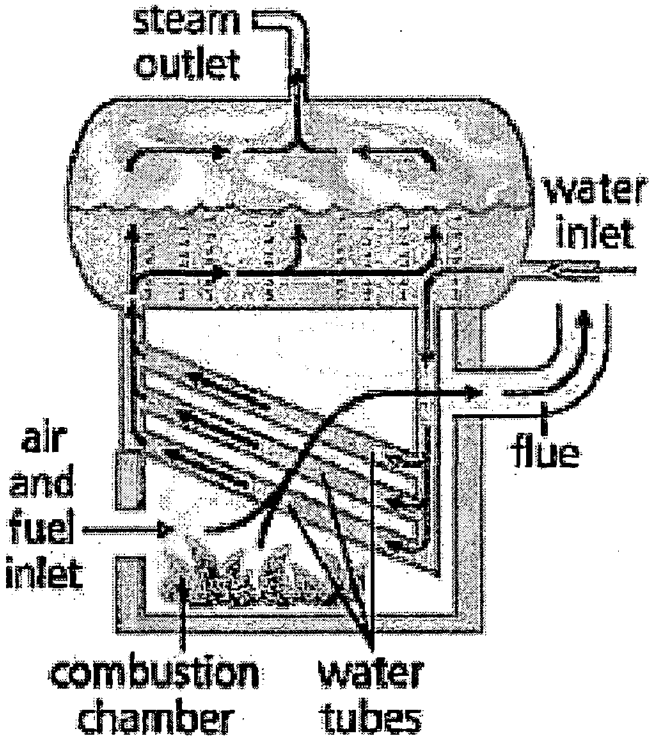


Fig .3.15: Simple Diagram of Water Tube Boiler [36].

Most modern water boiler tube designs are within the capacity range 4,500 – 120,000 kg/hour of steam, at very high pressures. Many water tube boilers nowadays are of "packaged" construction if oil and /or gas are to be used as fuel. Solid fuel fired water tube designs are available but packaged designs are less common [36].

As seen above the tubes are directly coming in contact with flame thus leading to very high temperature of tubes and at this high temperature there is hot corrosion taking place.

CHAPTER- 4

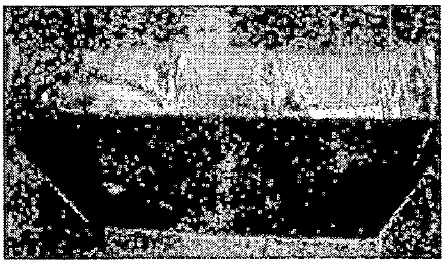
EXPERIMENTATION

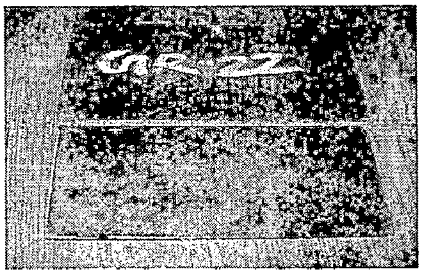
Base material of 2.25% Cr-1% Mo alloy steel of ASTM A387 Gr 22 is selected for the project. Electrodes of E9018 B_3 are selected which are low hydrogen electrodes. Electrodes are backed at 300 ^{0}C, base material is preheated at 280 ^{0}C in the furnace before use. Welding is done by choosing Double-V groove as joint design. Sectioning of the weldment is done as per the requirements for hardness, tensile, charpy impact test, microstructural study and hot Corrosion testing. The weldments are PWHT at 725 ^{0}C of holding temperature for different durations. The specimens are prepared for testing hardness, tensile strength, charpy impact toughness, microstructural study and hot corrosion testing to determine how the long duration PWHT affects the mechanical and microstructural properties of boiler and pressure vessel in thicker sections.

Experimentation Details

4.1. Base material

Base material of 2.25% Cr-1% Mo alloy steel of ASTM A387 Gr 22 is selected. Carbon content is normally less than 0.2%.Chromium provides corrosion resistance and Molybdenum increases strength at elevated temperatures. Cr-Mo alloy steels does not creep at long periods service conditions at high temperature and pressure. So these are widely used in boilers, pressure vessel, nuclear power plants and high elevated temperature applications. These steels are hardenable steels so preheating and PWHT is necessary to prevent hardening and cracking. The chromium–molybdenum alloy steel is obtained from ISGEC, Yamuna Nager for the thesis purpose in the form of plates in rolled condition.





Composition (wt %)

C	Si	Mn	P	S	Cr	Ni	Al	Cu	Mo	V	Nb	Fe
0.052	0.354	0.574	0.035	0.030	2.441	0.152	0.090	0.217	1.087	0.021	0.004	Balance

Table.4.1: composition of Base metal

The actual chemical composition of the above steel is determined with the help of Optical Emission Spectrometer (thermo jarrel Ash, TJA 181/81, U.S.) which is reported in the table.

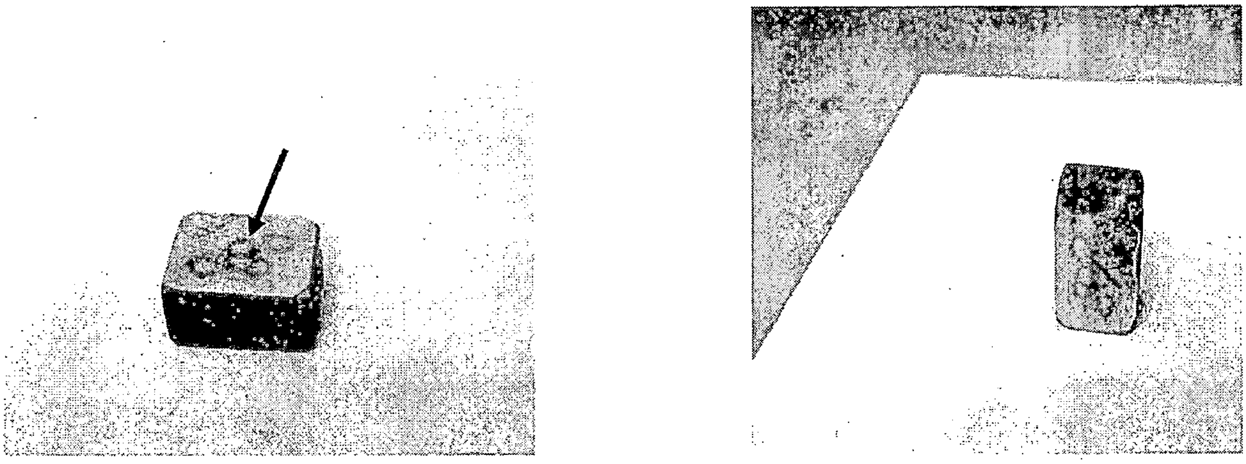


Fig.4.1: Specimen used for chemical analysis.

Physical properties of 2.25% Cr-1% Mo alloy steel

Density (×1000 kg/m^3)	7.7-8.03
Poisson's Ratio0.	27-0.30
Elastic Modulus (GPa)	190-210
Tensile Strength (MPa)	515-690
Yield Strength (MPa)	310
Elongation (%)	18
Reduction of Area (%)	45

Electrodes

Electrodes of AWS 5.5 E9018-B3 with 3.15 and 4 mm diameter are selected which are low hydrogen electrodes used commonly for pressure vessels, heat exchangers and boilers and high pressure pipe lines etc. These electrodes had been manufactured by D & H Secheron Company.

Composition (wt %)

C	Mn	Si	P	S	Cr	Mo
0.05-0.12	0.66	0.48	0.019	0.014	2.25	1.05

Table.4.2: Composition of Electrode

- Electrodes are backed at 300^0 C before use for one hour in the furnace.
- Preheat should be used on hardenable steels to prevent the formation of a hard heat-affected zone at temperature of 250 ^{0}C-300 ^{0}C.
- Preheat may also be required in welding heavy sections.

In all arc welding process it is necessary to closely match the weld metal deposit with the composition of the base metal. Filler metal of the same or slightly higher alloy content can be used for welding several Cr-Mo steels [37].

Designation of the Electrode: E9018 B3

E- MMAW Electrode manufactured from solid extrusion.

90- Indicates minimum tensile strength of 90,000 psi (650 MPa)

1-Indicates welding in all positions

8- Indicates power source (AC or DC), penetration (medium), low hydrogen, percentage of iron powder (30-50 %).

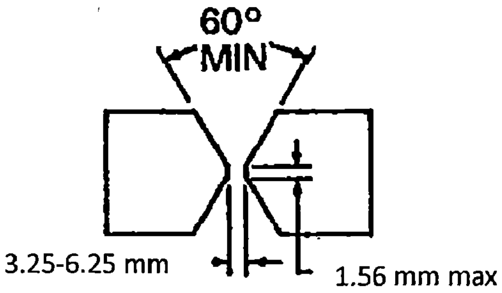
B3-Indicates the approximate alloy in the weld deposit [38].

Welding parameters

Electrode	E9018-B3
Joint geometry	double V (60^0 included angles)
Type of current& Polarity	DCEP
Preheating temperature	280 ^{0}C
Interpass temperature	340 ^{0}C
Electrode diameter for Root passes	3.15 mm
Welding current	100-140 A
Arc voltage	20 V
Electrode diameter for Filler passes	4.0 mm
Welding current (I)	150-190 A
Arc voltage (V)	25 V

Joint design

Double V Grove joint is made on both of the plates. Because the material is very thick these double V groove can prevent distortion due to the presence of less weldmetal.The design of the groove is as per ASTM standard.



All dimensions in mm

Fig.4.2: Double v groove joint design [27].

The double sided V-groove offer's 50% decrease in weld volume (and an implied 50% decrease in welding costs). Along with the decreased quantity of weld metal comes a corresponding decrease in residual stress and longitudinal shrinkage. Balancing the quantity of weld metal about the centreline of the plate can reduce angular distortion. If it is assumed that the probability of weld defects is proportional to the volume of weld metal deposited, reduced volume groove weld details should also result in higher quality weld deposits. Less residual stress, less distortion, higher quality and lower fabrication costs, all these are achievable with the use of double sided groove welds [39].

4.2. Preheating

Cr-Mo alloy steels are hardenable steels so preheating is necessary to prevent hardening and cracking. For Base material preheating temperature is selected as 280 ^{0}C as per ASTM standards [40].

Preheating is especially necessary in thicker carbon steel plates in order to guard against the high thermal conductivity of steel when the heat of the welding arc is absorbed rapidly by the metal adjacent to the weld. The rate of cooling is slowed by preheating and the hardness is decreased. This slowing of the cooling rate allows more time for metal transformation to a more favorable structure, minimizing hard zones and cracks; thus preheating retards the formation of undesirable metallurgical structures in metals having high hardness and susceptibility to cracking. Preheating dries up moisture, reducing the incidence of hydrogen and oxygen accumulation in a weld zone; consequently, it minimizes porosity, embrittlement, and cracking in the weld and heat-affected zone.

Preheating the metal before welding has proved a dependable method of securing a sound weld. Preheating helps equalize and distribute the stresses evenly by distributing the expansion and contraction forces over an enlarged area to which the stress differential is applied due to the heat of welding [24].

4.3. Welding process selected

Shielded metal arc welding

SMAW is a commonly used welding process which is most simple, flexible .This is a process that melts and joins metals by heating them with an arc established between a stick like covered electrode and the metals. It is often called stick welding. The electrode holder is connected through a welding cable to one terminal of the power source and the work piece is connected through a second cable to the other terminal of the power source the core of the covered electrode, the core wire, conducts the electric current to the arc and provides filler metal for the joint. The electrode holder is essentially a metal clamp with an electrically insulated outside shell for the welder to hold safely.

The heat of the arc causes both the core wire and the flux covering at the electrode tip to melt off as droplets. The molten metal collects in the weld pool and solidifies into the weld metal. The lighter molten flux, on the other hand, floats on the pool surface and solidifies into a slag layer at the top of the weld metal.

The welding equipment is relatively simple, portable, and inexpensive as compared to other arc welding processes. For this reason, SMAW is often used for maintenance, repair, and field construction. However, the gas shield in SMAW is not clean enough for reactive metals such as aluminum and titanium. The deposition rate is limited by the fact that the electrode covering tends to overheat and fall off when excessively high welding currents are used. The limited length of the electrode (about 35 cm) requires electrode changing, and this further reduces the overall production rate [41].

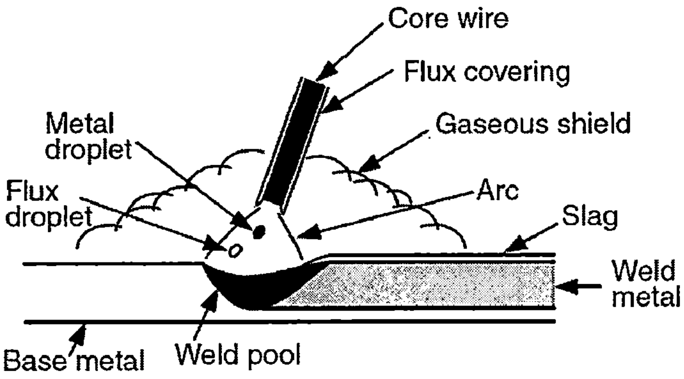


Fig.4.3. Schematic SMAW process [41].

4.4. Post Weld Heat Treatment

Cr-Mo alloy steels are hardenable steels so PWHT is necessary. Pressure vessels used in acid or caustic services must and should undergo PWHT in order to increase the corrosion resistance. The process of post weld heat treatment consists of uniform heating of a vessel or part of a vessel to a suitable temperature for the material below the critical range of the base metal, followed by uniform cooling. This process is used to release the locked-up stresses in a structure or weld in order to "stress-relieve" it. Because of the great ductility of steel at high temperatures, usually above 593 °C, heating the material to such a temperature permits the stresses caused by deformation or straining of the metal to be released.

Thus post weld heat treatment provides more ductility in the weld metal and a lowering of hardness in the heat-affected zone (HAZ). It also improves the resistance to corrosion and caustic Embrittlement. The proper heat treatment before and after fabrication is one that renews the material as near as possible to its original state. The requirement for post weld heat treatment is largely a function of the material and the thickness. The material (in terms of alloy content) and the thickness (in relation to the quench effect) control the microstructure that will be formed. Large section thicknesses in alloy steels can result in Martensitic, Pearlitic or Bainitic structures, depending on the cooling rate, the thicker the material that is welded, the greater the amount of residual stress that will be developed on cooling [41].

Due to the advantages mention above PWHT is selected as a tool to improve mechanical, microstructural and corrosion resistance properties of the boiler and pressure vessel steel. The steel plates are cut as per the requirements using band saw, welded using SMAW process and specimens are collected for tensile testing, hardness measurement,charpy-v-notch test, microstructural examination ,chemical analysis ,and hot corrosion studies, as per the dimensions mentioned in ASTM standards.

The heat treatment process of full annealing is selected with an holding temperature of 725 ^{0}C, as per the ASTM standards mentioned for Cr-Mo alloy steels. The welded specimens were heat treated in an electric resistance furnace with programmable control of temperature. The specimens were maintained at this temperature for continuous time periods of 0.5, 2, 10 and 50 hours of soaking time and cooled within the furnace up to the room temperature.
Holding temperature: 725 ^{0}C

Heat Treatment durations

SL.NO	1	2	3	4
TIME IN HOURS	0.5	2	10	50

Table.4.3: Heat Treatment durations selected

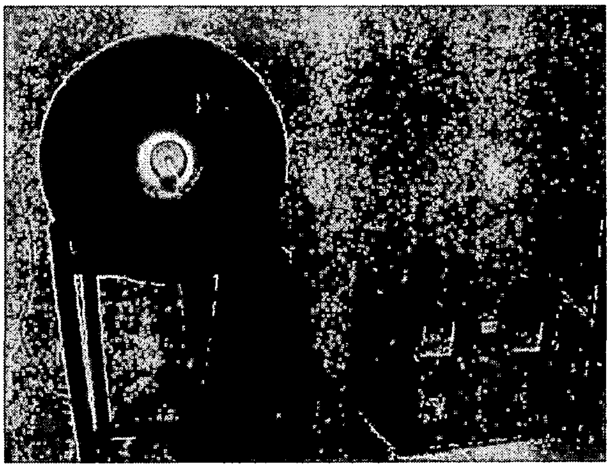


Fig.4.4: Furnace used for heat treatment process.

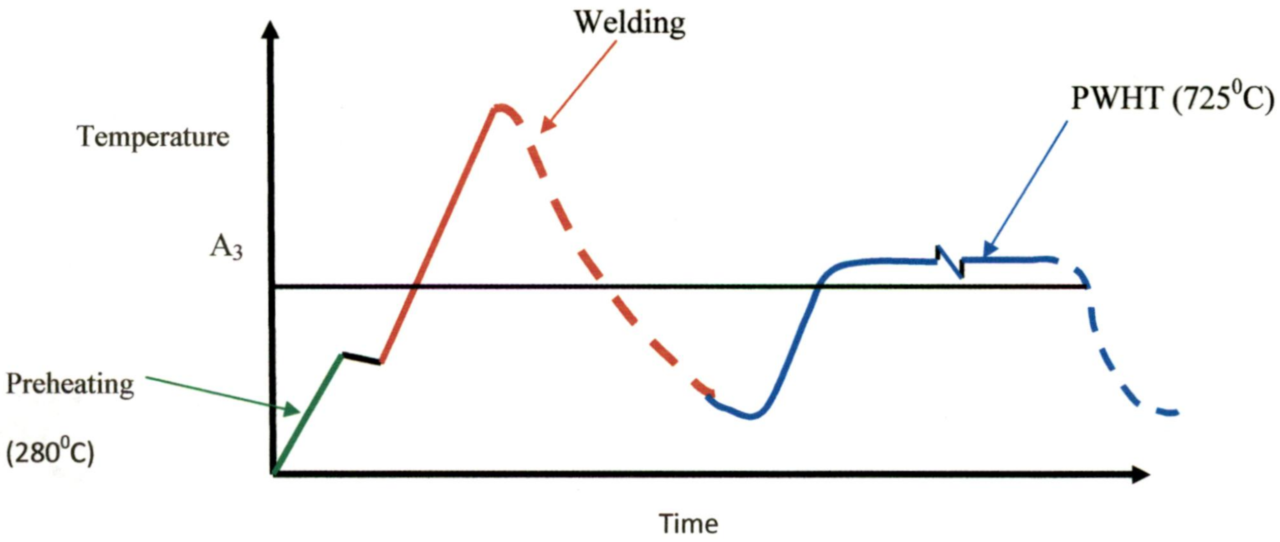


Fig.4.5: Schematic representation of Preheating, Welding and PWHT.

4.5. Collection of specimens from Weld Joints

Sectioning of the weld joints for fabrication of various test specimens was made with the help of band saw. The collection of specimens for mechanical testing, metallography and hot corrosion test from different locations of the weld joint has been schematically shown in **Fig.**4.6.

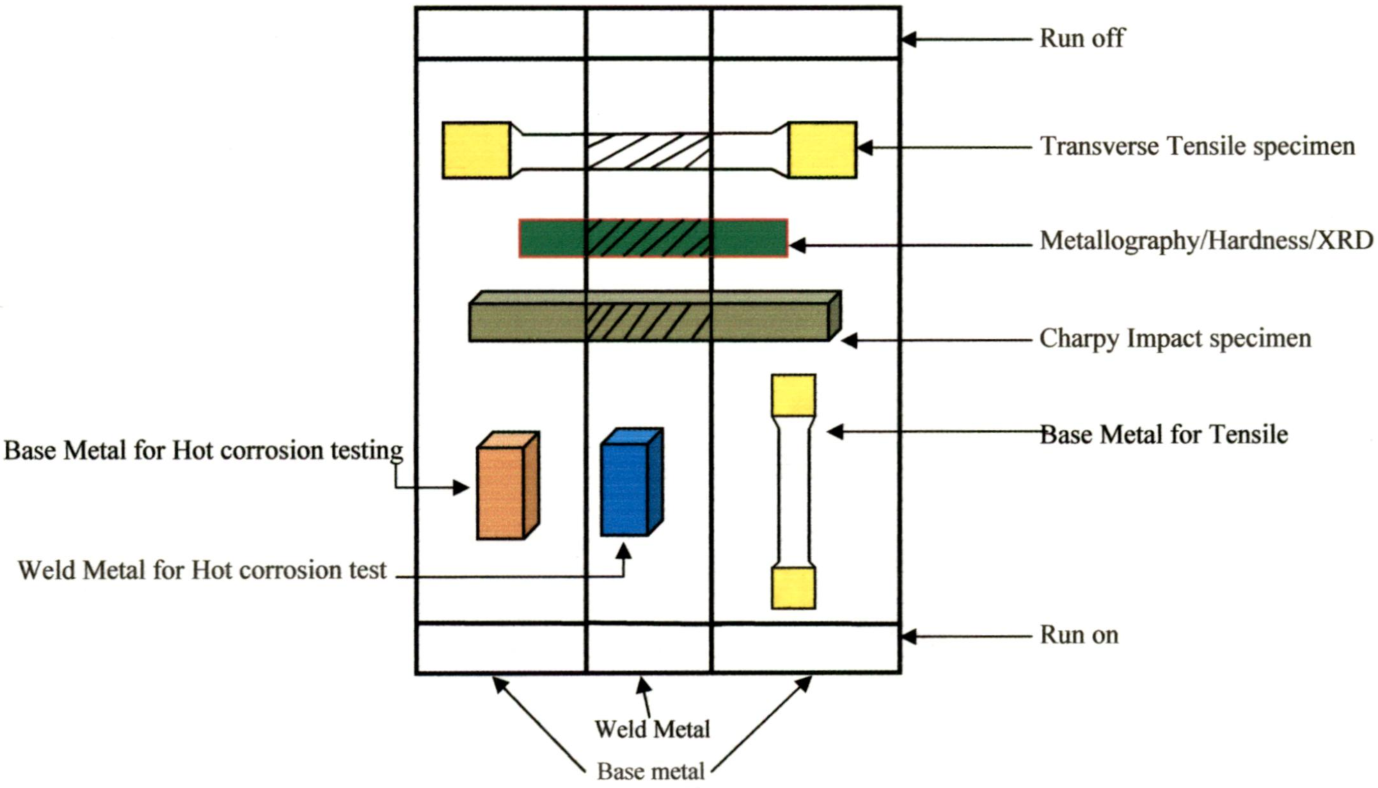


Fig.4.6: Collection of samples from weld joints.

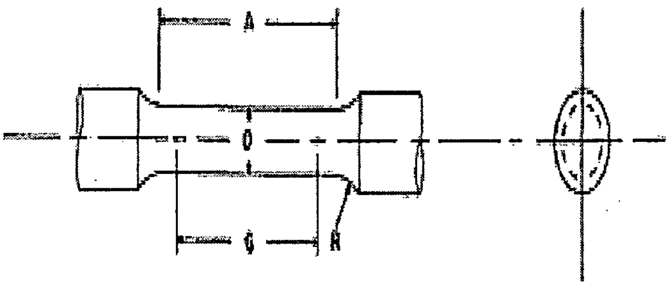
4.6. Testing of mechanical properties

The weld joints are prepared using the SMAW process, after the collection of material is done as shown in above **Fig.**4.6 as per the required dimensions. The specimens for testing's are sectioned using Band saw, undergone heat treatments for different soaking times like 0.5, 2, 10 and 50 hours. After PWHT specimen preparation is done as per ASTM standards.

4.6.1. Tensile test

Tensile properties of the base metal and weld joint ,with respect to their ultimate tensile strength(UTS), yield strength(YS), percentage area of reduction(%RA) and percentage of elongation were studied by using round tensile test specimens as per ASTM E8M [42] standard.

Tensile properties of weld joint were studied by using specimens from axial weld (weld at centre) the axial weld specimens are prepared by keeping the weld at centre of the gage length. The test is carried out on 6 tonn servo-hydraulic microprocessor controlled universal testing machine. The graph between stress and strain, yield strength, ultimate strength has been recorded. The percentage elongation in length and percentage reduction in area has been evaluated separately.



Dimensions of the specimen are:-

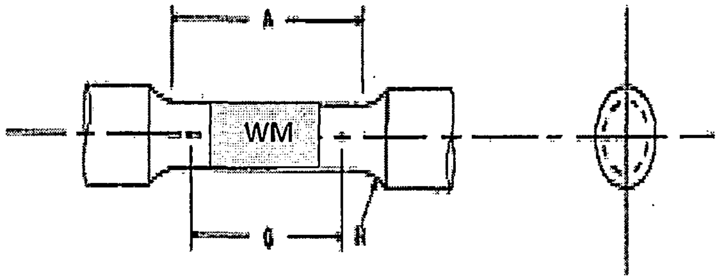
A-length of reduced section: 45 mm

G-gage length: 36 mm

D-diameter: 9 mm

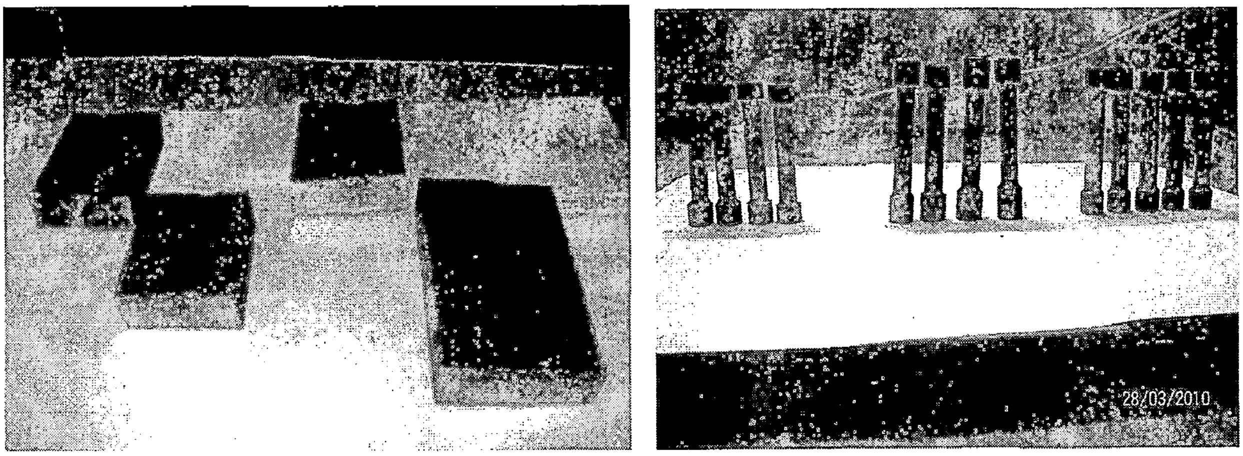
R-radius of fillet: 6.25 min

Tensile Base metal specimen



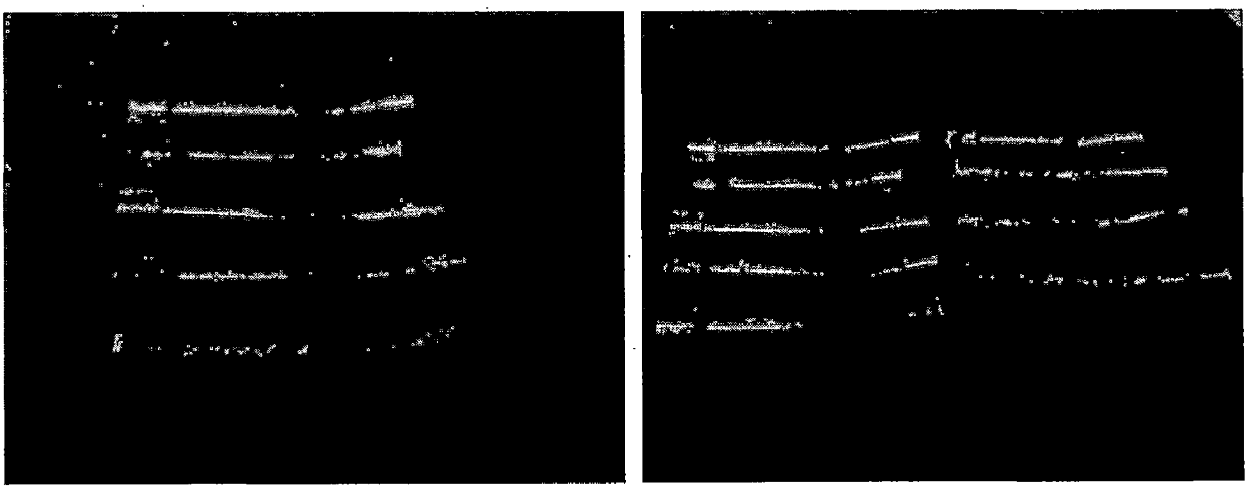
Transverse Tensile Specimen

Fig.4.7: Dimensions of tensile specimens selected [42].



Weld joints used for tensile testing

Tensile testing specimens



Specimens after testing

Fig.4.8: Tensile specimens before and after testing.

4.6.2. Hardness testing

Hardness test is conducted on the as welded and PWHT conditions. Samples of 15 mm width and 60 mm length were cut from the heat-treated material and are polished for measurement of hardness. Specimens having all the three portions of a weldment viz.weld metal, HAZ and base metal was tested using micro-hardness tester. The hardness is taken from the centre line toward base metal.

Parameters selected for hardness testing are:

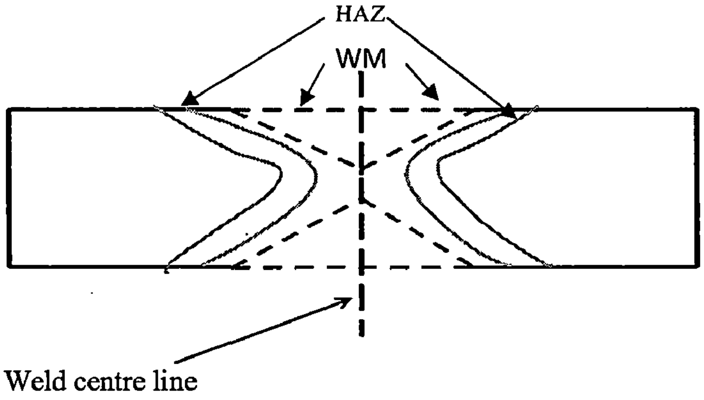
Hardness type: Micro hardness

Load: 100 gms

Dwell time: 30 seconds

Apparatus: LEITZ WETZLAR, GERMANY

The hardness measurements points are taken at a distance of 1 mm in weld metal, base metal and 0.5 mm in HAZ.



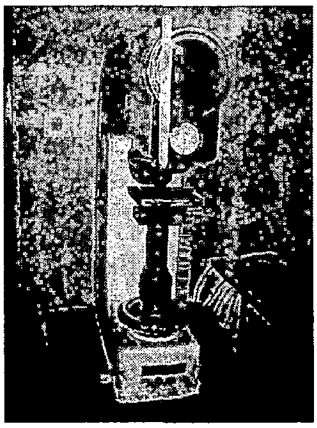


Fig.4.9: Hardness testing machine.

4.6.3. Charpy –V-Notch impact Testing

The standard charpy V-notch specimen were taken in transverse manner. The notch was made in transverse direction at the weld centre, so that the cracking plane lies along the direction of welding. Specimens were tested in as welded and PWHT conditions at higher temperature like 60 ^{0}C,120 ^{0}C and at sub zero temperatures like -90 ^{0}C,-60 ^{0}C and 0 ^{0}C. The material available does not permit making the standard impact test specimens so sub size specimens are prepared as per the dimensions mentioned in ASTM E23 standard [43].

The specifications of impact testing machine are:
Pendulum velocity: 5.4 meter per sec
Range: 0-300 joule
Least count: 2 joule
Specimen size: 55×5×5 mm

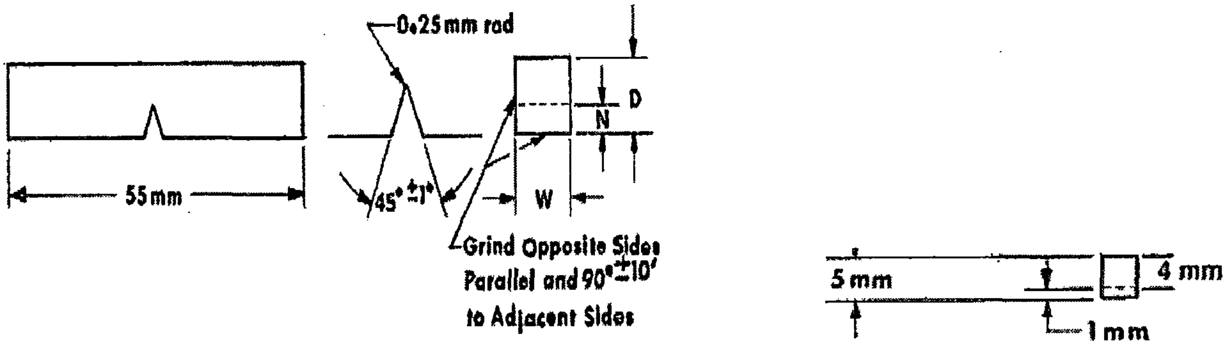
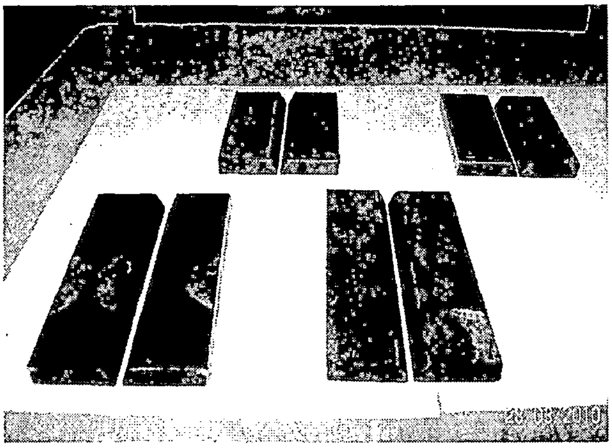
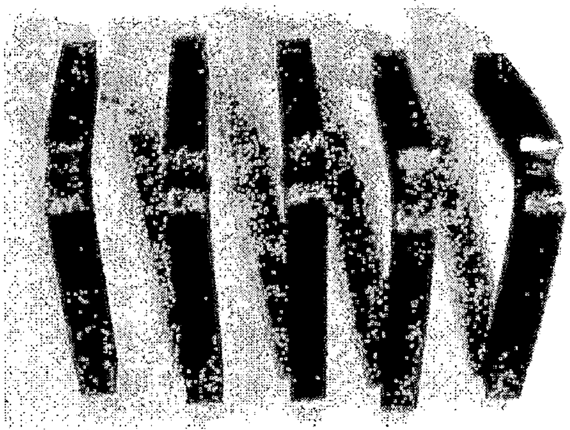


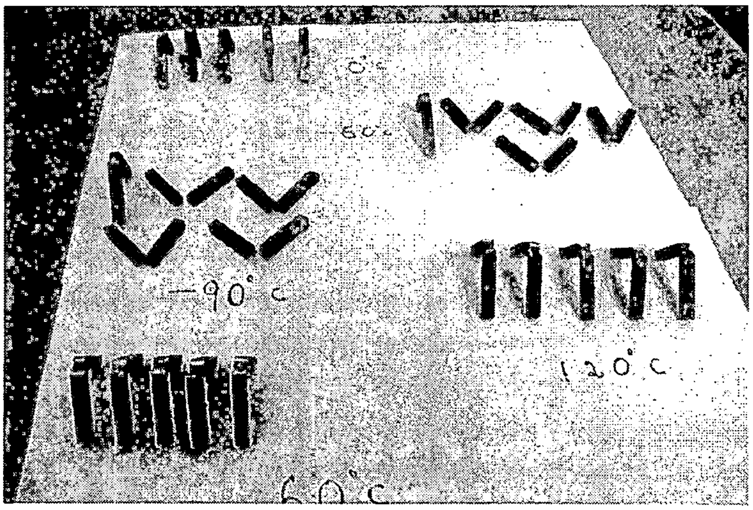
Fig.4.10: Dimensions of the Charpy-V-notch specimen [43].



Specimens used for charpy test

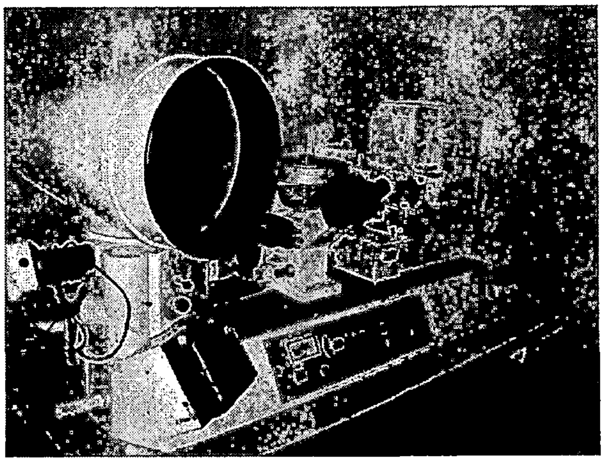


Specimens tested at different temperatures



4.7. Microstructural Examination

Microstructural examination is done on as welded and PWHT conditions. Samples of 15mm width and 60mm length were cut transverse to the weld for microscopic examination. The samples were polished and etched with 10 % Nital solution for 10-20 seconds in order to check the different locations in weld joint like weld metal ,HAZ and base metal.



Sample preparation

1-Paper Polishing

-220 Grit SiC paper

-400 Grit SiC paper

-600 Grit SiC paper

-1000 Grit SiC paper

(This step may be repeated 2-3 times)

2-Cloth polishing-

Soft Cloth allows the greatest shock absorbency and therefore allow for gentle polishing with little damage. Soft cloths allow the abrasives to abrade.

Soft cloth-

- a superior scratch removal
- risk generating surface ‘relief’.

Using Alumina (Aluminum oxide) Al_2O_3 as a paste on cotton cloth.

4.7.1. Etching

Etching delineates the grain structure. Etching may attack a second phase preferentially or attack grain boundaries excessively. Caution should be taken when choosing and using etchants. Impact the sample surface using a light microscope before and after etching to asses' effect. Materials that are difficult to polish may benefit from repeated etching and polishing.

After preparing samples, microstructural study had been done using optical microscopy and Scanning Electron Microscope (SEM) for getting microstructure of all processed samples. The microstructural study is focused on weld metal, HAZ and base metal. Briefly studied the microstructures at different magnifications like 100x, 250x, 500x and 1000x. Observed the microstructural changes taking place by the effect of PWHT in weld metal, HAZ and base metal.

4.8. Hot Corrosion study

Hot corrosion tests are conducted on 2.25% Cr-1% Mo alloy steel of ASTM A387 Gr 22, which are commonly used as boiler tube material in super heater, reheater and other parts especially when the service conditions are stringent from point of temperature and pressure. These materials are also used in pressure vessels, piping in the chemical industry, baffle plates/tubes in the fertilizer plants. The material is received from ISGEC, Yemuna nager in the form of plates in rolled condition. The composition is mentioned in initial pages. The hot corrosion test was conducted on Un- Weld sample, weld metal and base metal in order to study the corrosion effect on weld and un-weld condition.

4.8.1. Preparation of Specimens for Testing

Specimens with dimensions of approximately 20 mm × 15 mm × 5 mm were cut from the plates and polished using emery paper of 200, 400, 600, 800 and 1000 grit sizes and mirror polished using cloth polishing wheel machine with 1μm lavigated alumina powder suspension. The specimens were prepared manually and all care was taken to avoid any structural changes in the specimens.

4.9. Experimental procedure

After sample preparation, samples were placed on the ceramic boat. The experimental setup used for one has been shown in the following figure. Hot corrosion studies were conducted at 900 ^{0}C in laboratory silicon carbide tube furnace. The furnace was calibrated to an accuracy of ± 5 ^{0}C using platinum –rhodium thermocouple fitted with a temperature indicator. The physical dimensions of the specimens were recorded carefully with sylvac digital vernier caliper to evaluate their surface areas.

The specimens were washed properly with acetone and dried in hot air to remove moisture. During experimentation, the prepared samples were kept in an alumina boat and the weight of boat and specimen was measured. The alumina boats used for the studies were pre-heated at a constant temperature of 120^{0}C for 3 hours, and it is assumed that their weight would remain constant during the course of high temperature corrosion study.

Cyclic oxidation studies were performed in molten salt (Na_2SO_4–60% V_2O_5) and air for 50 cycles, with each cycle consisting of 1 hr heating at 900 ^{0}C, followed by 20 min cooling at room temperature. The aim of cyclic loading is to create severe Boiler environment conditions for testing. The hot corrosion tests are conducted in air and salt environment at 900 ^{0}C.The salt is coated uniform thickness of 3–5 mg/cm2 of Na_2SO_4–60% V_2O_5 was applied with a camel hairbrush on preheated samples (250 ^{0}C).

The boat containing the specimen was inserted in to hot zone of the furnace maintained at temperature of 900 ^{0}C. The weight of the boat loaded with the specimen was measured after each cycle during of the corrosion test. Weight-change measurements were taken at the end of each cycle using an electronic balance (model 06120) with a sensitivity of 1 mg. At the time of weighing, even the spalled scale was also included to determine the total rate of corrosion.

The specimens were subjected to visual observations carefully after the end of each cycle with respect to color, spalling and peeling of scale during cyclic corrosion. The above experiments were conducted for 50 cycles. The samples after corrosion were examined by characterization of SEM, EDAX, X-Ray Mapping and XRD analysis.

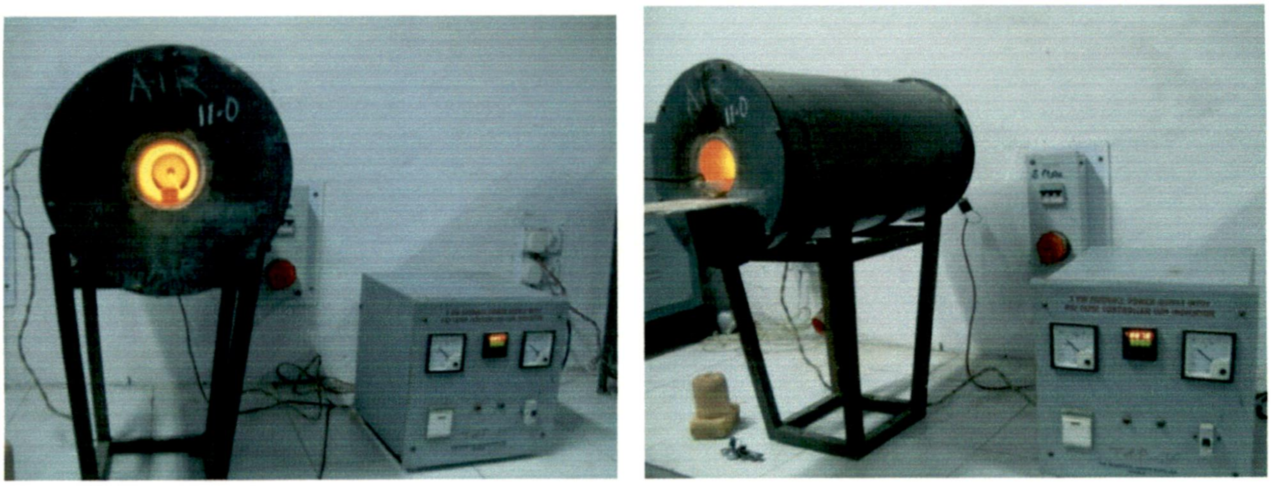


Fig.4.12: Tubular furnace used for corrosion test.

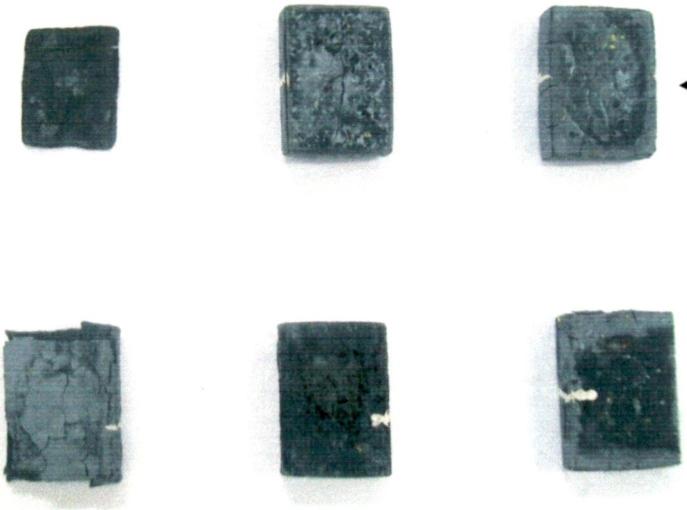
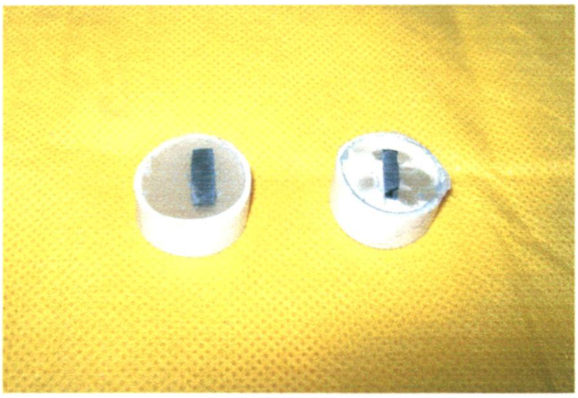


Fig.4.13: Samples after hot corrosion test from left un-weld sample, base metal and weld metal.

Ceramic boats containing samples.



Specimens used for cross-sectional analysis.

4.10. Analysis of Hot Corrosion test

All the specimens subjected to hot corrosion were analyzed for the characterization of corrosion products. The surface and cross-section of the corroded specimens were analyzed by using various analytical techniques such as XRD, SEM, E-DAX, and X-Ray Mapping analysis.

4.10.1. Visual observations

After every hot corrosion cycle surface of the specimen was visually examined for change in colour, luster, spalling tendency, and growth of cracks. The spalled scale was also included to determine the total rate of corrosion.

4.10.2. Weight change studies

The weight change values were measured at the end of each cycle with the aim to establish the kinetics of oxidation and hot corrosion. The weight change data was plotted with respect to number of cycles. The weight change is due to formation of the oxide scales, spalling and sputtering etc.

4.10.3. SEM/E-DAX

The surface morphology and chemical composition of the hot corroded specimen surfaces after 50 hours were studied with the help of field emission scanning electron microscope (FE-SEM) (FEI Quanta 200 F, made in Czech Republic).

4.10.4. X-Ray Diffraction (XRD) Analysis

For identification of different phases formed in the oxide scales of hot corroded specimens after 50 cycles X-Ray Diffractometer has been used.

4.10.5. Cross-sectional analysis

Cross-sectional analysis is done to find the elements, oxides and oxide layer thickness on the corroded sample, using X-Mapping and E-DAX analysis.

CHAPER-5

RESULTS AND DISCUSSIONS

This chapter presents the results of the various experiments described in the chapter 3 and demonstrate the analysis in order to understand the influence of PWHT in improving the mechanical and microstructural properties and providing resistance to hot corrosion. In these chapter results regarding microstructures, tensile testing, hardness measurement, charpy impact test and hot corrosion behaviour of boiler steel are reported.

5.1. Microstructural Examination

Microstructural study is focused on Weld metal, HAZ, Base metal and Fusion boundary using Optical Microscope and Scanning Electron Microscope (SEM).

5.1.1. Weld Metal Microstructures

Weld metal in As-Welded condition

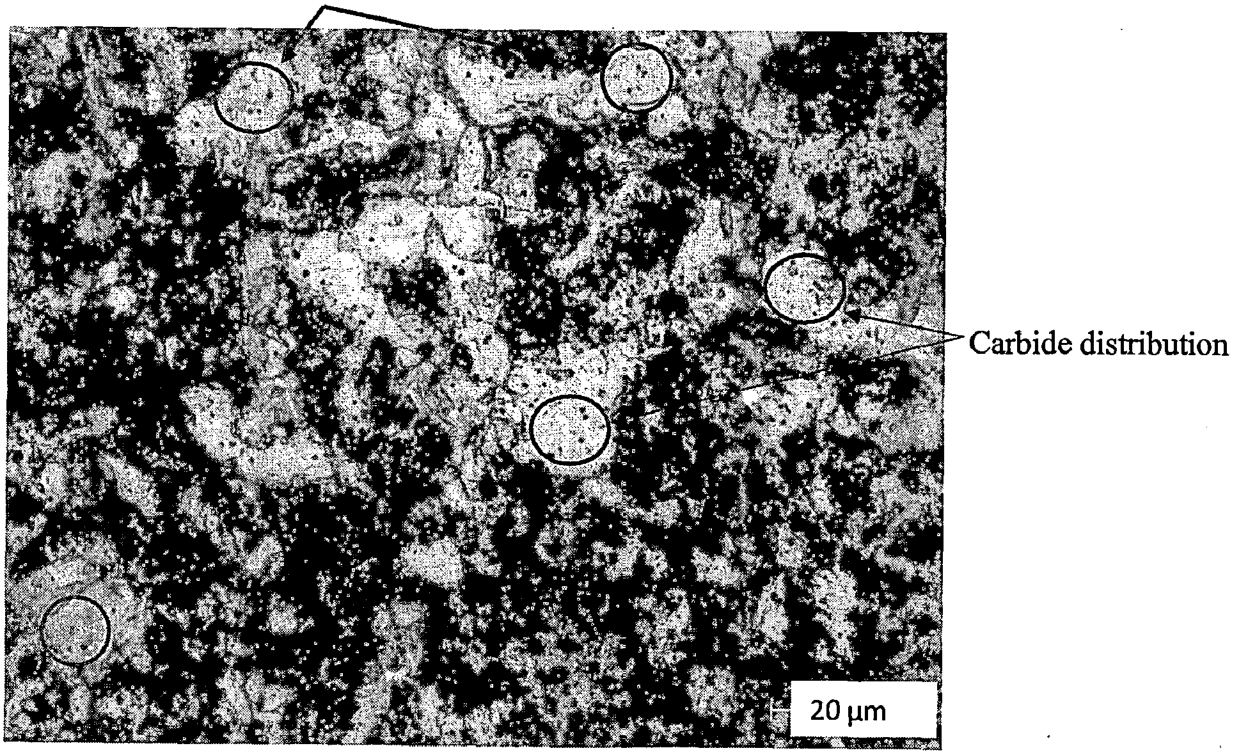


Fig.5.1: Lath bainite with fine distribution of carbides 1000x

The weld metal in as welded condition shows lath bainite with fine distribution of carbides in the ferrite grains. Due to the presence of carbides the micro hardness in this region is very high (300 HV). M.C. Murphy [44] Reported 2.25Cr-1Mo steel derives its creep strength from the fine distribution of acicular MO_2C particles in the weld regions so there may be chance of MO_2C carbide precipitation in weld metal.

Weld metal undergone 0.5 h PWHT

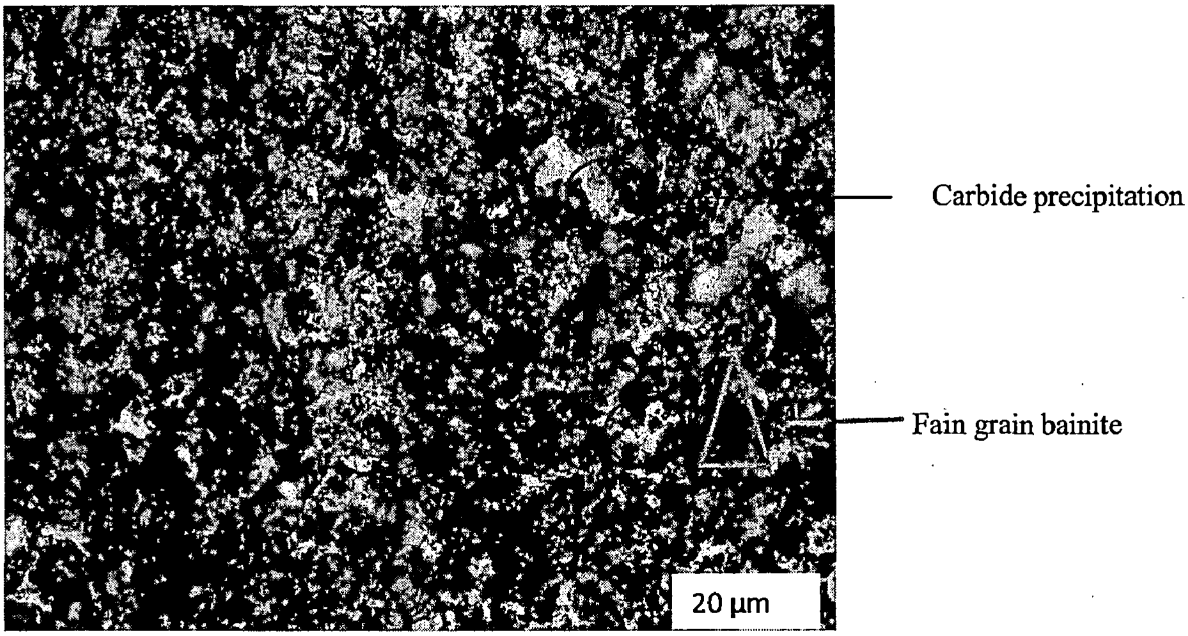


Fig.5.2: Fine grain bainite with small amount of carbide precipitation, 1000x

Weld metal undergone 2.0 h PWHT

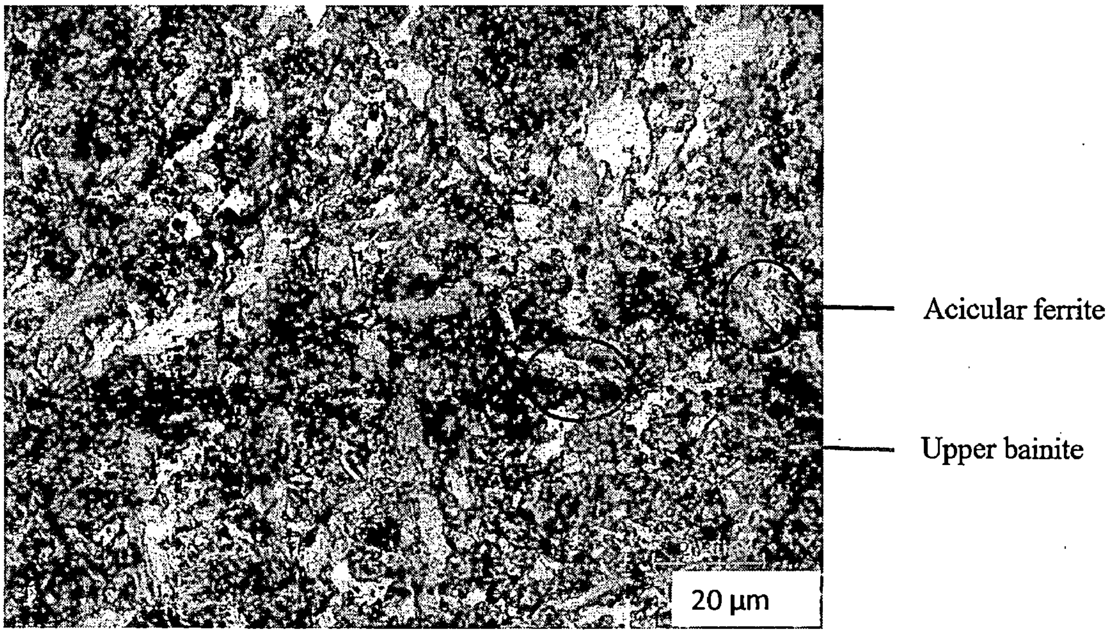


Fig.5.3: Upper bainite and acicular ferrite with dissolution of carbides, 1000x

In 0.5 PWHT the carbides which are generated during welding are not completely diffused. There is a slight reduction in hardness from 300-289 VHN. The upper bainite is identified by a lath like arrangement of ferrite. In 2.0 h of PWHT the carbides are fully diffused, so there is more hardness drop had occurred. This is conformed by the micro hardness testing.

Weld metal undergone 50 h PWHT

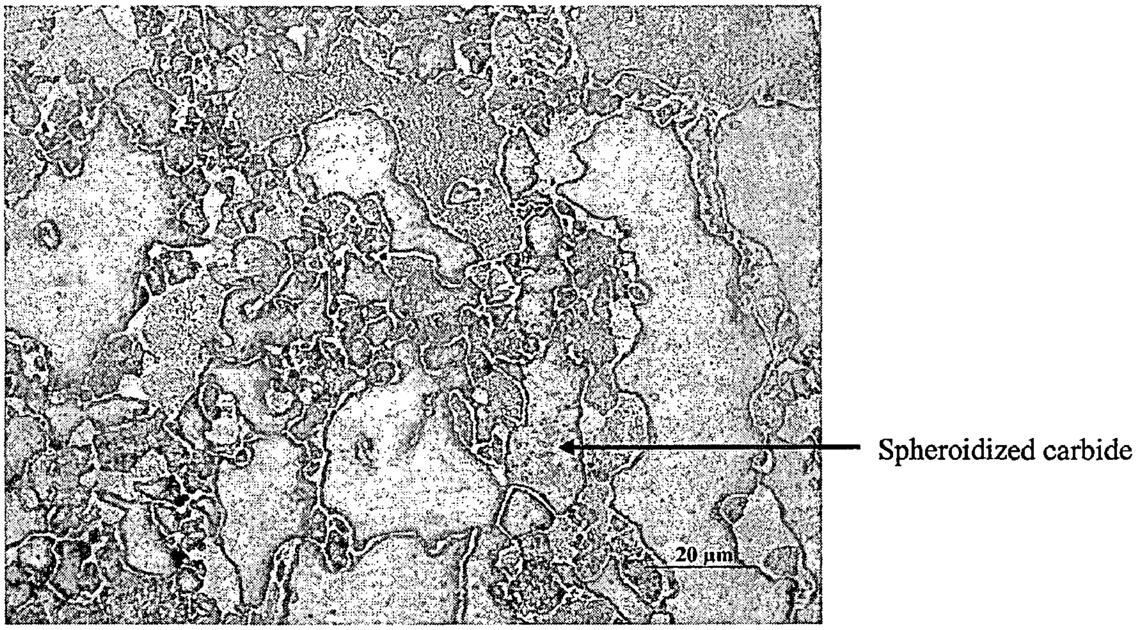


Fig.5.4: Microstructure showing spheroidized carbides and coarse dendrites, 1000x

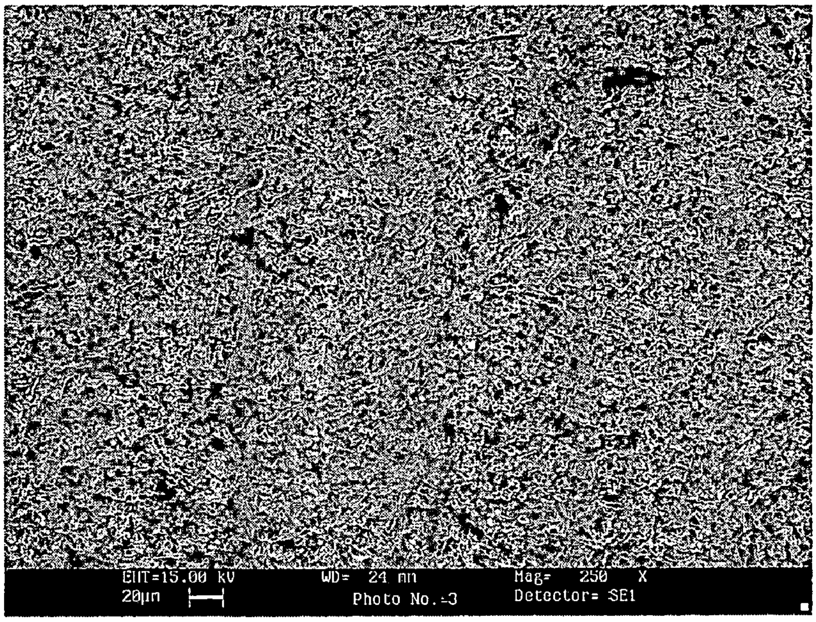


Fig.5.5: SEM microstructure showing dendrites

This specimen had undergone PWHT of 50 h at high temperature of 725^{0}C, so chances of formation of spheroidization of lath boundary carbides. From the SEM microstructure it is conformed that dendrites are present in the weld metal.

5.1.2. HAZ Microstructures

HAZ in As-Welded condition

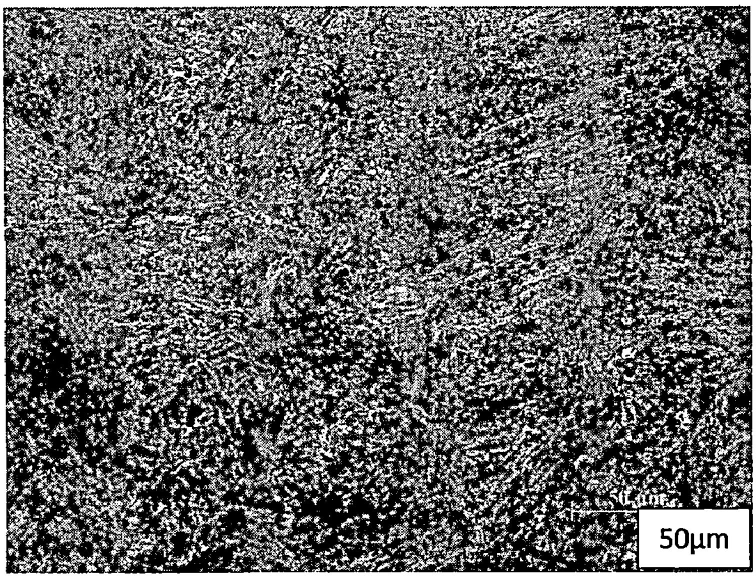


Fig.5.6: Lath martensite, 500x [45].

In As welded condition lath martensite is formed in HAZ, which is having high hardness. Chances for formation of lath martensite are due to high carbon percentage and rapid cooling. In Cr-Mo alloy steels carbon is very less (0.05 %), so rapid cooling may be the reason for the formation of lath martensite.

HAZ in 0.5 h PWHT

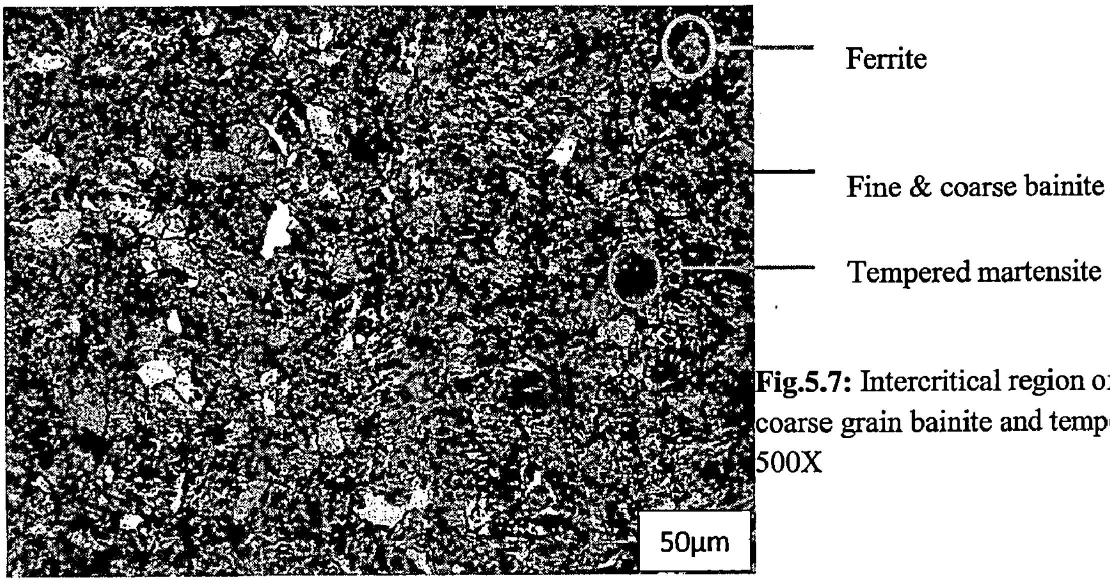


Fig.5.7: Intercritical region of ferrite with fine & coarse grain bainite and tempered martensite, 500X

Lath martensite which is formed in as welded condition is tempered in 0.5 h heat treatment and ferritic structure is formed with fine and coarse bainite. This intercritical region is having less hardness compared to as welded condition.

HAZ in 2.0 h PWHT

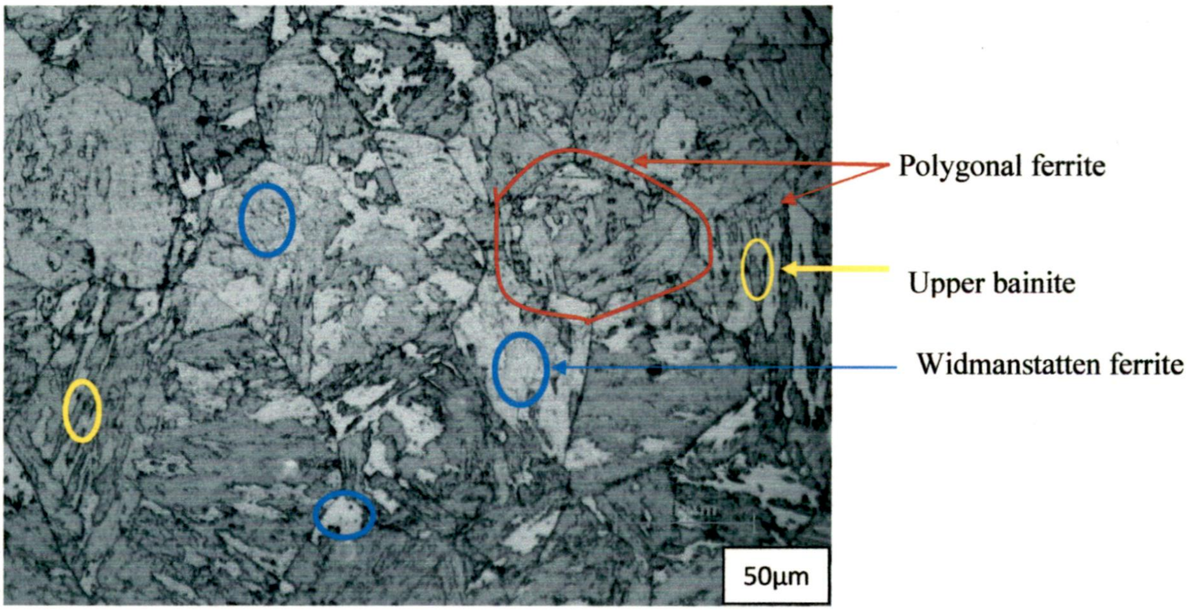


Fig.5.8: Mixed polygonal/Widmanstatten/upper bainitic structure, 500x

Specimen under gone 2.0h heat treatment has shown a mixed polygonal/ Widmanstatten/ upper bainitic structure. This structure is very coarse, so there is sharp drop in hardness, when compared to all PWHT'S.

HAZ in 10 h PWHT

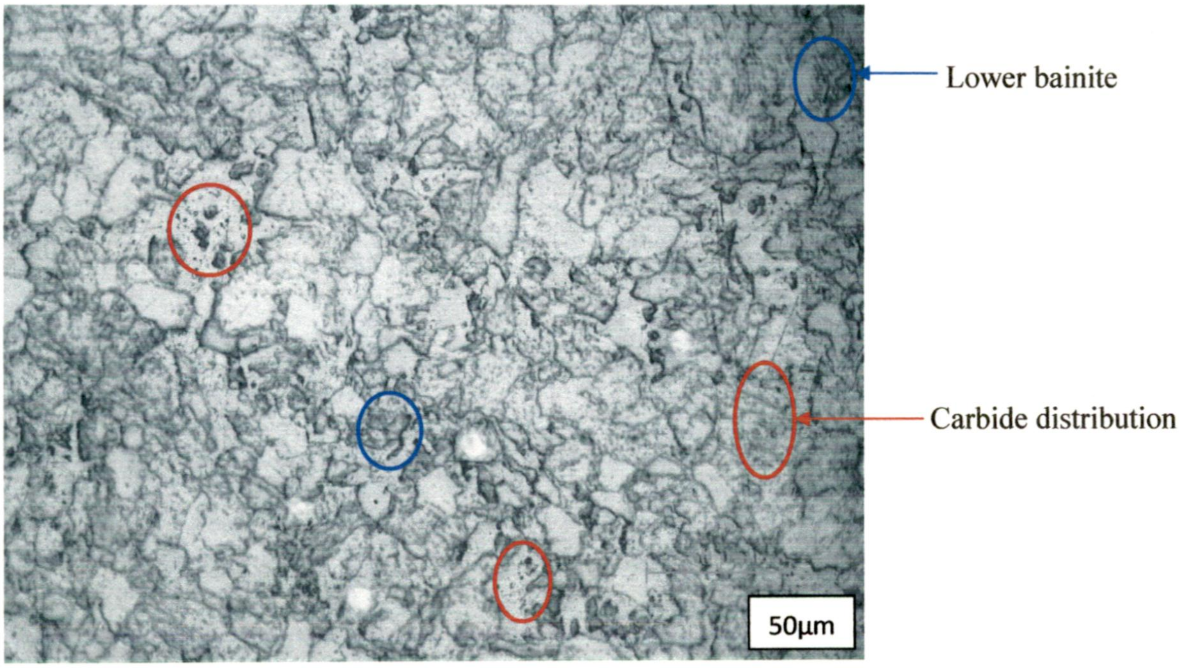


Fig.5.9: Fine dispersion of alloy carbides in a ferrite matrix and formation of lower bainite 500x [45].

Specimen heat treated at 10 h shown fine distribution of carbides in a ferrite matrix and lower bainite is formed in the structure, so their is increase in hardness which is slightly less than as welded condition.

HAZ in 50 h PWHT

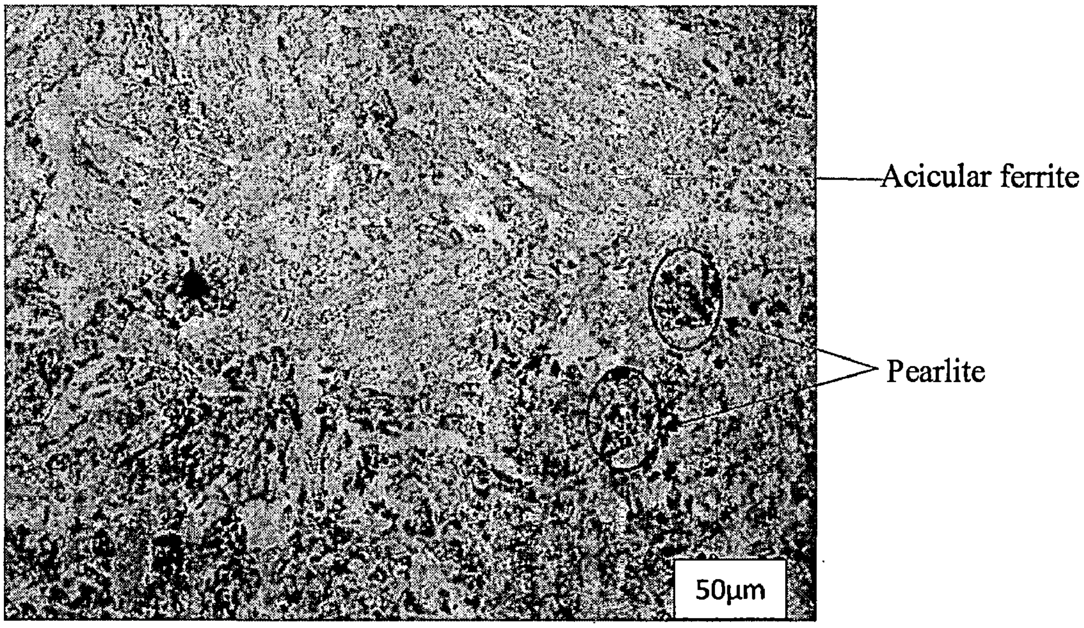


Fig.5.10: Microstructure contains pearlite and acicular ferrite, 500 x

5.1.3. Base Metal Microstructures

Base metal in as welded condition

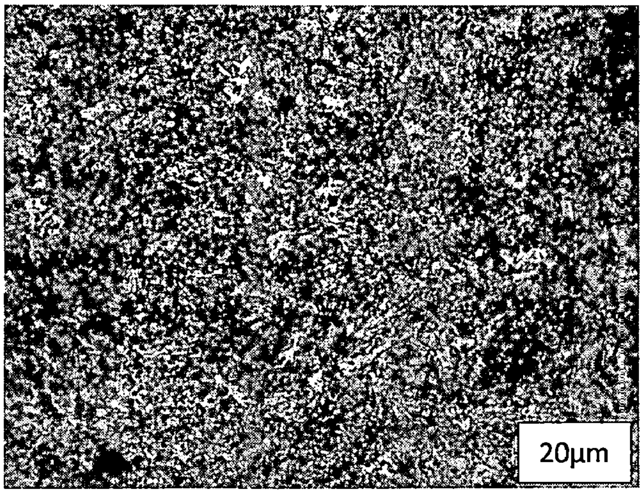


Fig.5.11: Carbide precipitation 1000, x

base metal in 0.5 h PWHT

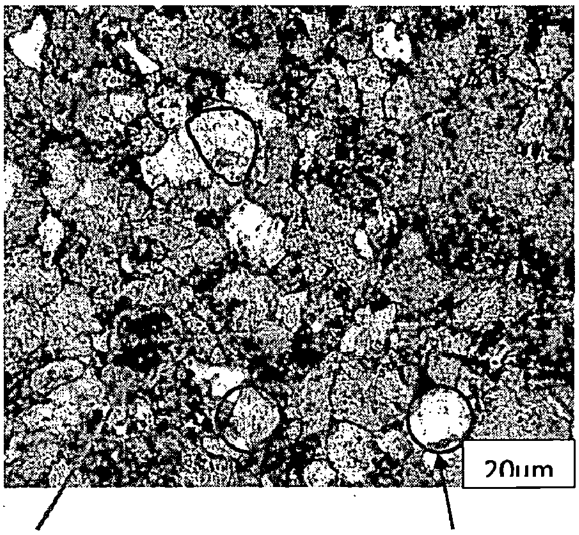


Fig.5.12: pearlite contains some widmanstatten plates of ferrite, 1000x [45].

In base metal as welded condition the ferrite matrix consist of extensive uniformly dispersed carbides were found. After heat treatment of 0.5 h the carbides were diffused and widmanstatten plates of ferrite are observed in between pearlite grains. Here grain coarsening was occurred.

Base metal in 2.0 h PWHT

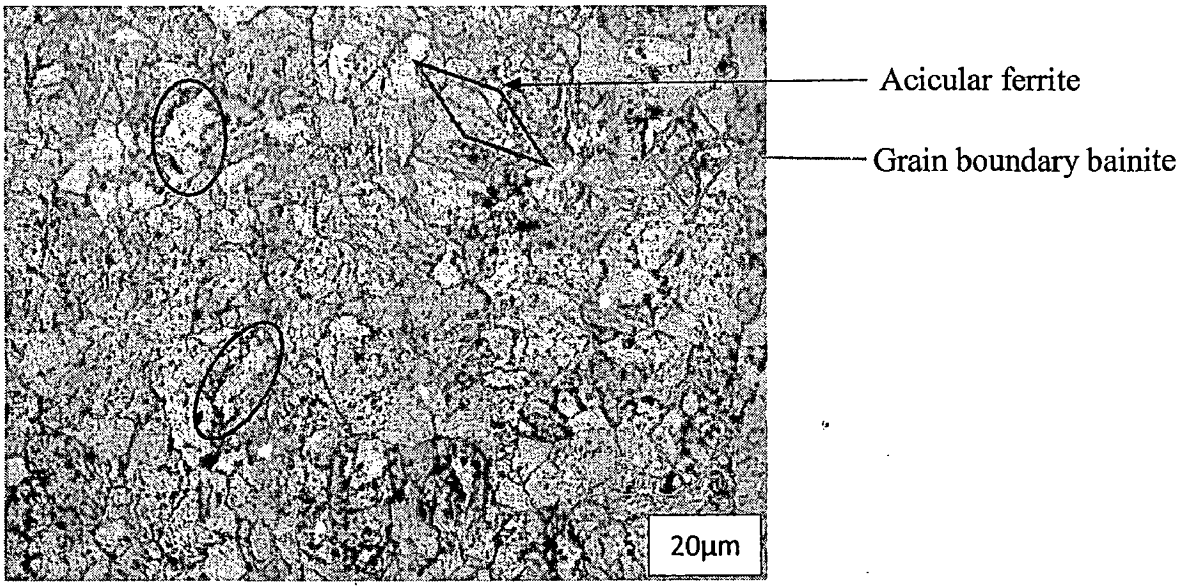


Fig.5.13: Acicular ferrite with grain boundary bainite, 1000x

base metal in 10 h PWHT

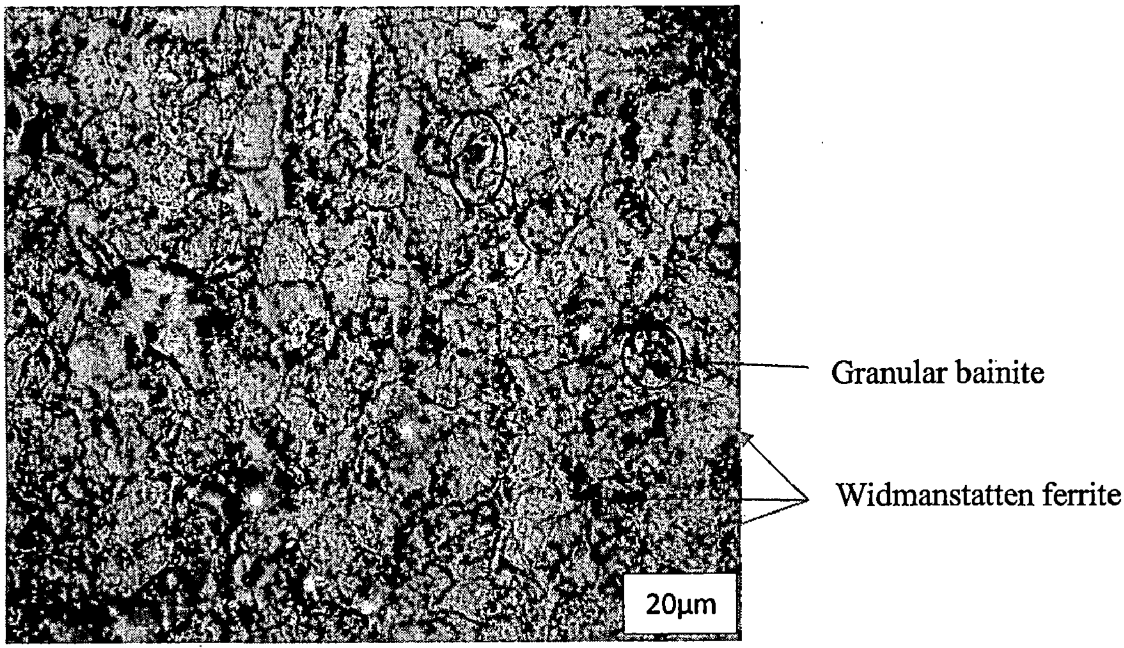


Fig. 5.14: Widmanstatten ferrite with granular bainite, 1000x

Base metal in 50 h PWHT

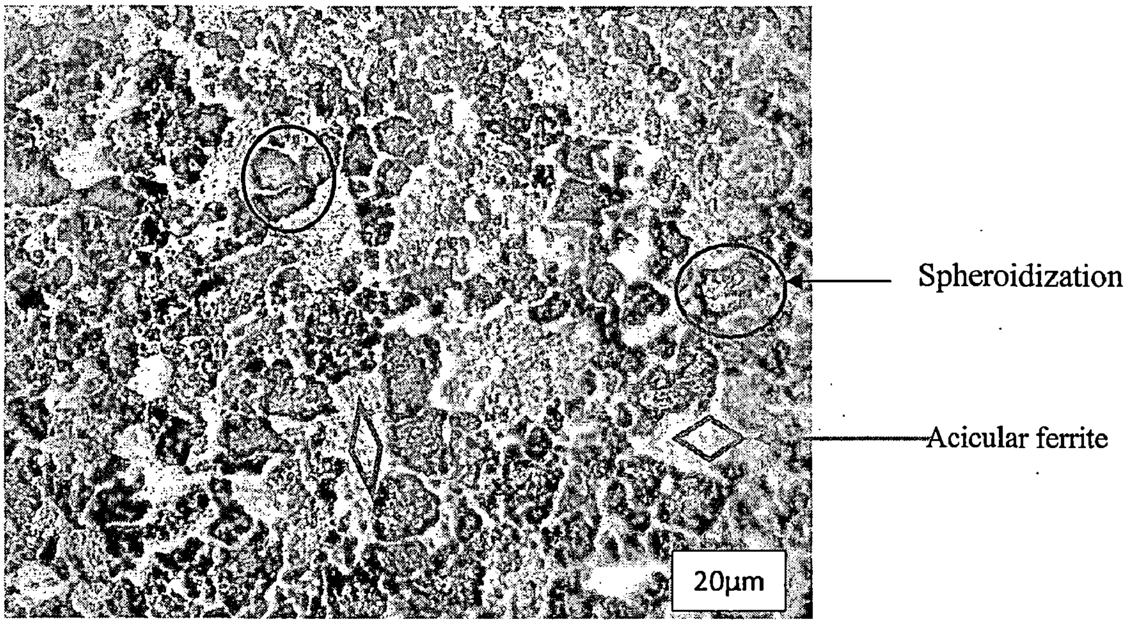


Fig.5.15: Acicular ferrite with pearlite and grain boundary bainite 1000x

A ferrite matrix with uniformly dispersed carbides was found in all the annealed base metals. In 50 h heat treatment due to long expose to high temperature spheroidization and grain coarsening was taken place.

5.1.4. HAZ and Weld Metal Interface

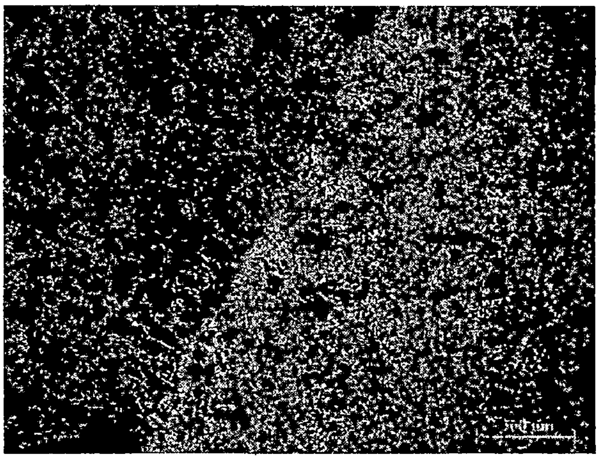


Fig.5.16: As- welded condition, 50x

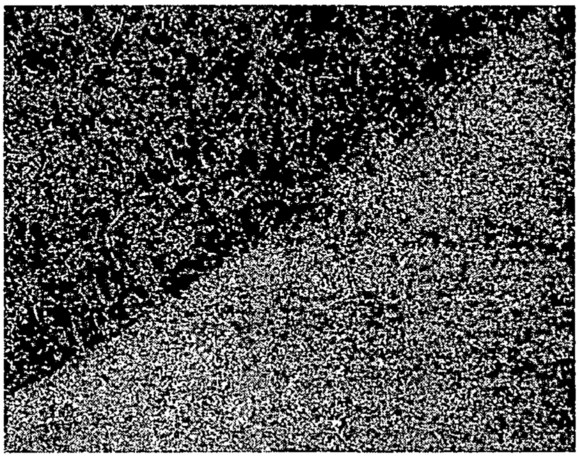


Fig.5.17: 0.5 h PWHT, 50x

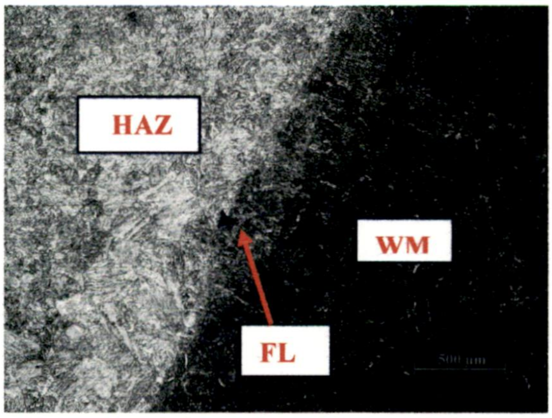


Fig.5.18: 2 h PWHT, 50x

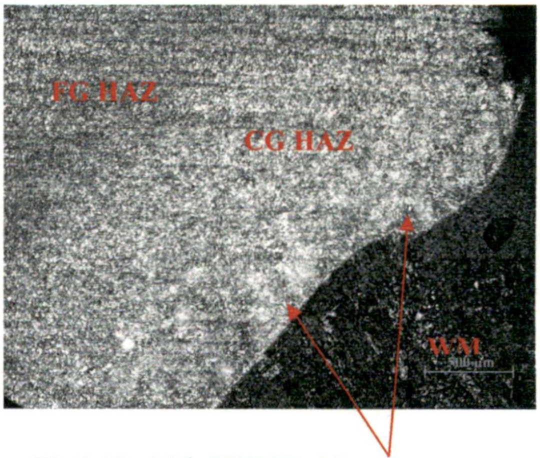


Fig5.19: 10 h PWHT, 50x

Fig.5.20: 50 h PWHT, 50x

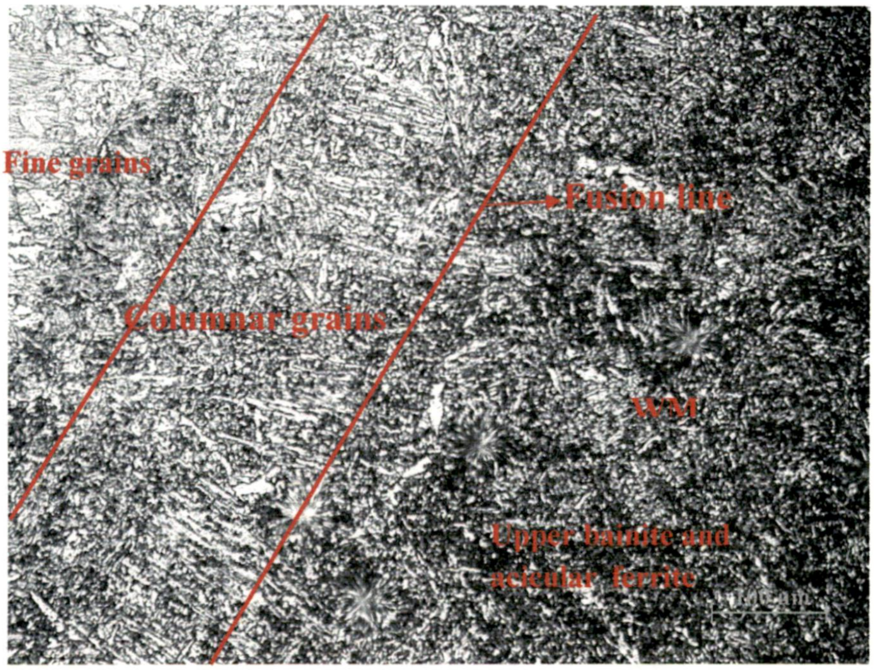


Fig.5.21: Interface at 2 h PWHT, 200x

5.1.5. Conclusions

Weld metal

In the as-welded WM, it was identified that upper bainite with fine distribution of carbides in the ferrite grains. A ferrite matrix with uniformly dispersed carbides was found in the annealed weld metal in 0.5h and 10 h PWHT. In 2 h the carbide dissolution had occurred and results also demonstrated that the lath boundary carbides present in the WM are coarsened and spheroidized after PWHT of 50 h due to exposure to high temperature (725^0C) at long durations.

HAZ

In as-welded HAZ consist of lath martensite, after 0.5 h heat treatment fine and coarse grain bainite is formed, which is further converted in to a mixed structure of polygonal/Widmanstatten/upper bainitic structure in 2 h PWHT, here very coarse grains are observed. And after 10 h carbide precipitation was observed in the ferritic matrix. All the CGHAZ and FGHAZ were composed of a bainitic structure with lath morphology.

Base metal

In as-welded base metal carbide precipitation was observed in a ferritic matrix. The microstructure of the annealed BM at different soaking times revealed a ferrite matrix with uniform dispersion of carbides.

The microstructures in various regions of the weld joint like weld metal, HAZ, base metal and interface region showed major changes in as-welded and after PWHT conditions when examined under optical microscope and SEM. These major changes had further shown changes in the mechanical and corrosion resistance properties.

5.2. Hardness Testing

Hardness testing is done using micro hardness tester LEITZ WETZLAR, Germany. Specimens having all the three portions of a weldment viz.weld metal, HAZ and base metal were tested .The hardness is taken from the weld centre line toward base metal.

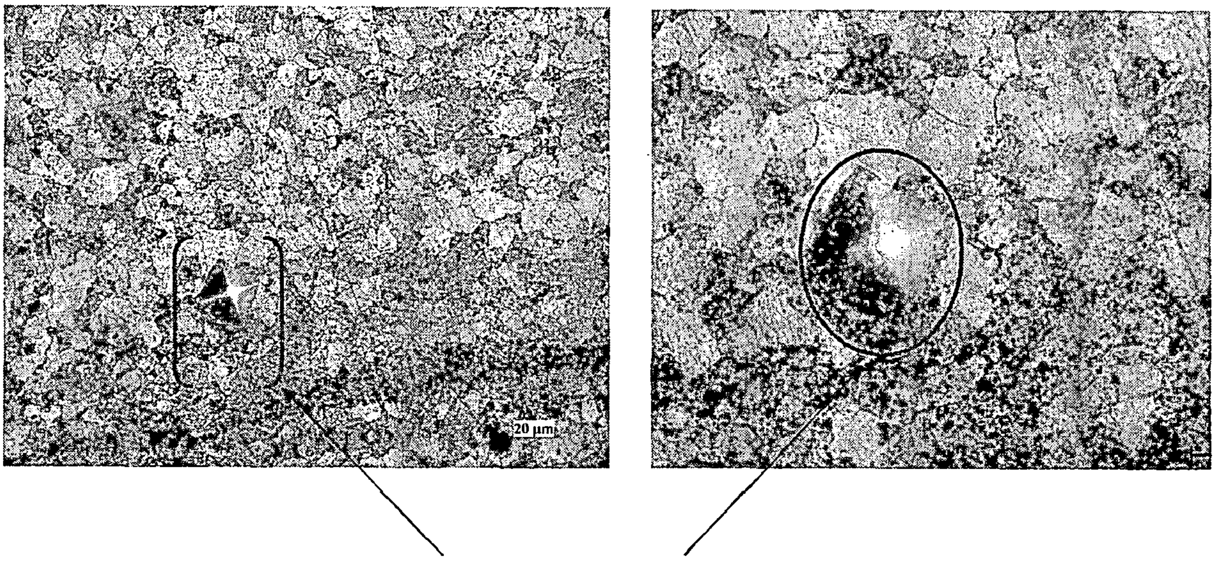


Fig.5.22: Hardness

Table.5.1: Hardness values for different heat treatments

Region	Average Hardness values (VHN at 100 gms) for various PWHT times (Hours)				
	As-Welded	0.5 h	2.0 h	10 h	50 h
	Hardness (VHN at 100 gms)				
WELD METAL	300	289	214	209	202
HAZ	348	293	190	310	210
BASE METAL	246	212	207	172	167

5.2.1. Observations

In weld metal more hardness is observed in as-welded condition. This is due to the precipitation of carbides. And as the heat treatment time goes on increasing hardness is reduced. Least hardness is observed in 50 h PWHT; this is due to the spheroidization of carbides.

In HAZ maximum hardness is observed in as-welded condition. This is due to the presence of lath martensite. The 2 h PWHT had shown a sharp drop in hardness from 384 VHN-190 VHN. This is due to the presence of mixed ferritic structure of polygonal/ Widmanstatten/ upper bainitic structure. This structure is very coarse. So 2 h PWHT is the favourable heat treatment which gives less hardness.

In base metal maximum hardness is observed in as-welded condition, this is due to the presence of carbides in the ferritic matrix. As the heat treatment time goes increasing the hardness is reduced. Minimum hardness is observed in 50 h PWHT.

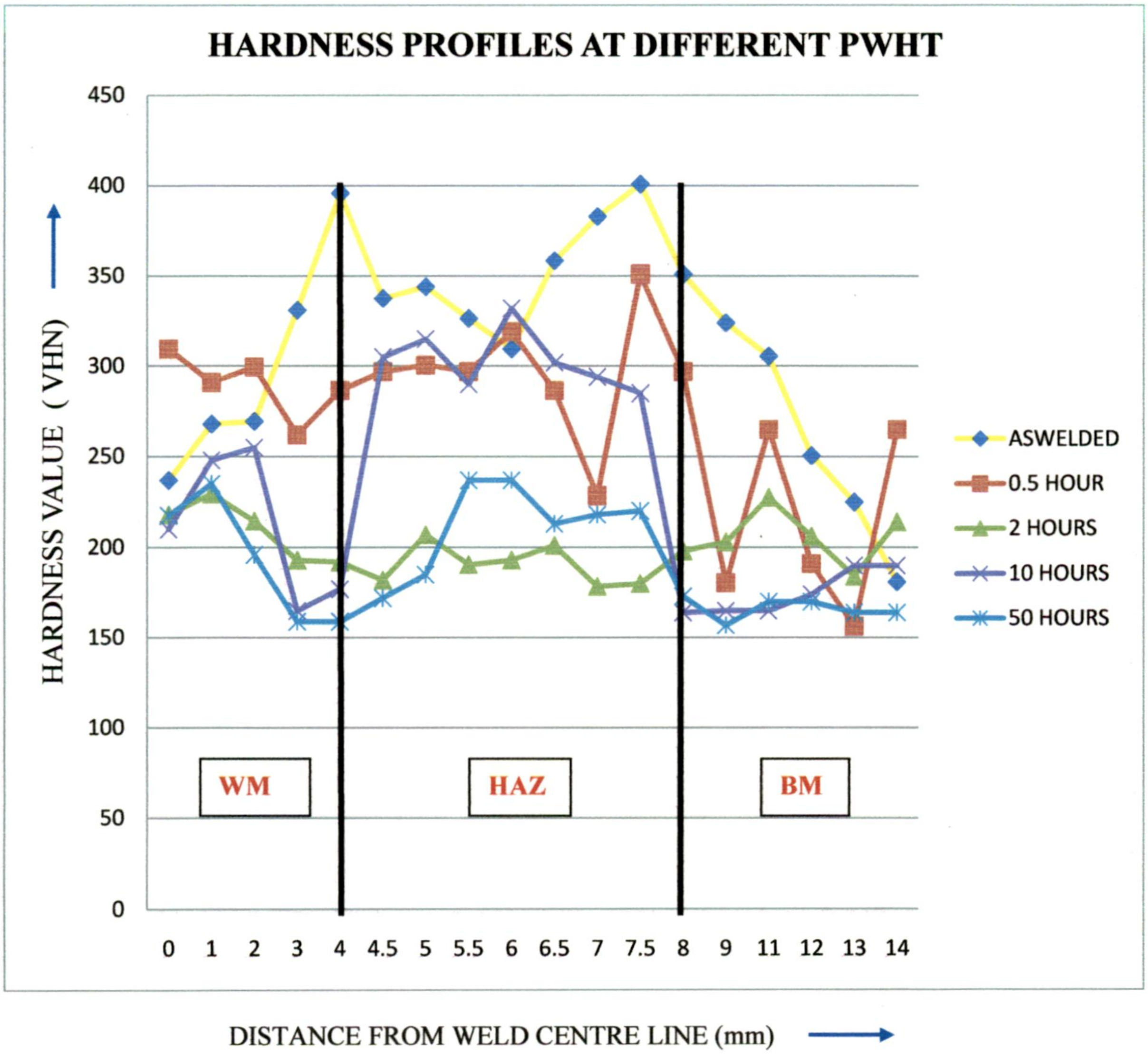


Fig. 5.23: Hardness profiles at different PWHT

5.2.2. Conclusions

By considering the average hardness values it is concluded that as the heat treatment time goes on increasing hardness is reduced in base metal and weld metal. But in HAZ least hardness is observed in 2 h of heat treatment due to the presence of coarse grains and less carbon percentage(conformed by E-DAX).The overall peak hardness is observed in HAZ portion, next to that is weld metal and the least in base metal as shown in fig.

From the above **Fig.**5.23 it is observed that HAZ hardness in 10 h of heat treatment is raised to large extent this is due to the effect of grain size in the haz which is confirmed by the SEM microstructure at the interface of WM and HAZ.

Hardness in HAZ

PWHT (hours)	As-welded	0.5	2	10	50
VHN	348	293	190	310	210

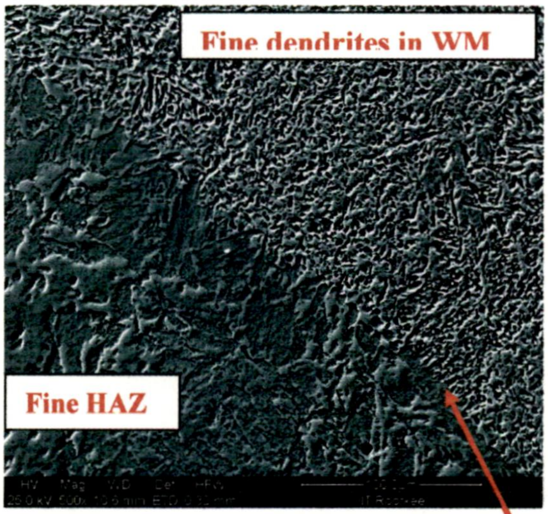


Fig.5.24: Interface in 10 h PWHT 500x

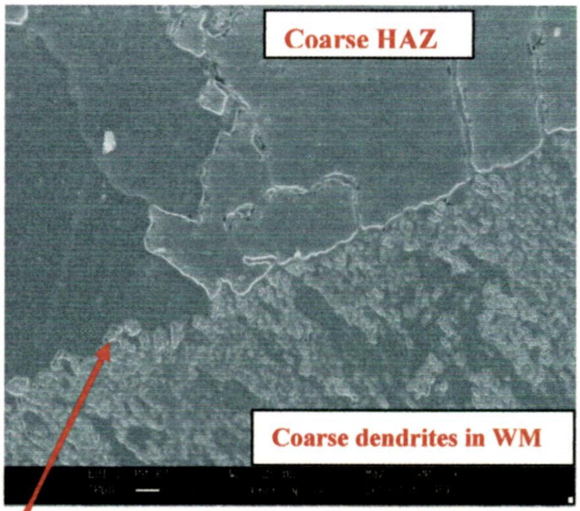


Fig.5.25: Interface in 50 h PWHT 500x

Fusion line

From the above micrographs it is observed that specimen undergone PWHT for 10 h have fine grains in the HAZ and weld metal and the specimen PWHT for 50 h consist of coarse grains in HAZ and weld metal. So the hardness is increased in the 10 h PWHT specimen.

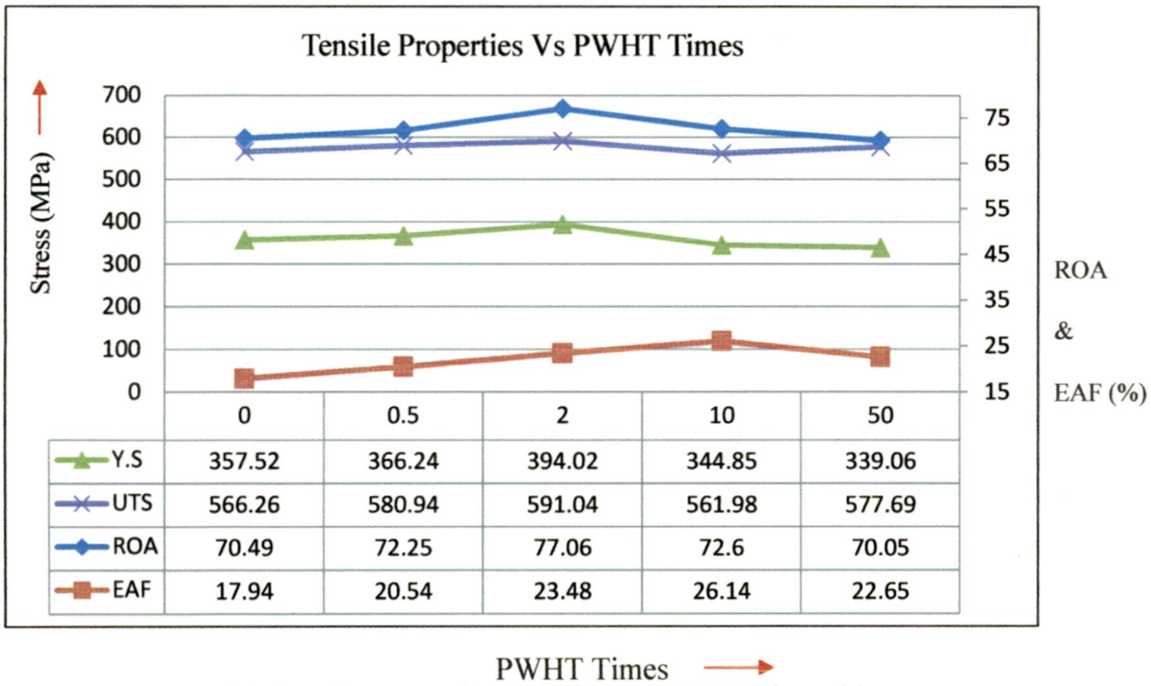
5.3. Tensile Testing

Tensile testing is conducted as per ASTM E 8M standard. The weld joints are taken in transverse direction and the base metal is taken in longitudinal direction of the weld joint. The mechanical properties such as yield strength (YS), ultimate strength (UTS), percentage of elongation (%EL) and percentage area of reduction (% ROA) are reported as follows.

5.3.1. Tensile properties in Transverse Weld Joints

PWHT (hours)	(ROA) %	EAF (%)	Y.S (MPa)	UTS (MPa)
As- Welded	70.49	17.94	357.52	566.26
0.5	72.25	20.54	366.24	580.94
2	77.06	23.48	394.02	591.04
10	72.60	26.14	344.85	561.98
50	70.05	22.65	339.06	577.69

Table.5.2: Tensile properties at different PWHT'S



	0	0.5	2	10	50
Y.S	357.52	366.24	394.02	344.85	339.06
UTS	566.26	580.94	591.04	561.98	577.69
ROA	70.49	72.25	77.06	72.6	70.05
EAF	17.94	20.54	23.48	26.14	22.65

Fig.5.26: Tensile properties at different PWHT'S in weldments

When tensile test is conducted in transverse weld joint's maximum ultimate tensile strength and yield strength are observed in specimens undergone 2 h PWHT.It is also observed that as the UTS and YS reduced as the heat treatment time goes on increasing. Percentage area of reduction is more in 2 h PWHT, which reveals that more ductility is there. In all the specimens failure is occurred in base metal closer to the end of the gage length, this is due to high strength in the weld metal and the constraint provided by the weld metal, but specimen undergone 10 h PWHT failed adjacent to the HAZ. This is due to the presence of coarse grains and carbide precipitation in this region.

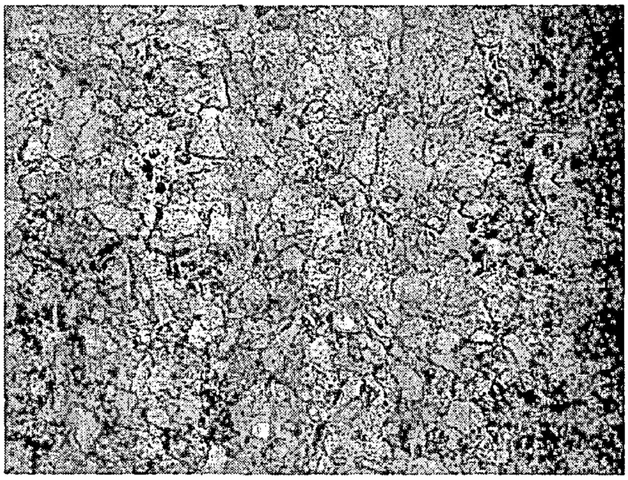


Fig.5.27: Showing grain coarsening and Carbide precipitation

It is concluded that low hardness and less tensile properties are observed in specimen undergone 50 h PWHT is due to the presence of oxygen in weld metal. Which is confirmed by the ELECTRON DISPERSIVE ANALYSIS (E-DAX).only in 50 h heat treated specimen had shown oxygen in the composition. Sindo kou [41] reported that presence of oxygen in the weld will reduce the tensile properties.

Table.5.3: E-DAX Analysis

Element	*Wt%*
CK	09.78
OK	02.07
AlK	00.58
SiK	00.59
PK	00.26
SK	00.00
VK	00.24
CrK	00.73
FeK	73.61
NiK	00.97
MoK	10.64

5.3.2. Tensile testing of Base Metal

PWHT (hours)	(ROA) %	EAF (%)	Y.S (MPa)	UTS (MPa)
0.5	68.80	26.45	358.75	538.27
2	71.65	27.77	377.14	580.75
10	73.50	33.25	337.03	541.07
50	72.01	27.20	314.38	562.78

Table .5.4: Tensile properties Vs PWHT in Base metal

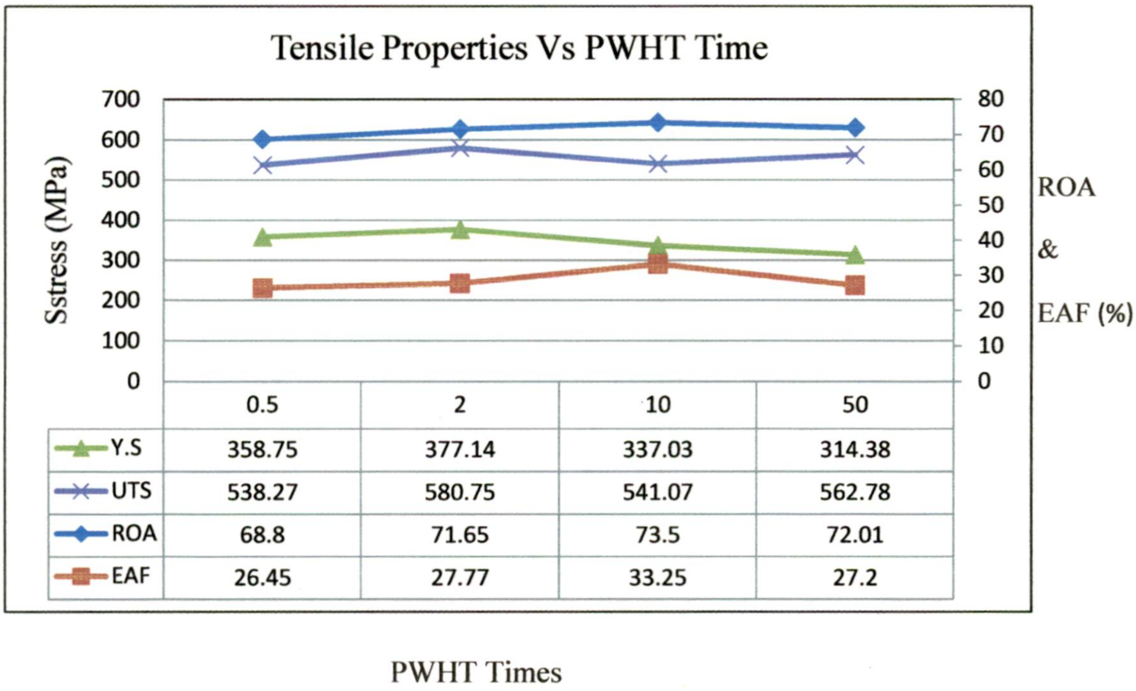


Fig.5.28: Tensile properties at different PWHT'S in Base metal

Tensile testing in base metal shown maximum yield strength and ultimate strength in specimen undergone 2 h PWHT. It is concluded from the above graph that the UTS and YS go on decreasing as the heat treatment time is increased beyond 2 h. Percentage of elongation and percentage area of reduction were increased in 10 h heat treatment, this reveals the specimen under gone ductile failure.AR and EL are least in 0.5 h PWHT, so these specimen may be failed in a brittle manner.

The reason for the fall of the strength of the BM in long durations of PWHT is due to spheroidization and coarsening of the pearlite as shown in **Fig.**5.29 this statement is supported by R.D.Stout [46].

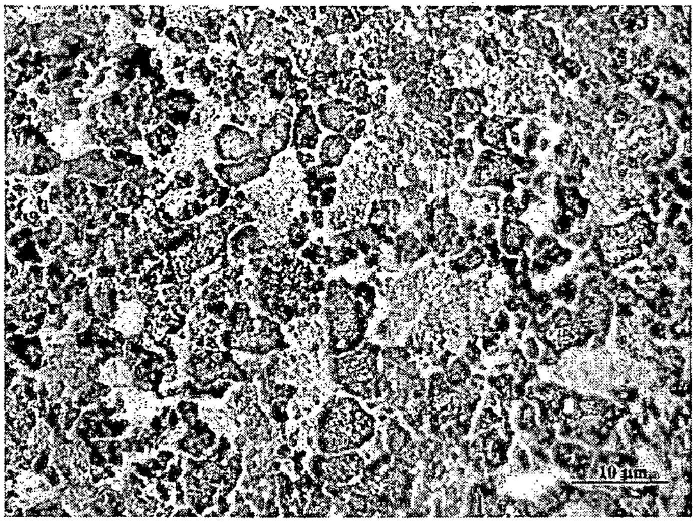


Fig.5.29: Base metal in 50 h PWHT 1000x

5.3.3. Conclusion

by conducting transverse (weld joint) and longitudinal (base metal) tensile test it is observed that 2 h PWHT gives good tensile properties like UTS and YS, when compared to long duration heat treatments. It is also observed that percentage of elongation is increased in 10h PWHT. This is due to the presence of refined grains in weld metal and HAZ as shown in the **Fig.**5.24 is it is also expected that there is another heat treatment time which is in between 2 h and 10 h which will improve tensile properties better than 2 h PWHT.

5.4. Impact test

PWHT(hours)	Testing Temperature ^{0}C				
	-90	-60	0	60	120
	Absorbed Energy (J)				
As Welded	2	11	16	20	18
0.5	10	17	18	20	24
2.0	8	11	18	22	20
10	12	20	22	24	26
50	8	6	24	26	30

Table.5.5: PWHT Vs Testing temperatures and Impact resistance

Charpy V-notch impact test is conducted on specimens in As-welded condition and PWHT at 0.5,2,10,50 hours, to determine the impact toughness. The notch is made in weld metal in transverse direction to the weld deposit. The impact test is done at higher temperature like 60^0 C, 120^0 C and at sub zero temperatures like -90^0 C,-60^0 C and 0^0 C. These temperatures are selected as per the industry applications. The notch design and specimen dimensions are shown in **Fig.**4.10.

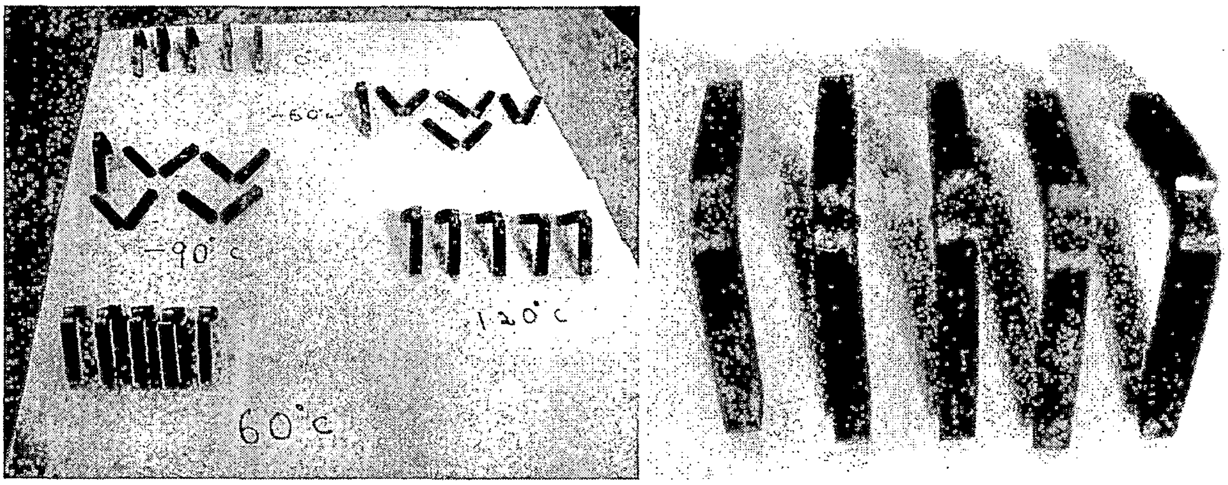


Fig.5.30: a) Samples tested at different temperature (b) samples not fractured completely

It is observed from the above **Table.**5.5 that samples tested at higher temperatures like 120^{0}C and 60 ^{0}C shown higher energy observation and these samples are not fractured completely. But the samples tested at sub zero temperatures like -90^{0}C and -60^{0}C shown less energy observation and these samples fractured completely as shown above.

That means these samples failed in a brittle manner, the reason is due to ductile-brittle transition. As it is known commonly that steels undergo ductile –brittle nature when exposed to low temperatures.

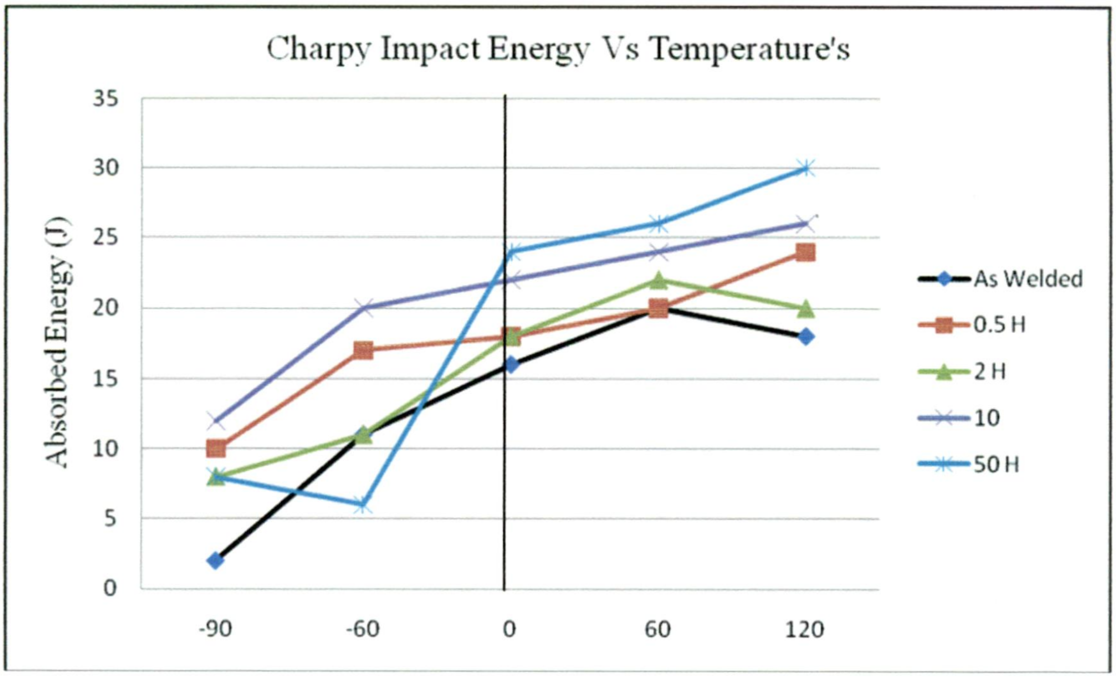


Fig.5.31: Impact energy at different PWHT's and Temperatures

5.4.1. Conclusion

The results obtained from the impact test allows the following observations

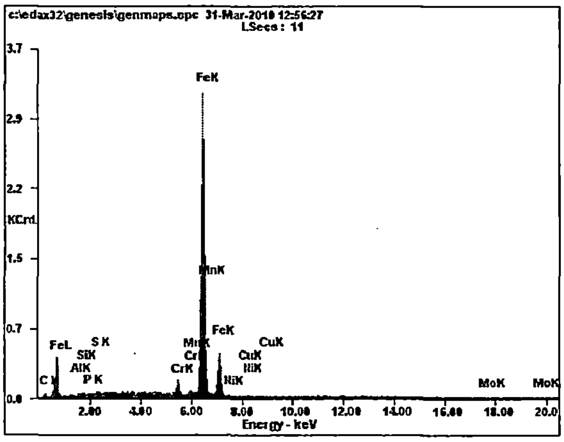
- It is concluded that impact resistance is very less in as-weld condition. The impact resistance increased after PWHT.
- It is also concluded that 10 h PWHT had shown higher impact resistance when tested in high temperatures and sub zero temperatures.
- For a given temperature, the impact resistance decreased with the increase of the heat treatment longer than 10 h. It means ductile-brittle transition temperature increased with the time of heat treatment.

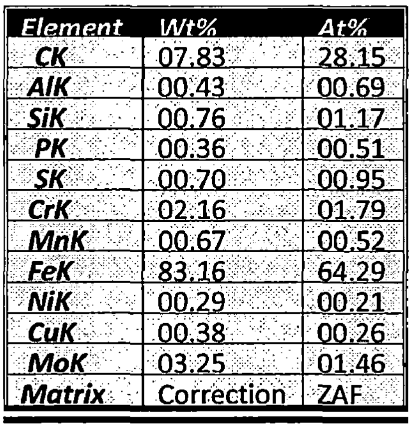
The reasons for the increase in impact resistance in 10 h PWHT is due to the presence of refined grains in weld metal and HAZ as shown in the **Fig.**5.24 the microstructure consists of fine dendritic structure. Fair Child [47] had reported that high toughness and smaller transition temperature in steels is achieved with finer microstructure and a small impurity content

From the above observations it is concluded that 10 h PWHT is the favourable heat treatment which gives higher impact resistance in higher temperatures and sub zero temperatures. And it is also expected that there is an another heat treatment which is in between 10h and 50 h which will improve impact resistance better than 10h.

5.5. E-DAX Analysis

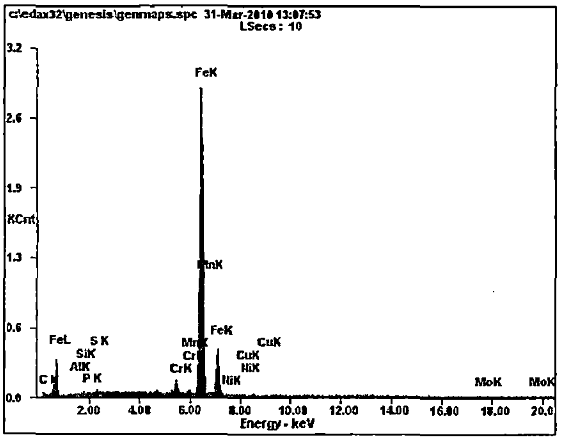
Energy-dispersive analysis (E-DAX) is used to determine the elemental compositions present at point/area of Interest. E-DAX analysis is focused on weld metal and the compositional variations are studied in as-weld and PWHT conditions. It is concluded that these variations will change the mechanical and microstructural properties.

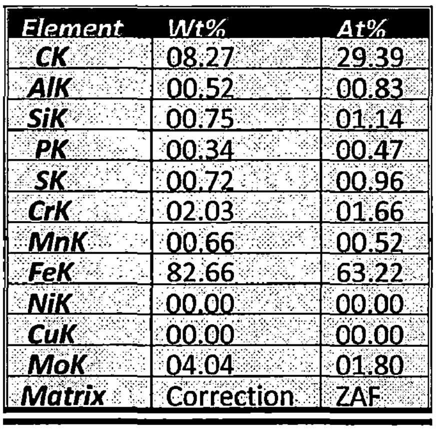




Element	Wt%	At%
CK	07.83	28.15
AlK	00.43	00.69
SiK	00.76	01.17
PK	00.36	00.51
SK	00.70	00.95
CrK	02.16	01.79
MnK	00.67	00.52
FeK	83.16	64.29
NiK	00.29	00.21
CuK	00.38	00.26
MoK	03.25	01.46
Matrix	Correction	ZAF

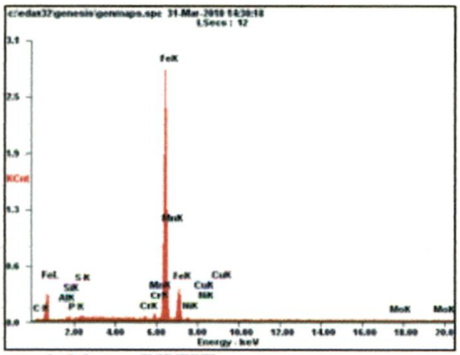
As- welded condition





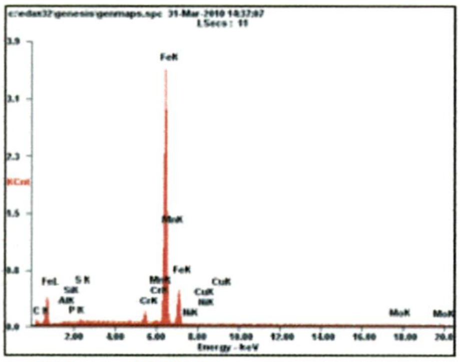
Element	Wt%	At%
CK	08.27	29.39
AlK	00.52	00.83
SiK	00.75	01.14
PK	00.34	00.47
SK	00.72	00.96
CrK	02.03	01.66
MnK	00.66	00.52
FeK	82.66	63.22
NiK	00.00	00.00
CuK	00.00	00.00
MoK	04.04	01.80
Matrix	Correction	ZAF

0.5 hour PWHT



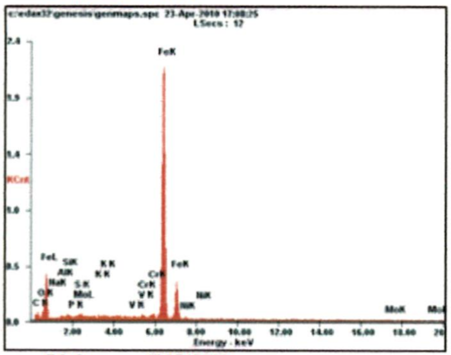
2.0 hour PWHT

Element	Wt%	At%
CK	04.99	19.76
AlK	00.64	01.13
SiK	00.79	01.35
PK	00.31	00.48
SK	00.53	00.79
CrK	00.63	00.58
MnK	01.37	01.18
FeK	82.48	70.21
NiK	01.20	00.97
CuK	00.24	00.18
MoK	06.81	03.37
Matrix	Correction	ZAF



10 hours PWHT

Element	Wt%	At%
CK	10.83	36.20
AlK	00.55	00.82
SiK	00.52	00.74
PK	00.18	00.23
SK	00.45	00.56
CrK	02.04	01.57
MnK	00.69	00.50
FeK	79.05	56.84
NiK	00.30	00.21
CuK	00.30	00.19
MoK	05.09	02.13
Matrix	Correction	ZAF



50 hours PWHT

Element	Wt%	At%
CK	09.78	32.86
OK	02.07	05.23
NaK	00.32	00.57
AlK	00.58	00.86
SiK	00.59	00.85
PK	00.26	00.34
SK	00.00	00.00
KK	00.20	00.21
VK	00.24	00.19
CrK	00.73	00.57
FeK	73.61	53.18
NiK	00.97	00.67
MoK	10.64	04.47
Matrix	Correction	ZAF

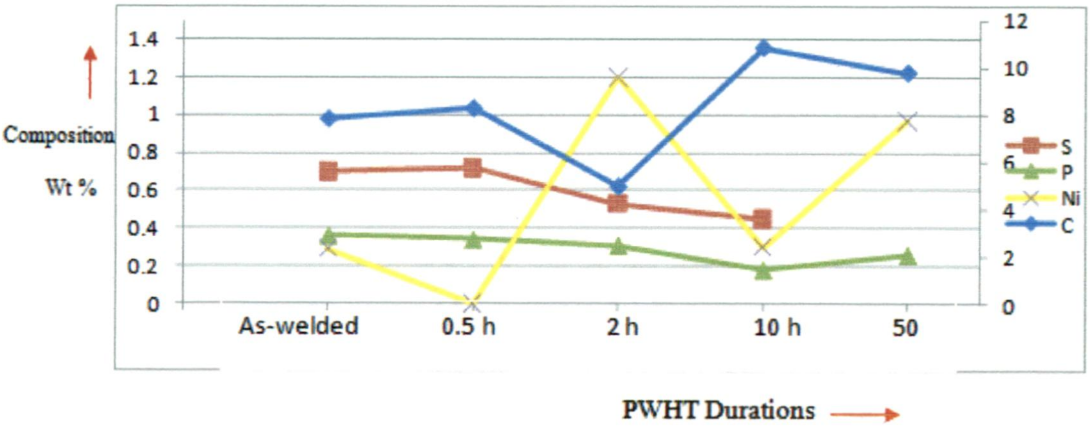


Fig.5.32: Composition in weld metal at different PWHT

5.5.1. CONCLUSIONS

Sulphur:

Decreases notch impact toughness especially in the transverse direction. Weldability decreases with increasing sulphur content. Sulphur is found primarily in the form of sulphide inclusions. As the PWHT is goes on increasing percentage of sulphur increases, in 10 h heat treatment less percentage of sulphur is their which improved the toughness.

Carbon:

Carbon has a major effect on steel properties. Carbon is the primary hardening element in steel. Ductility and weldability decrease with increasing carbon. It is observed that 2 h heat treatment has less amount of carbon in its composition when compared to as weld and other heat treatment conditions. From the **Fig.**5.26 it is observed that 2 h heat treatment is having high percentage area of reduction, indicating higher ductility

Phosphorous:

Phosphorous is kept to minimum and it is treated as impurity carbon steels contains phosphorus more than 0.03% increases strength and decrease notch impact toughness. These results are observed in 2 h heat treatment where high strength and low impact toughness is achived.in 10 h PWHT very less phosphorous is observed (0.18 %) which leads to high impact toughness.

Oxygen:

Oxygen is observed in 50 h heat treatment, its wt % is 2.07.sindo kou [41] reported that presence of oxygen in weld may increase the toughness and reduce its hardness. This is the reason why 50 h PWHT sample has high impact resistance **Fig.**5.31 when tested in high temperatures and low hardness compared to other PWHT samples.

Nickel:

It increases the hardenability and impact strength of steels at low temperatures. From the above composition obtained from E-DAX.10 h heat treated sample is having very less nickel when compared to other specimen compositions. From the **Fig.**5.31 it is observed that 10 h sample shown high impact resistance, and this may be the required composition of nickel in order to have high impact resistance in low temperatures.

5.6. Results and discussions for hot corrosion studies

Hot corrosion test is focused on un-welded specimen and different portions in weld specimen like weld metal and base metal. The hot corrosion test is carried at high temperature of 900^{0}C in Air and Salt (Na_2SO_4–60% V_2O_5) environments .The results are characterized by weight change measurements, visual observations, X-ray diffraction analysis(XRD) ,X-Ray Mapping, E-DAX analysis etc.

5.6. Weight change measurements

Weight change measurements are recorded for Un welded, Base metal and Weld metal exposed to air and salt oxidation. Graphs are plotted for weight gain per unit area (mg/cm$^{2)}$ verses time expressed in number of cycles.

5.6.1. Hot corrosion in Un-welded sample

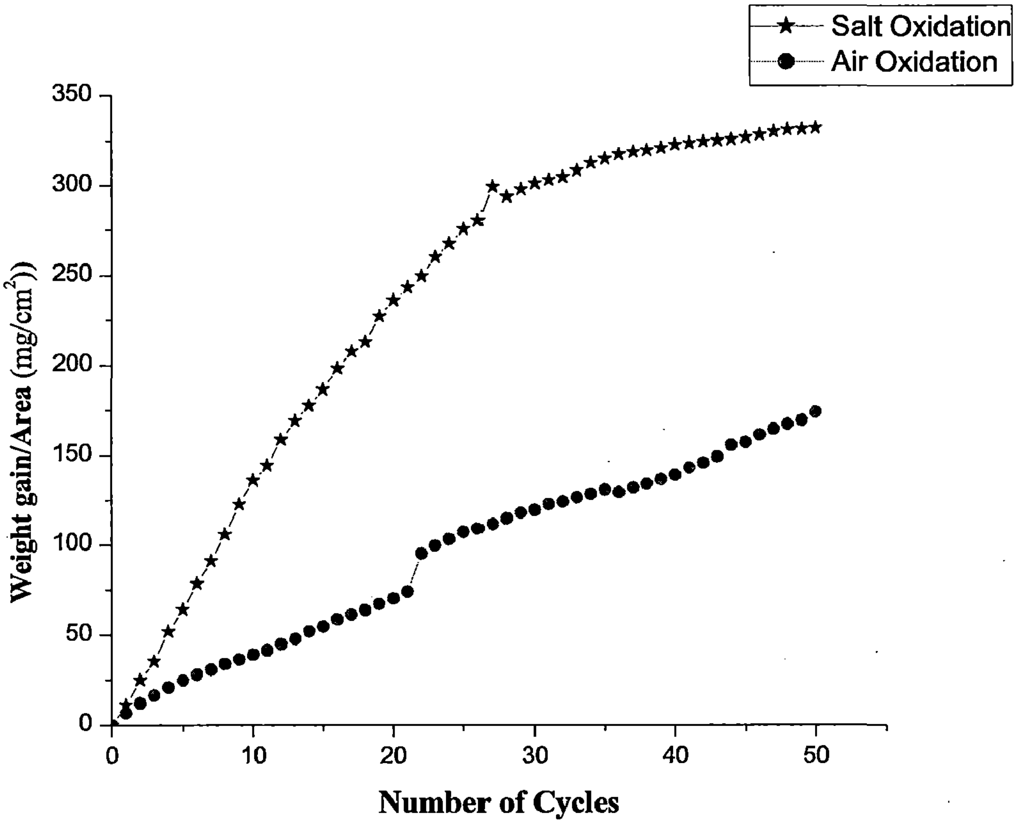


Fig.5.33: Combination of Salt and Air oxidation plots in Un-welded condition

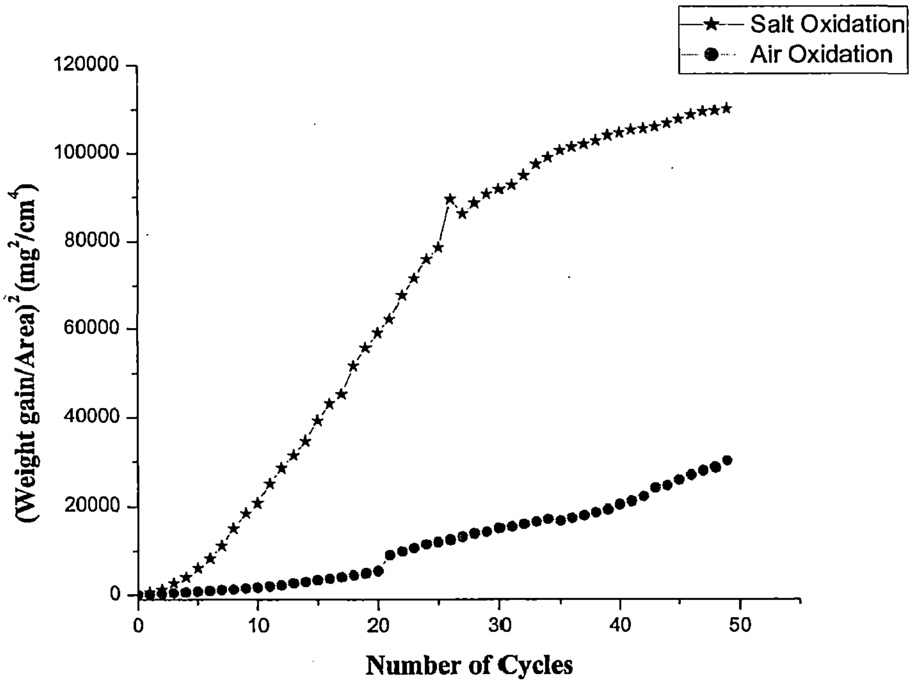


Fig.5.34: Weight gain square (mg^2/cm^4) plots for Un-welded specimen

The oxidation which occurred in salt and air at a temperature of 900°C is shown by plotting a graph. On x-axis "number of cycles" and on y-axis "total weight gain (mg/cm^2)" was taken. The salt oxidation has shown more weight gain after 27 cycles. The salt and air oxidation samples follow the parabolic kinetics when tested in hot corrosion. Un-welded sample undergone salt and air oxidation is highly affected which is confirmed by weight gain and XRD analysis.

5.6.2. Hot corrosion in Weld metal

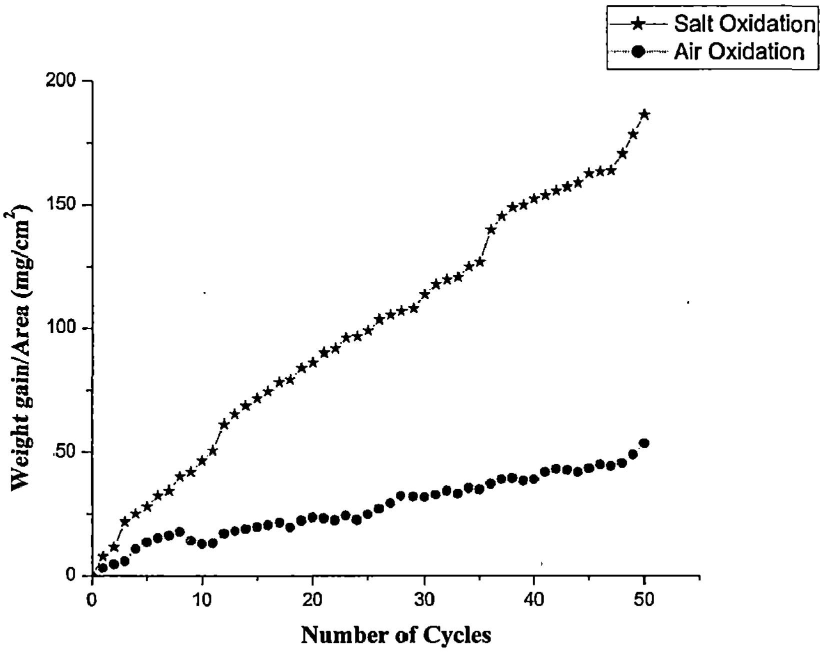


Fig.5.35: Combination of Salt and Air oxidation plots in Weld metal

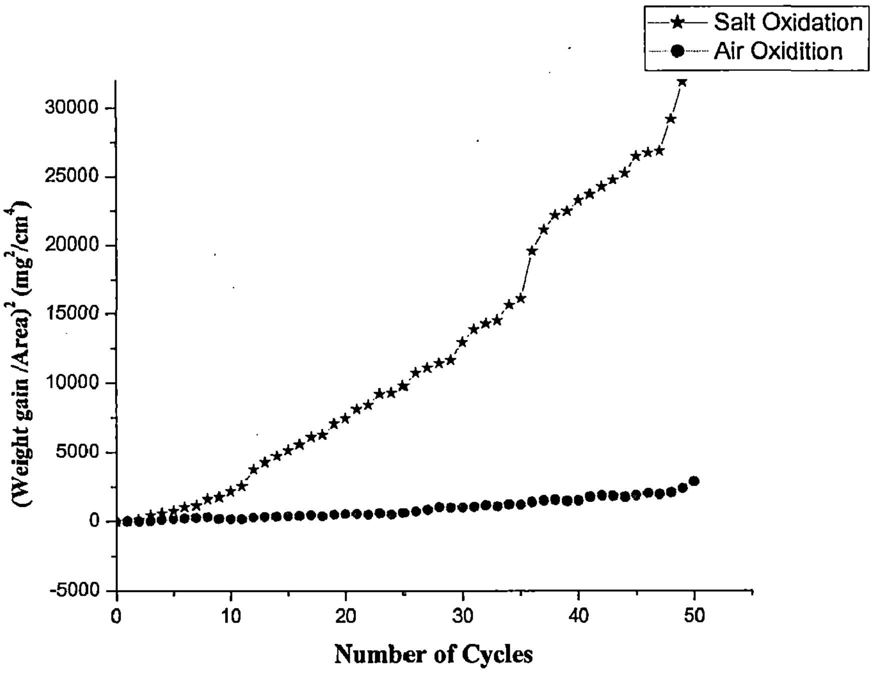


Fig.5.36: Weight gain square (mg^2/cm^4) plots for weld metal specimen

The oxidation which occurred in salt and air at a temperature of 900°C is shown by plotting a graph. On x-axis "number of cycles" and on y-axis "total weight gain (mg/cm^2)" was taken. The salt oxidation has shown more weight gain when compared to air oxidation. Less weight gains is observed in weld metal undergone hot corrosion. Weld metal in air oxidation follows linear rate of weight gain.

5.6.3. Hot corrosion in Base metal

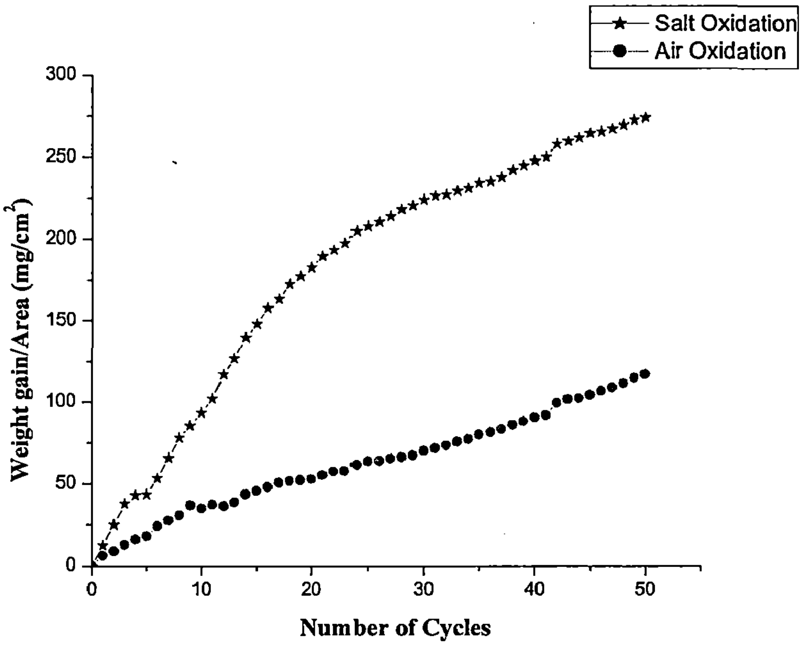


Fig.5.37: Combination of Salt and Air oxidation plots in Base metal

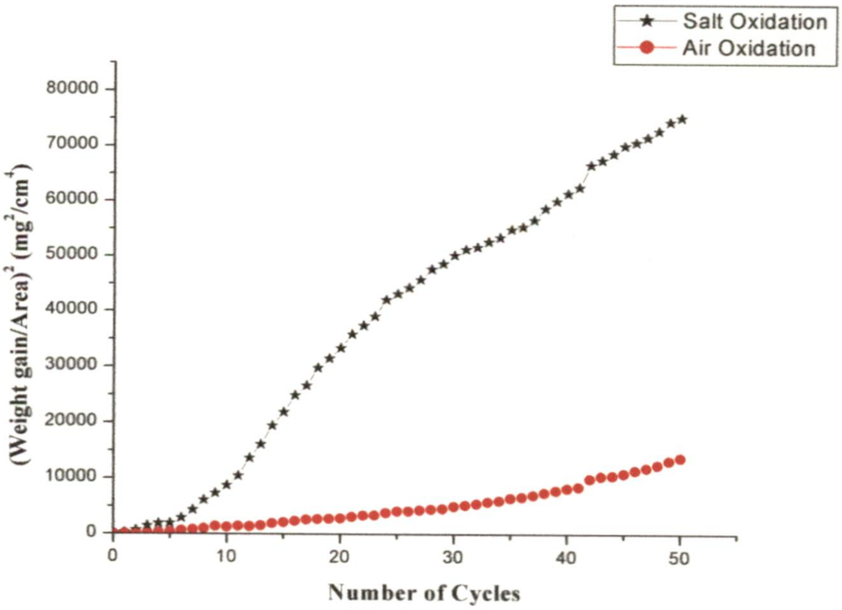


Fig.5.38: Weight gain square (mg^2/cm^4) plots for Base metal

The oxidation which occurred in salt and air at a temperature of 900°C is shown by plotting a graph. On x-axis "number of cycles" and on y-axis "total weight gain (mg/cm^2)" was taken. The salt oxidation has shown more weight gain when compared to air oxidation. Less weight gains is observed in Base metal undergone hot corrosion when compared to un-welded sample.

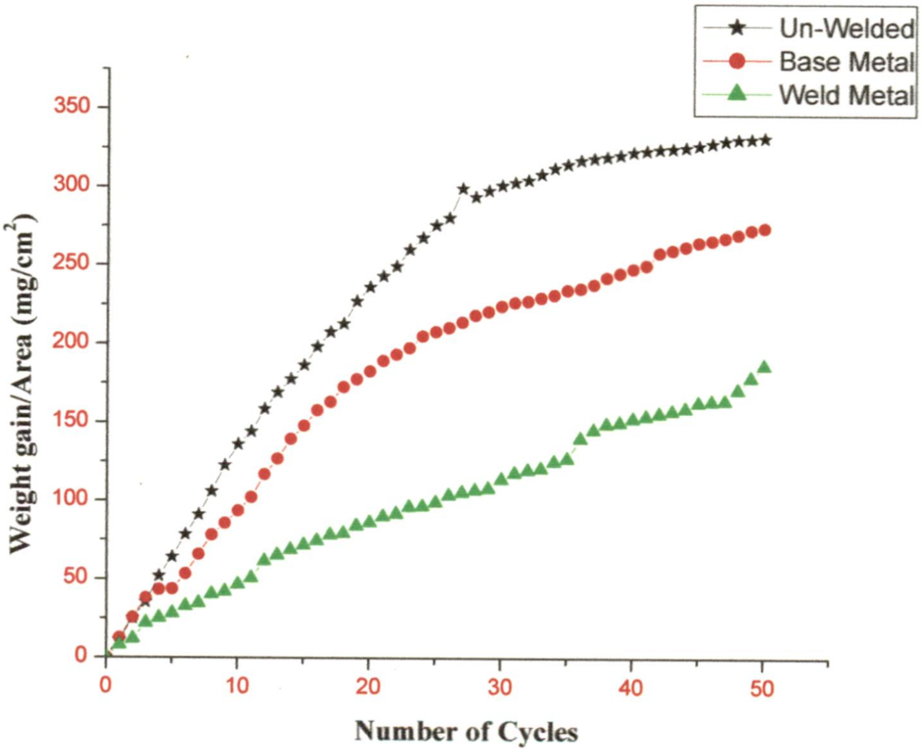


Fig.5.39: Combined graphs for Salt oxidations in Un-welded, weld metal and base metal

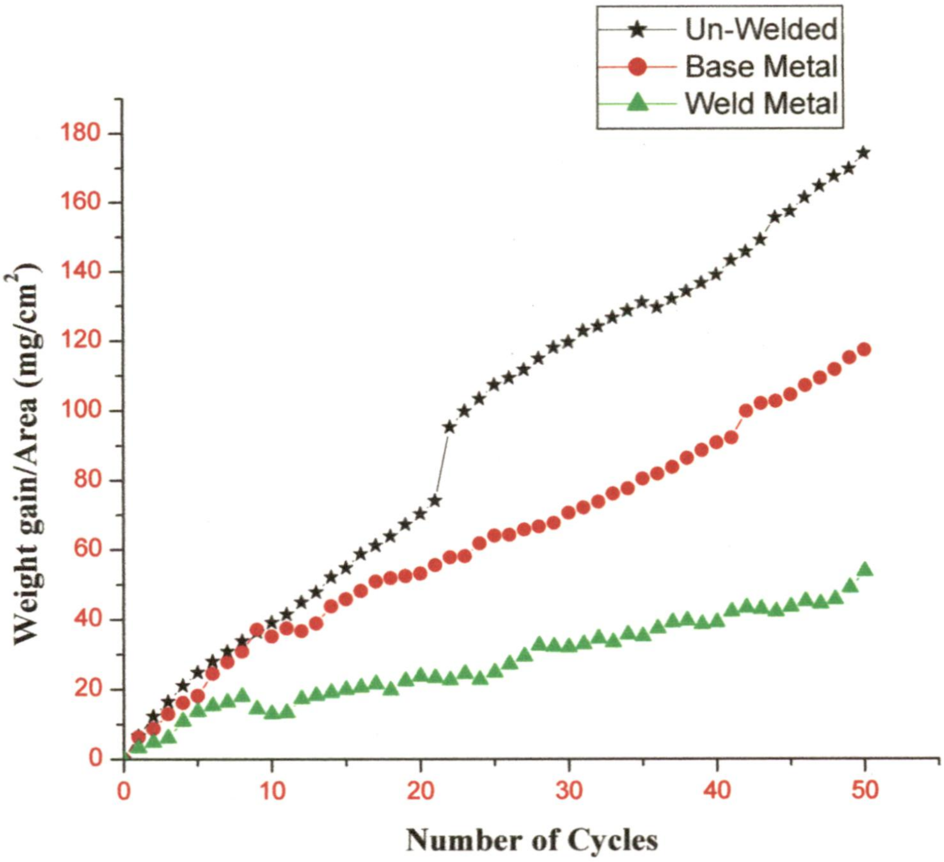
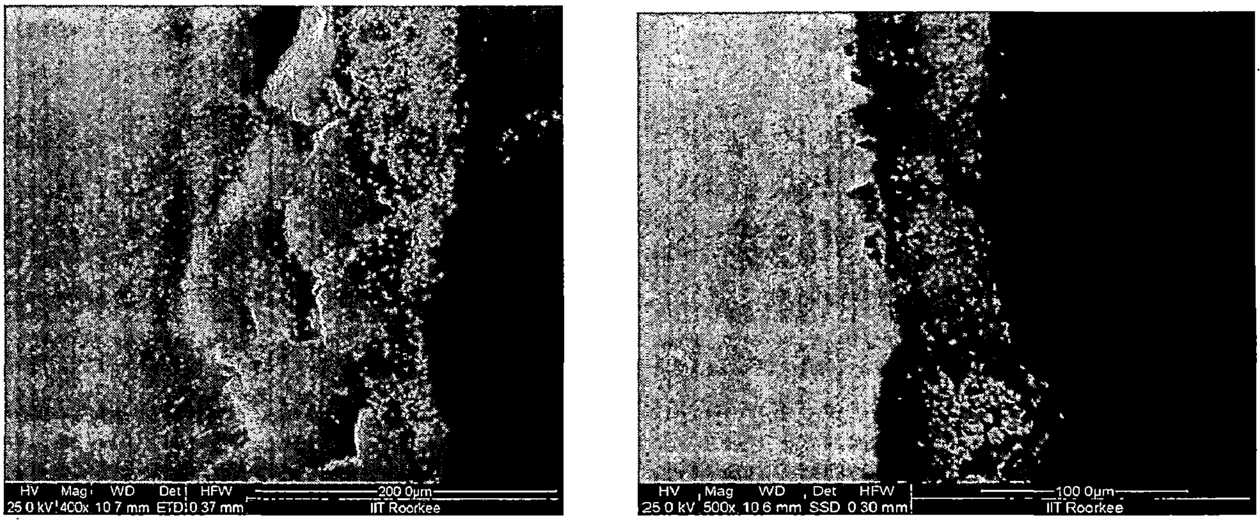


Fig.5.40: Combined graphs for Air oxidations in Un-welded, weld metal and base metal

5.6.4. Conclusions

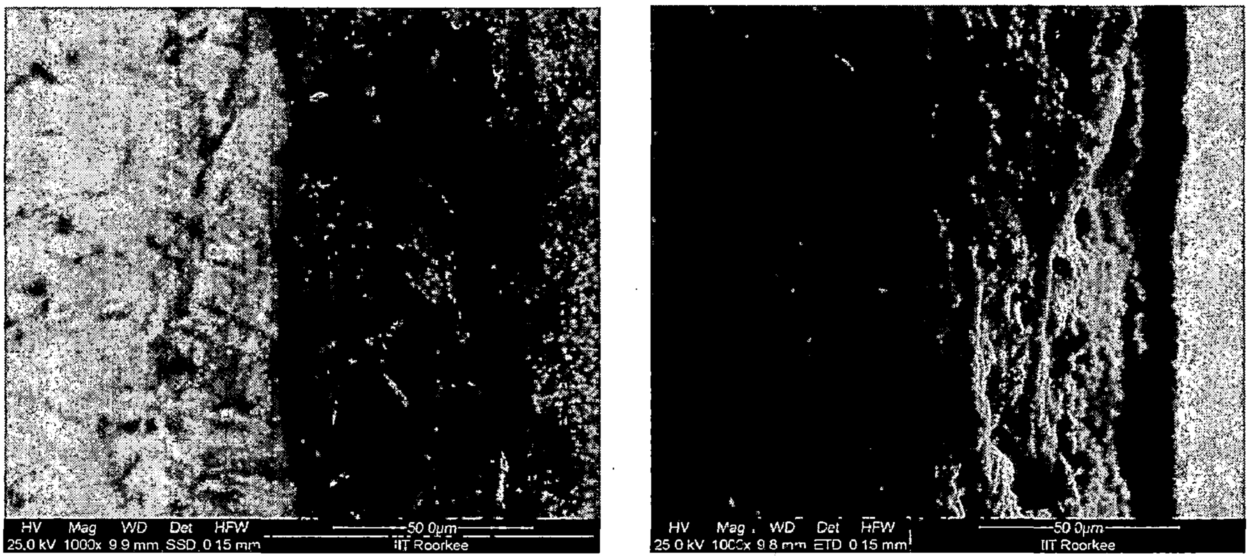
- Cumulative weight change (mg/cm^2) variation as a function of time expressed in number of cycles for un-welded, weld metal and base metal are shown in fig5.40
- Intense spalling was observed throughout the experimentation on un-weld specimen.
- It is concluded from the fig5.39That the weight gain for un-welded specimen is much larger in salt and air environment as compared to weld metal and base metal.
- All the specimens undergone hot corrosion test in air and salt environment followed the parabolic behaviour.
- It is also concluded that weld metal has shown high resistance to corrosion as compared to base metal and un-welded samples.
- The corrosion rate (in terms of weight gain) of un-weld specimen, weld metal and base metal has been found in the following order un-welded > base metal > weld metal.

5.7. Average Oxide Scale thickness



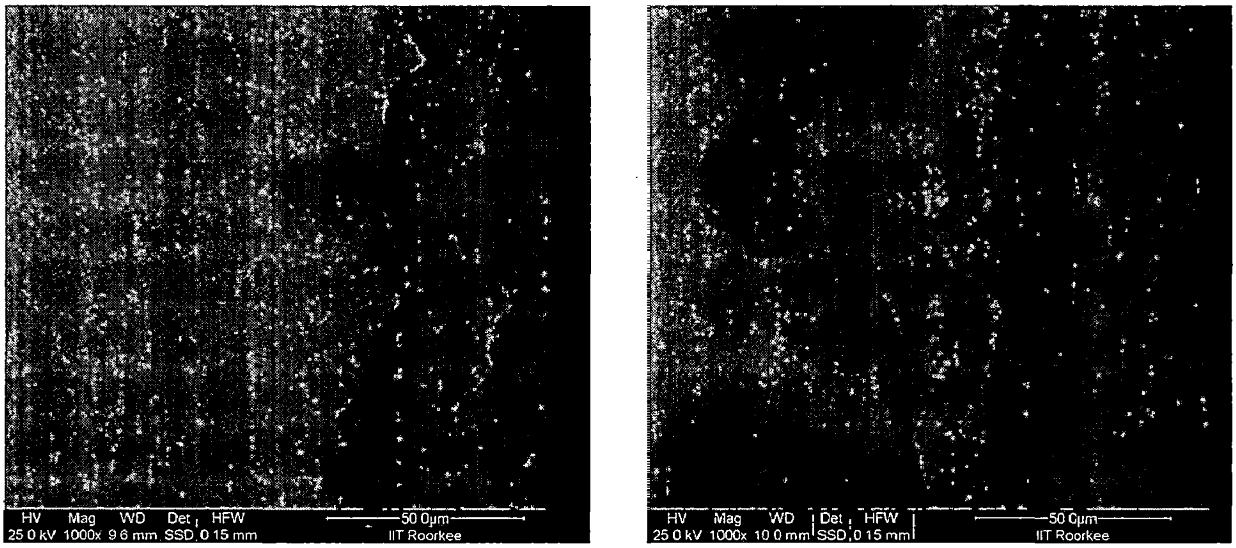
Un-Welded salt oxidation

Un-Welded air oxidation



Weld metal salt oxidation

weld metal air oxidation



Base metal salt oxidation

base metal air oxidation

Fig.5.41: oxides scales in Un-welded, Weld metal and Base metal samples

Oxide Scale thickness is measured on the BSE image of the corroded un-welded, base metal and weld metal. From the scale thickness measurement (BSEI), it is concluded that the un-weld sample is having more thickness indicating that the sample is highly affected by Hot corrosion.

specimen	Scale thickness (μm)	
	Salt oxidation	Air oxidation
Un-weld	140	116
Base metal	51	58
Weld metal	34	52

Table.5.6: Oxide scale thickness in Un-Welded, Weld metal and Base metal in air and salt.

5.8. X-Ray Diffraction analysis (XRD)

XRD analysis is carried out for the Un-weld, weld metal and base metal specimen's undergone salt and air oxidation of 50 cycles, in order to determine the oxide phases.

5.8.1. XRD analysis of salt oxidation

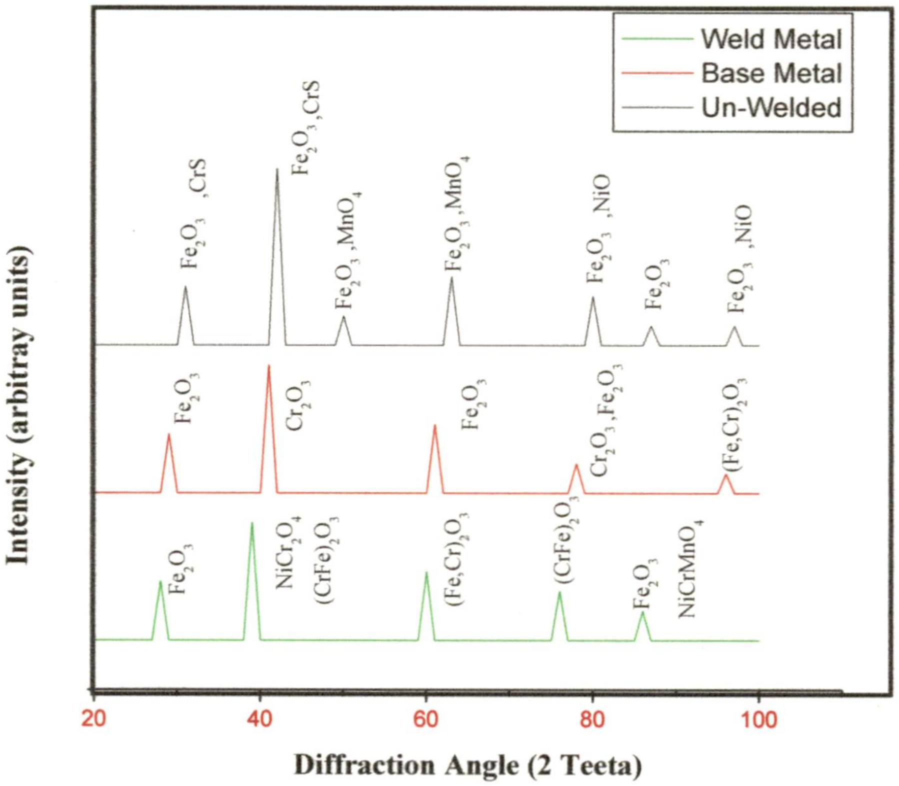


Fig.5.42: XRD patterns of Un-welded, Weld metal and Base metal in

The un-welded sample suffered corrosion in the form of intense spalling and peeling of the scale, which may be due to the formation of unprotective Fe_2O_3 oxide scale.fig.111 shows that the un –weld sample has Fe_2O_3 as the main constitute of scale.Fe_2O_3 is a unprotective layer so corrosion attack is more in un-weld sample[48]

In un-welded sample NiO is also formed in addition of Fe_2O_3, Nickel oxide (NiO) is not protective, rather than that NiO is said to be loose structured and thus lead to more pore size and thus results in more corrosion [49].The other oxides formed in un-welded sample are CrS and MnO_4.The weld metal and base metal are undergone PWHT before hot corrosion test, so the corrosion resistance is increased in weld metal and base metal which is confirmed by the weight change measurements and XRD patterns. XRD patterns show that weak peaks of Fe_2O_3 are observed in weld metal and base metal.

Weld metal shown high corrosion resistance when compared to base metal, the reason is the electrode used is a Austenitic stainless steel electrod, the austenitic stainless steel is known for its corrosion resistance due to the presence of chromium and molybdenum .Chromium adds to the overall resistance through a passivation process by forming a complex spinel-type (Fe,Ni)O and $(Fe,Cr)_2O_3$ passive film This complex produces a coherent, adherent insulating and regenerating chromium oxide protective film on the metal surface; while molybdenum increases the ability resist the localized corrosion in aggressive environments.[50].

The weld metal consist of strong peaks of $NiCr_2O_4$, $(Cr\ Fe)_2\ O_3$ $Fe\ Cr_2\ O_3$ etc. Ni concentration is more in weld metal which is confirmed by X-Ray Mapping, so iron combined with Ni to form $Ni\ Cr_2\ O_4$ and $Ni\ Mn_2O_4$ these helps to develop oxidation resistance as the spinel phases usually have much smaller diffusion coefficients of the ions.[51].

Base metal shown less resistance to hot corrosion when compared to weld metal but high resistance when compared to un-welded sample. Weak peaks of Fe_2O_3 are observed in base metal and other compounds like Cr_2O_3, $Fe\ Cr_2\ O_3$ and $Mn\ CrO_3$ are observed.Cr_2O_3 forms a protective oxides which contributed the better corrosion resistance by controlling the reactants entering the oxide layer. [52].

5.8.2. XRD analysis of Air oxidation

The un-welded, weld metal and base metal undergone air oxidation shown less weight gain compared to salt oxidation. The XRD patterns of air oxidation are shown in **Fig** 5.43. From the XRD patterns it was observed that un-weld sample suffered corrosion in the form of intense spalling and peeling of the scale, which may be due to the formation of unprotective Fe_2O_3 oxide scale. Strong peaks of Fe_2O_3 are observed in un-welded sample and other compounds like NiO and Fe Cr2 O3 are formed which are unprotective oxides.

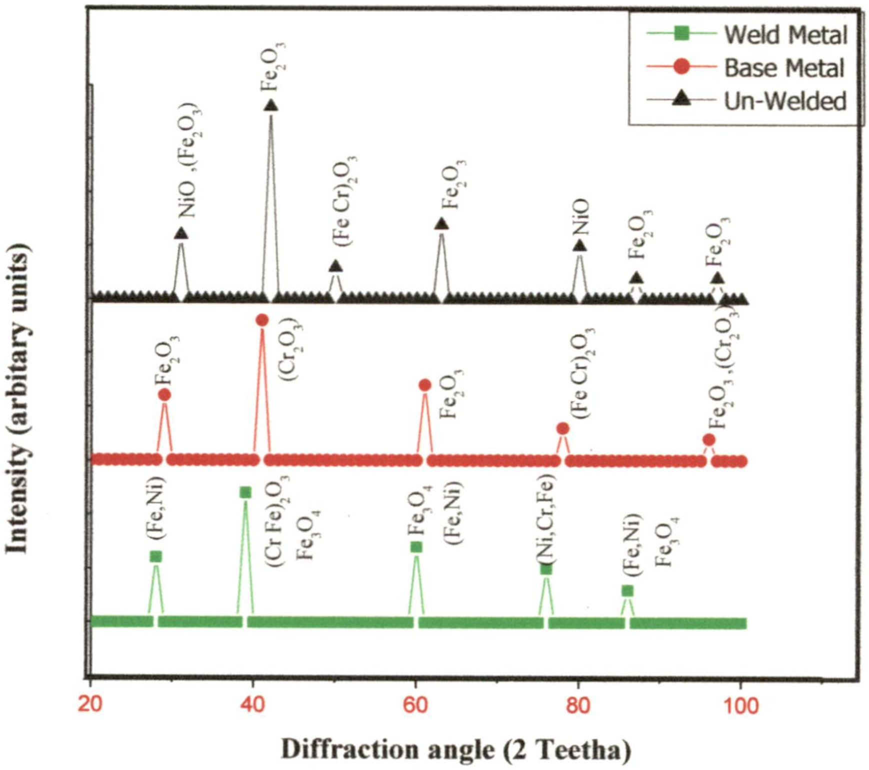


Fig.5.43: XRD patterns of Un-welded, Weld metal and Base metal in air oxidation

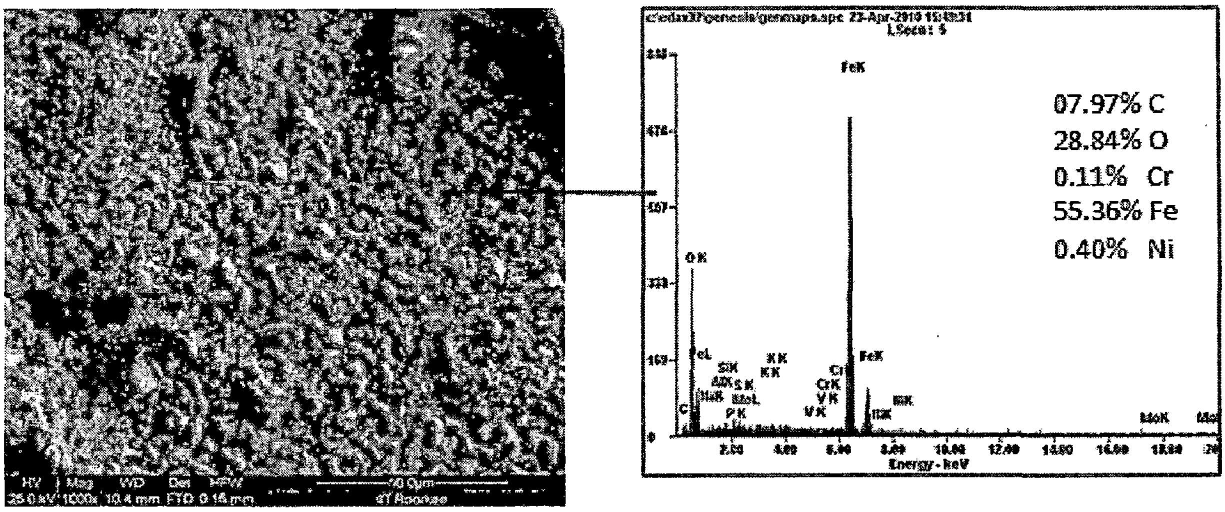
In weld metal there are no traces of Fe_2O_3 , so the weld metal had shown high resistance to hot corrosion which is confirmed by XRD analysis and weight gain measurements. Strong peaks of Fe3O4 are observed and other oxides formed in weld metal are (Fe, Ni), (Ni, Cr, Fe) and (Cr Fe)$_2$ O_3. Fe_3O_4 is a protective layer, it is having the tendency to be stable at high temperatures this layer will strongly resist the corrosion attack, So these is coated on water tubes of the boiler.

In base metal Cr_2 O_3 is observed in its phases in the oxide scale along with a few indications of Fe_2O_3 phase. Cr_2 O_3 forms protective oxides which contributed the better corrosion resistance [49].

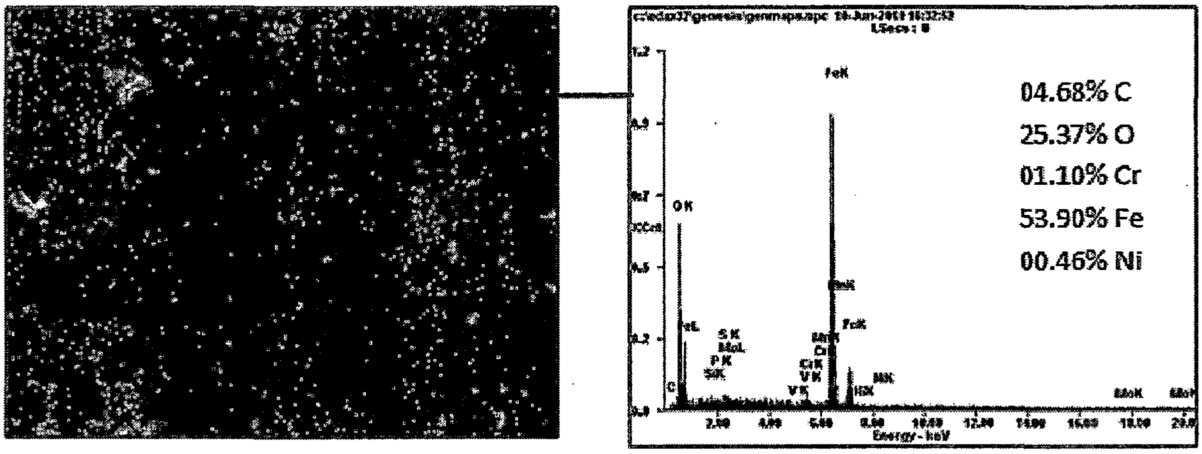
5.9. Surface scale morphology

SEM micrographs along with EDAX point analysis revels the surface morphology of the un-welded, weld metal and base metal of cr-mo boiler steel subjected to cyclic oxidation in Na_2SO_4-60%V_2O_5 (molten salt) and air environments at 900 ^{0}C for 50 cycles are shown below .

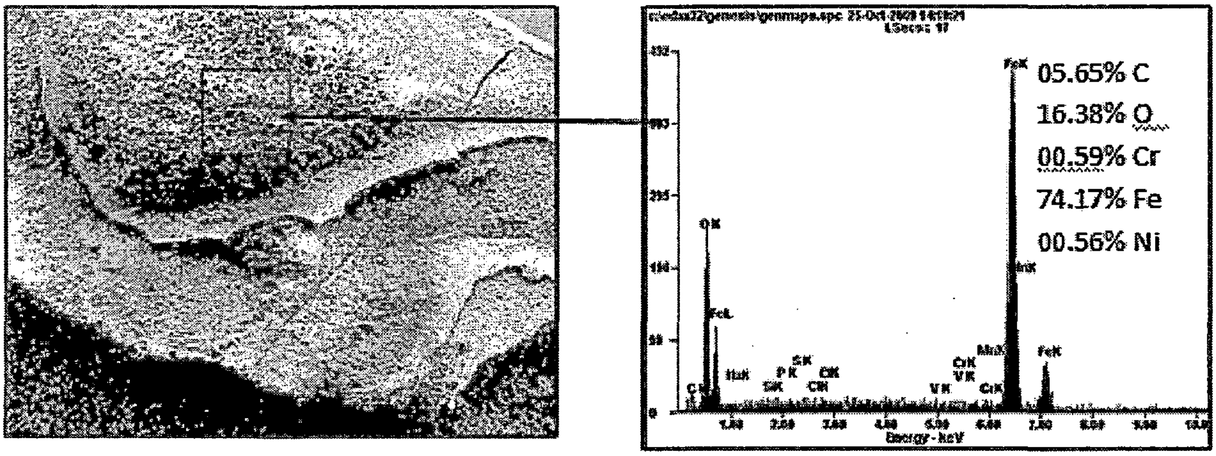
5.9.1. Surface morphology in salt oxidation



Salt Oxidation in Weld metal



Salt Oxidation in Base metal

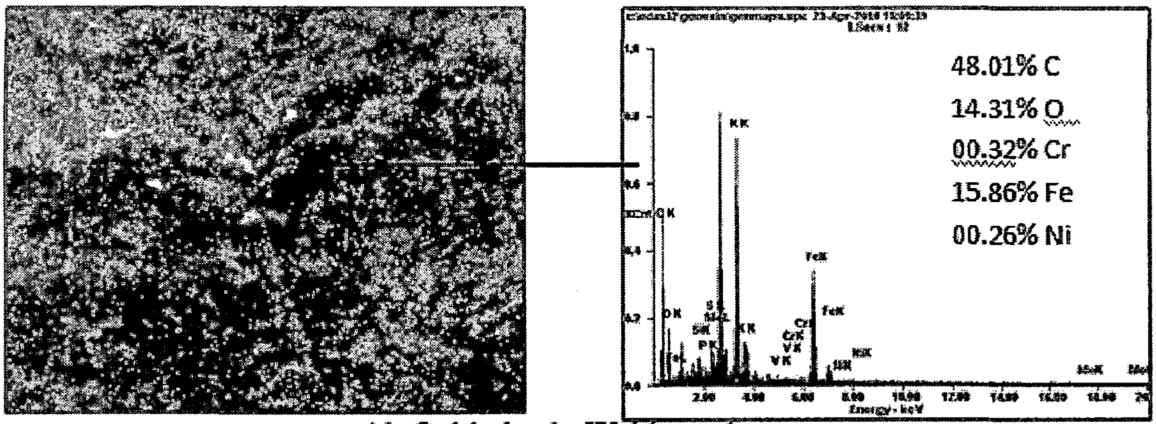


Salt oxidation in Un-welded sample

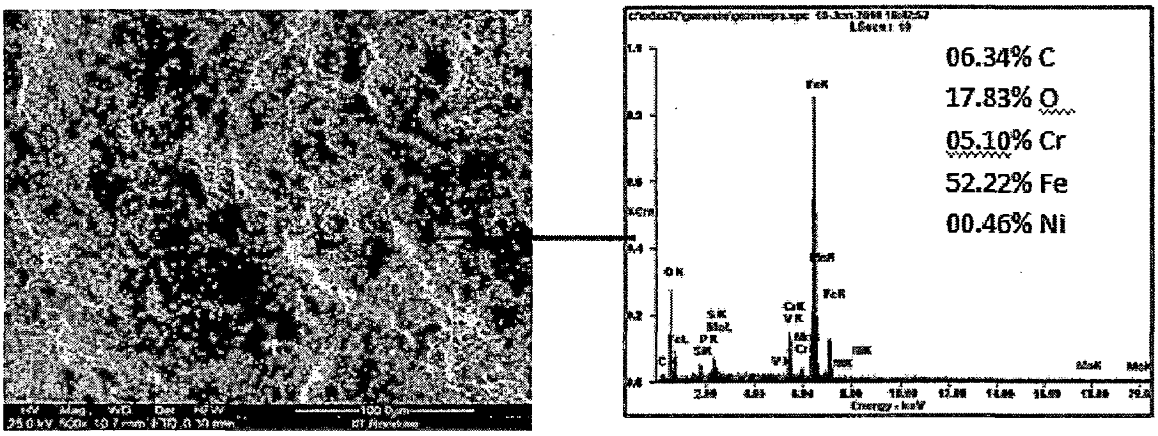
Fig.5.44: SEM micrographs in salt oxidation

SEM micrographs of specimen's undergone hot corrosion test in salt are shown above. The oxide scale in weld metal indicates the dominance of Fe and O. Small amount of nickel is also found in the scales. Oxygen percentage is more in weld metal so oxides of Fe, Ni and Cr are formed like $NiCr_2O_4$, $Cr\,Fe_2\,O_3$ $Fe\,Cr_2\,O_3$ which were confirmed by XRD analysis. In base metal chromium percentage is more so $Cr_2\,O_3$ is formed which is a protective layer.

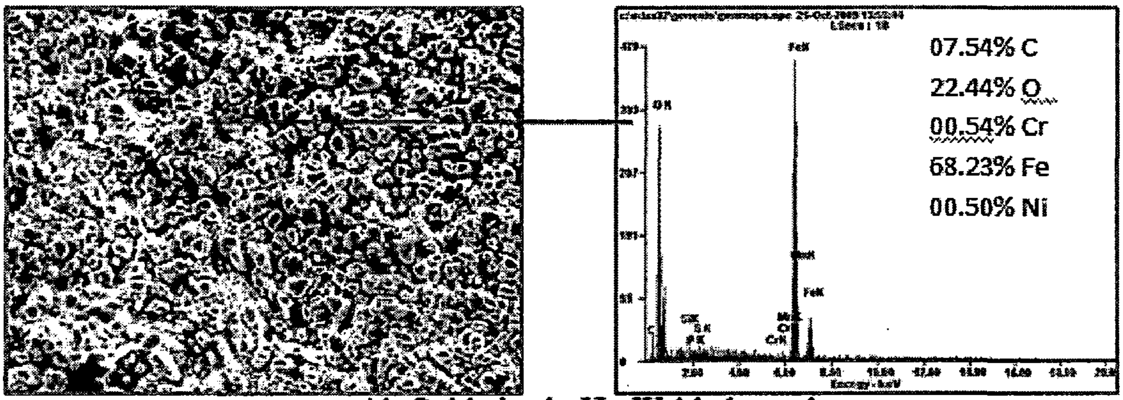
5.9.2. Surface morphology in air oxidation



Air Oxidation in Weld metal



Air Oxidation in Base metal



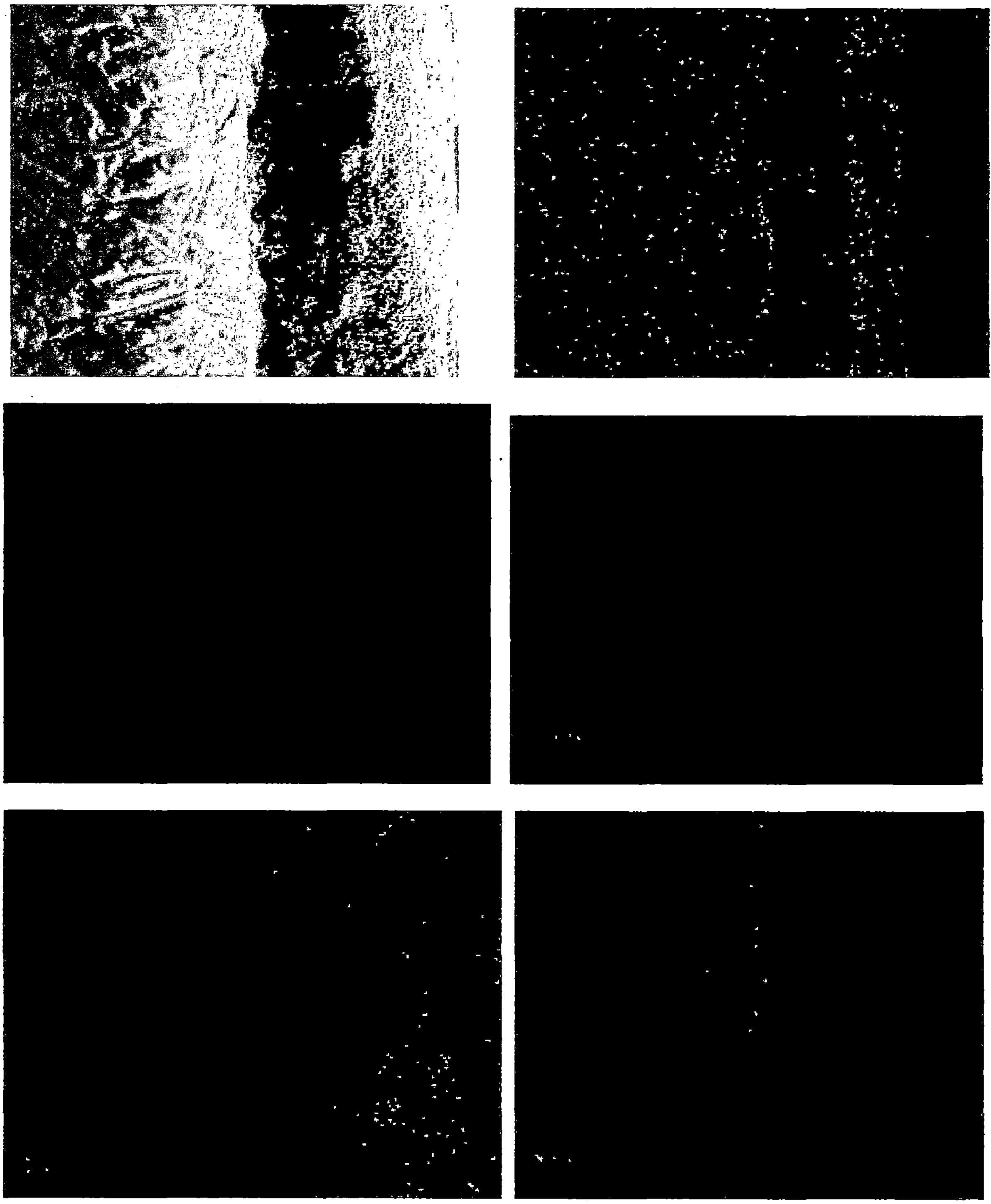
Air Oxidation in Un-Welded sample

Fig.5.45: SEM micrographs in air oxidation

5.10. X-Ray Mapping

X-Ray Mapping analysis is conducted on specimens undergone hot corrosion test in salt and air environments. X-Ray Mapping is used to determine the elemental distribution.

5.10.1. X-Ray Mapping of Weld metal in salt oxidation



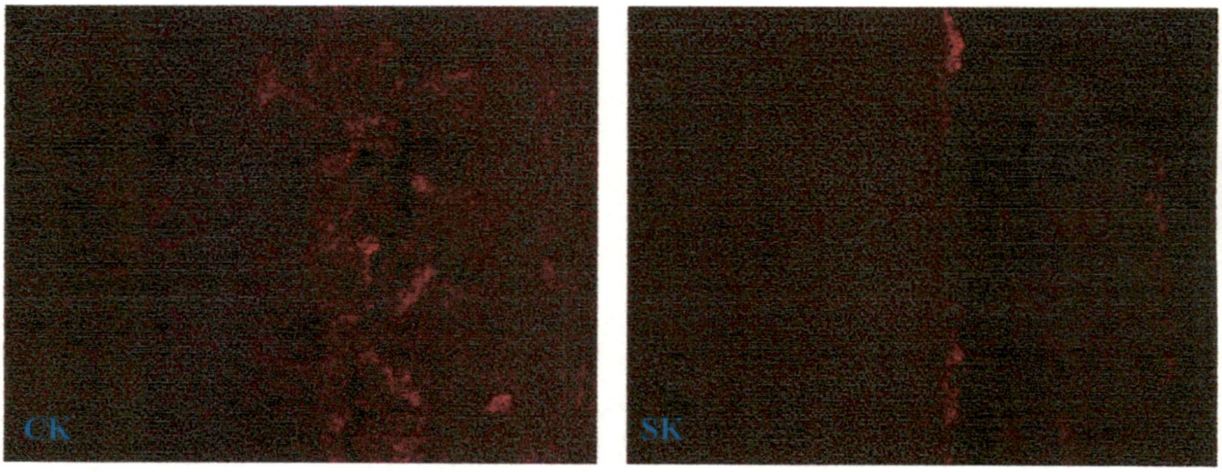
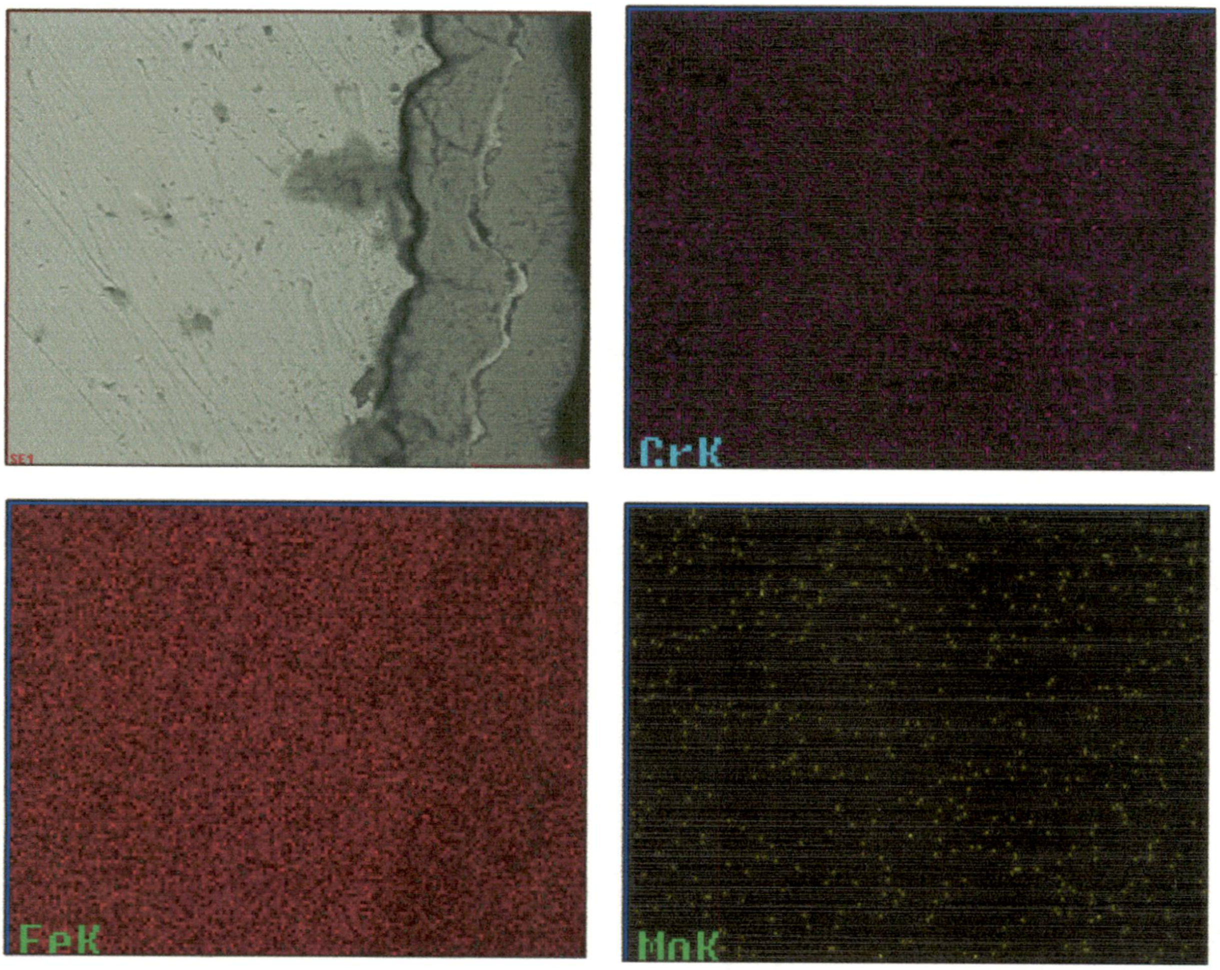


Fig.5.46: X.ray mappings of weld metal

5.10.2. X-Ray Mapping of Base metal in salt oxidation



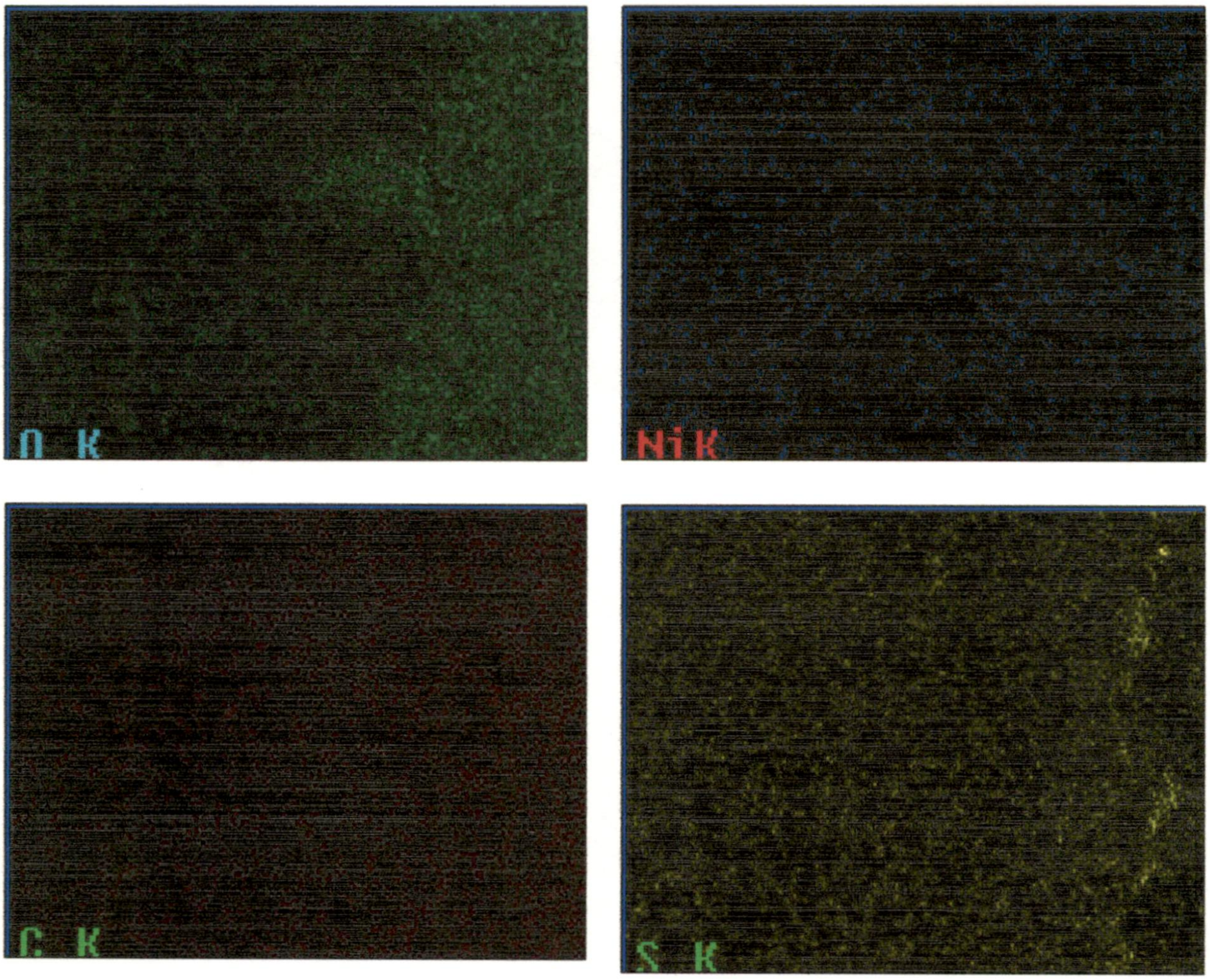
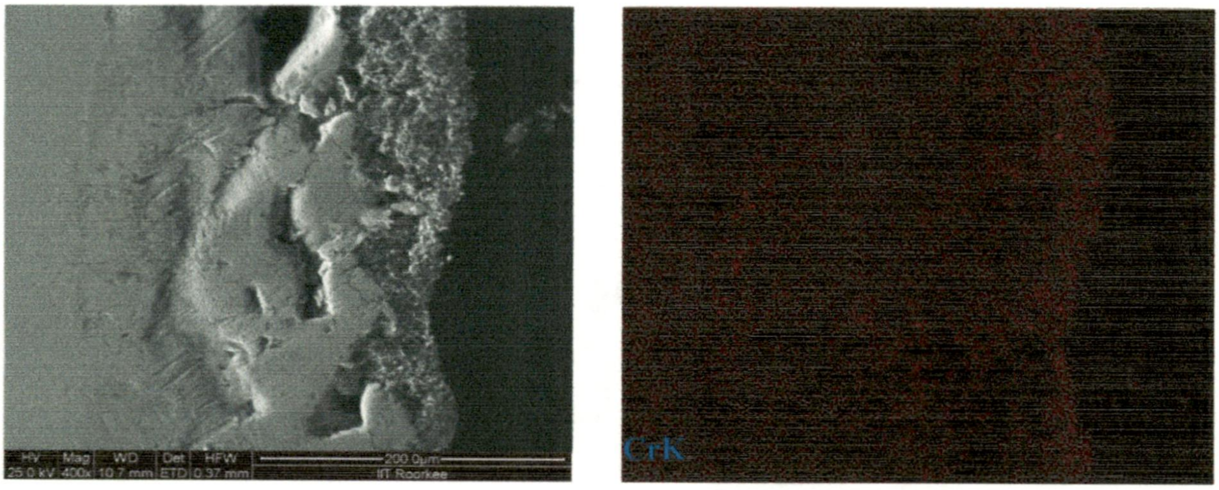


Fig.5.47: X.ray mappings of base metal

5.10.3. X-Ray Mapping of Un-welded sample in salt oxidation



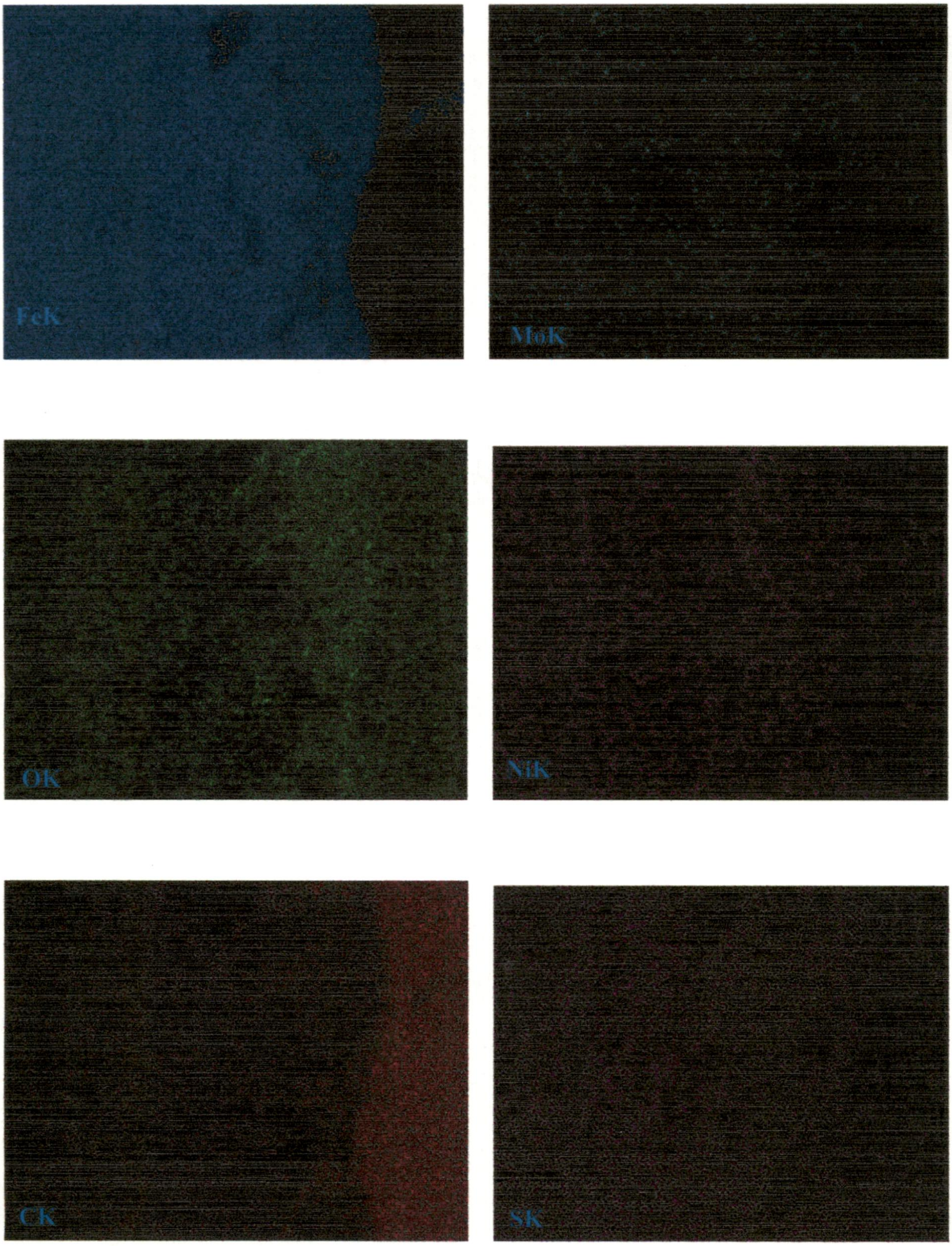


Fig.5.48: X.ray mappings of Un-welded sample

From the above X-Ray mappings it is observed that chromium (Cr), oxygen (O) and iron (Fe) is dense in weld metal and base metal. These are less in un-welded specimen. So in weld metal and base metal the oxides of Cr and Fe are formed like $(Cr Fe)_2 O_3$, $(Fe Cr)_2 O_3$, Cr_2O_3 are formed these are protective oxides which contributed the better corrosion resistance by controlling the reactants entering the oxide layer In weld metal Ni is more so Ni concentration is more in weld metal which is confirmed by X-Ray Mapping, so iron combined with Ni to form $Ni Cr_2 O_4$ and $Ni Mn_2O_4$ these helps to develop oxidation resistance as the spinel phases usually have much smaller diffusion coefficients of the ions

The corrosion resistance of un weld sample is less due to the presence of a NiO layer in the scale. During investigation, the NiO layer was observed in the oxide scale of T-22 Un-welded sample. This layer has been suggested to be loose structured by X. Wu [49], which may not be able to provide effective protection.

CHAPTER- 6

Technical problems faced in Thesis

The technical problems which were faced in thesis work are Improper Penetration and Reheat Cracking. Failure analysis is conducted and the problem is solved. The details are reported in the form of case studies in this chapter.

6.1. Improper Penetration

6.1.1. Case study: Failure of tensile specimens in a brittle manner while tensile testing.

Description: Specimens for tensile testing are prepared after welding with SMAW process and the specimens are PWHT at 0.5, 2.0, 10 and 50 hours. Transverse tensile specimens are prepared as per ASTM Standard E-8M. Tensile testing is performed in As-welded condition and PWHT condition. While testing all the specimens failed in the brittle manner, indicating a defect.

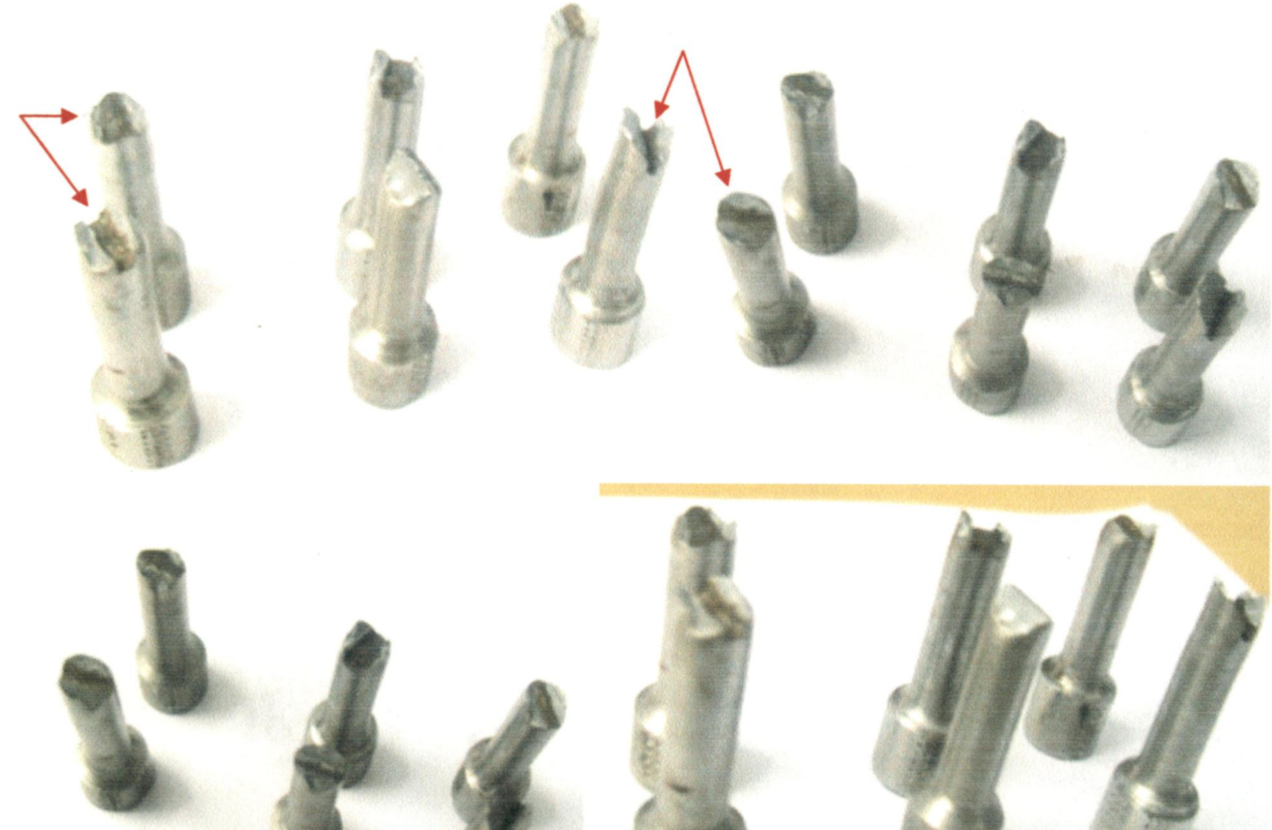


Fig.6.1: Specimens failed in a Brittle manner with 45^0 in slant direction

6.1.2. Investigation

Investigation is focused to determine the type of defect and its cause. It is concluded that the defect is improper penetration. Penetration is the distance from base plate top surface to the, maximum extent of the weld nugget. Various Causes for Incomplete Penetration in welding are:

[1] Too large root face.
[2] Root gap too small.
[3] Too small bevel angle.
[4] Less arc current.
[5] Faster arc travel speed.
[6] Too large electrode diameter.
[7] Incorrect polarity when welding with direct current.
[8] Wrongly held electrode. It should be in the centre of the joint.

The above causes are considered initially and it is concluded that the main reason of the defect is due to use of too large electrode. Diameter of electrode used is 4 mm, it is expected that the molten material is not penetrated properly. So the improper penetration had occurred. During tensile testing the fracture occurred in the Weld metal, due to the presence of defect. As it clearly observed from the figure **Fig.**6.1 there is no Necking at the fracture. The fracture surface appears smooth suggesting that the fracture was brittle

6.1.3. Microstructural Examination

Microstructural examination is focused on defect to determine its orientation. From the microstructueral examination it is concluded that molten metal is not penetrated up to the Nugget.

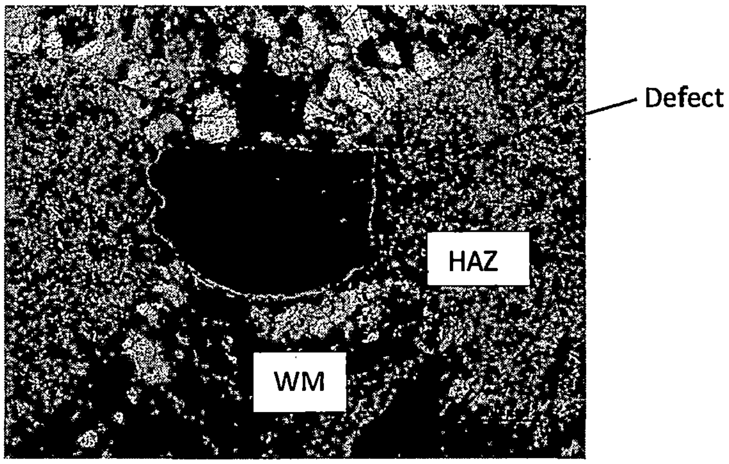


Fig.6.2 (a): Improper penetration in the weld joint.

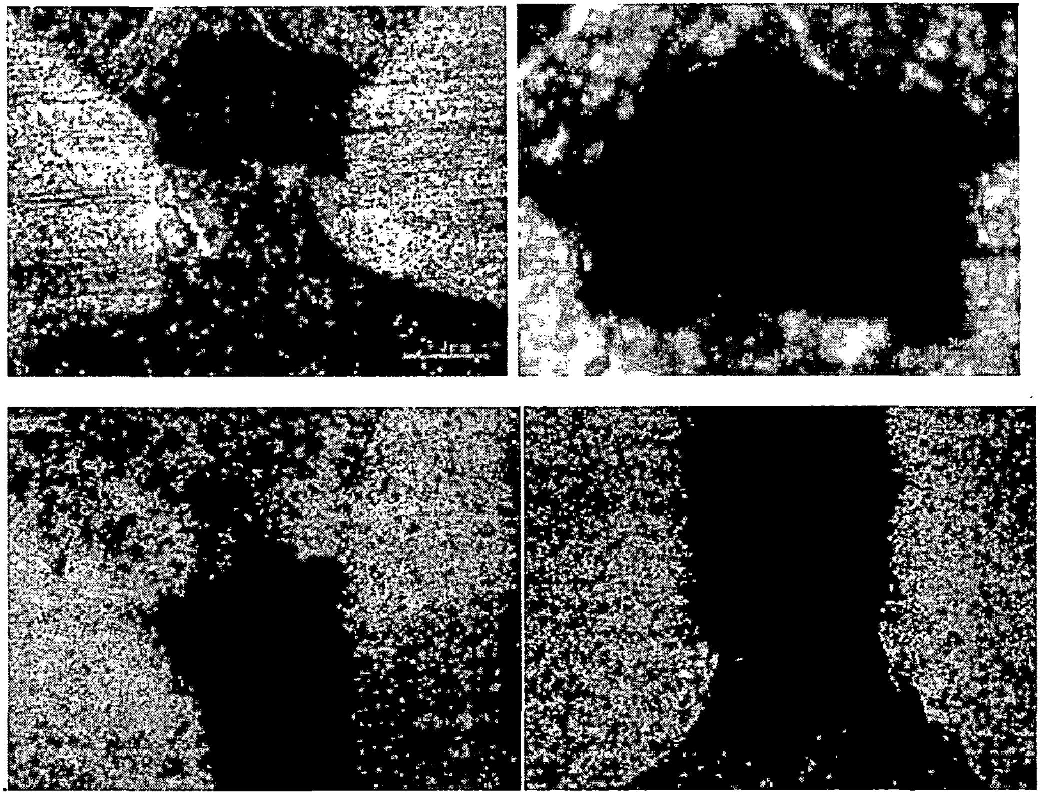


Fig.6.2.(b): Microstructures showing improper penetration

From the above results it can be concluded that due to use of large diameter electrode improper penetration occured,which causes the tensile specimens to fail in a brittle manner.

6.1.4. Preventive technique used

The problem of improper penetration is solved by welding with a small diameter electode at the Root pass, the electrode diameter selected is 3.15 mm and for filler passes 4 mm diameter electrode is used.

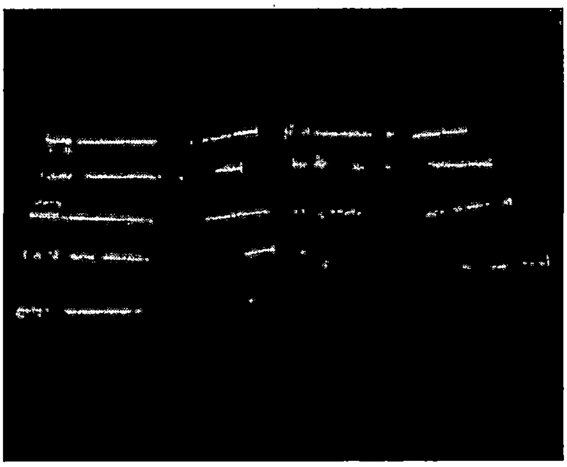


Fig.6.3: Specimens fractured in ductile manner

6.2. Reheat Cracking in Weld Joint

Reheat Cracking

Reheat cracking may occur in low alloy steels containing alloying additions of chromium and molybdenum or chromium, molybdenum and vanadium when the welded component is being subjected to post weld heat treatment, such as stress relief heat treatment, or has been subjected to high temperature service Cracking is almost exclusively found in the coarse grained regions of the heat affected zone (HAZ) beneath the weld

6.2.1. Case study: Reheat cracking is observed in 0.5 hour PWHT

Description: Specimens for Microstructural Examination are prepared by welding with SMAW process, PWHT at different soaking times (0.5, 2.0,10,50 hours) polished and etched with Nital 10%. The specimen undergone 0.5 h PWHT had shown reheat cracking in HAZ near to the interface.

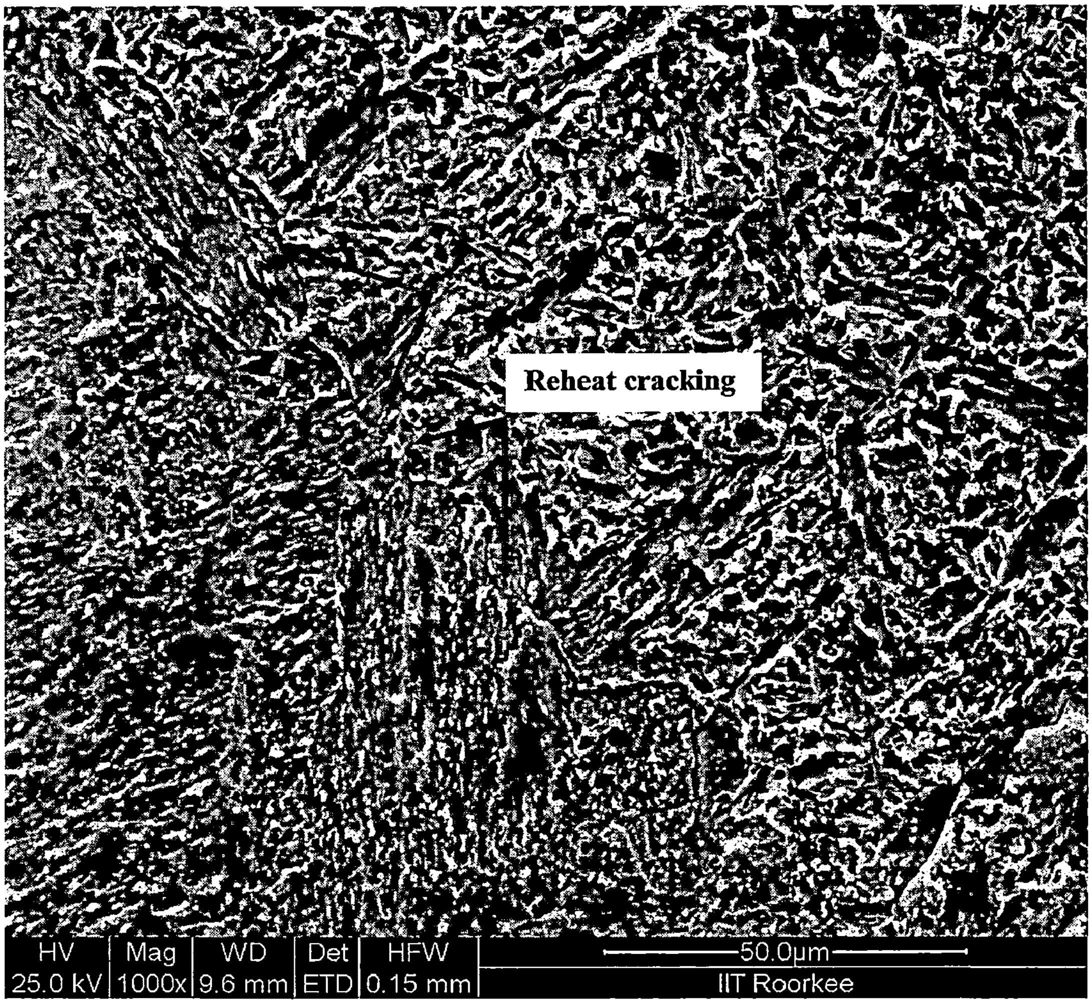


Fig.6.4: Microstructure showing reheat cracking

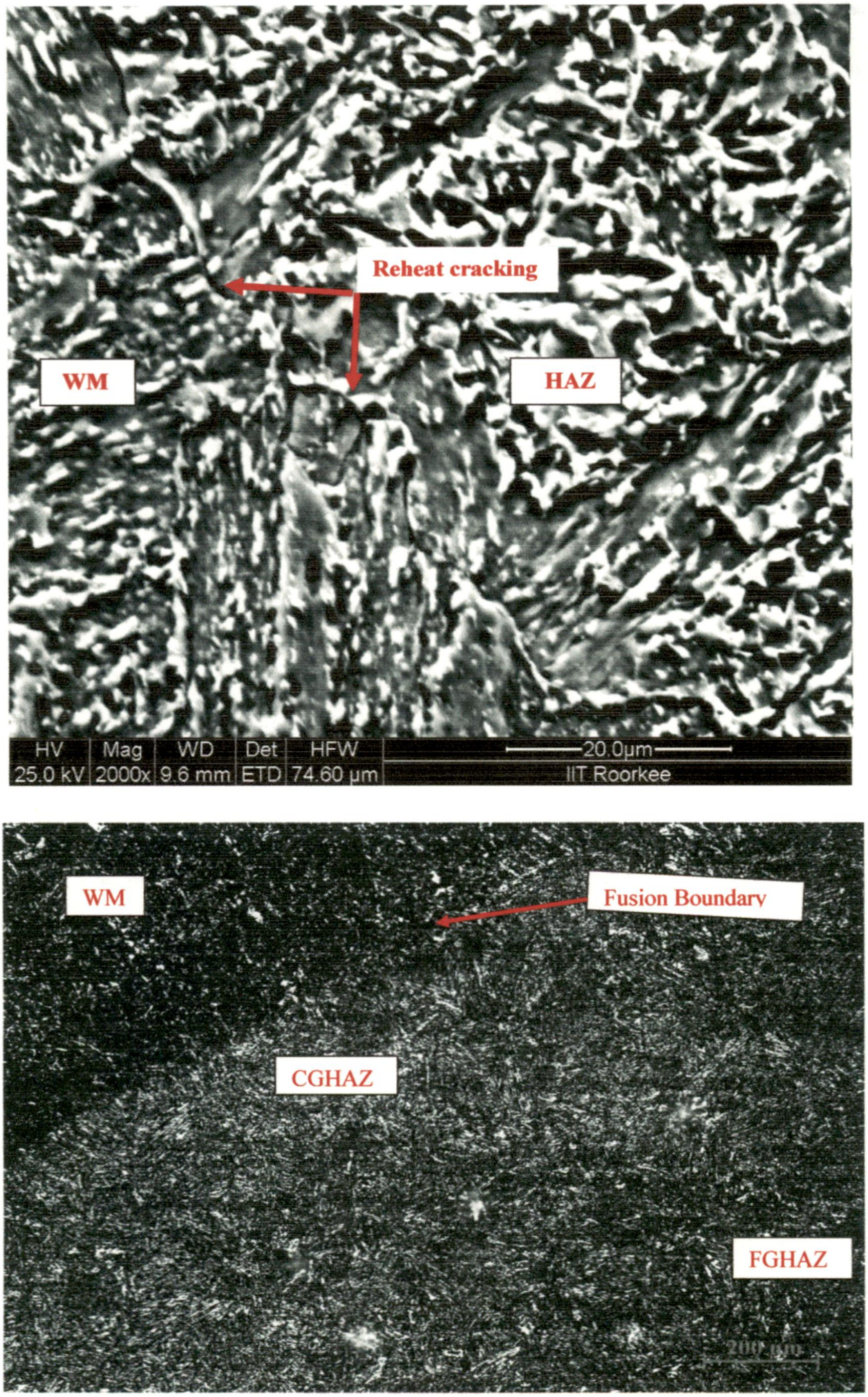


Fig.6.5: a) SEM Micrograph showing reheat cracking b) Interface in 0.5 PWHT

6.2.2. Investigation

- Investigation is carried out to find the reason why the reheat cracking is occurred in 0.5 h PWHT. And it is concluded that the cracking was present in HAZ near the weld metal interface, where coarse and columnar grains are present.

- The microstructure in weld metal consists of Fine grain bainite with small amount of carbide precipitation. M.C. Murphy [44] Reported 2.25Cr-1Mo steel derives its creep strength from the fine distribution of acicular MO_2C particles in the weld regions so there may be chance of MO_2C carbide precipitation in weld metal.

Weld metal undergone 0.5 h PWHT

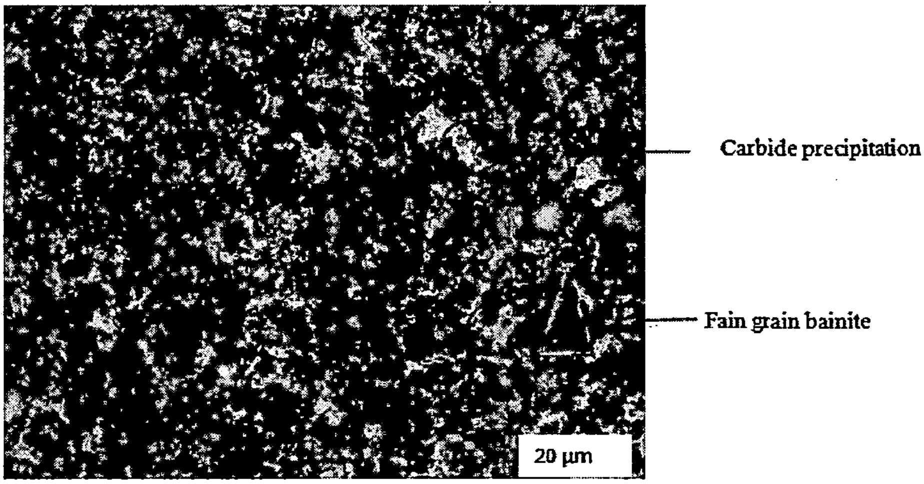


Fig.5.2: Fine grain bainite with small amount of carbide precipitation, 1000x

- The principal cause of reheat cracking is grain interior becomes strengthened by carbide precipitation, forcing the relaxation of residual stresses by creep deformation at the grain boundaries. The grain interior is having high strength when compared to coarse grain boundrys. So grain boundry tearing occurred which is called reheat cracking [24].

Conclusions

From the above results it can be concluded that 0.5 h PWHT is not suitable for Cr-Mo alloy steels, because chances of reheat cracking is more which leads to further failure of weldments.

6.2.3. Preventive technique used

- The welding procedure used to minimize the risk of reheat cracking by producing the maximum refinement of the coarse grain HAZ. This procedure is aimed to refine the coarse grained HAZ by subsequent passes.
- Two layer technique is used to prevent the reheat cracking which is reported by N.Bailey [24] as shown in the **Fig.**6.5. In this technique two electrodes of diameter 3.15mm and 4mm are used to refine the HAZ.
- Refinement of the HAZ can be promoted by first buttering the surface of the susceptible plate with a thin weld metal layer using a small diameter (3.15mm) electrode. The joint is then completed using a larger diameter (4 mm) electrode, which is intended to generate sufficient heat to refine any remaining coarse grained HAZ under the buttered layer[24].

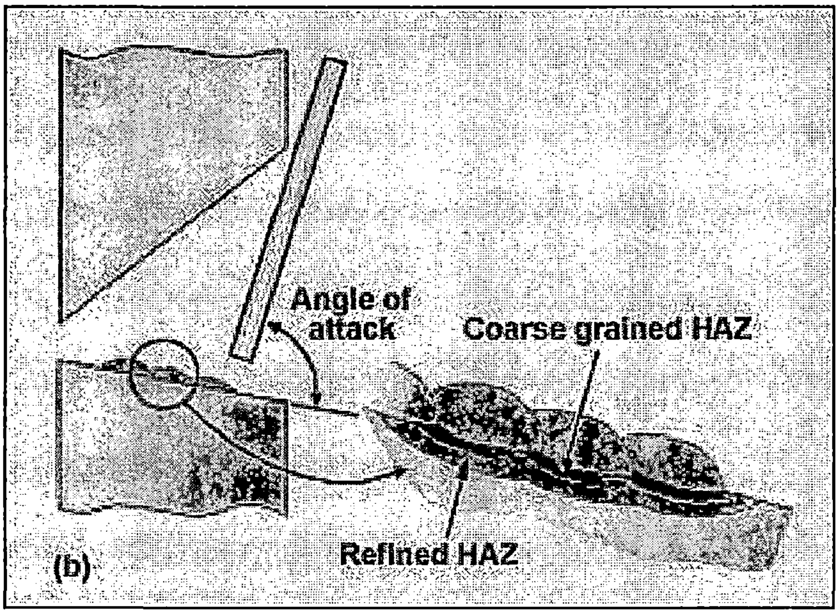


Fig.6.5 Two layer technique for HAZ refinement

CHAPTER- 7

CONCLUSIONS

PWHT is used as a tool to impart mechanical and microstructural properties and to improve corrosion resistance in weldments. PWHT is carried out for 0.5, 2.0,10 and 50 hours. Mechanical characterization and microstructural study is done to determine the effect in As-welded and PWHT conditions, from the investigations it concluded that:

1) The microstructure of the annealed BM at different soaking times revealed a ferrite matrix with uniform dispersion of carbides. In 50 h heat treatment due to long exposure to high temperature spheroidization and grain coarsening was taken place in base metal. In weld metal carbide precipitation is observed in as-welded and 0.5 h, PWHT. These carbides are diffused in 2 h heat treatment. In HAZ All the CGHAZ and FGHAZ were composed of a bainitic structure with lath morphology at all PWHTs.

2) The microstructures in various regions of the weld joint like weld metal, HAZ, base metal and interface region showed major changes in as-welded and after PWHT conditions when examined under optical microscope and SEM. These major changes had further shown changes in the mechanical and corrosion resistance properties, which can be concluded as:

- lath martensite is observed in HAZ **Fig.**5.6 in As-welded condition the Hardness observed is (348 VHN), after PWHT at 2.0 h a mixed structure **Fig.**5.8 consist of polygonal/ Widmanstatten/ upper bainitic structure is formed in the microstructure with Micro Hardness of 190 VHN. This structure is very coarse, so there is sharp drop in hardness, when compared to all PWHT'S.
- The weld metal in As-welded condition shows lath bainite with fine distribution of carbides in the ferrite grains, so the impact resistance is very less and Hardness is maximum (300 VHN). After the specimen undergone PWHT for 10 h, the hardness was dropped to (209 VHN) and impact resistance is maximum. This is due to the presence of due to the presence of refined grains in weld metal and HAZ as shown in the **Fig.**5.24 the microstructure consists of fine dendritic structure.

- When the Hot Corrosion test is done on Un-welded sample and PWHT sample's like Weld metal and Base metal. The Un-welded sample shown less resistance to Hot Corrosion compared to PWHT samples.

3) By considering the average hardness values it can be concluded that as the heat treatment time is increasing the hardness is reducing in Base metal and Weld metal. But in HAZ minimum hardness of 190 VHN is observed in 2 h of PWHT due to the presence of coarse grains as shown in **Fig.**5.8.

4) The overall peak hardness is observed in HAZ portion, next to that is weld metal and the minimum in base metal as shown in **Fig.**5.23.

5) When tensile test is conducted in transverse direction of the weldment and in longitudinal direction in base metal, the maximum ultimate tensile strength and yield strength are observed in specimens undergone 2 h PWHT. It is also observed that as the UTS and YS reduced as the PWHT time goes on increasing beyond 2 h of PWHT as shown in **Fig.**5.26.

6) It is also observed that percentage of elongation and impact resistance is increased in 10h PWHT. This is due to the presence of refined grains in weld metal and HAZ as shown in the **Fig.**5.24. From this result it can be expected that there is another heat treatment time which lies within 2 h and 10 h which would improve tensile properties better than 2 h PWHT.

7) It is concluded that Impact resistance is very less in As-welded condition. The impact resistance increased after PWHT. From the **Fig.**5.31 it is also concluded that 10 h PWHT had shown higher impact resistance when tested in high temperatures and sub zero temperatures (0^0C,-60^0C and -90^0C).For a given temperature, the impact resistance decreased with the increase of the heat treatment longer than 10 h. It means ductile-brittle transition temperature increased with the time of PWHT.

8) The reasons for the increase in Impact resistance in 10 h PWHT is due to the presence of refined grains in weld metal and HAZ as shown in the **Fig.**5.24 the microstructure consists of fine dendritic structure. Fair Child [47] had reported that high toughness and smaller transition temperature in steels is achieved with finer microstructure and a small impurity content.

9) From the above observations it is concluded that 10 h PWHT is the favourable heat treatment which gives higher impact resistance in higher temperatures and sub zero temperatures. Samples PWHT at 50 h shown high impact resistance when tested at higher temperatures. From this result it is expected that there is an another heat treatment which is in between 10h and 50 h which will improve impact resistance better than 10h.

10) From the E-DAX analysis Oxygen is observed in 50 h heat treatment, its wt % is 2.07. Sindo Kou [45] reported that presence of oxygen in weld may increase the toughness and reduce its hardness. This is the reason why 50 h PWHT sample has high impact resistance **Fig.**5.31 when tested in high temperatures and low hardness **Fig.**5.23 compared to other PWHT samples.

11) From the **Fig.**6.1 it can be concluded that the transverse tensile specimen fractured in the weld metal in a brittle manner, due to the presence of defect improper penetration. It is also concluded that this defect can be overcome by using a small diameter electrode at the root passes.

12) From **Fig.**6.5 it is can be concluded that 0.5 h PWHT is not suitable for Cr-Mo alloy steels, because there are more chances of reheat cracking in the coarse HAZ. It is also concluded that two layer technique is used as a preventive technique to overcome reheat cracking problem.

Based on the overall weight gain after 50 cycles in air and an aggressive Na_2SO_4-60%V_2O_5 (molten salt) environment at 900^0C temperature ,the oxidation resistance of weld metal, base metal and un-welded sample are studied in the present investigation and it is conclude that:

13) Intense spalling was observed throughout the experimentation in un-welded specimen in air and salt environment. All the specimens undergone hot corrosion test in air and salt environment followed the parabolic behaviour but the weld metal in air oxidation shown linear rate of weight gain.

14) It is also concluded that weld metal has shown high resistance to corrosion as compared to base metal and un-weld samples. The corrosion rate (in terms of weight gain) of un-welded specimen, weld metal and base metal has been found in the following order Un-welded > Base metal > Weld metal.

15) From the oxide scale measurement it is concluded that the un-welded sample is having more thickness of oxide layer indicating that the sample is highly affected by hot corrosion.

16) XRD analysis had shown that protective oxides like $(Cr\ Fe)_2\ O_3$, $(Fe\ Cr)_2\ O_3$, Cr_2O_3, $(Ni\ Cr_2\ O_4)$ and $(Ni\ Mn_2O_4)$ are formed in weld metal and base metal in air and salt oxidation but in un-welded sample oxides like Fe_2O_3, NiO and CrS are formed which are unprotective oxides so corrosion effect is more in Un-welded sample.

17) X-ray mapping shown that elements like Cr,Fe,Ni are dense in weld metal and base metal so the protective oxides of Cr, Fe and Ni are formed and so these specimens had shown better corrosion resistance when compared to un-weld sample.

18) The un-welded sample suffered corrosion in the form of intense spalling and peeling of the scale, which may be due to the formation of unprotective Fe_2O_3 oxide.

19) The Weld metal has shown high resistance to corrosion because the electrode used is a austenitic stainless steel electrode consisting of Chromium of 2.25% .Chromium adds to the overall resistance through a passivation process by forming a complex spinel-type (Fe, Ni) O and $(Fe, Cr)_2O_3$ passive film This complex produces a coherent, adherent insulating and regenerating chromium oxide protective film on the metal surface.

20) The main reason why the Weld metal and Base metal shown higher corrosion resistance when compared to Un-welded samples is after welding these samples under gone PWHT (Annealing) for 10 h. So due to the effect of heat treatment corrosion resistance is increased. Heat treatment dissolves chromium carbides, converts delta ferrite to austenite in equilibrium phase-fractions, spheroidizes the remaining ferrite, thus imparting corrosion resistance [53].

FUTURE SCOPE FOR WORK

The present work gives a new direction to further improvement of mechanical, microstructural properties and increasing resistance to Hot Corrosion in Boilers, Pressure vessels, Turbine parts, Diesel engines etc, in which Cr-Mo alloy steels are used. Following suggestions may be considered for the scope for future work.

- Similar investigations can be focused on other boiler steels to determine the favourable PWHT time which gives better mechanical and microstructural properties.
- It can be concluded from the results that PWHT can improve the mechanical and microstructural properties and resistance to corrosion. It can also be concluded from tensile and charpy test that there can be another PWHT time which lies in between 2 h and 10 h. Hence further investigation in this direction has to be carried out which could give better mechanical properties better than 2h PWHT.
- Similar investigations can be focused on Hot Corrosion studies by comparing the different boiler Grade materials.
- Further studies can be made to improve resistance to hot corrosion by applying coatings like high–chromium, nickel chromium, NiCrAlY, Ni–20Cr, NiCrAlY-Al_2O_3 coatings on the super heater tubes used in boilers.

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Behaviour of Cr-Mo Un-welded sample in salt at 900° C

Sample = Cr-Mo alloy steel in un-welded condition

Boat weight after cleaning with methanol = 46.727 gms

Sample size = 21.19 x 16.15 x 2.92 mm

Salt used = 40%wt Na_2SO_4 + 60%wt V_2O_5

Initial Sample weight = 8.382 gms

Sample weight after salt apply = 8.417 gms

Boat+ sample after pre heating = 55.152 gms

Furnace used = Tubular furnace

Controller of furnace = PID Temperature controll

Cycle of 1 hr heating at 900°C and then cooling to 20 min and taking weight along with boat Initially keep sample and boat in oven for 2hr 30 min at 250° C after application of salt and then place it in furnace

Table.5.7: Weight gain observations of Un-welded sample in salt

No. of Cycles	Sample + Boat wt (gms)	Wt. gain (gms)	Remarks
1	55.251	0.099	Small tong sound while cooling. Lusture disappeared while salt was white in colour
2	55.377	0.225	Sample colour changed to dark greyish & salt colour changed to reddish black
3	55.470	0.318	Salt colour changed to black and it formed a layer & cracked . While cooling small noise occurred
4	55.620	0.468	Tong sound occurred and bottom layer separates
5	55.730	0.578	…………………………………
6	55.860	0.708	Salt was still intact on surface of sample
7	55.974	0.822	Sample while cooling no sound occurred and no of layers formed on the substrate are increasing
8	56.108	0.956	Sample while cooling no sound occurred
9	56.258	1.106	From downside a small patch of oxide layer formed separated from sample & other part of it peeled out & was hanging
10	56.380	1.228	Sample while cooling small cracking sound occurred
11	56.454	1.302	Sample while cooling small cracking sound occurred

12	56.584	1.432	Sample while cooling small cracking sound occurred
13	56.679	1.527	Oxide layer remained rough
14	56.755	1.603	Oxide layer remained rough
15	56.835	1.683	Small patch of oxide got separated
16	56.942	1.790	Oxide layer which was formed got cracked from all side & while cooling sound occurred
17	57.026	1.874	While cooling sound occurred
18	57.074	1.922	The oxide layer cracked from 2 side i.e. from up & down side & while cooling sound occurred
19	57.203	2.051	A small portion of oxide got removed and small cooling sound occurred
20	57.284	2.132	Oxide layer cracked from various point but there was no cooling sound
21	57.347	2.195	Oxide layer wholly cracked & separated from surface but it did not got wholly separated as it was attached from bottom edge & while cooling no sound occurred
22	57.404	2.252	While cooling small cracking sound occurred
23	57.500	2.348	While cooling small cracking sound occurred
24	57.568	2.416	Thin & medium size oxide layer got seperated from upper surface & some in form of small pieces & while cooling small cracking sound occurred
25	57.639	2.487	Oxide layer got cracked from side also & tiny oxide pieces got separated
26	57.683	2.531	Small cracking sound observed while cooling
27	57.853	2.701	Small cracking sound observed while cooling
28	57.804	2.652	Little bit more oxide layer got removed horizontally
29	57.840	2.688	Oxide scale is peeled off from the surface
30	57.870	2.718	Small cracking sound observed while cooling
31	57.887	2.735	Little bit more oxide layer got removed at the edges
32	57.901	2.749	Small cracking sound observed while cooling
33	57.934	2.782	Small cracking sound observed while cooling
34	57.970	2.818	
35	57.992	2.840	....
36	58.015	2.863	....
37	58.026	2.874	....

38	58.035	2.883	No sound occurred while cooling.
39	58.046	2.894	No sound occurred while cooling.
40	58.062	2.910	Less oxide layer is removed
41	58.070	2.918	No sound occurred while cooling.
42	58.079	2.927	No sound occurred while cooling.
43	58.083	2.931	No sound occurred while cooling.
44	58.089	2.937	No sound occurred while cooling.
45	58.100	2.948	....
46	58.113	2.961	....
47	58.128	2.976	....
48	58.137	2.985	....
49	58.140	2.988	....
50	58.146	2.994	Layers separated and fall in boat

Behaviour of Cr-Mo steel in Un-welded condition in air at 900^0 C

Sample = Cr-Mo steel in un weld condition

Furnace used = Tubular furnace

Controller of furnace = PID Temperature controller

Sample size = 21.19 x 16.15 x 2.92 mm

Sample + Boat weight = 57.340 gms

Boat weight after heating = 49.785 gms

Cycle of 1 hr heating at 9000 C and then cooling to 20 minutes and taking weight along with boat.

Table.5.8: Weight gain observations of Un-welded sample in air

No of cycles	**Sample+ Boat wt (gms)**	**Wt. gain (gms)**	**Remarks**
1	57.400	0.060	Bubble layer formed on surface .Tong sound while cooling
2	57.451	0.111	sample colour turned to dark greyish .Cracks on bubbled surface layer
3	57.489	0.149	Cracking sound observed while cooling, small pores were observed
4	57.530	0.190	Oxide colour changed to black

5	57.564	0.224	Excess black carbon deposition at corners but bottom portion is still greyish
6	57.593	0.253	Thick carbon layer cracked & splashed separating with noise but at lower side small patches of oxide layer fallen out
7	57.618	0.278	While cooling small noise occurred & black oxide layer separated with sound , then very dark black oxide layer formed on surface
8	57.645	0.305	While cooling small noise occurred & black oxide layer separated with sound
9	57.668	0.328	A very thick layer of black colour oxide exploded with noise & while cooling small noise occurred
10	57.693	0.353	All oxide layer was separated
11	57.714	0.374	While cooling whole oxide layer formed and splashed with cling sound and another dark blackish oxide layer adherent to surface was observed, from corners also oxide layer splashed
12	57.745	0.405	On both surface rough thick black oxide formed but from 2 big edges oxide layer peeled out but still intact at edge
13	57.771	0.431	Black oxide at corner side was observed latter on which exploded
14	57.810	0.470	....
15	57.834	0.494	While cooling small noise occurred
16	57.870	0.530	Side oxide layer got removed while cooling sound was observed
17	57.892	0.552	....
18	57.916	0.576	From various places on both surface oxide layer seperated by explosion & whole surface observed as very rough
19	57.946	0.606	Very thick layer of black oxide got seperated but it remained overlying on surface as it was not detached from bottom side
20	57.974	0.634	Thick oxide layer formed separated from all side completely. Oxide layer formed at edge side was thick as compared to big surface
21	58.008	0.668	New shabby oxide layer was formed & the previous oxide layer of edge got completely removed
22	58.199	0.859	Shabby layer formed got removed from most of the upper surface & then new bubbled oxide layer was formed .Little bit more squeezing sound occurred while cooling
23	58.240	0.900	The second layer which was which was formed also got cracked & got removed from various point
24	58.272	0.932	Oxide layer got seperated with large sound & on other hand a blackish thin layer of oxide was present from before only
25	58.308	0.968	All oxide layer was removed & later a black colored oxide was formed for 3-4 mm from top which peeled of later for some patch & other part was grayish oxide
26	58.326	0.986	The new oxide layer which was formed also got exploded thus leading to small oxide fragments
27	58.347	1.007	For a small portion plane grayish oxide layer formed & rest remained shabby & after 5 min the plane oxide formed also

			got seperated from surface of substrate
28	58.376	1.036	Oxide layer from side of more thickness were seperated
29	58.404	1.064	Loud noise occurred and oxide got detached from corner side
30	58.418	1.078	Loud noise occurred and oxide layer explode & seperated
31	58.447	1.107	While cooling sound was observed & a thin layer of oxide formed & cracked
32	58.459	1.119	Oxide layer got seperated by mild explosion
33	58.481	1.141	Oxide layer got seperated very shabbly in nature
34	58.499	1.159	While cooling sound occurred & oxide layer got seperated from surface
35	58.520	1.180	While cooling sound occurred & oxide layer got seperated from surface
36	58.507	1.167	Oxide layer got removed from side also
37	58.529	1.189	....
38	58.549	1.209	Top layer fall down and new layers got exposed to atmosphere
39	58.571	1.231	....
40	58.593	1.253	....
41	58.630	1.290	....
42	58.653	1.313	....
43	58.684	1.344	....
44	58.742	1.402	A voice occurred as if water evaporated very rapidly (squeezing voice)
45	58.758	1.418	....
46	58.794	1.454	....
47	58.824	1.484	Metallic sound occurred while cooling
48	58.849	1.509	....
49	58.869	1.529	....
50	58.909	1.569	....

Behaviour of Cr-Mo Weld metal in salt at 900^0 C

Sample = Cr-Mo alloy steel in weld and Heat Treated condition

Furnace used =tubular furnace

Controller of furnace = PID Temperature controller

Sample dimensions = 20.44 x 15.43 x 5.08 mm

Sample weight = 12.189 gms

Sample weight after salt coating = 12.228 gms

Boat +sample weight after pre heating =59.288 gms

Salt applied = 40 % Na_2So_4-60%V_2O_5

Cycle of 1 hr heating at 900°C and then cooling to 20 min and taking weight along with boat Initially keep sample and boat in oven for 2hr 30 min at 250° C after application of salt and then place it in furnace

Table.5.9: Weight gain observations of weld metal sample in salt

No. of Cycles	Sample + Boat wt (gms)	Wt. gain (gms)	Remarks
1	59.367	0.079	Small tong sound while cooling. Salt was appeared in white colour
2	59.405	0.117	Sample colour changed to dark greyish & salt colour changed to reddish black
3	59.506	0.218	Sample colour changed to dark greenish and salt colour to reddish
4	59.538	0.25	Tong sound occurred and bottom layer separates
5	59.567	0.279	....
6	59.613	0.325	Salt was still intact on surface of sample
7	59.632	0.344	....
8	59.689	0.401	Sample while cooling no sound occurred
9	59.707	0.419	From downside a small patch of oxide layer formed separated from sample & other part of it peeled out & was hanging
10	59.752	0.464	Sample while cooling small cracking sound occurred
11	59.792	0.504	....
12	59.900	0.612	Sample while cooling small cracking sound occurred
13	59.939	0.651	Tong sound while cooling, more scale fall in boat
14	59.973	0.685	Oxide layer remained rough
15	60.003	0.715	Small patch of oxide got separated
16	60.031	0.743	Oxide layer which was formed got cracked from all side & while cooling sound occurred
17	60.067	0.779	While cooling sound occurred
18	60.078	0.79	The oxide layer cracked from 2 side i.e. from up & down side & while cooling sound occurred
19	60.125	0.837	A small portion of oxide got removed and small cooling sound occurred
20	60.147	0.859	Oxide layer cracked from various point but there was no cooling sound
21	60.185	0.897	Oxide layer wholly cracked & separated from surface but it did not got wholly separated as it was attached from bottom edge & while cooling no sound occurred
22	60.202	0.914	While cooling small cracking sound occurred
23	60.245	0.957	While cooling small cracking sound occurred
24	60.249	0.961	Thin & medium size oxide layer got separated from upper surface

			& some in form of small pieces & while cooling small cracking sound occurred
25	60.274	0.986	Oxide layer got cracked from side also & tiny oxide pieces got separated
26	60.319	1.031	Small cracking sound observed while cooling
27	60.337	1.049	Small cracking sound observed while cooling
28	60.352	1.064	Little bit more oxide layer got removed horizontally
29	60.363	1.075	Small cracking sound observed while cooling
30	60.420	1.132	Small cracking sound observed while cooling
31	60.460	1.172	Small cracking sound observed while cooling
32	60.478	1.19	Small cracking sound observed while cooling
33	60.488	1.2	Small cracking sound observed while cooling
34	60.532	1.244	Small cracking sound observed while cooling
35	60.551	1.263	....
36	60.680	1.392	....
37	60.734	1.446	....
38	60.770	1.482	No sound occurred while cooling.
39	60.780	1.492	No sound occurred while cooling.
40	60.805	1.517	No sound occurred while cooling.
41	60.819	1.531	No sound occurred while cooling.
42	60.837	1.549	No sound occurred while cooling.
43	60.853	1.565	No sound occurred while cooling.
44	60.869	1.581	No sound occurred while cooling.
45	60.906	1.618	....
46	60.914	1.626	....
47	60.918	1.63	....
48	60.987	1.699	....
49	61.064	1.776	....
50	61.142	1.854	Layers separated and fall in boat

Behaviour of Cr-Mo weld metal in air at 900^0 C

Sample = Cr-Mo alloy steel in weld and heat treated condition

Furnace used = tubular furnace

Controller of furnace = PID Temperature controller

Sample size = 21.41 x 15.10 x 5.28 mm

Sample weight = 12.835 gms

Boat + sample weight = 62.472 gms

Cycle of 1 hr heating at 9000 C and then cooling to 20 minutes and taking weight along with boat.

Table.5.10: Weight gain observations of weld metal sample in air

No of cycles	Sample+Boat wt (gms)	Wt. gain (gms)	Remarks
1	62.505	0.033	Tong sound while cooling, upper layer is removed in form of powder
2	62.521	0.049	sample colour turned to dark greyish .Cracks on bubbled surface layer
3	62.534	0.062	Cracking sound observed while cooling, small pores were observed
4	62.584	0.112	Oxide colour changed to black
5	62.613	0.141	....
6	62.630	0.158	Thick carbon layer cracked & splashed separating with noise but at lower side small patches of oxide layer fallen out
7	62.640	0.168	....
8	62.657	0.185	While cooling small noise occurred & black oxide layer separated with sound
9	62.620	0.148	A very thick layer of black colour oxide exploded with noise & while cooling small noise occurred
10	62.606	0.134	All oxide layer was separated
11	62.609	0.137	While cooling whole oxide layer formed and splashed with cling sound and another dark blakish oxide layer adherant to surface was observed, from corners also oxide layer splashed
12	62.650	0.178	
13	62.660	0.188	Black oxide at corner side was observed latter on which exploded
14	62.669	0.197	....
15	62.677	0.205	While cooling small noise occurred
16	62.684	0.212	Side oxide layer got removed while cooling sound was observed
17	62.694	0.222	....
18	62.675	0.203	From various places on both surface oxide layer seperated by explosion & whole surface observed as very rough

19	62.703	0.231	Very thick layer of black oxide got separated but it remained overlying on surface as it was not detached from bottom side
20	62.716	0.244	Thick oxide layer formed separated from all side completely. Oxide layer formed at edge side was thick as compared to big surface
21	62.712	0.24	....
22	62.705	0.233	Shabby layer formed got removed from most of the upper surface & then new bubbled oxide layer was formed .Little bit more squeezing sound occurred while cooling
23	62.724	0.252	The second layer which was which was formed also got cracked & got removed from various point
24	62.706	0.234	Oxide layer got separated with large sound & on other hand a blackish thin layer of oxide was present from before only
25	62.728	0.256	....
26	62.752	0.28	The new oxide layer which was formed also got exploded thus leading to small oxide fragments
27	62.775	0.303	For a small portion plane greyish oxide layer formed & rest remained shabby & after 5 min the plane oxide formed also got separated from surface of substrate
28	62.807	0.335	Oxide layer from side of more thickness were separated
29	62.804	0.332	Loud noise occurred and oxide got detached from corner side
30	62.802	0.33	....
31	62.811	0.339	While cooling sound was observed & a thin layer of oxide formed & cracked
32	62.828	0.356	Oxide layer got separated by mild explosion
33	62.816	0.344	Oxide layer got separated very shabbly in nature
34	62.839	0.367	While cooling sound occurred & oxide layer got separated from surface
35	62.834	0.362	While cooling sound occurred & oxide layer got separated from surface
36	62.857	0.385	Oxide layer got removed from side also
37	62.875	0.403	....
38	62.880	0.408	Top layer fall down and new layers got exposed to atmosphere
39	62.869	0.397	....
40	62.875	0.403	....
41	62.906	0.434	....
42	62.918	0.446	....
43	62.914	0.442	....
44	62.906	0.434	A voice occurred as if water evaporated very rapidly (squeezing voice)
45	62.920	0.448	....
46	62.937	0.465	....
47	62.930	0.458	Metallic sound occurred while cooling

48	62.942	0.47	Metallic sound occurred while cooling
49	62.977	0.505	....
50	63.025	0.553	....

Behaviour of Cr-Mo Base metal in salt at 900^0 C

Sample = Cr-Mo alloy steel in weld and heat treated condition

Furnace used =tubular furnace

Controller of furnace = PID Temperature controller

Sample dimensions = 20.25 x 15.84 x 4.99 mm

Sample weight = 12.158 gms

Boat +sample weight after pre heating =55.330 gms

Cycle of 1 hr heating at 900°C and then cooling to 20 min and taking weight along with boat Initially keep sample and boat in oven for 2hr 30 min at 250° C after application of salt and then place it in furnace

Table.5.11: Weight gain observations of Base metal sample in air

No. of Cycles	Sample + Boat wt (gms)	Wt. gain (gms)	Remarks
1	55.487	0.124	Small tong sound while cooling. Salt was appeared in white colour
2	55.616	0.253	Sample colour changed to dark greyish & salt colour changed to reddish black
3	55.742	0.379	Sample colour changed to dark greenish and salt colour to reddish
4	71.661	0.432	Tong sound occurred and bottom layer separates
5	71.666	0.437	....
6	71.764	0.535	Salt was still intact on surface of sample
7	71.888	0.659	....
8	72.012	0.783	Sample while cooling no sound occurred
9	72.087	0.858	From downside a small patch of oxide layer formed separated from sample & other part of it peeled out & was hanging
10	72.167	0.938	Sample while cooling small cracking sound occurred
11	72.253	1.024	....
12	72.400	1.171	Sample while cooling small cracking sound occurred
13	72.500	1.271	Tong sound while cooling, more scale fall in boat

14	72.627	1.398	Oxide layer remained rough
15	72.712	1.483	Small patch of oxide got separated
16	72.811	1.582	Oxide layer which was formed got cracked from all side & while cooling sound occurred
17	72.865	1.636	While cooling sound occurred
18	72.959	1.73	The oxide layer cracked from 2 side i.e. from up & down side & while cooling sound occurred
19	73.008	1.779	A small portion of oxide got removed and small cooling sound occurred
20	73.060	1.831	Oxide layer cracked from various point but there was no cooling sound
21	73.127	1.898	Oxide layer wholly cracked & separated from surface but it did not got wholly separated as it was attached from bottom edge & while cooling no sound occurred
22	73.167	1.938	While cooling small cracking sound occurred
23	73.210	1.981	While cooling small cracking sound occurred
24	73.285	2.056	Thin & medium size oxide layer got separated from upper surface & some in form of small pieces & while cooling small cracking sound occurred
25	73.312	2.083	Oxide layer got cracked from side also & tiny oxide pieces got separated
26	73.339	2.11	Small cracking sound observed while cooling
27	73.374	2.145	Small cracking sound observed while cooling
28	73.418	2.189	Little bit more oxide layer got removed horizontally
29	73.441	2.212	Small cracking sound observed while cooling
30	73.476	2.247	Small cracking sound observed while cooling
31	73.499	2.27	Small cracking sound observed while cooling
32	73.508	2.279	Small cracking sound observed while cooling
33	46.137	2.3	Small cracking sound observed while cooling
34	46.155	2.318	Small cracking sound observed while cooling
35	46.186	2.349	………………………………………………..
36	46.196	2.359	………………………………………………..
37	46.222	2.385	………………………………………………..
38	46.266	2.429	No sound occurred while cooling.
39	46.294	2.457	No sound occurred while cooling.

40	46.322	2.485	No sound occurred while cooling.
41	46.344	2.507	No sound occurred while cooling.
42	46.424	2.587	No sound occurred while cooling.
43	46.441	2.604	No sound occurred while cooling.
44	46.463	2.626	No sound occurred while cooling.
45	46.490	2.653	…………………………………………………
46	46.502	2.665	…………………………………………………
47	46.519	2.682	…………………………………………………
48	46.541	2.704	…………………………………………………
49	46.571	2.734	…………………………………………………
50	46.619	2.748	Layers separated and fall in boat

Behaviour of Cr-Mo Base metal in Air at 900^0 C

Sample	= Cr-Mo alloy steel in weld and heat treated condition
Furnace used	=tubular furnace
Controller of furnace	= PID Temperature controller
Sample dimensions	= 20.27 x 15.80 x 4.94 mm
Sample weight	= 12.131 gms

Boat +sample weight after pre heating =55.584 gms

Cycle of 1 hr heating at 9000 C and then cooling to 20 minutes and taking weight along with boat.

Table.5.12: Weight gain observations of Base metal sample in air

No of cycles	Sample+Boat wt (gms)	Wt. gain (gms)	Remarks
1	55.654	0.063	Tong sound while cooling, upper layer is removed in form of powder
2	55.678	0.087	sample colour turned to dark greyish .Cracks on bubbled surface layer
3	49.918	0.129	Cracking sound observed while cooling, small pores were observed
4	49.950	0.161	Oxide colour changed to black

5	49.970	0.181	....
6	50.033	0.244	Thick carbon layer cracked & splashed separating with noise but at lower side small patches of oxide layer fallen out
7	50.066	0.277	....
8	50.096	0.307	While cooling small noise occurred & black oxide layer separated with sound
9	50.158	0.369	A very thick layer of black colour oxide exploded with noise & while cooling small noise occurred
10	50.139	0.35	All oxide layer was separated
11	50.162	0.373	While cooling whole oxide layer formed and splashed with cling sound and another dark blackish oxide layer adherent to surface was observed, from corners also oxide layer splashed
12	50.154	0.365	
13	50.176	0.387	Black oxide at corner side was observed latter on which exploded
14	50.225	0.436	....
15	50.245	0.456	While cooling small noise occurred
16	50.269	0.48	Side oxide layer got removed while cooling sound was observed
17	50.295	0.506	....
18	46.903	0.516	From various places on both surface oxide layer separated by explosion & whole surface observed as very rough
19	46.923	0.522	Very thick layer of black oxide got separated but it remained overlying on surface as it was not detached from bottom side
20	46.942	0.529	Thick oxide layer formed separated from all side completely. Oxide layer formed at edge side was thick as compared to big surface
21	46.966	0.553	....
22	46.988	0.575	Shabby layer formed got removed from most of the upper surface & then new bubbled oxide layer was formed .Little bit more squeezing sound occurred while cooling
23	41.557	0.578	The second layer which was which was formed also got cracked & got removed from various point
24	41.594	0.615	Oxide layer got separated with large sound & on other hand a blackish thin layer of oxide was present from before only
25	41.616	0.637	....
26	32.734	0.639	The new oxide layer which was formed also got exploded thus leading to small oxide fragments
27	32.749	0.654	For a small portion plane greyish oxide layer formed & rest remained shabby & after 5 min the plane oxide formed also got separated from surface of substrate
28	41.723	0.663	Oxide layer from side of more thickness were separated
29	41.733	0.673	Loud noise occurred and oxide got detached from corner side
30	41.762	0.702	....
31	41.777	0.717	While cooling sound was observed & a thin layer of oxide formed & cracked
32	41.794	0.734	Oxide layer got separated by mild explosion

33	41.817	0.757	Oxide layer got separated very shabbly in nature
34	41.832	0.772	While cooling sound occurred & oxide layer got separated from surface
35	41.860	0.8	While cooling sound occurred & oxide layer got separated from surface
36	41.874	0.814	Oxide layer got removed from side also
37	41.893	0.833	....
38	41.919	0.859	Top layer fall down and new layers got exposed to atmosphere
39	41.941	0.881	....
40	41.963	0.903	....
41	41.977	0.917	....
42	42.053	0.993	....
43	42.075	1.015	....
44	42.081	1.021	A voice occurred as if water evaporated very rapidly (squeezing voice)
45	42.100	1.04	....
46	41.126	1.066	....
47	42.147	1.087	Metallic sound occurred while cooling
48	42.171	1.111	....
49	42.205	1.145	....
50	42.272	1.167	....

DETAILS OF PUBLICATIONS

- S.Riyaz Ahmed, Ajai Agarwal, B.S.S.Daniel, "Some investigations on Mechanical and Microstructural properties of SMA Welded Cr-Mo alloy steel subjected to different Post Weld Heat Treatment times", National Welding Meet 2010, The Indian Institute of Welding, Indira Gandhi Centre for Atomic Research, Kalpakkam, 06 August 2010. (Paper accepted for presentation, Presentation date is on 06-08-2010).

- S.Riyaz Ahmed, Ajai Agarwal, B.S.S.Daniel, "Characterization of Mechanical properties and hot corrosion behaviour in SMA Welded Cr-Mo alloy steel", 3rd International and 24th AIMTDR Conference, Andhra University, Visakhapatnam, December 13-15, 2010. (Communicated for Abstract acceptance)