

**GRADUATE SCHOOL OF
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**REDUCTION OF WELD DECAY EFFECT OF STAINLESS
STEELS USED IN MEDICAL AND FOOD SECTOR THAT
WELD BY TIG PROCESS**

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**BY
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**Reduction of Weld Decay Effect of Stainless Steels Used in
Medical and Food Sector that Weld by TIG Process**

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December, 2013

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I declare that the related thesis is written properly according to academic and ethical rules and using all literature information referenced in the related thesis.

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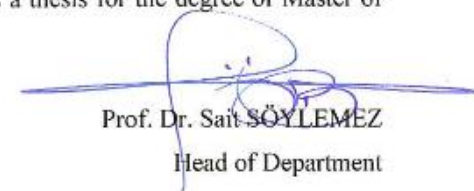
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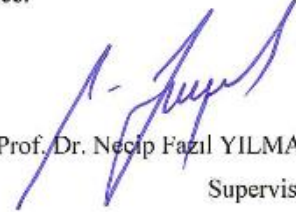
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Prof. Dr. Sait SÖYLEMEZ
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This is to certify that we have read this thesis and that in our opinion it is fully adequate, in scope and quality, as a thesis for the degree of Master of Science.


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CHAPTER 1

INTRODUCTION

1.1 INTRODUCTION

Stainless steel is one the most important material in the medical and food industries. Especially 304 and 316 type austenitic stainless steels have the best corrosion resistance, strong, easy to clean and sterilized properties [1,2]. Austenitic stainless steels exhibit a single-phase, face-centered cubic (FCC) structure that is maintained over a wide range of temperatures. The common welding problem with this type of stainless steel is known as weld decay which occurs in the heat-affected zone (HAZ) [3].

1.2 STAINLESS STEELS AND WELD DECAY PROBLEM

Stainless steels based on the safety of a thin passive film to preserve low corrosion rates in moist solutions. Any damage of this film is probable to effect in localized corrosion, at pits and crevices [4]. The weld decay problem consists of localized attack at grain boundaries, causing relatively little corrosion of grains, but resulting in the disintegration of the alloy and loss of strength. The impurities at the grain boundaries, enrichment of one of the alloying element or depletion of one of the elements in the grain boundary areas causes weld decay. This form of corrosion has been observed in the case of 18-8 stainless steels heating to temperatures of 500-800°C. It is surmised that depletion of chromium in the grain boundary location of the steels results in intergranular corrosion. When the carbon content of the steels is 0.03% or higher, the chromium carbide (Cr_{23}C_6) being insoluble precipitates out of the solid solution and results in depletion of chromium in areas adjacent to the grain boundaries. The chromium carbide remains intact while the chromium-depleted areas near the grain boundary corrode. Weld decay of austenitic stainless steels can be controlled or

minimized by quench annealing or by the addition of small amounts of Niobium or Tantalum which form carbides readily or by lowering the carbon content of the steel to less than 0.03%. By heating the steel to 1000-1100°C followed by water quenching, the chromium carbide dissolves and results more homogeneous alloy resistant to corrosion [4,5].

1.3 TYPES OF STAINLESS STEEL AND THEIR APPLICATION

There are five major classes of stainless steels, designated in accordance with their crystallographic structure. Each class consists of several alloys of somewhat differing composition having related physical, magnetic and corrosion properties.

1.3.1 Martensitic

The name derives from the similar martensite phase in carbon steels. Martensite is created by a shear type phase transformation on fast cooling by quenching from the austenite region structure (face centered cubic) of the phase diagram. It is the characteristic hard component of quenched carbon steel, as well as of the martensitic stainless steels. In stainless steels, the structure is body centered cubic and the alloys are magnetic. Typical applications include steam turbine blades, cutlery, and tools [1-3].

1.3.2 Ferritic

Ferritic steels come from after the analogous ferrite phase or relatively pure iron component of carbon steels cooled slowly from the austenite region. Alpha, or the ferrite, phase of pure iron is the steady phase existing below 910°C. For low carbon Cr-Fe alloys, the high temperature austenite (gamma) phase exists only up to 12% Cr; above this Cr content, the alloys are ferritic at all temperatures up to the melting point. By cold working they be hardened moderately, but not by heat treatment. Ferritic stainless steels are magnetic and are body centered cubic in structure [1-3].

1.3.3 Austenitic stainless steels

Austenitic stainless steels are named after the austenite, or γ phase, which for pure iron exists as a stable structure between 910°C and 1400°C. They have a face centered cubic and nonmagnetic, single-phase structure at room temperature. They are able to be strengthened and hardened by cold working process. The standard grades can

be further subdivided into 300-series and 200-series stainless steels. The 300-series alloys are Fe-Cr-Ni austenitic grades containing 16 to 26% Cr and 6 to 22% Ni. Stainless steel AISI type 304 which contains nominally 18% Cr and 8% Ni as alloys. AISI type 316 contains 2% molybdenum for improved corrosion resistance. Both these types are permitted to have a maximum carbon content of 0.08%. Typical applications include pharmaceutical and food materials, surgical equipment, cookware, flatware and food transport [2-5].

1.3.4 Duplex stainless steels

Duplex is a common definition for many dual-phase material systems. Within the stainless steel nomenclature, it can be named as the definition for combinations of the structures ferrite and austenite. In common sense duplex stainless steels are defined as steels with an austenitic– ferritic crystal structure with a ratio of 60: 40. Duplex stainless steel can be used in many applications such as pressure vessels, Storage tanks and heat exchangers [3,5].

1.3.5 Precipitation Hardening Steels

The precipitation hardening steels achieve high hardness and strength through low temperature heat treatment after quenching from high temperatures. These Cr –Fe alloys contain less nickel (or none) than is necessary to stabilize the austenite phase. In addition, they contain alloying elements, such as copper or aluminum that produce high hardness through formation and precipitation of intermetallic compounds along slip planes or grain boundaries. Typical applications include heat exchangers and boiler tubes [3-5].

1.4 ORGANIZATION OF THE WORK

In this work, it is aimed to reduce weld decay in austenitic stainless steel, because of the size, type or location of the part to be welded, Weld decay problem cannot be overcome by conventional methods. So a new approach has been proposed by acquiring a model (copper block), from which to reduce the time required to formation of sensitization, and do not allow adequate time for the interaction of carbon

with chromium for the configuration of chromium carbide which leads to hindering weld decay.

This thesis organized in six chapters.

The following chapter, Chapter 2, denotes the most related works on the “welding decay problem in austenitic stainless steel” through special heat treatment by a control on grain boundary or added some element to decrease weld decay.

Chapter 3 deals with characteristic of austenitic stainless steel especially used in medical and food sector and the general knowledge on austenitic stainless steel welding. Theoretical approach to weld decay is explained in detail.

The experimental procedure and newly proposed method is explained in Chapter 4. Procedure for microstructural analysis is also provided in this chapter.

In chapter 5, microscopic analysis and microhardness results are discussed according to different experimental cases.

Chapter 6 gives the conclusion gathered from the work results.

CHAPTER 2

LITERATURE SURVEY

2.1 INTRODUCTION

Many researches have been carried out some effects on the effect of heat-treatment, added element and controlled distribution of grain boundary on weld decay defect. This chapter presents a comprehensive up to date literature survey.

2.2 LITERATURE RELATED WITH THE WORK

E. A. Trillo [6] tested samples of 304 stainless steel heat treated at 670°C for 50 h to study on carbide precipitation behavior of the varying grain boundary. It was shown that increasing the carbon content leads to an increment in the degree of sensitization and lead to an increment in the density of precipitation. High carbon steels will nucleate and grow larger sized precipitates on lower angle boundaries (where the energy is the highest) than lower carbon steels. This means that there is a direct correlation between carbide precipitation and energy boundary.

K Nishimoto & K Ogawa [7] classified the type of corrosion in welding of stainless steel in HAZ region into three groups. The first thought is considered in the HAZ far from the interface. This type is called “Weld Decay” and found in the HAZ of austenitic group stainless steel, caused by the sensitization induced by the precipitation of Cr carbide at the grain boundary. The second, called “knife-line attack” that which exists in the nearness of the interface on both sides, occurs at the HAZ of ferritic Stainless Steel which is caused by the deterioration of intercrystalline corrosion resistance and pitting corrosion resistance induced by the precipitation of chromium (Cr) carbide at the grain boundary and the obstetrics of martensite in this zone. The third that which occurs in the vicinity of the interface on one side. This type is found in the

welding of different materials and occurs owing to the galvanic corrosion caused by the difference in electrochemical actions between dissimilar materials.

Hiroyuki Kokawa [8] proved that twin boundaries have a little flair to corrosion and carbide precipitation because atomic structure is more coherent and regular compared to irregular or random grain boundary structure.

H. Kokawa [9] designed and studied “grain boundary engineering (GBE)” to prevent weld decay of austenitic stainless steel by heat treatment. He proved that “low total coincidence site lattice (CSL) boundaries” need more time for intergranular chromium carbide precipitation and corrosion than random boundaries.

Sen Yang [10] improved the weld decay resistance of 304 stainless steel by using “laser surface re-melting (LSM)”. He showed that combination of LSM and annealing treatment resulted in a high frequency of twin boundaries and continuous random grain boundary network by thermo-mechanical processing.

H. Kokawa [11] researched the prevention of weld decay during arc welding. He had achieved by a very high frequency of coincidence site lattice (CSL) boundaries in 304 to resist weld decay. Distributed low energy segments in the grain boundary network made a discontinuous chain of chromium depletion and could prevent weld decay of the surface.

C. Garcia [12] studied the influence of the small-scale electrochemical cell to describe the degree of sensitization and weld decay on welded joints of austenitic stainless steel materials. It is shown that the AISI 316L weldment has better weld decay resistance than AISI 304. Weld decay resistance of AISI 316L can be strongly affected by post-welding heat treatments. Annealing prior to welding was clearly deleterious, especially for the base material. For both weld regions the HAZ was determined as the most critical region in every heat treatment condition for all materials.

Pilar. De .Tiedra [13] studied the resistance spot welding end analyzed degree of sensitization and weld decay. It was proved that the weld decay increased with increasing heat input, which removes signs of prior cold work and twins inside austenite

grains of the HAZ. The involvement between heat input and the degree of sensitization, made very difficult to predict the degree of sensitization from heat input, because of the degree of sensitization was the combined effect of weld decay in the HAZ.

F.F. Curiel [14] explored the effect of magnetic fields by gas metal arc welding on corrosion of the HAZ in AISI 304 stainless steel. The application of magnetic field during welding induced a lower degree of sensitivity and better resistance to pitting and weld decay in 3.5% NaCl solution than samples welded without magnetic fields. It was detected that magnetic fields enable Cr re-distribution in the austenitic base metal. Welding current promoted Cr redistribution, reducing the Cr depletion and provided that continuous and corrosion resistant passive film in the HAZ.

Subodh. Kumar [15] showed the influence of low heat input on mechanical and microstructure properties during the TIG welding process by using different welding currents (120,150,180A) in 304 stainless steel. There was a direct correlation between heat input and the fusion zone and HAZ area increasing together. Significant grain coarsening was found in the HAZs of all the joints. It was also observed that the extent of grain coarsening increase with increasing heat input. The results showed that low heat input to welding has higher “ultimate tensile strength (UTS)” than others because of different arc energy inputs, and less grain size in HAZ region.

Majid Laleh [16] prevented weld-decay and improved grain refinement of twin's formation in 304 stainless steel for gas tungsten arc welding by using “surface mechanical attrition treatment (SMAT)” by producing nanostructured surface layers of bulk materials. It was found the degree of sensitization in heat-affected zone as high as 50 times than the untreated sample because of the formation of high density of twins as well as grain refinement.

E. Ranjbarnodeh [17] developed a thermal model to simulate heat transfer and carbide precipitation during welding of 304 stainless steel. There were four effective factors of sensitivity (chemical composition, thermal cycle, internal or external stresses and welding operations). It was proved that when the current is increased the weld heat input and dimensions of weld pool increased. It is found that certain temperature range

(650-850 °C) the chromium carbide precipitation occurs. The cooling rate of points adjacent to weld was increased and there is not enough time for the formation of chromium carbide. The carbide band doesn't occur because of maximum temperature limit doesn't approach to 650-850°C.

2.3 PLACE OF THIS WORK IN THE LITERATURE

From the above discussion we can discover that there are some various attempts to amend the “welding decay in austenitic stainless steel”. But most of the attacks were based on heat treatment of the specimens or add some elements to increase weld decay resistance. The purpose of the present study is to reduce welding decay in “austenitic stainless steel”, especially those parts which do not make heat treatment, because of the type or position the part to be welded. So, a new approach has been proposed by acquiring a copper block model. It is aimed to reduce the time required to formation of sensitization, and don't allow adequate time for the interaction of carbon with chromium.

CHAPTER 3

THEORETICAL WORK

3.1 INTRODUCTION

The aim of this chapter is to present general knowledge on weld decay problem at austenitic stainless steels, especially those types used in medical and food sector.

3.2 STAINLESS STEEL USED IN MEDICAL AND FOOD SECTOR

Stainless steel is firstly considered metal when corrosion or oxidation is considered as a problem. The function that they perform cannot be duplicated by other materials for their cost. Since more than 50 years, it was discovered that a minimum of 12% chromium would impart corrosion and oxidation resistance to steel. Hence the definition “Stainless Steels” are those ferrous alloys that contain a minimum of 12% chromium for corrosion resistance. This development was the start of a family of alloys which has enabled the advancement and growth of chemical processing and power generating systems.

Austenitic stainless steels represent the largest groups of stainless steels and are produced in higher quantities than any other group and the common one in medical and food application. Because, in many instances, they provide very predictable levels of corrosion resistance with excellent mechanical properties. Using them wisely can save the design engineer significant costs for product. They are a user-friendly metal alloy with life-cycle cost of fully manufactured products lower than many other materials. The austenitic alloys can have compositions anywhere in the portion of the Delong diagram labeled austenite shown in Figure 3.1. This diagram was designed to show which phases are present in alloys in the as solidified condition, such as found in welds. The salient feature of austenitic alloys is that as chromium and molybdenum are increased to

increase specific properties, usually corrosion resistant, nickel or other austenite stabilizers must be added if the austenitic structure is to be preserved [18].

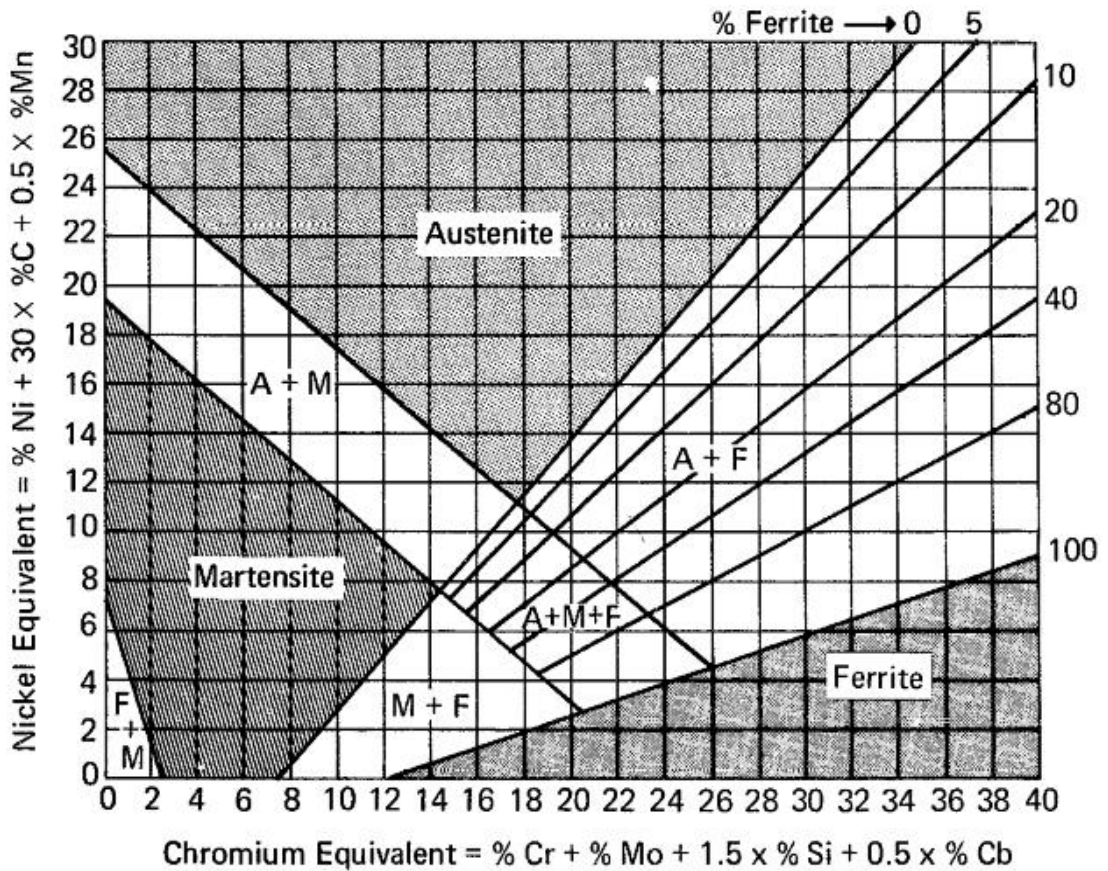


Figure 3.1 Schaeffler-Delong Stainless Steel (constitution) Diagram [19].

The traditional way of displaying the austenitic stainless steels is to present 304 as a base. Figure 3.2 shows diagrams such as these treat alloys as an evolutionary family tree and subtly mislead. Austenitic stainless steels have a strength equivalent to those of mild steels, approximately 210 Mpa minimum yield strength at ambient temperature, and are not transformed hardenable.

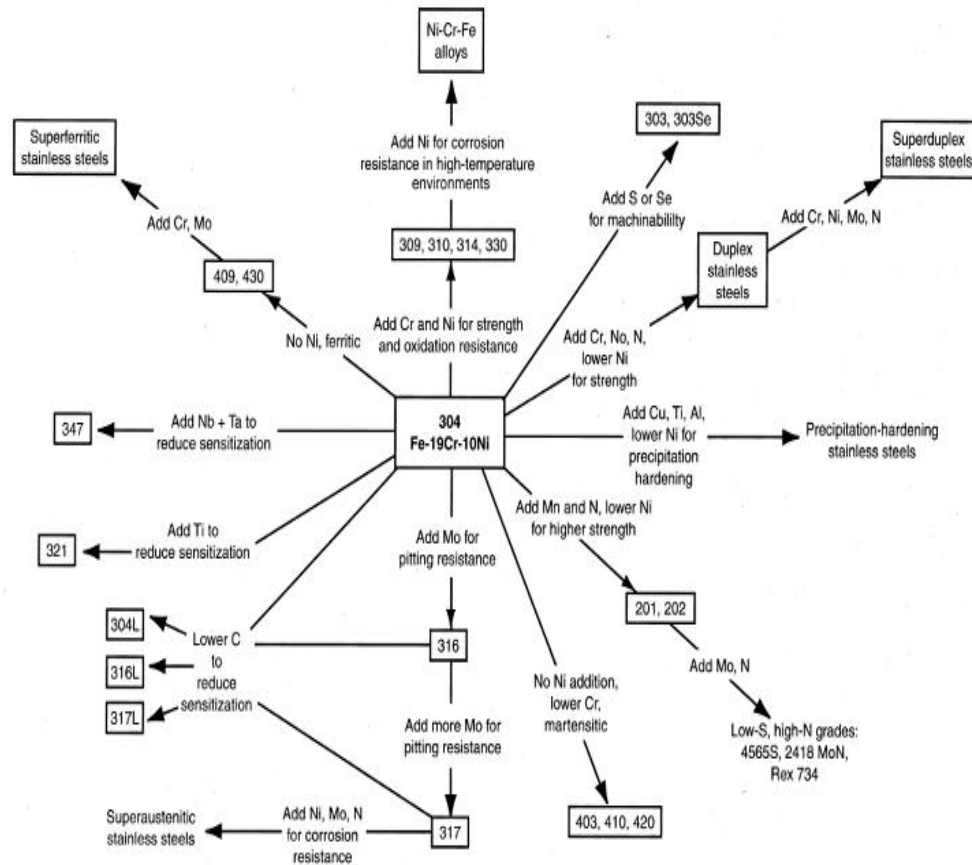


Figure 3.2 The Austenitic Stainless Family [3]

Low temperature impact properties are good for these alloys, making them useful in cryogenic applications. Service, temperatures can be up to 760 °C or even higher, but the strength and oxidation resistance of these steels are limited in such high temperatures. Austenitic stainless steels can be strengthened significantly by cold working. They are often used in applications requiring good atmospheric or elevated temperature corrosion resistance. Austenitic stainless steels are generally considered to be weldable, if proper precautions are followed. Elements that promote the formation of austenite, most notably nickel, are added to the austenitic stainless steels in large quantities (generally over 8%). Other austenite-promoting elements are added in small, but sufficient, quantities. Such elements are C, N and Cu. Carbon and nitrogen are strong austenite promoters. Carbon is added to improve the creep resistance at high

temperatures. Nitrogen is added to some austenitic alloys in order to improve strength, mainly at ambient and cryogenic temperatures, sometimes more than doubling it. Nitrogen-strengthened alloys are designated with a suffix N added to their AISI 300 series designation (e.g., 316LN) [18].

Austenitic stainless steels generally have good ductility and toughness and exhibit significant elongation during tensile loading. They are more expensive than the martensitic and low to medium Cr ferritic grades, due to their higher alloy content. Despite the cost, they offer distinct engineering advantages, particularly with respect to formability and weldability, which often reduce the overall cost, compared to the other groups of stainless steels. Although there are a wide variety of austenitic stainless steels, the oldest and most commonly used are the 300 series. Most of these alloys are based on the 18Cr-8Ni system, with additional alloying elements or modifications to provide unique or enhanced properties. Type 304 is the foundation of this alloy series and along with 304L represents the most commonly selected austenitic grade. While type 316 substitutes approximately 2%Mo for a nearly equal amount of Cr to improve pitting corrosion resistance. The stabilized grades, 321 and 347, contain small additions of Ti and Nb, respectively, to combine with carbon and reduce the tendency for intergranular corrosion due to Cr-carbide precipitation. The L grades became popular in the 1960s and 1970s with the introduction of AOD (argon-oxygen decarburization) melting practice that reduced the cost differential between standard (not low carbon) and L grades. These low-carbon grades (304L, 316L) have been widely used in applications where intergranular attack and intergranular stress corrosion cracking are a concern. Austenitic stainless steels are used in a wide range of applications, including structural support and containment, architectural uses, kitchen equipment and medical products. They are widely used not only because of their corrosion resistance but because they are readily formable, fabricable and durable. Some highly alloyed grades are used for very high temperature service (over 1000°C) for applications such as heat-treating baskets. In addition to higher chromium levels, these alloys normally contain higher levels of silicon (and sometimes aluminum) and carbon, to maintain oxidation and/or carburization resistance and strength, respectively. It should be pointed out, that the common austenitic stainless steels are not an appropriate choice in some common

environments such as seawater or other chloride-containing media, or highly caustic environments. This is due to their susceptibility to stress corrosion cracking, a phenomenon that afflicts the base metal, HAZ and weld metal in these alloys. Finally, care should be taken when selecting stainless steels that will be under significant stress in these environments. [19,20]

3.3 WELDING OF STAINLESS STEEL

Although all welding processes can be used, there are three most popular processes for welding stainless steels which are shielded metal arc welding (SMAW), tungsten inert gas (TIG) and metal inert gas (MIG) welding. Welding of stainless steels are slightly more difficult to weld than mild steels [20]. This is due to the fact that the physical properties of stainless steels exhibit several differences from those of the mild steels.

These differences are:

1. Lower melting temperature
2. Lower coefficient of thermal conductivity
3. Higher coefficient of thermal expansion
4. Higher electrical resistance

The above physical properties are not the same for all stainless steel grades. The metallurgical features are those that determine the physical properties and the weldability characteristics. In general, the weldability of martensitic stainless steels is affected greatly by hardenability that can result in cold working. Welded joints in ferritic stainless steels have low ductility as a result of grain coarsening that is related to the absence of allotropic phase transformation. The weldability of austenitic stainless steels is governed by their susceptibility to hot cracking, similar to other single-phase alloys with an FCC structure. Weldability of stainless steels does not work only change in mechanical properties, but also the chemical characteristics that affect corrosion resistance. Because of reactions of chromium with carbon and oxygen at welding temperatures make the choice of welding processes is limited. Common welding of stainless steels is done by manual shielded-arc methods rather than any other process [21].

3.4 WELDING AUSTENITIC STAINLESS STEELS BY GAS TUNGSTEN ARC WELDING (GTAW), “TIG”

Gas tungsten arc welding process (Figure 3.3) uses an electric arc, established by a non-consumable tungsten electrode and the base metal, to join the metal being welded. A separate welding filler rod is fed into the molten base metal, if needed. The torch holding the tungsten electrode is connected to a shielding gas cylinder and to one of the terminals of the power source. The tungsten electrode is usually in contact with a water-cooled copper tube, called the contact tube, which is connected to the terminal as well. This allows both the welding current to enter the electrode and the electrode to be cooled to prevent overheating. The workpiece is connected to the other terminal of the power source through a different cable. The shielding gas goes through the torch body and is directed by a nozzle toward the weld pool to protect it from the surrounding atmosphere. The gaseous protection in GTAW is much better than SMAW, because an inert gas such as argon or helium, is directed toward the weld pool. Tungsten Arc Welding is a famous way to weld stainless steel in factories and maintenance work according of American Welding Society (AWS) specification [21-23].

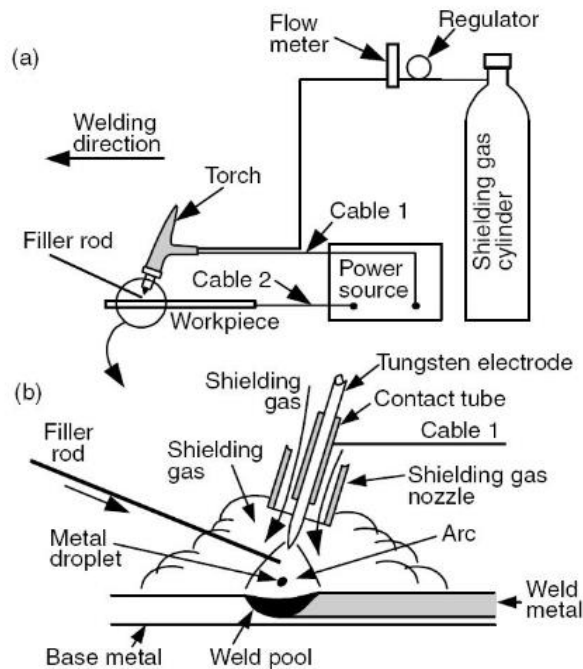


Figure 3.3 Gas Tungsten Arc Welding Process [22]

a) Overall Process b) Welding Area

3.5 CORROSION AT STAINLESS STEEL WELDMENT

The corrosion which occurs at the stainless steel weldment can be examined by three different categories;

1. **Base metal corrosion:** Base metal corrosion is a mode of corrosion which occurs when the corrosion resistance of the weld metal is superior to that of the base metal. An example can be found in the welding of carbon steel with the use of austenitic group of stainless steel electrodes as shown in Figure 3.4.



Figure 3.4 Base Metal Corrosion

2. **Weld metal corrosion:** The type corrosion occurs when a welding electrode with low corrosion resistance and not matching with material as shown in Figure 3.5.



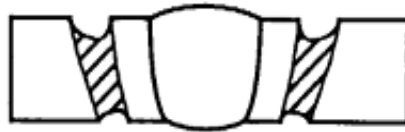
Figure 3.5 Weld Metal Corrosion.

3. **Heat affected zone (HAZ) corrosion:** Every position in the HAZ relative to the fusion line experiences during welding, in terms of both maximum temperature and cooling rate. Thus, each position has its own microstructural features and corrosion susceptibility.

There are three types of heat affected zone (HAZ) corrosion as shown in Figure 3.6.

- a) Corrosion in the HAZ far from the interface
- b) Corrosion in the vicinity of the interface on both sides

c) Corrosion in the vicinity of the interface on one side



(a) (HAZ) corrosion type



(b) (HAZ) corrosion type



(c) (HAZ) corrosion type

Figure 3.6 Modes of Corrosion at HAZ of Stainless Steels [7].

A representative example of the (a) type corrosion is ‘Weld Decay’ found in the HAZ of austenitic stainless steels. This is induced by the sensitization caused by the precipitation of Cr carbide at the grain boundary.

The (b) type corrosion is found at the HAZ of ferritic stainless steels and induced by the deterioration of intercrystalline corrosion resistance. Pitting corrosion resistance caused by the precipitation of Cr carbide at the grain boundary and the generation of martensite in this region. Another example of corrosion, which occurs in the HAZ vicinity of the interface is ‘knife-line attack’, occurs in stabilized steels. This phenomenon is named as sensitization because of the precipitation of Cr carbide at the grain boundary. Its characteristic is that the area of corrosion occurrence is narrower than that found in the HAZ of a group stainless steels and much closer to the interface [25,26].

The (c) type corrosion is found in the welding of different materials and occurs owing to the galvanic corrosion caused by the difference in electrochemical actions between dissimilar materials.

3.6 INTERGRANULAR CORROSION

Intergranular attack is defined as the selective dissolution of grain boundaries, or closely adjacent regions, without appreciable attack of the grains themselves. This is caused by potential differences between the grain boundary region and any precipitates, intermetallic phases, or impurities that form at the grain boundaries. The actual mechanism differs with each alloy system. Intergranular corrosion is focused attack on or at grain boundaries. The attacks propagate into the material. This is a risky form of corrosion because the cohesive forces between the grains may be poor to resist tensile stresses, the toughness of the material is seriously reduced at a relatively early stage, and fracture can occur without warning [27].

Intergranular corrosion has always been a subject of investigation and research due to the implications that can cause to the welded structures in service conditions. Figure 3.7 presents a schematic representation of a weld that has undergone intergranular attack in the HAZ.

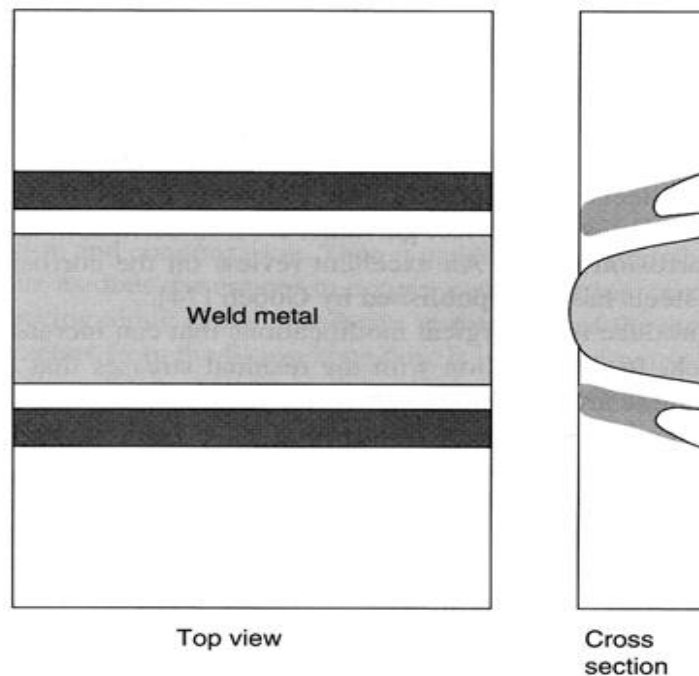


Figure 3.7 Schematic Representation of Intergranular Attack in the HAZ [23].

3.7 WELD DECAY PROBLEM

Weld decay is a form of intergranular corrosion, usually of stainless steel or certain nickel-base alloys, which occurs as the result of sensitization in the heat affected zone (HAZ) during the welding operation. The corrosive attack is restricted to the heat affected zone (HAZ). Positive identification of this type of corrosion usually requires microstructure examination under a microscopy although sometimes it is possible to visually recognize weld decay if parallel line is already formed in the heat affected zone along the weld region as shown in Figure 3.8. Cr diffuses relatively slowly, and therefore, the amount of Cr forming carbide is taken from the very nearest region. The area that is most liable to intergranular corrosion is usually located some millimeters from the fusion zone of the weld [27-30]

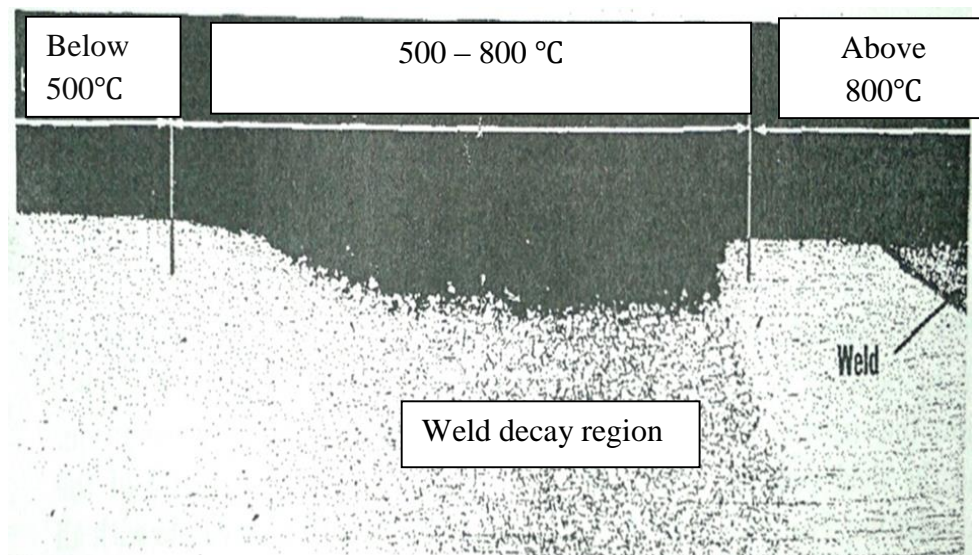


Figure 3.8 Positions of Weld Decay Occurrence in 304 steel HAZ and Curve of welding Thermal Cycle [24].

When austenitic stainless steels are heated or cooled through the temperature range of about 500–800°C, where the temperature is kept within the critical range for the longest time intervals, the chromium along grain boundaries tends to combine with carbon to form carbon carbides (Cr_{23}C_6). It is called carbide precipitation or weld decay defect (sensitization). This effect is a depletion of chromium and the lowering of

corrosion resistance in areas adjacent to the grain boundary which leads to failure of the metals Figure 3.9 shows temperature range of weld decay [31].

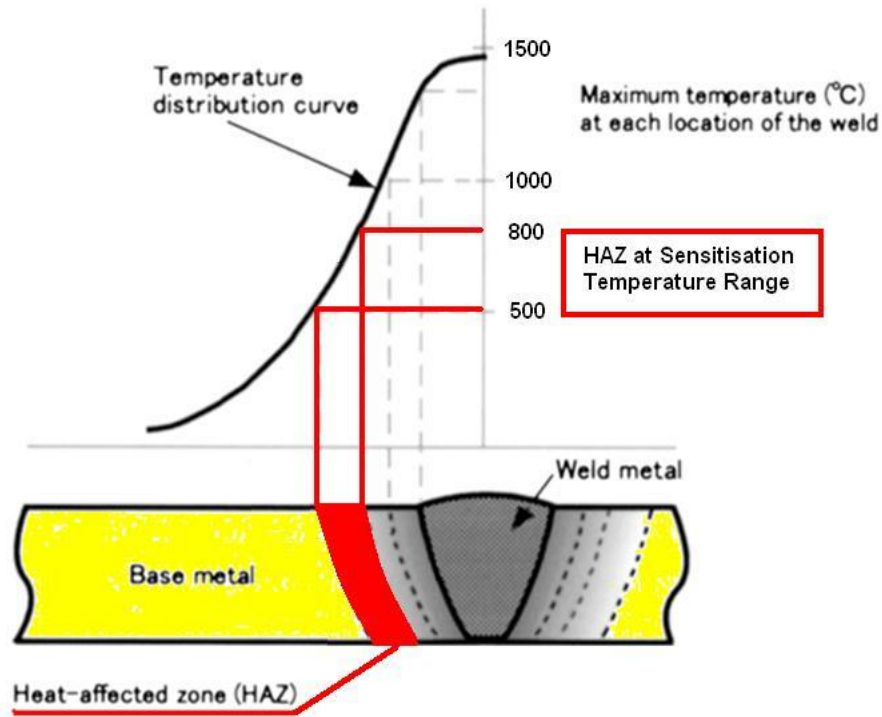


Figure 3.9 Temperature Distribution Curve and Temperature Range of Weld Decay [32].

The sensitization produced by the formation of a Cr carbide near the grain boundary places parallel to the weld metal in the HAZ. The region in the HAZ which is heated above or lower sensitization temperature is not sensitized as shown in Figure 3.9. Because, the temperature does not reach the temperature region for sensitization.

Required sensitization time of stainless steels is usually shown as C-shaped curves in which Carbon-Time- Temperature is being shown in Figure 3.10. Carbon content has the most profound influence on susceptibility to intergranular corrosion cracking (IGC) in austenitic stainless steels. The use of low-carbon (L grades) alloys minimizes the risk of sensitization by slowing down the carbide precipitation reaction. Demonstrate the effect of carbon content on the time of precipitation. For low carbon content (e.g. $C < 0.04$ wt %), the nose of the curve is beyond 1 hour, while for carbon

levels from 0.06 to 0.08 wt % the time of precipitation may be less than a minute. This difference demonstrates the benefit of the low-carbon alloys for reducing or eliminating HAZ grain boundary sensitization during welding. The presence of residual stresses in the HAZ may also serve to accelerate the precipitation reaction [33].

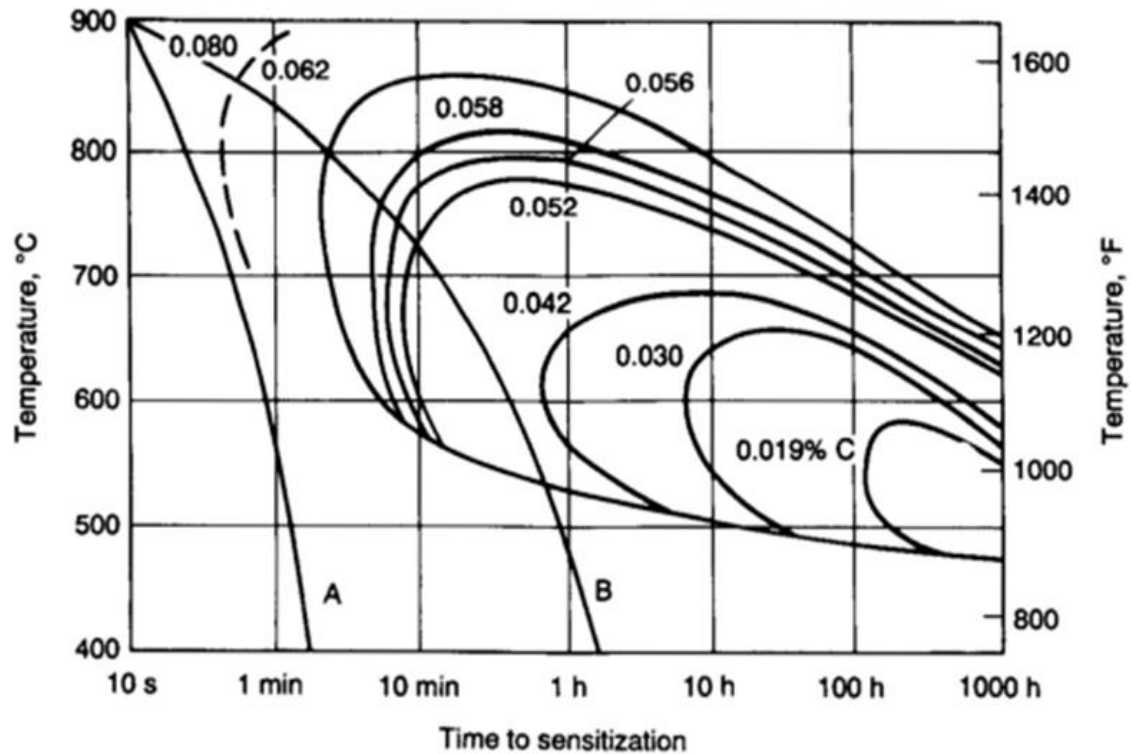
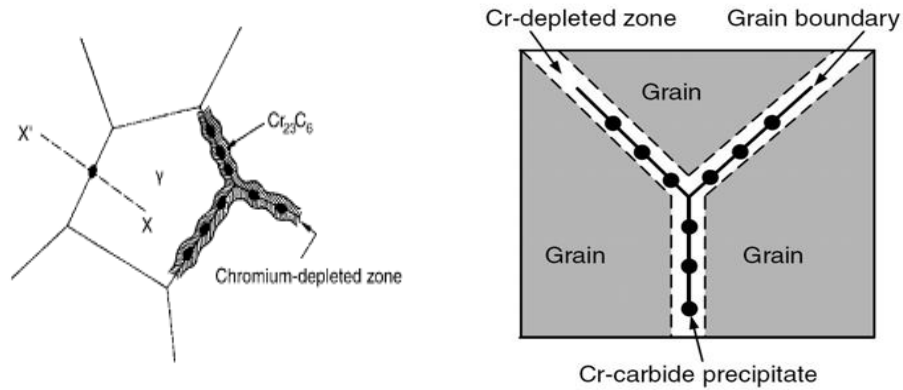


Figure 3.10 Cr₂₃C₆ Time-Temperature-Precipitation Curves for “18-8” Alloys with Variable Carbon Content [2]

When the austenitic steels are heated in the range of 500 to 800°C and cooled slowly through that range, carbon is precipitated from solid solution and unites with chromium to form chromium carbides. In the HAZ of most austenitic stainless steels, Cr-rich Cr₂₃C₆ carbides form preferentially along grain boundaries, as shown Figure 3.11. This results in a chromium-depleted zone along the grain boundary that is “sensitive” to corrosive attack. Hence, the term sensitization is often used to describe the metallurgical condition leading to intergranular attack [27,34].

Intergranular corrosion results from the localized precipitation of Cr-rich carbides, or carbonitrides, at the grain boundary.



Chromium carbides at grain boundary

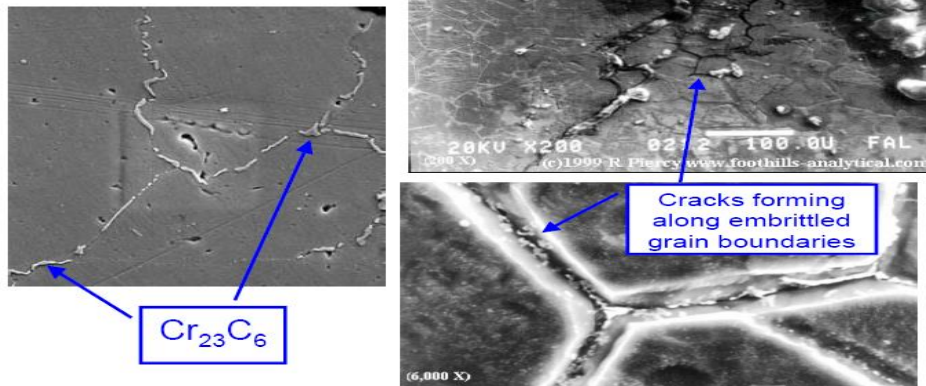
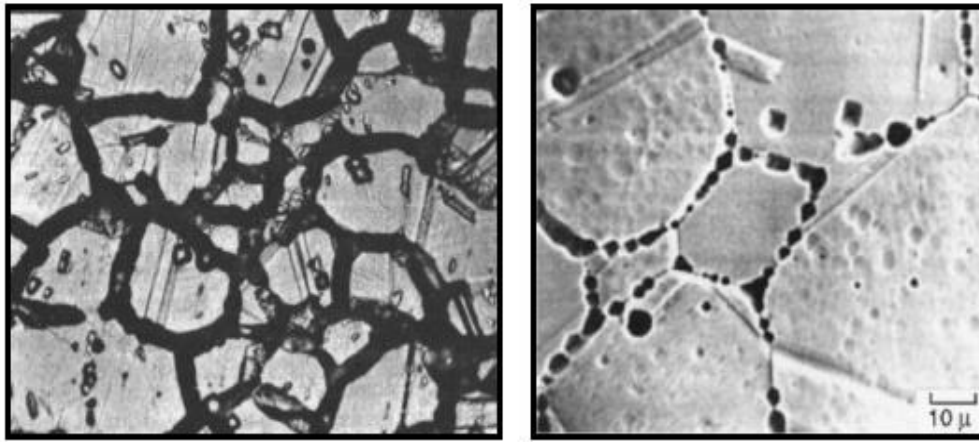


Figure 3.11 Grain Boundary Carbide Precipitation and Local Chromium Depletion [21]

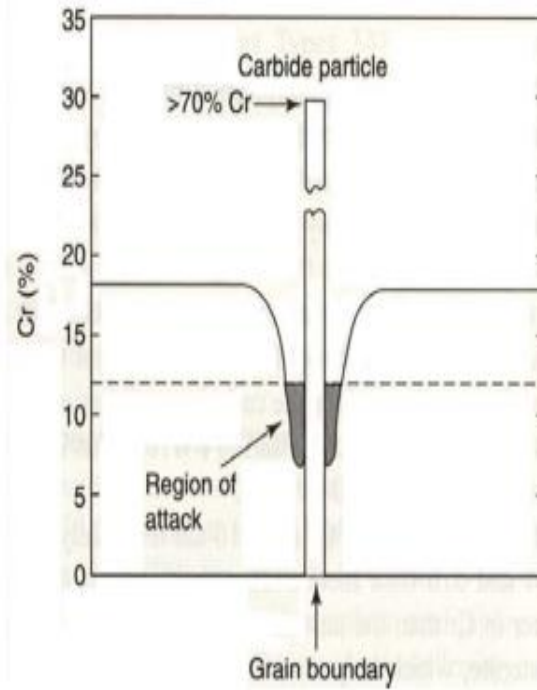
This precipitation requires short-range diffusion of Cr from the adjacent matrix and produces a Cr-depleted region surrounding the precipitate. This reduces local corrosion resistance of the microstructure and promotes rapid attack of the grain boundary region. In certain corrosive environments the effect is a local “ditching” at the grain boundary, as shown in Figure 3.12 (a). In extreme cases, the grains will actually

drop out of the structure because of the complete grain boundary attack and dilution [28,35].



a) With C 0.06 wt%

b) With C 0.05 wt%



c) Chromium depletion adjacent to the grain boundary carbide

Figure 3.12 Intergranular corrosion: Grain boundary attack in the Type 304 [21].

3.8 AVOIDING WELD DECAY DEFECT

Susceptibility to weld decay defect in austenitic stainless steels can be avoided by limiting their carbon contents or by adding elements whose carbides are more stable than those of chromium [3]. There are three conventional methods to avoid risk of weld decay defect.

3.8.1 Thermal Treatments

Various methods are used to reduce or prevent carbide precipitation in austenitic stainless steels. One of it is to heat the weldment to the range of 1050 to 1150°C and rapidly cool it (by quenching) through the 800 to 500°C range. This thermal treatment re-dissolves the precipitated carbides (puts the carbon back into solution in the austenite and restores the chromium at the grain boundaries) and the rapid quench prevents precipitation from recurring. However, this method has disadvantages. The high-temperature treatment may cause distortion in welded assemblies and large welded structures cannot be accommodated in heating furnaces [29].

3.8.2 Low-Carbon Stainless Steels

Another remedy to the carbide-precipitation problem is to use stainless steels and electrodes having such a low carbon content that no carbides can be precipitated. The 18-8 austenitic steels retain about 0.03% carbon in solid solution under all conditions. With a carbon content increased to about 0.08%, the amount of carbon that can be precipitated increases slowly; above that amount, precipitation can occur rapidly (when the material is exposed to the sensitizing temperature). Extra-low-carbon plate and electrodes cost slightly more than the regular grades, but the results are well worth the cost difference, particularly when weldments are to be used in the as-welded condition. Carbide precipitation decreases significantly in changing, from AISI type 304 (0.08% C) to 304L (0.03% C). Low-carbon welding electrodes are also available. Use of low-carbon plate and electrodes may produce a small amount of carbide precipitation, but usually not enough to be harmful. However, when the temperature (either during welding or in service) and corrosion conditions are severe, stabilized electrodes and base metal should be used [24]

3.8.3 Stabilized Steels

Inter-granular corrosion is formed if the product is subjected to a corrosive medium. The most common way of preventing inter-granular corrosion, especially where critical temperatures will be reached in service or where environmental conditions are severe, is to prevent the formation of chromium carbides by using stainless steels that contain Columbium or Titanium. These elements have a greater affinity for carbon than chromium does; thus they form columbium carbide or titanium carbide, leaving the chromium in solution where it can do its intended job of providing corrosion resistance. Stainless steels that contain Columbium or Titanium (AISI types 347 and 321) are called “stabilized” steels since they are not made susceptible to intergranular corrosion by heating. They can be used in the 500 to 800°C range with no effect on corrosion resistance, and no heat treating is required. Welding of stabilized stainless steels should be done with stabilized electrodes for best results. Since Columbium transfers through an arc much more effectively than titanium, stabilization of electrodes is achieved with Columbium additions [24,30].

3.9 CONTINUES COOLING

The aim of this work is to reduce welding decay in austenitic stainless steel, particularly those sections which do not make any conventional methods (Thermal Treatments, Low-Carbon Stainless Steels and Stabilized Steels), because of the size, type or location of the part to be welded. In this work, a new method has been proposed to eliminate the weld decay problem in stainless steels that is used in a medical and food sector that welded by TIG welding.

By using copper block, it is the aimed to reduce the time required for formation of sensitization, and do not afford adequate time for the interaction of carbon with chromium to configuration of chromium carbide, which leads to hindering of weld decay. Copper block and thermal properties of some materials are presented in Figure 3.13 and Table 3.1, respectively.



Figure 3.13 Copper Block Model

Table 3.1 Thermal Conductivity of Some Materials

| Material | Thermal Conductivity |
|--------------------|----------------------|
| Copper | 2724 |
| Aluminum | 1536 |
| Steel | 314 |
| Cupro-nickle 90/10 | 310 |
| Cupro-nickle 70/30 | 200 |
| Stainless steel | 108 |

This method characterized by the followings:

- It is easy method that any welder can use during welding or maintenance
- Can be done in any position after appropriate preparation of block copper
- Expensive equipment is not needed
- Proposed method offers cheaper material

CHAPTER 4

EXPERIMENTAL PROCEDURE

4.1 INTRODUCTION

This chapter describes the experimental procedure to examine the influence of continues cooling by different medium conditions during welding to reduce weld decay defect in austenitic stainless steel.

4.2 MATERIALS AND METHOD

A commercial type 304 austenitic stainless steel was used throughout this study. The chemical composition of the steels used in the experiments are indicated in Table 4.1.

Table 4.1 Chemical Composition of Stainless Steel Used in Experiments

| C% | Si% | Cr% | Ni% | Mn% | S% | Mo% | P% | V% |
|-------|-------|-------|------|------|-------|------|-------|------|
| 0.058 | 0.668 | 18.00 | 9.09 | 1.10 | 0.002 | 0.07 | 0.013 | 0.03 |

Samples were tested by portable CCD (Charge Coupled Device) spectrometer metals analysis systems, made by ARUN serial number 7221 as shown in Figure 4.1. All samples were prepared in 3mm thickness and surfaces were cleaned with acetone to remove any grease or any dust. Cleaning was done just prior to welding to prevent the formation of oxides. The base metal surface must be free of grease, oil, paint, dirt, etc. to provide a smoother and stronger joint.



Figure 4.1 Portable CCD Spectrometers.

4.3 EXPERIMENTAL AND SET-UP

Tungsten Arc Welding (TIG) as Figure 4.2 is the common methods to weld stainless steel in industry.

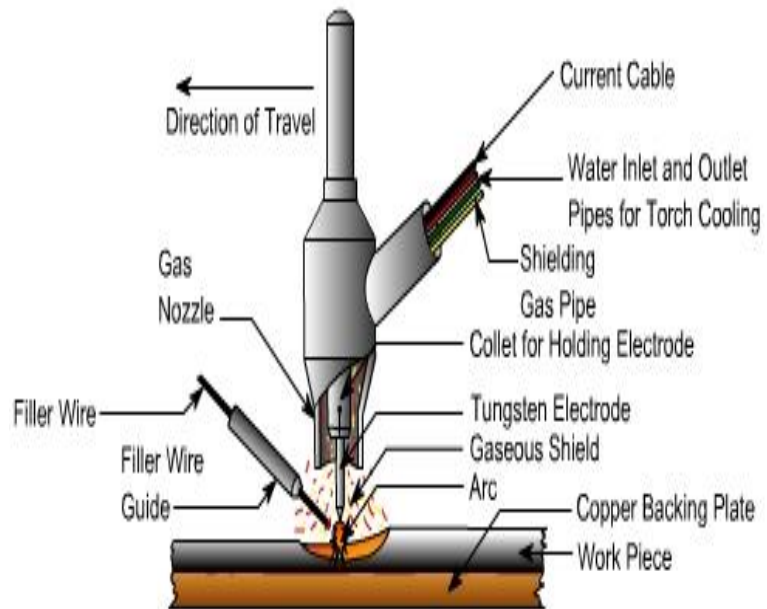


Figure 4.2 Principle of TIG Welding

TIG welding parameters used in the experiments are as follows:

- Filler materials : No
- Non-consumable electrode type : W-%2Th
- Electrode diameter : 1.6 mm
- Internal diameter of ceramic nozzle : 12mm
- Current : 150 A
- Welding speed : 5.3 mm/Sec
- Shielded gas : Argon
- Gas flow : 5 liter/min

In this experiments 20 mm thickness copper block was placed under the weldment shown in Figure 4.3. 8 mm diameter holes were used to transmit the water from channels to sustain the continues cooling as shown in Figure 4.4.

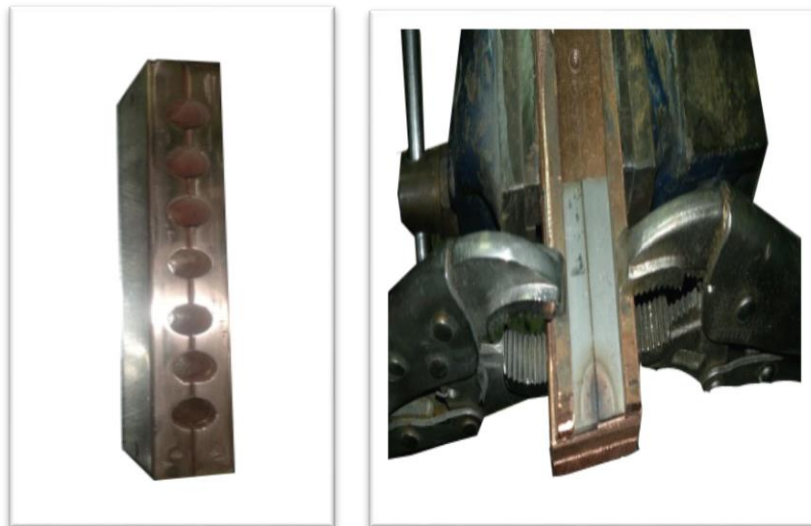


Figure 4.3 Copper Block Placed Under Weldment

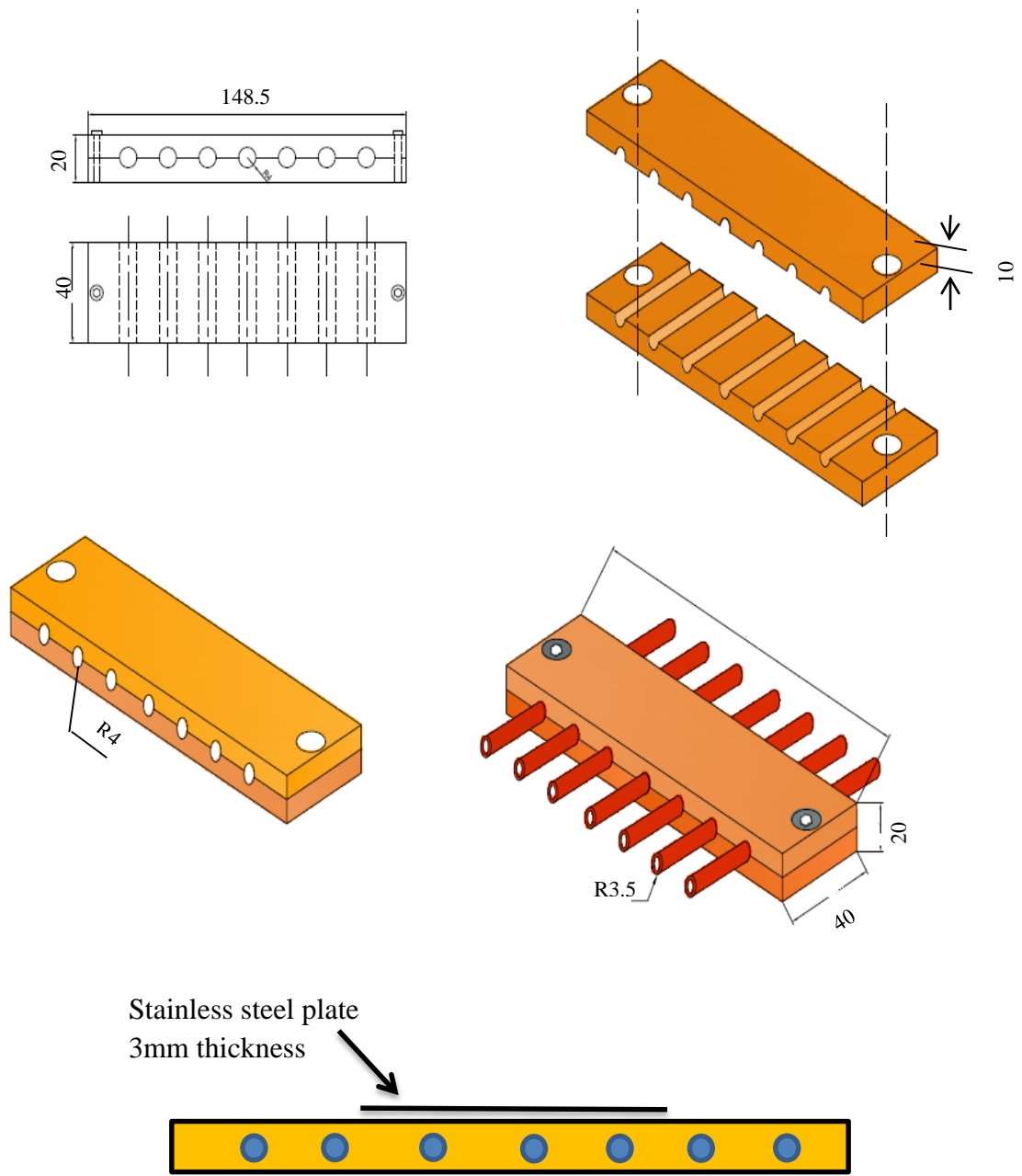


Figure 4.4 Sketchy Copper Block

Welding, without any filler metal, was made at center of samples with gas tungsten arc processes. Figure 4.5 shows the tungsten torch used in welding.



Figure 4.5 Tungsten Torch used in Welding Stainless Steel

4.4 CONTINUES COOLING CONDITIONS

Four different welding conditions were performed to investigate the cooling conditions. All these conditions are presented in Table 4.2.

Table 4.2 Continues Cooling Conditions

| Case No | Type of samples | Water Inlet temp. (°C) | Water Outlet temp. (°C) | Base metal Temp. (°C) |
|---------|---------------------------------|------------------------|-------------------------|-----------------------|
| 1 | Without cooling | - | - | 248 |
| 2 | Copper as cooling | - | - | 150 |
| 3 | Using copper and water at 25 °C | 25 | 28 | 110 |
| 4 | Using copper and water at 5 °C | 5 | 8 | 70 |

- The first sample was welded without copper backup to make this sample as stander and compare it with other samples. Temperature of base metal was measured by thermocouples 10 mm away from the weld center.
- The second sample has been welded by using copper block as backup strip. Sample was fixed to avoid distortion as shown in Figure 4.6.



Figure 4.6 Fixing Stainless Steel to Avoid Distortion

- The third sample was welded by using copper block as backup strip. Water at 25°C was passed through the copper block by using a pump with flow rate $V=1\text{m/Sec}$, as shown in Figure 4.7.
- The fourth sample has the same welding conditions with the third condition. The only difference is inlet water temperature is 5°C instead of 25°C. During welding, temperature of the welding zone was measured instantly by thermocouple as shown in Figure 4.8.

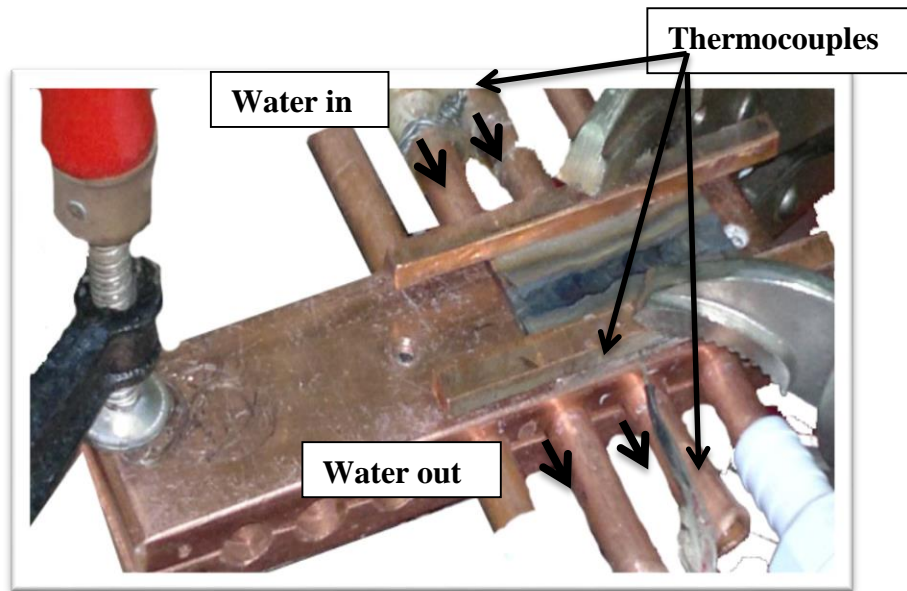


Figure 4.7 Water Passes Through a Copper Block at 25°C and 5°C.



Figure 4.8 Temperature Measured by a Thermocouple.

4.5 MICROSCOPIC ANALYSIS

4.5.1 Grinding

Surface layers damaged by cutting and welding must be removed by grinding. Mounted specimens are ground with rotating discs of abrasive paper. All samples were cleaned with acetone and grinded metallic papers from 60 grit to 1200 grit respectively as shown in Table 4.3.

Table 4.3 Abrasive Paper Sequence Used in the Search

| NO. | Standard ANSI grit | Diameter (microns) | Surface Roughness |
|------------|-------------------------------|-------------------------------|------------------------------|
| 1 | 80 | 180 | 1140 |
| 2 | 180 | 75 | 880 |
| 3 | 240 | 63 | 300 |
| 4 | 400 | 25.8 | 120 |
| 5 | 600 | 15.3 | 110 |
| 6 | 800 | 6.5 | 25 |
| 7 | 1200 | 2.5 | 20 |

4.5.2 Polishing

After the grinding step, specimens were polished by polishing cloths with lubricant and progressively smaller diamond abrasives.

4.5.3 Etching the specimen

Microscopic examination of a properly polished, un-etched specimen will reveal only a few structural features such as inclusions and cracks or other physical imperfections. Etching is used to highlight, and sometimes identify, microstructural features or phases present. The time required for etching as much as ten minutes after being etched the specimen is washed in running hot water at 40°C to remove the chemicals used in the etching. After that immersion the sample in ethanol and then dried by drier as shown in Figure 4.9.

All samples were chemically etched with;

- 10 cm³ Nitric acid
- 20 cm³ Hydrochloric acid
- 20 cm³ Glycerol

- 10 cm³ Hydrogen peroxide mixture



Figure 4.9 Dryer is Used for Drying Samples

4.6 MICROHARDNESS TEST

The term microhardness test usually refers to static indentations made with loads not exceeding 1 kgf. The indenter is either the Vickers diamond pyramid or the Knoop elongated diamond pyramid. The procedure for testing is very similar to that of the standard Vickers hardness test, except that it is done on a microscopic scale with higher precision instruments. The surface being tested generally requires a metallographies finish; the smaller the load used, the higher the surface finish required. Precision microscopes are used to measure the indentations.

To investigate the hardness value of welding area and HAZ, microhardness tests were carried out. The microhardness device used in the experiment is shown in Figure 4.10. From the center of weldment to the outwards, seven different measurement were taken to analysis the hardness distribution.



Figure 4.10 Micro Hardness Device

These have a magnification of around 200X and measure to an accuracy of +0.5 micrometers. Also with the same observer differences of + 0.2 micrometers can usually be resolved.

Diamond Pyramid of microhardness device indentation was grounded in the form of a squared pyramid with an angle of 136° between faces. The depth of indentation is about $1/7$ of the diagonal length.

CHAPTER 5

RESULTS AND DISCUSSION

5.1 INTRODUCTION

In this chapter, microstructural and hardness analysis of the weldment are presented and discussed.

5.2 MICROSTRUCTURAL ANALYSIS OF WELDMENT

Figure 5.1 shows the positions of weld decay found in the HAZ of austenitic stainless steels. Point (B) represent the start of weld decay defect and ended at point (C) at specific time.

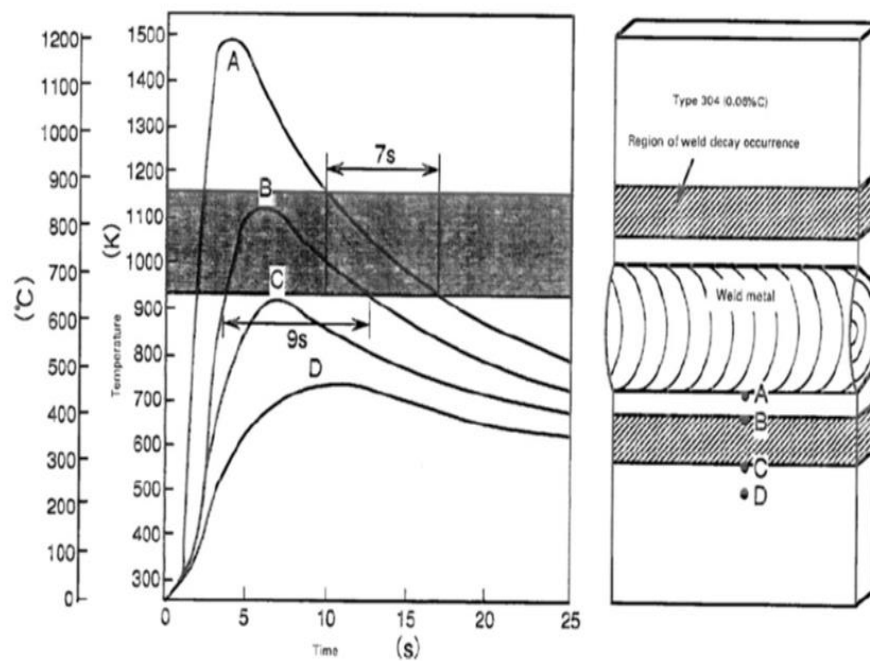


Figure 5.1 Positions of Weld Decay Occurrence in 304 Steel HAZ and Curve of Welding Thermal Cycle [31]

First case: In this work, four different cases of TIG welding were performed. In order to clarify the weld decay defect, 3mm thick stainless steel was welded with conventional method. This is named as "case 1".

Weldment were cut transversely to examine the microstructural changes. Photograph of the specimen is shown in Figure 5.2.

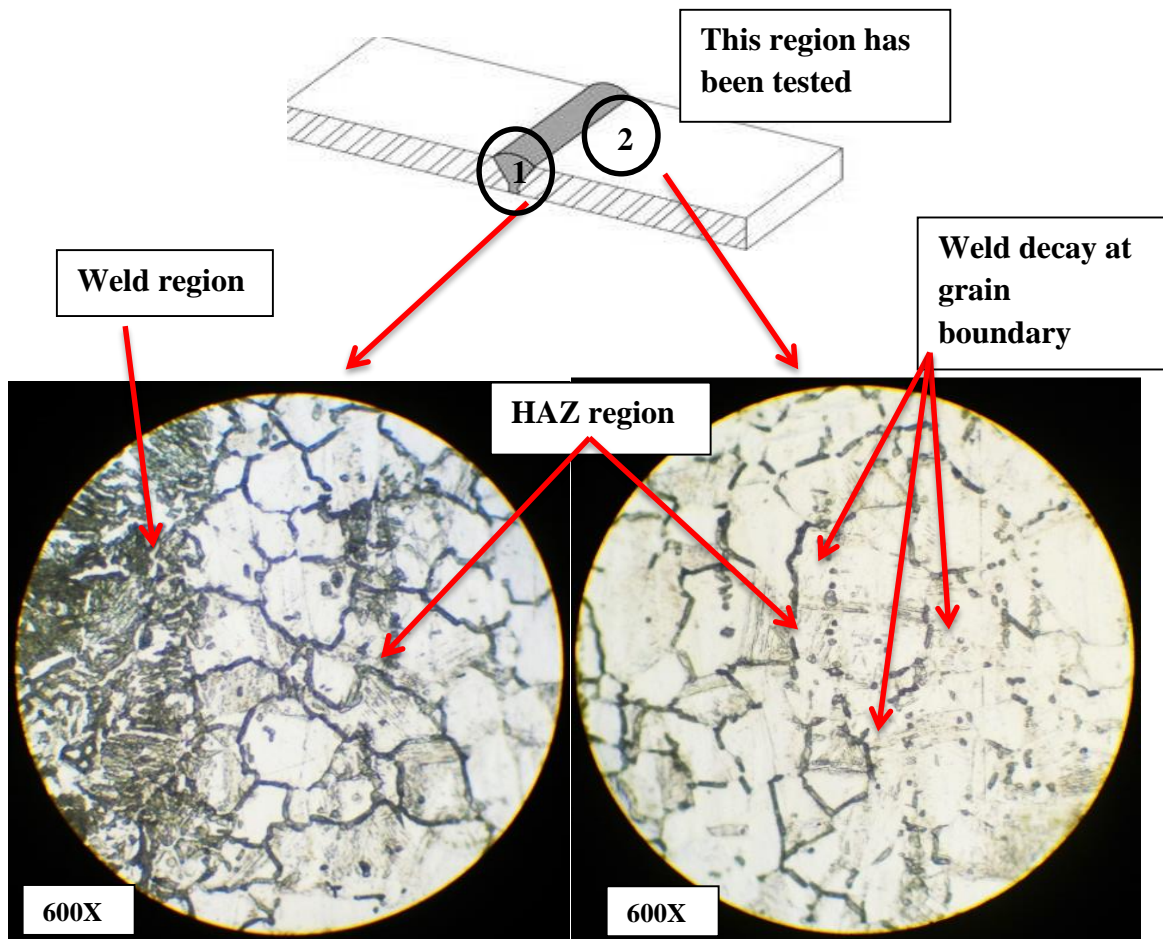


Figure 5.2 Weld Sample Without any Cooling in Weld & HAZ Region

Microscopic views were taken from the surface noted by number 2.

The samples welded without any cooling to made comparison with other samples. Weld decay at the grain boundaries is seen very clearly. Temperature in the base metal was 248°C.

Second case, copper block was placed at the bottom of the weldment to make easy the heat distribution. Weld decay again was observed, but it was less than the first case as shown in Figure 5.3. It means that the time has been reduced to prevent the sensitization or forming Cr_{23}C_6 .

It was noted that the base metal temperature was 248°C in case 1. But, by placing a copper block under the weldment, the temperature was reduced to 150°C .

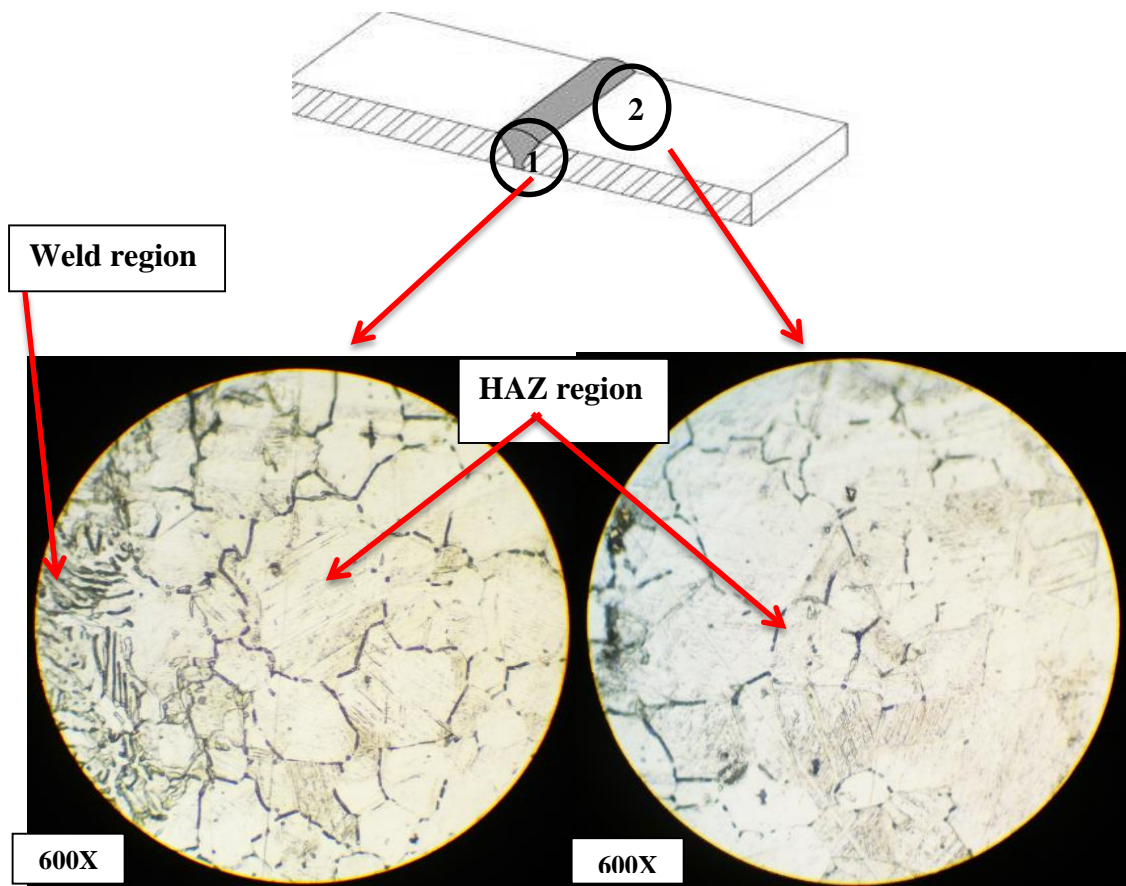


Figure 5.3 Weld Sample Without any Cooling HAZ Region

Third case, 25°C water was passed through from the channels made in the copper block previously.

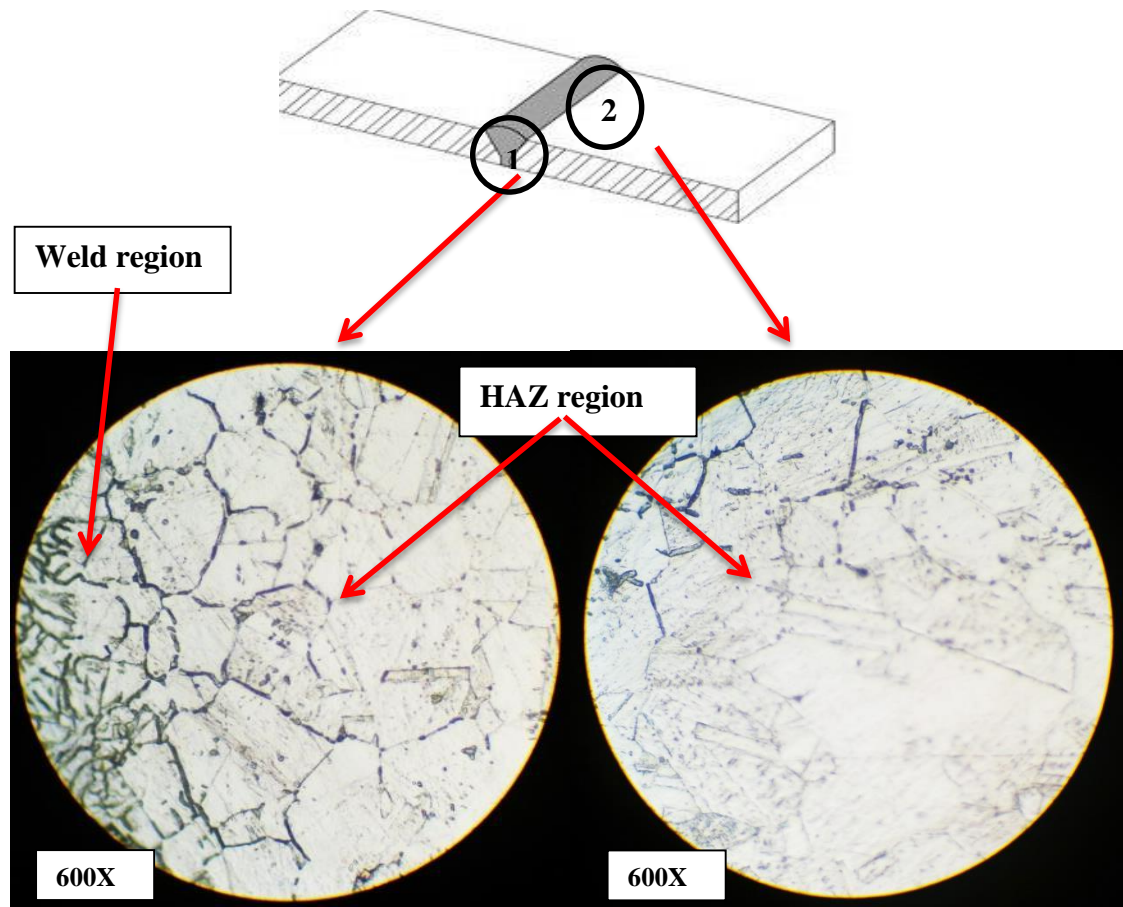


Figure 5.4 Weld Sample by Using Copper and Water at 25°C as Cooling Medium Weld & HAZ Region

The aim was to reduce the temperature in order to not allow enough time for the formation of weld decay. Weldment temperature was measured and recorded as 110°C. In Figure 5.4 it is clearly shown that weld decay is partly eliminated.

Fourth case, Water inlet temperature was reduced from 25°C to 5°C. This caused reduction in base metal temperature to 70°C. Figure 5.5 shows the microstructure of case 4. It is very obvious that, there is no formation of Cr_{23}C_6 at grain boundaries.

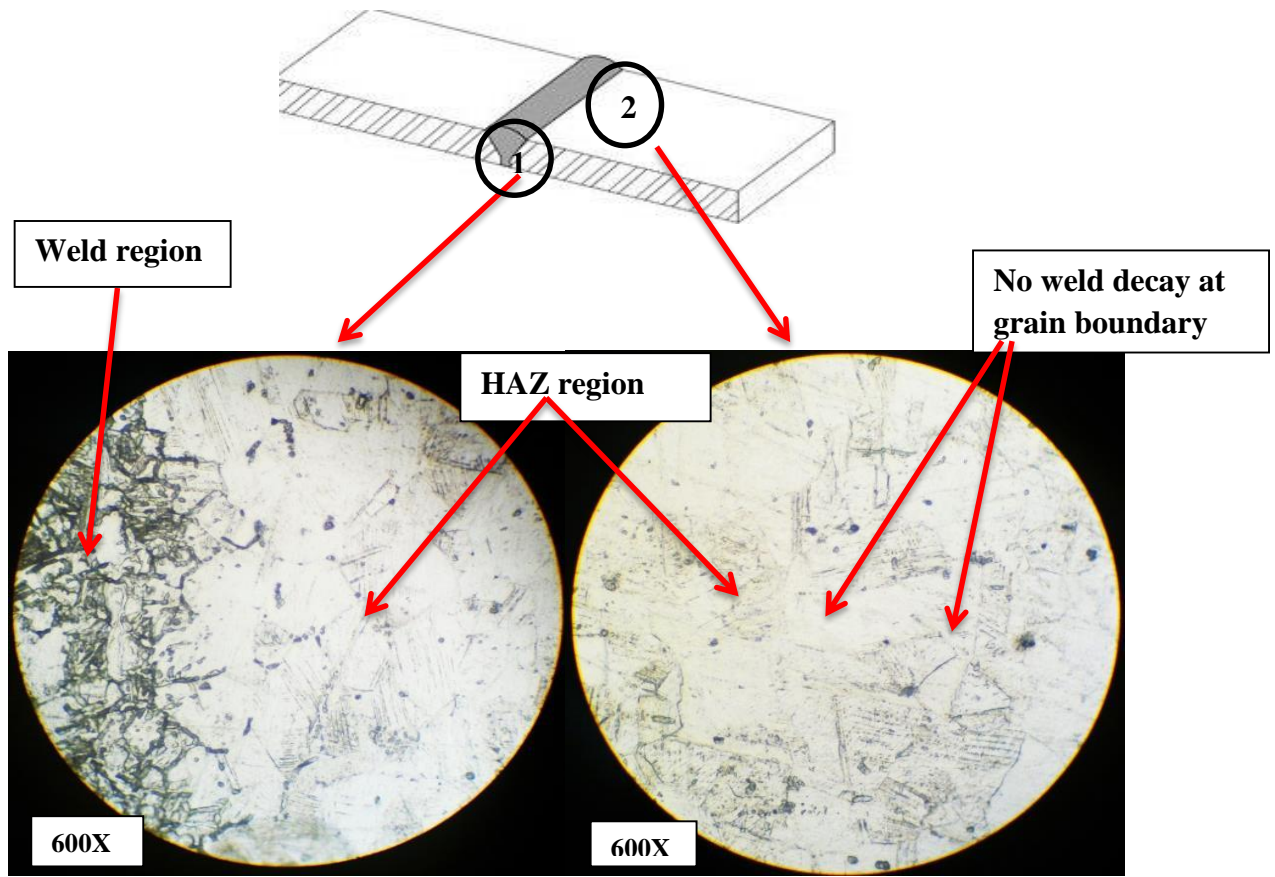


Figure 5.5 Weld Sample by Using Copper and Water at 5°C as Cooling Medium Weld & HAZ Region

Figure 5.6 Shows stages of reduction of weld decay effect of stainless steels used in medical and food sector that welded by TIG process by using continues cooling method.

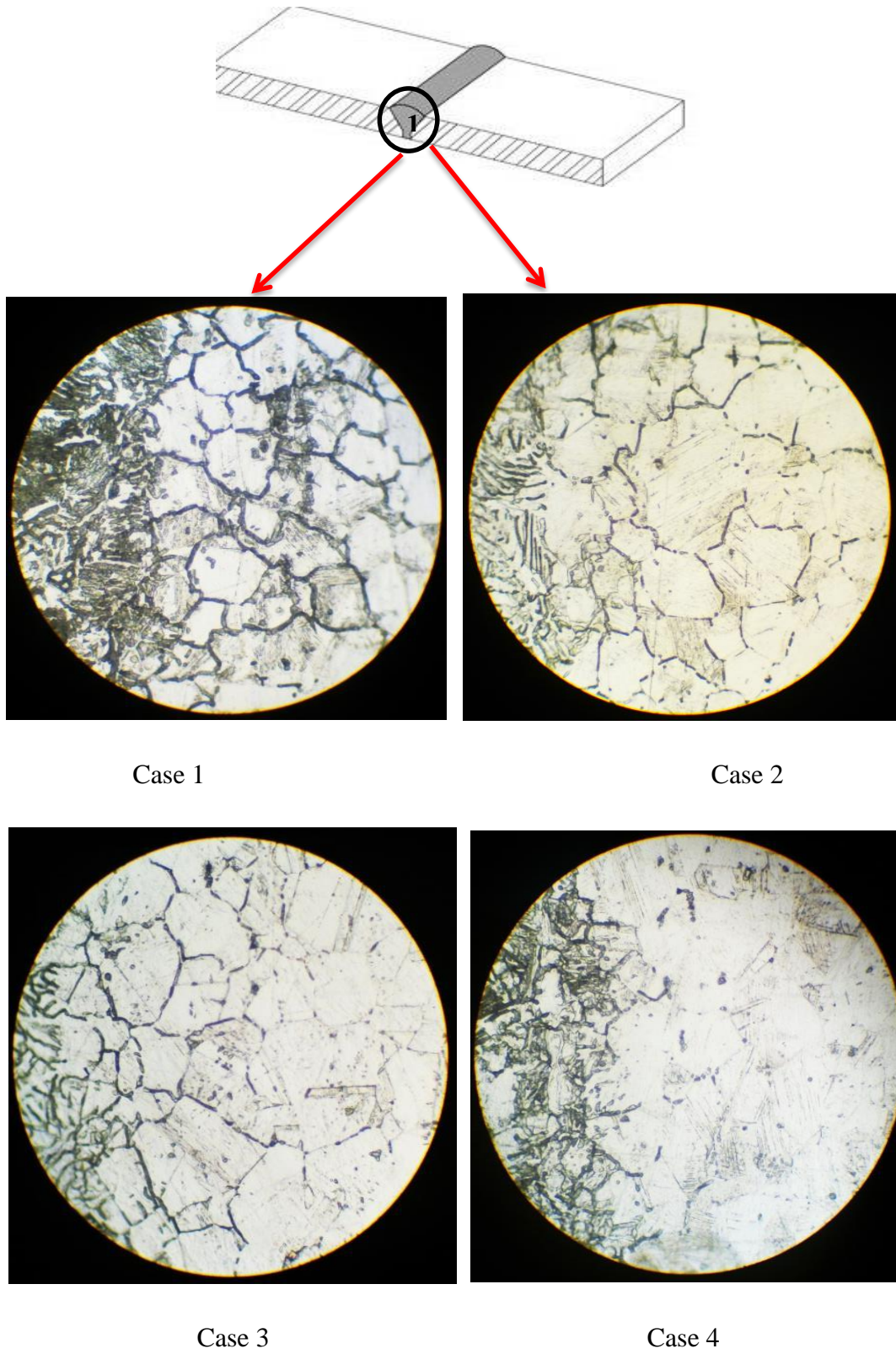
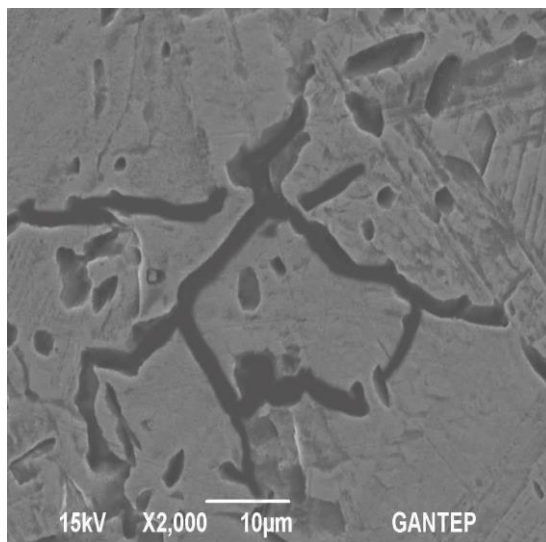
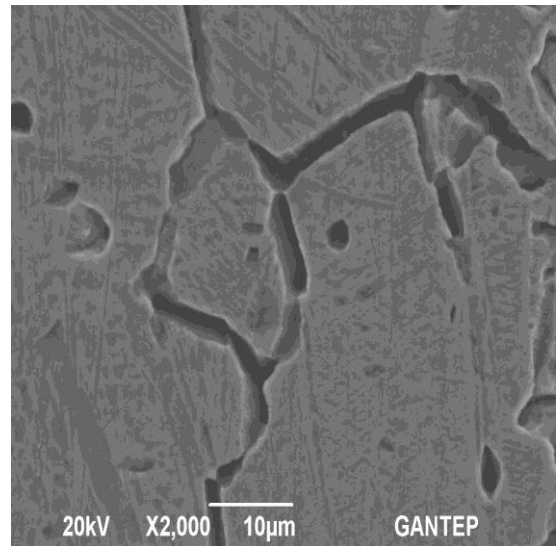


Figure 5.6 Stages of Reduction of Weld Decay Effect by Using Continues Cooling Method

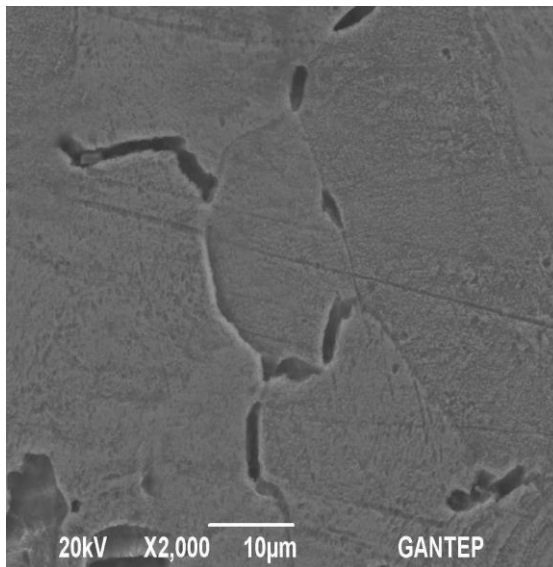
Figure 5.7 shows scanning electron microscope (SEM) views for the stages of reduction of weld decay effect of stainless steels that welded by TIG process.



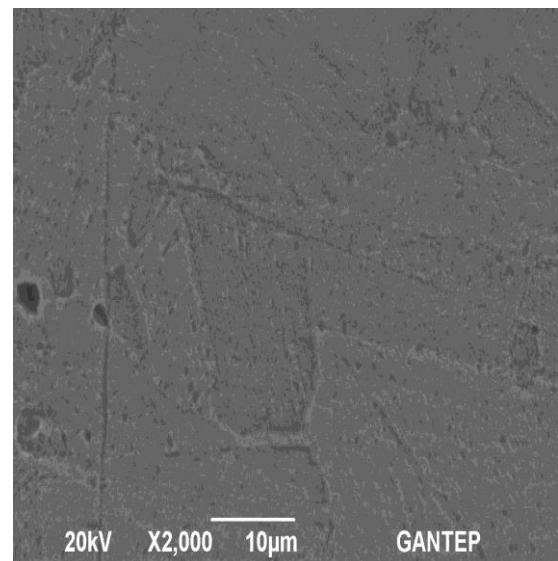
a) Case 1



b) Case 2



c) Case 3



d) Case 4

Figure 5.7 SEM Views Illustrating the Stages of Reduction of Weld Decay

Along with observations on random grain boundaries, Figure 5.7 (a) shows typical carbide precipitation behavior. Figure 5.7.b also displays carbides nucleated at grain boundaries but relatively fewer amounts than Figure 5.7.a. Lower tendency for nucleation and growth is observed when the copper block is attached to the base metal

(Figure 5.7.b). No carbide precipitation behavior was observed on the grain boundaries as the cooling rate increased (Figure 5.7.d).

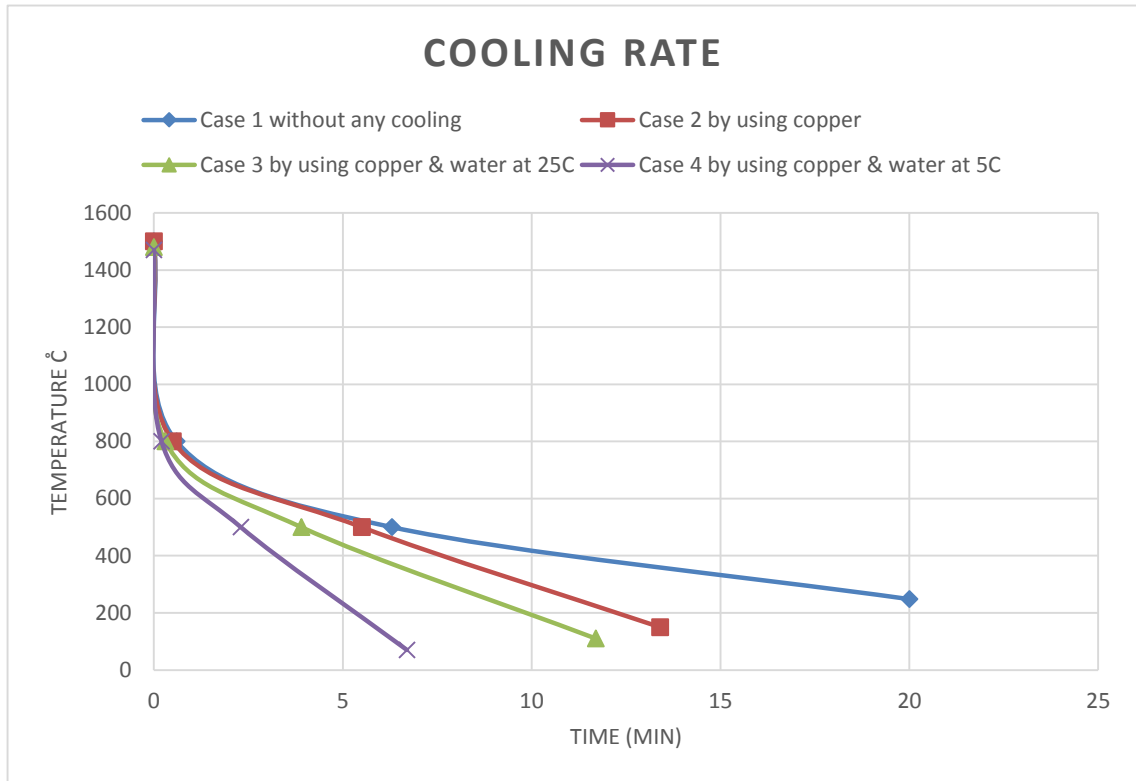


Figure 5.8 Critical Time To Forming Weld Decay Between (500-800°C) In HAZ Region

Figure 5.8 shows the critical time in the HAZ region between 500°C-800°C, which indicate temperature kept more time in this range that mean weld decay had a good chance for forming. In case 1, (500°C-800°C) temperature range requires more than 6 minutes while it is 2,3 minutes for case 4. Thus, it was sustained that carbide particles have no enough time to form. Figure 5.8 implies that forming weld decay decrease with increase cooling rate at (500-800°C) in HAZ region.

5.3 MICROHARDNESS TEST

All tested all samples were subjected to micrhardness test. Microhardness measurements were taken from the center to outwards of the welding as shown in Figure 5.9 Table 5.1 presents the microhardness data. Figure 5.10 shows that the microhardness in the center of the weld is high and decreases as it approaches to base metal gradually.

Case four, 5°C waters passed through from the channels made in the copper block previously, has the highest hardness value which is due to the fact that cooling rate is relatively higher than the other cases.



Figure 5.9 Microhardness from the center to outwards of the welding zone

Table 5.1 Micro-hardness HV data

| Distance from weld center mm | Case 1 | Case 2 | Case 3 | Case 4 |
|---|---------------|---------------|---------------|---------------|
| 1 | 204.3 | 206 | 210 | 221 |
| 2 | 204 | 205 | 209 | 220 |
| 3 | 203.4 | 203.5 | 207 | 212 |
| 4 | 202 | 202.5 | 204 | 207 |
| 5 | 201 | 201.2 | 202 | 204 |
| 6 | 200.5 | 200 | 200.2 | 200 |
| 7 | 200.5 | 200 | 200.2 | 200 |

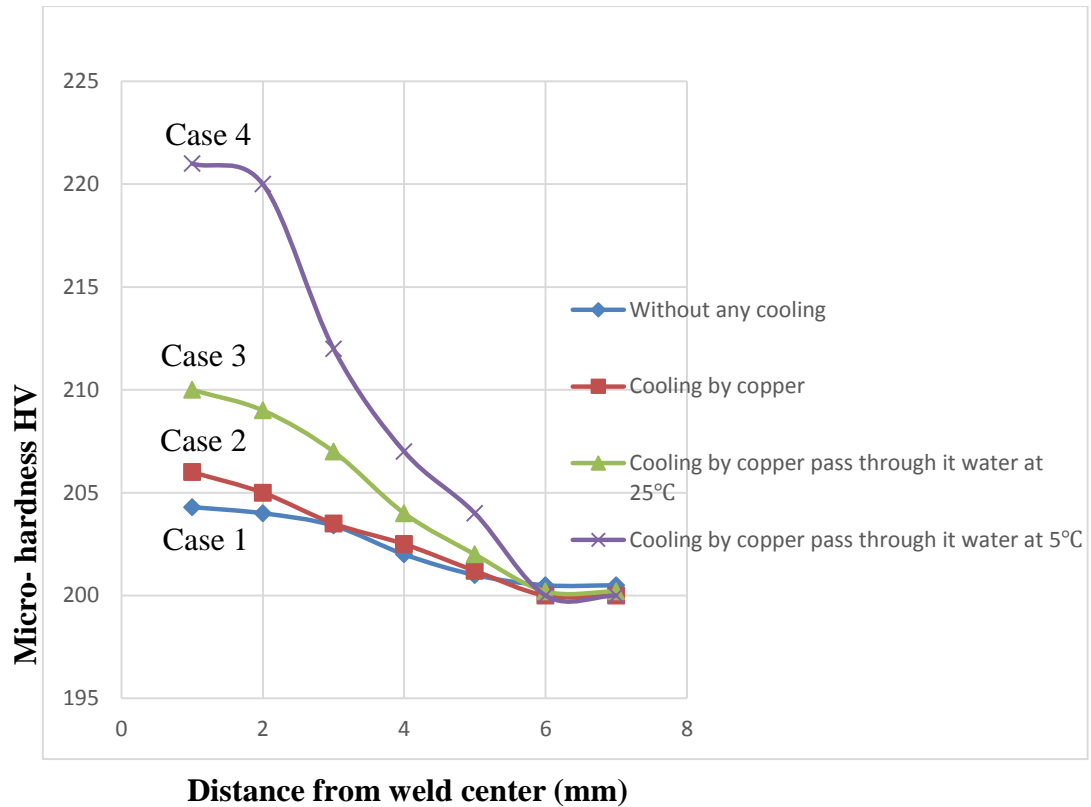


Figure 5.10 Micro-hardness Profile Showing Micro-hardness Values at Different Points.

By observing the Figure 5.9, the values of microhardness increases with cooling rate in the weld and HAZ region, and it is almost constant in the base metal. Case 4 has the highest hardness value, because of the fast cooling rate, which effects on the grain size.

CHAPTER 6

CONCLUSIONS

Following conclusions can be drawn from this work;

- A new method has been proposed to eliminate the weld decay problem in stainless steels that is used in a medical and food sector that weld by TIG welding.
- Continues cooling method helps in solving the weld decay problem, especially in the maintenance work that needs to welding operations in large appliances that cannot make transactions by heat treatment.
- Continues cooling method, doesn't need high equipment and it is low cost.
- Copper block was used to assist the reduction of base metal temperature.
- If the temperature of the base metal is uniformly reduced, weld decay at the grain boundaries are reduced (or eliminated) considerably. By decreasing the cooling rate, nucleation starts and sensitization may occur. At very low cooling rates, the formation of chromium carbide occurs at higher temperatures and allows for more nucleation and growth, resulting in a more extensive chromium-depleted region.
- Corresponding hardness values were increased with cooling rate. This came from the change in grain size.
- Grain size is getting smaller as cooling rate increases, which leads to increase hardness values.

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