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Microstructure and toughness of welded A710 steel

Qin, Weifang, Ph.D.

Lehigh University, 1991

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Ann Arbor, MI 48106

**MICROSTRUCTURE AND TOUGHNESS
OF
WELDED A710 STEEL**

**By
Weifang Qin**

A Dissertation
Presented to the Graduate Committee of
Lehigh University
in Candidacy for the Degree of
Doctor of Philosophy
in
The Department of Materials Science and Engineering
Lehigh University
March, 1991

Approved and Recommended for acceptance as a dissertation in partial fulfillment of the requirement for the degree of Doctor of Philosophy.

14 MARCH 1991

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ABSTRACT

HSLA copper-bearing, precipitation-aged A710 steels are of increasing interest in the Navy ship-building and structural applications. In this investigation, weldments of ASTM A710 steel plates (grade A, class 3) were studied through mechanical testing and microstructural characterization (including optical, scanning and transmission electron microscopy). From the experimental observations the relationship between the microstructure and mechanical properties of both base materials and welded (or Gleeble simulated) heat affected zones (HAZ) are presented.

As for A710 base metal, its high strength and toughness were a result of fine ferrite grain size, less amount of pearlite compared with conventional HSLA steels, and uniformly distributed small Cu-containing precipitates. In the welded structure of both 19 mm and 32 mm thick A710 plates, the coarse grained heat affected zone had a lower impact toughness than the base metal, and this effect was greater at higher welding heat inputs. The cause for the HAZ toughness deterioration was mainly due to the formation of coarse bainitic microstructures in the coarse grained region. In spite of this toughness loss in the HAZ compared with the base metal, the weldability of A710 steels is very satisfactory, and the impact toughness levels of the HAZ (as well as the weld metal), especially for those made by heat input of 3 KJ/mm or less, are adequate for structural applications.

The effect of post weld heat treatment (PWHT) on the HAZ toughness is not consistent. PWHT provides a small improvement on the HAZ toughness for higher heat inputs (4.9 or 5 KJ/mm), but decreases

the toughness for lower heat inputs. From TEM studies, it was found that some of the Cu-containing precipitates are probably in the form of Fe – Cu compounds , and it is suggested that Cu – containing precipitates are responsible for the HAZ toughness changes (compared with as welded condition) after PWHT.

1 Introduction

In the modern construction industry, welding is one of the most important and versatile means of material fabrication. In fact, the ability to produce welded joints of high performance is essential for utility of most steels in engineering applications. Since most welded structures such as off - shore drilling platforms, pressure vessels, gas pipelines and off - highway mining truck frames are often exposed to severe service conditions, demands for materials with improved mechanical and fabrication properties are high and increasing. These demands include raising the yield strength to higher levels while maintaining good toughness values at various environmental temperatures, and at the same time satisfying other requirements such as weldability, formability, and economy of production. Consequently, high - strength low - alloy (HSLA) steels have been and are still being developed to meet these contradictory requirements. The advantage of HSLA steels, compared with carbon structural steels, is that they can provide: (1) higher strength, (2) improved toughness, ductility and formability, and (3) increased weldability.

For a welded structure, it is always desired that the mechanical properties of the welded joint region and the base metal be closely matched so that the welded structure will possess the structure integrity for the specific service conditions. However, this is not true in reality. When two metals are joined together by fusion welding, the original microstructures formed by prior thermomechanical processing both in the fusion zone and in the region next to the fusion line, i.e., the coarse grained heat affected zone (HAZ) are substantially destroyed as a result

of the very severe thermal cycles during welding. Even though the width of the HAZ is very narrow, its microstructure and property may govern the integral properties of a welded structure. In most instances, a welded structure with good impact toughness properties in the HAZ at the operating temperature is essential to the service of the entire structure.

For microalloyed HSLA steels, the toughness properties of the base metal are usually better than the HAZ (Deb, 1985). Among the HAZ problems are toughness deterioration due to the formation of coarse and/or hardened microstructures, and HAZ cracking. Both of the problems are often caused by the carbon and alloy content necessary to attain high strength levels using conventional steel hardening treatments. As a result, special welding procedures, extensive preheat or sometimes heat treatment have to be used in order to avoid these problems. Therefore, the development of new low alloy steels has been encouraged by the need for higher strength steels with improved toughness and resistance to hydrogen induced cracking in the weld HAZ. It has been recognized that these HAZ problems, especially HAZ cracking, can be reduced significantly when a precipitation-strengthened steel is used. This is because in these materials the strengthening is provided by precipitation hardening instead of carbon, thus carbon content could be reduced to the lowest amount possible. One such steel is ASTM specification A710 (Iron and Steel Products, 1986), a low carbon ($< 0.07\%$), copper containing (1.0 – 1.3%) steel. It was introduced more than a decade ago to the family of HSLA steels but did not receive acceptance because of the relatively high cost and potential hot shortness problems caused by copper. It is not until recent years that these steels are becoming popular for specific structural applications.

It has been shown in the literature that A710 base plates possess very high toughness, and the coarse grained heat affected zone almost always has a higher transition temperature (or lower toughness) than the base plate (Jesseman, 1984; Kvidahl, 1985; Jesseman, 1983; Wilson, 1987). However, the weldability of these steels is very satisfactory, and they are capable of being welded with little or no preheat. Cracking problems in the weldments have not been reported (Kvidahl, 1985; Anderson, 1987; Czuryca, 1988) although some recent work (McNutt, 1988; Lundin, 1987) has indicated some susceptibility to cracking in the HAZ associated with post – weld stress relief treatment.

In those published research there is also some evidence indicating that short time post weld heat treatment (PWHT) does not improve the toughness of the HAZ. However the existing testing data are not enough to clarify this suggestion. In addition, systematic investigations of microstructures, especially the fine structures in the HAZ, such as the behavior of copper precipitates in the base material as well as in the HAZ using transmission electron microscopy (TEM), are still limited. Much more detailed examinations, both qualitatively and quantitatively, are needed. There have been several studies of general defect structures in A710 base metal or weldments in the literature, but few correlations to mechanical properties were made. Thus in order to better understand these steels and to establish the suitability of A710 steels for more critical applications, there is a need for further research to obtain more results on the structure/property relationships for the base metal, HAZ and weld metals.

Therefore, the objectives of this research work have been: (1) to study the weldability and mechanical properties of A710 (Class 3)

weldments (both 19 mm and 32 mm plates) made by various heat inputs through impact toughness testing and microhardness measurements from real welds and weld simulation specimens respectively; (2) to examine the effect of post – weld heat treatment (PWHT) on the impact toughness and the microstructure of the HAZs; and (3) to study in detail the microstructural changes in the HAZ under as welded conditions and after PWHT, through optical, scanning electron microscopy (SEM) and TEM observations. Special emphasis has been on the observations of defect structures as well as the behaviour of copper precipitates in both base metal and HAZs under various welding and heat treatment conditions. Through these extensive studies, the relationship between microstructures and the impact toughness in the HAZ of A710 steels under various welding parameters, and also the role of Cu – containing precipitation in affecting the mechanical properties in the HAZ, have been more clearly understood. It is desired that these experimental results can be useful in developing methods for successful welding of A710 and similar steels.

2 Background

2.1 Description of the investigated A710 steels

Low-carbon, copper precipitation-aged plate steels were introduced in the late 1960's by the International Nickel Company, and the current specifications A710 for structural applications and A736 for pressure vessel applications were developed over 10 years ago by ASTM. A710 steels can be produced in three different classes: Class 1, as - rolled, class 2, normalized, and class 3, quenched and aged. All these classes are precipitation hardened at 540 to 665°C for about 30 to 60 minutes, and exhibit a wide range of tensile strength (450 – 650 MNm⁻²) as well as good impact toughness at low temperatures (Jesseman, 1983). In the present study, only class 3 (grade A) steels are involved, thus the following presentation will be focused mainly on class 3 steel, that is, quenched and tempered materials.

Figure 1 is the continuous cooling transformation diagram for A710 steels (Wilson, 1988). It can be seen that a range of microstructural constituents can be formed in this low carbon steel, but in practice proeutectoid ferrite is the primary microstructural component. Very small amounts of acicular ferrite, pearlite and martensite might also be formed. Detailed discussions on the material microstructures will be made in the experimental sections. The compositions of A710 steels used for this study are listed in Table 1 (Lukens Steel Company, 1986; Bolliger, 1986). The carbon content is restricted to below 0.07% so that high toughness levels can be developed, and cold cracking problems can be essentially eliminated even if the welding consumables could generate

**Table 1: ASTM A710 Grade A Class 3
Chemical and Mechanical Properties**

<i>Chemical Composition</i> (wt%)	<i>Specified</i>	<i>Measured</i>	
		<i>32 mm plate</i>	<i>19 mm plate</i>
C	0.070 Max.	0.050	0.050
Mn	0.400 to 0.70	0.530	0.500
P	0.025 Max.	0.008	0.010
S	0.025 Max.	0.003	0.002
Si	0.400 Max.	0.240	0.350
Ni	0.700/1.00	0.980	1.000
Cr	0.600/0.900	0.720	0.720
Mo	0.150/0.250	0.210	0.230
Cu	1.000/1.300	1.130	1.170
Nb	0.020 Min.	0.034	0.032
Al	not specified	0.027	0.018
N	not specified	0.008	—
V	not specified	—	0.003
Ti	not specified	—	0.005

Tensile Requirements

(thickness = 4.80 to 50.8 mm)

Minimum 0.2% Yield Strength	515 MPa(75 KSi)	602 MPa	560 MPa
Minimum Tensile Strength	585 MPa(85 KSi)	673 MPa	620 MPa
Elongation	20%		26%

Impact Properties (All thickness)

Longitudinal at -62°C (-80°F)	69J(50 ft-lbs)
Transversal at -62°C	47J(35 ft-lbs)

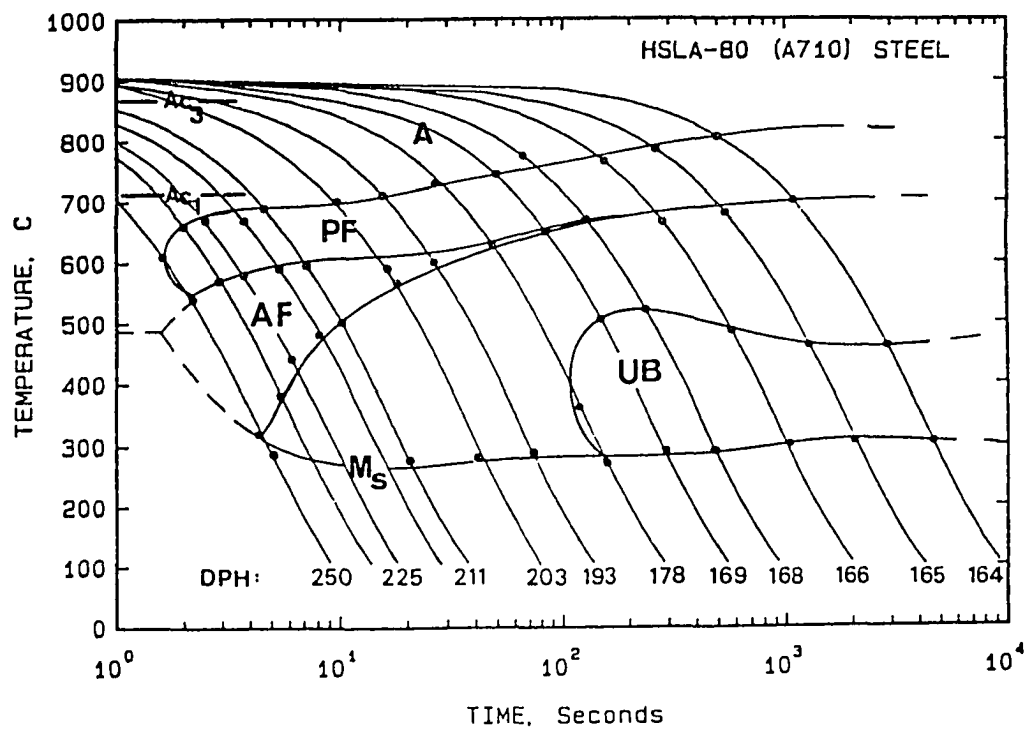


Figure 1: Continuous cooling transformation diagram for A710 steel

A = Austenite, PF = Proeutectoid Ferrite, AF = Acicular Ferrite, UB = Upper Bainite, M_s = Martensite start.

high hydrogen potentials. The 1.0/1.3 wt.% copper is the primary strengthener since copper can be precipitated from the supersaturated ferrite as a fine ϵ -Cu phase. Niobium, besides giving some precipitation hardening, helps to retard austenite recrystallization during hot rolling and makes grain refinement possible just as in conventional control – rolled C – Mn steels microalloyed with Nb (DePaul, 1970; Hydrean, 1971). Grain size control during the austenitizing of heat treated plates (class 2 and class 3) is also provided by the Nb carbon – nitride precipitates. Nickel prevents the potential hot shortness problem normally associated with Cu – containing steels (DePaul, 1970). The usual beneficial toughness effect of Ni is also obtained. Chromium and Molybdenum are added to retard copper – precipitation during cooling from the austenite range. Both elements are important in influencing the transformation kinetics of these steels, and particularly they increase bainitic hardenability by delaying the onset of proeutectoid ferrite formation. They are also important in retarding tempering during the aging treatment. This steel is deoxidized by Si, and contains Al for grain refinement.

2.2 The mechanisms of precipitation strengthening

Generally the yield strength of a alloy steel can be increased by solid solution hardening, dispersion and precipitation hardening, strain hardening and grain refinement. Chemistry, processing, and heat treating parameters can be varied to control the complex interactions between these mechanisms to produce a material optimized for a specific use. For microalloyed steels, their strength can be gained not only from

the direct effects of lattice friction, solute elements, and grain boundaries as the conventional predictions by the Hall – Petch equation (Hall, 1951; Petch, 1953), but mainly through additions of strong carbide and nitride formers which contribute to grain refinement as well as providing a fine distribution of precipitates for strengthening (DeArdo, 1985; Gray, 1973). As mentioned in the previous section, A710 steel is a material in which precipitation plays the major role in strengthening, therefore, it is worthwhile to make a brief review on the mechanisms of precipitation strengthening in order to provide a theoretical basis for the experimental results in the following sections.

Precipitation is produced by a fine distribution of a second phase in the material matrix in which the solute atoms exhibit decreasing solubility with decreasing temperature. Precipitation strengthening results when a metal, with the second phase atoms in solution, is quenched and then given an aging treatment to precipitate the second phase. The precipitates act to increase the yield stress by creating obstacles for dislocation motion. If the precipitate is coherent, the dislocation can cut through it, but requires a stress much greater than that required for the dislocation to move through the matrix (Lloyd, 1986). If the precipitate is incoherent and relatively strong, it acts as an impenetrable obstacle which the dislocation can move past only by changing the shape. For particles which can be sheared by dislocation passage, strengthening arises from the matrix strain resulting from any mismatch between the particle and matrix, including elastic moduli mismatch, the increase in surface area of the particle caused by passage of the dislocation, and the work done to move the dislocation through the particle.

The phenomenon of precipitation hardening was discovered in the first decade of this century by Wilm (1911), and the first plausible explanation for this “aging” process was presented by Merica *et al.* (1920), who postulated that age hardening occurred in alloys in which the solid solubility increased with increasing temperature, thereby enabling a new phase to form at the lower temperature by precipitation from an initially supersaturated solid solution. Since this discovery, extensive research on the mechanisms of precipitation strengthening has been undertaken through the following decades. The earliest attempted explanation of age hardening using the concept of dislocations was that of Mott and Nabarro (1940), who suggested that strengthening arose through the interaction between dislocations and the internal stresses produced by misfitting coherent precipitates. They determined that the yield stress was dependent on the magnitude of the internal stress and the mean wavelength over which it fluctuated. The wavelength L , which was found to be equal to the separation of solute atoms, was determined in relation to the radius of curvature, R , to which the internal stress, τ_i , was able to bend a dislocation. This curvature was given by

$$R = \frac{T}{b\tau_i} = \frac{\alpha Gb^2}{\tau_i} \quad (1)$$

where the tension T , equaled αGb^2 with α , a constant approximately equal to 1.0, b , the burgers vector, and G , the shear modulus. For increasing coarseness of the dispersion of solute atoms or precipitates, three regimes were distinguishable: (1) $L \ll R$, which is typical of solid solution; (2) $L = R$, which represents a precipitation hardened alloy at maximum hardness; (3) $L \gg R$, which represents a dispersion

strengthened material. In the last case ($L \gg R$), the dislocation moves forward by passing between obstacles leaving a closed dislocation loop behind. This mechanism was suggested by Orowan in 1947 (Orowan, 1984) to describe strengthening of alloys by non-deformable particles. The increase in yield stress due to particle “pinning” of a dislocation was determined to be given by

$$\Delta\sigma_y = \frac{2Gb}{L} \quad (2)$$

The stress to move dislocations between particles increases as each dislocation leaves a loop around the particles it has passed, which exerts a back stress on other dislocations in the slip plane. This mechanism of dislocation motion causes rapid strain hardening of the matrix. Based on the most widely accepted model of dislocation cutting by Ashby – Orowan (Ashby, 1966), the increase in tensile stress ($\sigma \approx 2\tau$) due to precipitation hardening can be quantitatively expressed as:

$$\sigma(MPa) = \frac{5.9\sqrt{f}}{\bar{X}} \ln\left(\frac{\bar{X}}{2.5 \times 10^{-14}}\right) \quad (3)$$

Where $\bar{X}$ is the mean planar intercept diameter of the precipitates, and f is the volume fraction of the precipitates. Since $(1 / \bar{X})$ is the predominant function of $\bar{X}$, the strengthening increment is inversely proportional to precipitate size, indicating that a fine dispersion of precipitates for a given precipitate fraction is most desirable for high strength.

Impressive contributions to the understanding of the mechanisms of precipitation hardening have been made since the 60's, when the progress was measurably accelerated by the application of transmission electron

microscopy to the examination of the microstructures of aged alloys. Based on the concept of precipitation/dislocation interaction, so far the following precipitation strengthening mechanisms have been suggested: (1) chemical strengthening, which results from the additional matrix - precipitate interface created by the dislocation when it shears through a coherent particle; (2) stacking fault strengthening, which occurs when the stacking fault energies of the precipitate and matrix phases differ; (3) modulus hardening, which occurs when the shear moduli of the matrix and precipitate phases differ; (4) coherency strengthening, which arises by virtue of the elastic interaction between the strain fields of a coherent misfitting precipitate and the dislocation; and (5) order strengthening, which operates when the crystal structure of a coherent precipitate is a superlattice and the matrix is a disordered solid solution.

2.3 The role of copper in Fe - Cu alloys

The precipitation hardening phenomenon in high - copper alloys has been recognized for over 60 years (Smith, 1933). Figure 2 (Speich, 1966), which is part of the Fe - Cu phase diagram, shows that over 3% copper is soluble in pure iron at 900°C. However, the presence of even 0.10% carbon reduces copper solubility to about 2%. Ferrite formed at high temperatures has a high copper solubility, but the amount of copper which can remain in solid solution drops to 0.35% or less at room temperature. Copper which is not soluble in ferrite forms fine particles which are copper - rich (96% Cu + 4% Fe). This Cu-Fe phase is called ϵ -Cu, and is responsible for the precipitation hardening effect of copper - bearing steels.

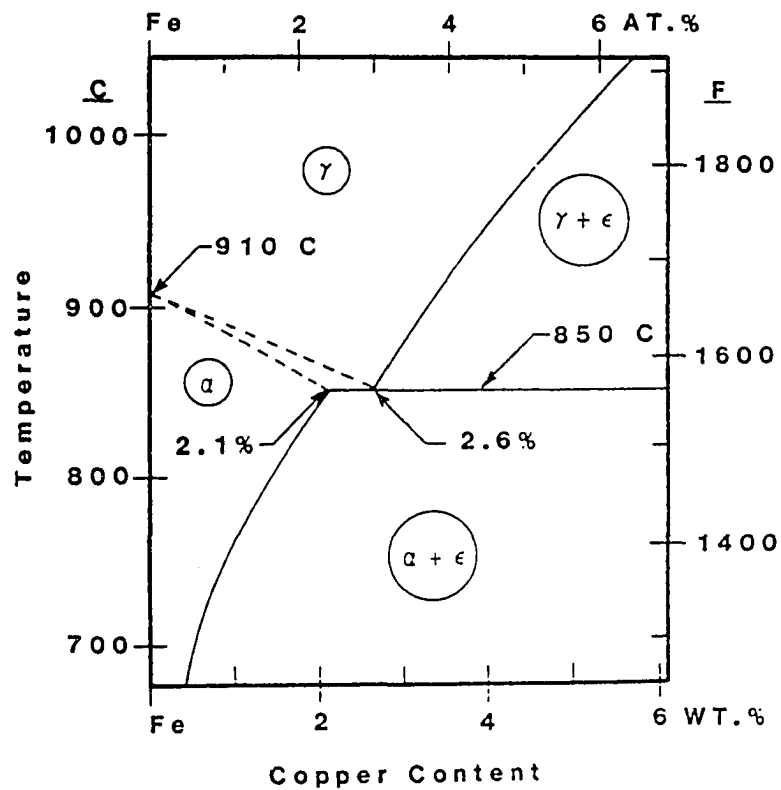


Figure 2: Iron rich portion of the Fe – Cu phase diagram

The presence of face centered cubic (fcc) ϵ -Cu phase in aged super saturated Fe - Cu alloys was first detected by X-ray diffraction (Wasserman, 1958), but soon confirmed by electron microscope replica techniques (Rickett, 1959; Hornbogen, 1960). It was found that considerable precipitation of copper takes place in steels containing 0.6% Cu or more when the steels are held at 1100 - 1300°F. In the early 1960's, Hornbogen and Glenn (Hornbogen, 1960) suggested that copper precipitates from α -Fe as a ϵ -phase with a fcc structure without formation of intermediate compounds. During aging, coherent, body - centered cubic (bcc) Cu - rich clusters are first formed in the underaged condition. The clusters at this stage would not be visible in the electron microscope since they are coherent and thus lack a strain field of sufficient intensity. Upon further aging, these Cu - rich precipitates grow and transform to an incoherent fcc structure when reaching the size of $\sim 90 \text{ \AA}$ in diameter. In the subsequent work on copper precipitation, Speich and Oriani (Speich, 1966) confirmed that the equilibrium shape of the particle is that of rods with hemispherical caps. This shape results from the large anisotropy due to the very poor atomic fit at the ends of the particle, which contrasts with the excellent atomic fit along the cylindrical rods. They also observed that these copper rods have a Kurdjumov - Sachs orientation relationship, $[011]_{\text{Cu}} // [111]_{\text{Fe}}$ and $(111)_{\text{Cu}} // (011)_{\text{Fe}}$. This relationship is frequently found for the fcc to bcc martensitic transformation in iron based alloys.

In the early 1970s, copper precipitation in iron was studied in detail by Goodman, Brenner, and Low (1973) using field ion microscopy. They observed that coherent, copper-rich clusters were first formed in ferrite during aging and grew by bulk diffusion. These bcc clusters contained

less copper than the equilibrium fcc ϵ -phase which eventually forms from these clusters upon further aging. The maximum strength was reached while the precipitates were still bcc with an average diameter of $24 \text{ \AA}$ and an average copper content of 40 to 60 percent. The strengthening is caused by the interaction between the glide dislocations and the elastic coherency stresses surrounding the coherent particles. In the overaged, incoherent stage, strengthening by these copper precipitates may be caused by a combination of coherency strains and Orowan strengthening as described in the previous section (Speich, 1966; Hornbogen, 1960; Speich and Oriani, 1966). In their subsequent study on Fe – Cu alloy, Hornbogen *et. al* (Hornbogen, 1974) suggested that when the size of ϵ -Cu particles was less than $50 \text{ \AA}$ (still coherent), precipitation hardening was through particle shearing by dislocations, and when the size exceeded $150 \text{ \AA}$, precipitation hardening was achieved by dislocation bypassing of particles, i.e., by the Orowan mechanism. Some researchers (Kathryn, 1972) also suggested that copper precipitation strengthening results from the difference in elastic modulus between copper and iron (since Cu precipitates are elastically softer than the Fe matrix), thus when a dislocation moves from the matrix into a particle of different shear modulus there is a change in dislocation energy and hence an interaction force between the dislocation and the particle.

2.4 The effect of welding on the structure and properties of the material joined

When two metals are joined by fusion welding, the materials are heated to their melting points and then cooled again rapidly under the restraint imposed by the geometry of the joint. As a result of this severe thermal cycle, the original microstructures and properties of the metal in the region close to the weld are changed. This region is called the heat – affected zone (HAZ). It is composed of a number of sub - zones as illustrated in figure 3 (Easterling^a, 1983). Each sub - zone refers to a different type of microstructure and, perhaps more importantly, each structure type is likely to possess different mechanical properties.

The microstructural changes in the HAZ depend on the composition of the materials to be welded, and the prior thermal and mechanical history of the material. The extent of these changes as well as the zone width is also determined by the welding heating and cooling cycles. The following two parameters which are related to the welding thermal cycle can affect the microstructure in the HAZ:

1. the time at high temperatures during which grain growth can occur;
2. the cooling rate or the cooling time. For welding of steels, the cooling time from 800 to 500°C ($\Delta t_{800-500^{\circ}\text{C}}$) is widely accepted as an index for the thermal conditions under which the austenite to ferrite transformation takes place (Easterling^b, 1983).

The weld thermal cycle is mainly controlled by the energy input (or

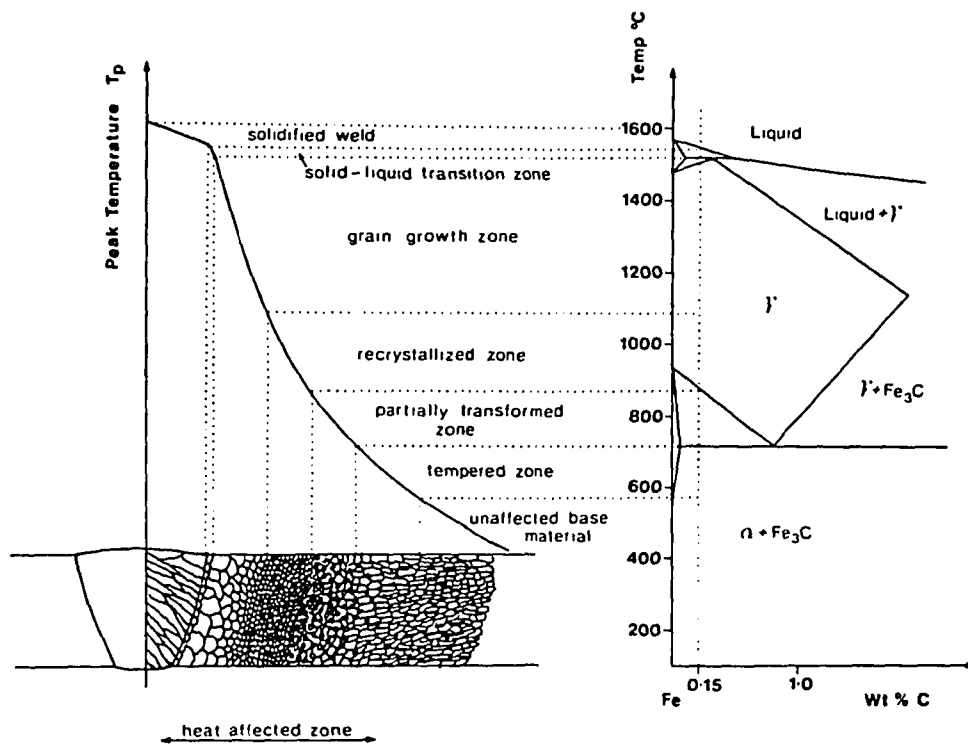


Figure 3: A schematic diagram of the various sub-zones of the HAZ (Easterling, 1983)

heat input) and also by the section thickness of the plates to be welded. These parameters determine the peak temperature, holding time in the temperature range, and the subsequent cooling rate. The heat input is controlled by the electrical current (A) and voltage (V) applied during welding, and the travel speed of the torch that holds the electrode. For a given material (given composition), the microstructure and grain size of the HAZ in the as - welded condition are governed by the proper choice of heat input and also the preheat temperature which is used when reducing the cooling rate is necessary. High heat input (lower cooling rate) will promote grain growth and softer microstructures. Lower heat input (higher cooling rate), on the other hand, will keep the grain size to a more favorable level, but may cause martensitic transformation. For each material to be welded, the proper combination must be chosen to obtain the required strength and toughness. The economical requirements are generally to avoid unnecessary preheating and to increase the feasible weld deposit size (higher heat input).

When welding heat treated (quenched and tempered) steels such as A710 class 3, the weld heat cycle will eliminate the effect of the prior heat treatment (quenching and precipitation hardening) in the HAZ, since the heat cycle will reaustenitize totally the grains in the grain growth zone and also the grain refined zone and partially in the partially transformed zone (see figure 3). In the lower temperature zones of the HAZ, there may be an overtempering and overaging effect. Thus the main concern in welding quenched and tempered steels is to have a sufficiently rapid cooling rate to create the microstructure necessary for the required strength. Consequently, lower heat input and low preheat (when needed) temperatures are most desirable for these steels.

2.5 Weldability

The weldability of a steel is a property which includes both the sensitivity to weld cracking and the toughness required by service conditions and test temperatures. It is a very complex property of steels, since in practice it is associated with many factors such as:

- solidification (hot) cracking,
- hydrogen (cold) cracking,
- lamellar tearing,
- weld metal brittleness,
- HAZ brittleness,
- stress relief embrittlement,
- stress relief cracking.

Among these categories, solidification cracking and hydrogen cracking are fabrication problems, while HAZ and weld metal toughness are service problems.

From the viewpoint of hydrogen induced cracking (HIC), the weldability of a steel is good when the steel has a low carbon equivalent, i.e., a factor incorporating the effects on hardenability of the common alloying elements. A simple empirical relationship, as a rough guide is (Easterling^c, 1983):

$$C_{equiv} = C + \frac{Mn}{6} + \frac{Cr+Mo+V}{5} + \frac{Cu+Ni}{15} \quad (in \text{ wt}\%)$$

where a steel with a carbon equivalent of less than 0.45 should be

weldable with modern techniques. The concept of carbon equivalent is introduced because the main hazard in welding is the formation of martensite in the HAZ which can lead to microcracks, and when a sufficient amount of hydrogen is present, hydrogen induced cracking is likely to occur in the HAZ as well as in the weld metal. This cracking can be avoided not only by control of the hardenability but also by preheating the welded area to provide slower cooling after welding. However, in some high strength steels, slower cooling may result in the formation of upper bainite in the HAZ which encourages cleavage fracture.

The weldability of microalloyed steels has been greatly improved, compared with traditional steels, in that the carbon contents in microalloyed steels have been progressively lowered to below 0.10%. The desired strength is largely achieved through a refinement of the ferrite grain size, which is produced by the additions of microalloying elements such as Al, V, Nb and Ti in combination with various forms of thermo – mechanical processing (Pickering, 1977). This procedure has made it possible to improve the resistance of steels to hydrogen – assisted cold cracking, stress corrosion cracking, and brittle fracture initiation in the weld HAZ region, without sacrificing base metal strength, ductility, or low temperature toughness (Suzuki, 1982).

Studies of the weldability of A710 steels have been progressing in the last decade. As mentioned in the introduction, A710 steels have been found to possess very good weldability, and they can be readily fabricated under shop or field conditions using a wide variety of welding processes. Little or no preheat is required for the steel because the low carbon content results in little quench hardening. This is shown schematically in the Graville diagram in figure 4 (Graville, 1986). Therefore, excellent

resistance is obtained to hydrogen – induced cold cracking in the HAZ of A710 steel weldments. These welding characteristics of A710 steel have been reported by several researchers in the literature (Kvidahl, 1985; Jesseman, 1983).

2.6 Material microstructure and impact toughness

For microalloyed steels, the mechanical properties such as the yield stress (σ_y), toughness (impact transition temperature, ITT) and ductility (strain to fracture, ε_F), are closely related to the microstructural features of the materials. These relations can be expressed as follows (Gladman, 1973):

$$\sigma_y = \sigma_1 + K_y(d^{-1/2}) + K_1(\text{dispersion}) \quad (4)$$

$$ITT = C_1 - K_1(d^{-1/2}) + K_2(\text{dispersion}) + K_3(\text{pearlite}) \quad (5)$$

$$\varepsilon_F = C_2 - f_1(\text{volume fraction})f_2(\text{shape}) \quad (6)$$

where d is the grain size. Hence the evaluation of mechanical properties as a function of microstructural changes is the basis for the development of high – strength notch – ductile steels and high strength formable steels.

Among the various techniques of ITT toughness testing, the Charpy V – notch impact test is the most widely used technique. Although the notch impact energy has a limited practical use compared with fracture toughness, Charpy testing is simple to perform and the test bars are cheap to produce. It is widely used in the welding industry since it can provide a useful quality control test for ranking the toughness of materials, and also provide a fairly reliable value of the transition

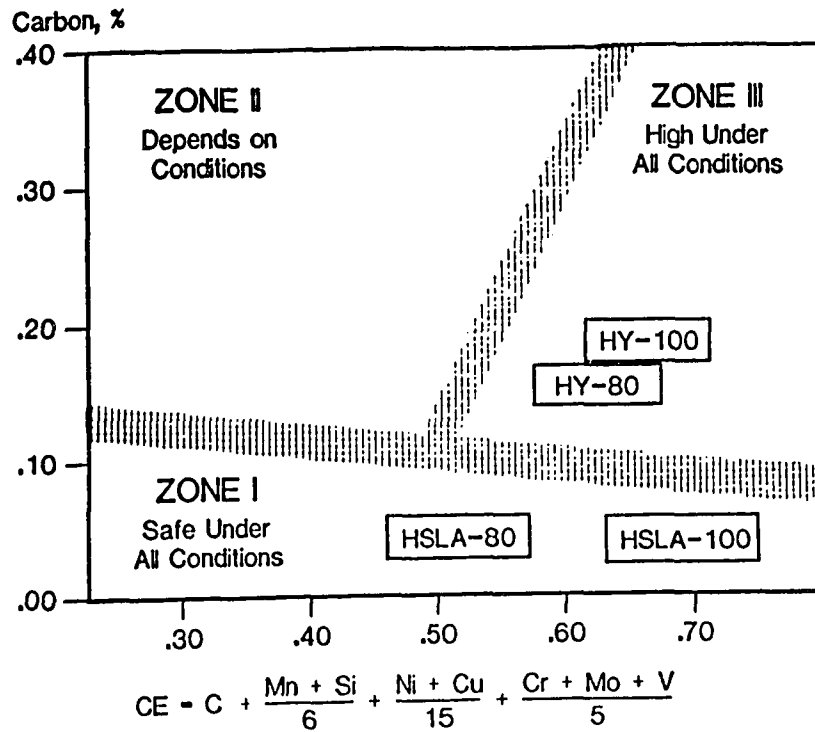


Figure 4: Weldability of steel as a function of carbon content and carbon equivalent from Graville

temperature. It will be shown from the following experimental procedure section that through the Charpy testing, a curve of energy absorbed in fracturing a notched specimen (related to the specimen impact toughness) as a function of testing temperature can be obtained. There is a transition from low - to - high energy fracture over a relatively narrow temperature range and this is associated with a change in micromechanisms of material fracture from brittle cleavage fracture to dimpled or ductile fracture. Thus material quality can be defined in terms of this ductile to brittle transition temperature (DBTT), which in the welding literature is usually specified as the lowest temperature at which a certain minimum energy (e.g., 47J or 35 ft-lbs in this study) can be absorbed.

2.6.1 Cleavage fracture

Any metal that exhibits strong temperature and strain – rate dependence of the yield strength is susceptible to brittle cleavage fracture. For example, all body - centered cubic metals including iron show a marked temperature dependence of the yield stress, i.e., the stress necessary to move dislocations. Under this condition of high stress, cracks will be likely nucleated by dislocation pile - ups (or dislocation coalescence) at the intersection of slip bands on two different slip systems, or by the intersection of a slip band with a grain boundary (Cottrell, 1958). This is the case for single phase materials, e.g., relatively pure iron. In multiphase materials such as steels, cleavage fracture (in ferrite) can be initiated by the cracks of a second phase such as cementite films (McMchon Jr., 1962). Plastic deformation can crack grain boundary cementite particle or cementite lamellae in pearlite so as

to produce micro-cracks which, in certain circumstances, propagate to cause catastrophic cleavage failure. Therefore, to prevent brittle fracture, that is, to lower the ductile - to - brittle transition temperature, all the factors which make crack nucleation and propagation possible must be minimized or eliminated. From the metallurgical view points, factors that can be controlled are:

1. austenite and ferrite grain size,
2. elements in solid solution in ferrite,
3. dislocation density and distribution,
4. second phases and their composition, amount, shape, and number density,
5. segregation of impurities of limited solubility.

Among these factors, grain size refinement is the most effective technique for lowering DBTT, and at the same time increasing the yield strength as indicated in equation (5) and (6). Petch (1959) has shown that for each unit increment in $d^{-1/2}$ ($\text{mm}^{-1/2}$), the transition temperature is decreased by 11.5°C for C – Mn steels. The smaller the grain size, the smaller the number of dislocations piling - up where a slip band arrives at a boundary, thus the local stress concentrations at grain boundaries will be correspondingly less. This situation will lead to less crack nuclei regardless of whether they are formed by dislocation coalescence or by dislocation pile-ups causing carbides to crack or by twinning interactions.

The best example to show the effect of a second phase on brittle fracture behavior is the pearlite constituent in conventional mild steels.

The toughness, as measured by DBTT decreases dramatically with carbon content, i.e., with increasing volume fraction of pearlite in the steel. The low toughness arises from the fact that the presence of pearlite in the microstructure provides sites of easy nucleation of cracks, particularly at the ferrite - cementite interfaces, together with the high work hardening rate, which restricts plastic deformation in the vicinity of the crack.

In microalloyed steels, small concentrations ($< 0.10\%$) of Nb, V or Ti are added, and the carbon level is usually less than 0.15% (and often below 0.10%), so that the carbide phase occupies only a small volume fraction. In this case, cementite is partly replaced by much finer dispersion of alloy carbides, NbC etc. On the other hand, precipitation also leads to some loss of toughness to the extent similar to that resulting from increasing the density of dislocations. Fine carbides of Nb or V increase the DBTT by about 0.3 to 0.5 °C for each 1 MPa increase in yield stress as a result of precipitation strengthening (Gladman, 1965). However, precipitation strengthening is probably the least detrimental of the strengthening mechanisms other than grain refinement.

In bainitic and martensitic steels, the austenite grains transform to lath structures where the lath width is usually between 0.2 and 2.0 μm . The laths occur in bundles or packets with low angle boundaries between the laths. Larger misorientations occur across packet boundaries. In such structures, the packet width (instead of austenite grain size) is the main microstructure feature controlling cleavage crack propagation.

Nickel in solid solution is generally known to increase toughness by lowering the transition temperature. This is because Ni can promote cross slip of screw dislocations (Leslie, 1981). Therefore, besides lower

carbon and grain refinement, the only way to maintain a low transition temperature is to increase the Ni content as the yield strength increases.

As will be shown later, most of the studies relating the roles of inclusions to fracture have been concerned with ductile fracture. However, a number of investigators (Morrison, 1975; Lowes, 1973; Chilton, 1980) have shown that nonmetallics can also influence the ductile - brittle transition. Toughness may be improved or deteriorated by the presence of inclusions. Large brittle inclusions can enhance brittleness by promoting cleavage crack initiation. On the other hand, a stress relaxation may be produced by interfacial decohesion in the case of elongated inclusions.

2.6.2 Ductile fracture

The higher temperature side of the ductile/brittle transition is associated with a much tougher mode of failure, which absorbs much more energy in the impact tests. This failure mode is often referred to as ductile fracture. It is now well established that ductile fracture is initiated by the nucleation of voids at second phase particles. In steels these particles are either carbides, sulphide or silicate inclusions. The voids form either by cracking of the particles, or by decohesion at the particle/matrix interfaces. Growth and joining of the voids leads to final fracture. So it is clear that the volume fraction, distribution and morphology of both carbides and inclusions are important in determining the ductile behavior of steels. Ductile failure can be associated with inclusions from the brittle alumina type to the much more ductile sulphide inclusions. However, the inclusions are more effective in initiating ductile cracks when they are above a critical size range. The

presence of particles in the size range 1 – 35 μ m broadens substantially the transition temperature region and also lowers the energy absorbed during ductile failure, the shelf energy. A fine dispersion of non - brittle type inclusions can delay cleavage fracture by localized relaxation of stresses with a concomitant increase in yield stress (Honeycombe, 1981). The addition of rare - earth metal is also effective in improving the toughness since rare earth elements can change the sulfide shape from stringers to equiaxed particle. Overall, any procedure that improves steel cleanliness will improve toughness in the ductile range.

3 Experimental Procedure

3.1 Material

For this research project, A710 (grade A) steel plates in the form of 244 cm $\times$ 762 cm with thickness of 32 mm (1.25 inch) and 19 mm (0.75 inch) respectively, were supplied by Lukens Steel Company. All the plates were in the class 3 heat treatment condition, that is, they had been solutionized at 1660°F, quenched in water and then aged at 1160°F (627°C) (60 minutes for 19 mm plates, and longer for 32 mm plates). The compositions of the plates were listed in Table 1 in section 2.1.

3.2 Aging treatment of A710 base metal

In order to understand the precipitation hardening behavior of A710 steel during the aging treatment, one piece (26 cm $\times$ 20 cm) of the steel plate was cut from the bulk plate (19 mm plate), and it was re-solutionized in a heavy duty furnace at 1650°F (910°C) for 60 minutes, and then quenched in cold water. The heat treated plate was then cut into many small specimens ($\sim$ 1.5 cm cube) for aging treatment in five small furnaces which were held at temperatures 455°C, 500°C, 550°C, 600°C and 650°C respectively. For each temperature, a number of specimens were tempered for different length of time, ranging from 0.25 to 100 hours. They were all air cooled following tempering. Finally macrohardness (Rockwell) testing was performed on all of these heat treated specimens.

3.3 Weldment preparations

In this investigation, all the welding was carried out by the flux – cored arc welding (FCAW) with a type T-75 electrode (AWS A5.20 E70 T-5), and 75% argon + 25% CO₂ gas shield. No preheat was used. Heat inputs of 3.0 KJ/mm and 5.0 KJ/mm, respectively, were employed for the welding of 19 mm plates, and heat inputs of 1.77 KJ/mm and 4.9 KJ/mm, respectively, for the welding of 32 mm plates. The welding parameters were summarized in Table 2. The welded plate geometry is shown in figure 5.

Table 2: Welding Parameters (with type T-75 electrode)
Used in This Study

<i>Plates</i>	<i>Heat Input (KJ/mm)</i>	<i>Wire Dia. (mm)</i>	<i>Voltage (V)</i>	<i>Current (A)</i>	<i>Travel Speed (in. /min)</i>
19 mm	3.00	1.6	30	350	8.30
	5.00	1.6	30	450	6.38
32 mm	1.77	1.6	30	250	10.00
	4.90	1.6	30	450	6.50

The welding joint was designed as a single bevel groove (with a bevel angle 32.5°) welded from one side with a back up strip. It was so chosen as to result in a fusion line which should be fairly straight and the fusion plane perpendicular to the plate surface, so that Charpy specimens for HAZ testing can be extracted from the welds. This was attempted by adjusting the angle of the torch, and by prepositioning the weld groove such that a flat HAZ perpendicular to the plate surface was maintained on one side of the weld joint. To achieve this result a number of

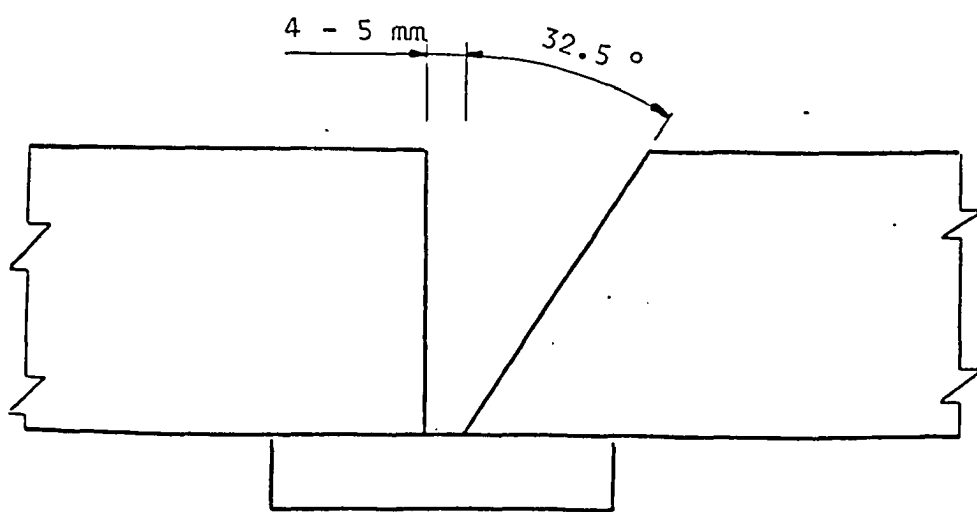


Figure 5: A schematic drawing of the weld chamfer

preliminary welds were made for practice prior to the primary weldments used for the following mechanical property testing. Figure 6 is a example of the final welded joint, and figure 7 shows how the Charpy specimens for HAZ toughness testing were extracted. The welding direction for all welds was parallel to the rolling direction of the plates in order to produce transverse orientation Charpy specimens. For each welding condition (each heat input), at least two welds (each 18 inches long) were made so that there was sufficient material for Charpy test specimens for the HAZ as well as for the weld metal under conditions of as - welded and various post - weld heat treatments.

3.4 Post - weld stress relief heat treatment

The purpose of post - weld heat treatment is to reduce the residual stresses caused by welding, and to change the metallurgical properties, and accordingly the mechanical properties, of the weldments.

The heat treatment for both thickness plates was performed only on the welds for HAZ testing. The welds were heated in a furnace at 620°C (1150°F), with one half of the welds held for 2 hours and another half held for 10 hours, followed by air cooling.

3.5 Weld simulation

Weld simulation is to duplicate the weld thermal cycle experienced by the real welded materials on a properly prepared “over size” Charpy blank. This is especially helpful in studying the microstructural and property changes in the HAZ. In the case of Charpy specimen preparations for HAZ testing, it is difficult to locate the notch exactly on

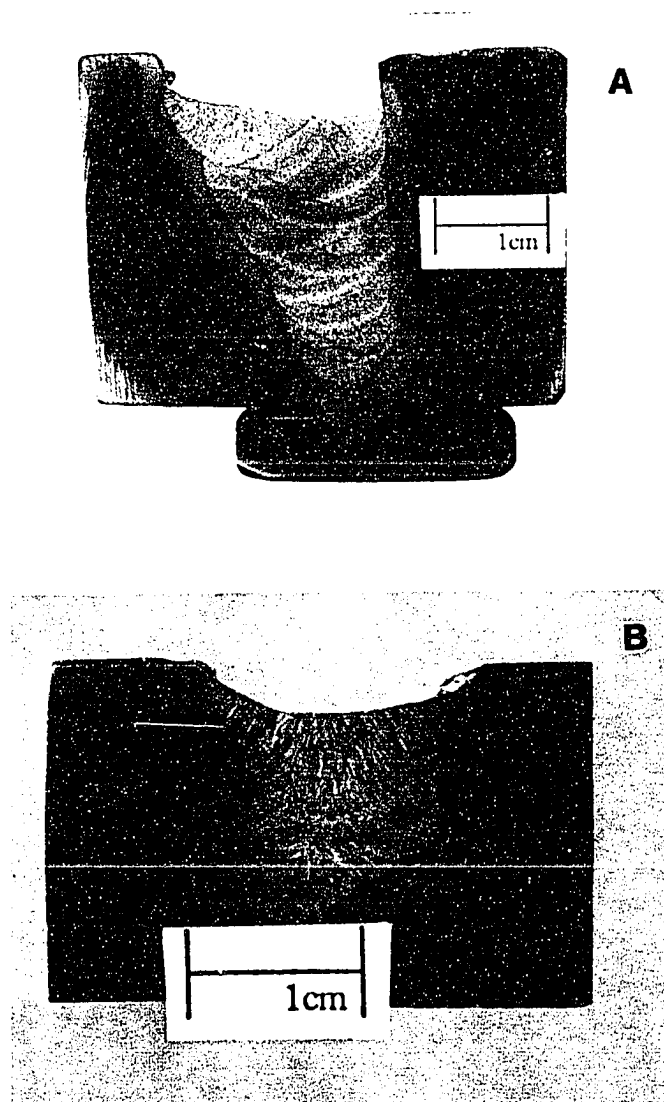


Figure 6: The cross section of the weldments

A) 32 mm plate, 1.77 KJ/mm,

B) 19 mm plate, 3.0 KJ/mm.

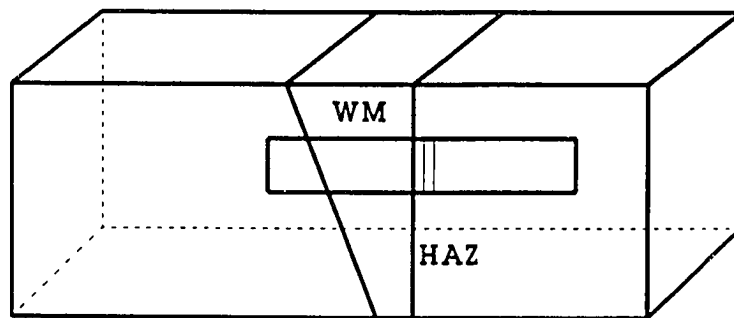
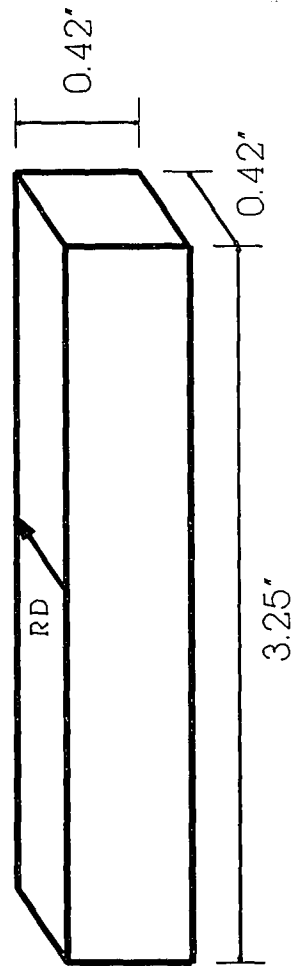


Figure 7: A schematic diagram showing how the Charpy specimens for HAZ toughness testing are extracted through thickness.

the grain coarsened zone in the weld because this zone is very narrow, and the fusion line is not perfectly straight. Therefore it is more convenient to work with test specimens with a relatively large width of a single microstructure and grain size. Consequently a weld simulation technique using a Gleeble machine has been developed to serve these purposes (Easterling^d, 1983).

The choice of the weld thermal cycle for simulation of the coarse - grained zone is not very easy, since it is difficult to obtain an experimental thermal cycle corresponding to a given peak temperature representative of the coarse - grained zone and then to choose a practical cooling rate in the 800 – 500°C range for the given plate thickness and joint geometry. For this study, the thermal cycle, which was expected to simulate the microstructures in the HAZ 0.5 mm from the fusion line in the real weld, was calculated empirically using the calculation methods provided in the literature (Nippes, 1958; Adams Jr., 1958). A “Gleeble” computer – controlled thermal simulation device was employed for this experiment. A chromel – alumel thermocouple welded onto the midpoint of the specimen provided the feedback signal for control of the thermal cycle. The gleeble specimens were mounted in water – cooled copper jaws. The parameters which were used in the simulation experiment, along with the size and geometry of the gleeble specimen are indicated in figure 8. All the gleeble simulation work was conducted in the Department of Materials Science and Engineering , The University of Tennessee. Only the specimens from 19 mm thick plate were tested for this purpose.



Parameters used in the Gleeble testing:

Peak temperature T_p : 1293°C (2360°F)

Heating rate: 121°C/S

$\Delta t_{800-500^\circ\text{C}}$: 12 seconds

Figure 8: Shape and dimensions of a gleeble specimen

3.6 Mechanical property testing

3.6.1 Charpy impact testing

As has been indicated earlier, Charpy V – notch (CVN) impact testing is a rapid and inexpensive way of material toughness measurement. Thus this testing was employed for material toughness evaluation in this study.

Standard size Charpy specimens were machined from the base metal, weld metal and HAZ respectively. For HAZ testing specimens (as has been drawn in Fig. 7), firstly the notch was placed at a distance of 0.5mm from the fusion line on etched specimen blanks which were sliced from the welds, then the specimens of standard Charpy specimen length were cut from these blanks. For the gleeble specimens, the surface layer of the simulation – treated blanks were removed by grinding to a cross section size of a standard Charpy specimen, and then the notch was located at the center of the specimen where the thermocouple had been placed during the thermal cycle. For the HAZ specimens from the 32 mm thick plates, two specimens were obtained through the plate thickness as shown in figure 7.¹ Only one layer of specimens was obtained from the 19 mm thick plates. Impact testing was conducted in accordance with the standard method described in ASTM A 370 and ASTM E 23. Alcohol or Methylbutane (for very low temperatures) was used for coolant.

¹Some of the specimens from the bottom portion of the welds are subsize Charpy specimens due to the limited thickness of weld with straight fusion lines.

3.6.2 Microhardness measurements

Microhardness testing is a quick and convenient way to characterize the microstructural and mechanical property changes across a narrow region, such as the HAZ. In this study, the hardness tests were used to determine the HAZ microhardness profile and also the location of the maximum HAZ hardness under as – welded condition, and any possible changes of this maximum value after post - weld heat treatments. Vickers hardness measurements were made using a Leco 400 – 1 microhardness tester. A load of 100 gram was applied and loading time for each testing point was 20 seconds. Prior to the hardness testing, all the specimens were polished and then etched by nital. The measurements were made by taking a microhardness traverse across the HAZ on each specimen. The value for each point on the microhardness figures (in the following section) was the average of at least 3 measurements.

3.7 Microstructural characterization

3.7.1 Optical microscopy

Optical examinations were conducted on the base metal, weld metal, and HAZ under all welding conditions. The Objectives of the examination by optical microscopy were:

- to identify the gross microstructures in terms of type and amount of transformation products;
- to observe the extent of the heat affected zone from different thermal cycles (heat inputs);

- to produce a measure of the grain size in the base metal and the prior austenite grain size in the coarse grained HAZ.

For optical examinations, sections were taken through the thickness of the weldment, and they were ground and polished using standard metallographic techniques. The microstructures were revealed by etching in 2% nital solution.

In examining the HAZ, the emphasis was placed on characterizing the structure of the coarse grain region immediately adjacent to the fusion boundary. This region is the least tough with respect to HAZ fracture toughness properties, and the tip of the Charpy V-notch in the impact test specimens is positioned within the coarse grain region.

During the optical examinations the grain size of the base metal and the HAZ next to the fusion line were measured by using ASTM standard E112 method.

3.7.2 Transmission electron microscopy

Transmission electron microscopy (TEM) has become a very important tool for fine structure study in the field of material science. In this study, TEM was employed to study in detail the defect structures and precipitation behavior in A710 base metal and in the HAZ respectively. Since A710 class 3 is a copper precipitation strengthened steel, most of the TEM work has involved the examinations of copper – containing precipitates, including: (1) precipitate identification by selected area diffraction (SAD) in the TEM mode, and by X-ray analysis from energy dispersive spectrums (EDS) in the mode of scanning transmission electron microscopy (STEM); (2) precipitate size measurements in the thin foil specimens from base metal as well as the

HAZs; (3) observations on the interactions between dislocations and precipitates, and (4) examining the effects of weld thermal cycle and PWHT on the changes of size and morphology of the precipitates.

In this study, all the TEM work was conducted on thin foil specimens. Three mm discs for HAZ specimens were made by first slicing a thin section (~0.5 mm thick) from the bulk weld using a diamond saw, then this section was ground to a thinner foil, polished and etched to reveal the HAZ, and then the fusion line was marked by a sharp point. Three mm disks were punched out of this foil by using a electrical discharge machine (EDM). Since the discs must contain the coarse grain zone (hopefully in the center), when punching the disks, the copper tube, which determines the location of the disc, was very carefully positioned by following the mark on the fusion line. This is shown in figure 9. The disks were further thinned by mechanical grinding to about 50 μm . Final electropolishing of the disks was carried out in a twin jet electro - polisher using a solution of 98% methanol and 2% perchloric acid. Polishing conditions were – 45°C, 80 volts and 40~50 mA. After electropolishing, the specimens were “dusted” on the ion beam thinner (IBT) for 8~10 minutes on hot stage.²

The three mm disks for base metal were made by first machining a long rod (along the rolling direction) in 3 mm diameter, then slicing it with a diamond saw. The final thin foils were also “dusted” on IBT.

The thin foils were examined on the Philips 400 with an EDAX – 9900 X – ray microanalytical system attached. TEM study was conducted

²This was done because it was found that, after being electropolished, the surface of the thin foil specimens was covered by many large, apparently artificial particles which were also found by other investigators (Michael, 1988).

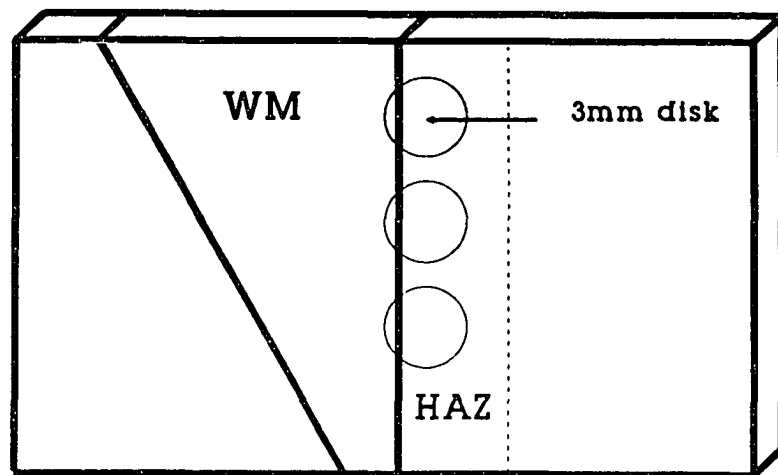


Figure 9: A schematic drawing showing the method of 3 mm disc extraction from the HAZ

only on the 19 mm thick plates including base metal, weldments and gleeble specimens.

After precipitation identification and general defect structure observations, the sizes of the precipitates in the base metal and in the HAZ after various types of PWHT were measured by using the Micro Plan II Image Analysis System. Measurements were all made on enlarged photographs of TEM bright field images. For each curve of size distribution (shown later), measurements on at least three photographs with the most uniformly distributed precipitates were made.

3.7.3 Scanning electron microscopy (SEM)

The fracture surfaces of selected Charpy specimens from the 32 mm and 19 mm plate weldments as well as gleeble specimens were examined using the ETEC SEM equipped with an EDAX detector, operating at 25 KV. The purposes of the SEM examination were to examine the microstructures (on polished and nital etched specimen surface) in comparison with the optical structure, and to identify the microstructural factors (such as inclusions, grain boundaries and bainitic or martensite laths) that are important in controlling the brittle fracture of the coarse-grained HAZ.

4 Experimental results and discussion

4.1 A710 base metal

4.1.1 Optical microstructures

The microstructures of A710 base metal were predominantly ferritic in nature, consisting of polygonal ferrite and a small amount of acicular ferrite and pearlite. Typical optical microstructures of the as – received A710 class 3 steels for the present study were shown in figure 10. For the thinner plate (19 mm), some bainite or martensite was expected to be present. However, these phases have not been found. They were either not existed or the amount was too small to be detected under optical microscope. Even with the level of alloying elements (Cu, Ni, Cr, and Mo) present in the steel, the characteristics of this steel, i.e., fine grain size and low carbon content, limit the transformation of low temperature products.

The average grain size of both thickness plates (in the direction perpendicular to the rolling direction) has been measured under the optical microscope at 400× magnification. The grain size was 7.1 μm (in diameter) for 19 mm plate, and 5.9 μm for 32 mm plate.

4.1.2 Aging behaviour

The aging response of 19 mm thick base plate of A710 steel at five aging temperatures is shown in figure 11.³ The hardness values were measured from samples in the as – quenched condition as well as from

³ The measured hardness numbers Rockwell C or B were converted to Brinell hardness numbers.

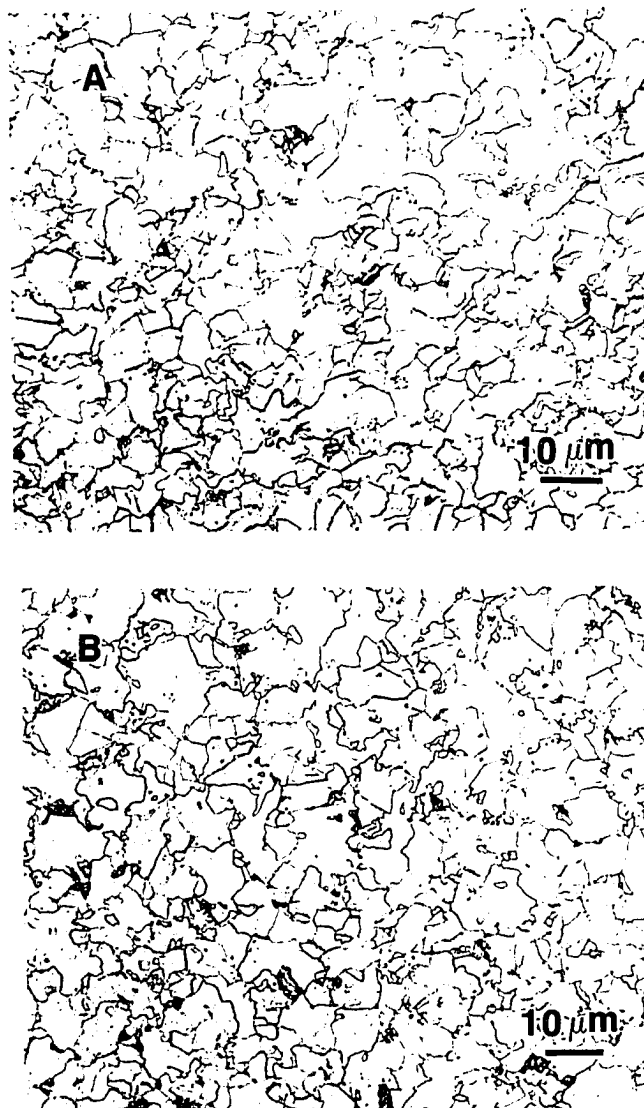


Figure 10: Optical microstructures of as received A710 base metal
(a) 19 mm plate, (b) 32 mm plate.

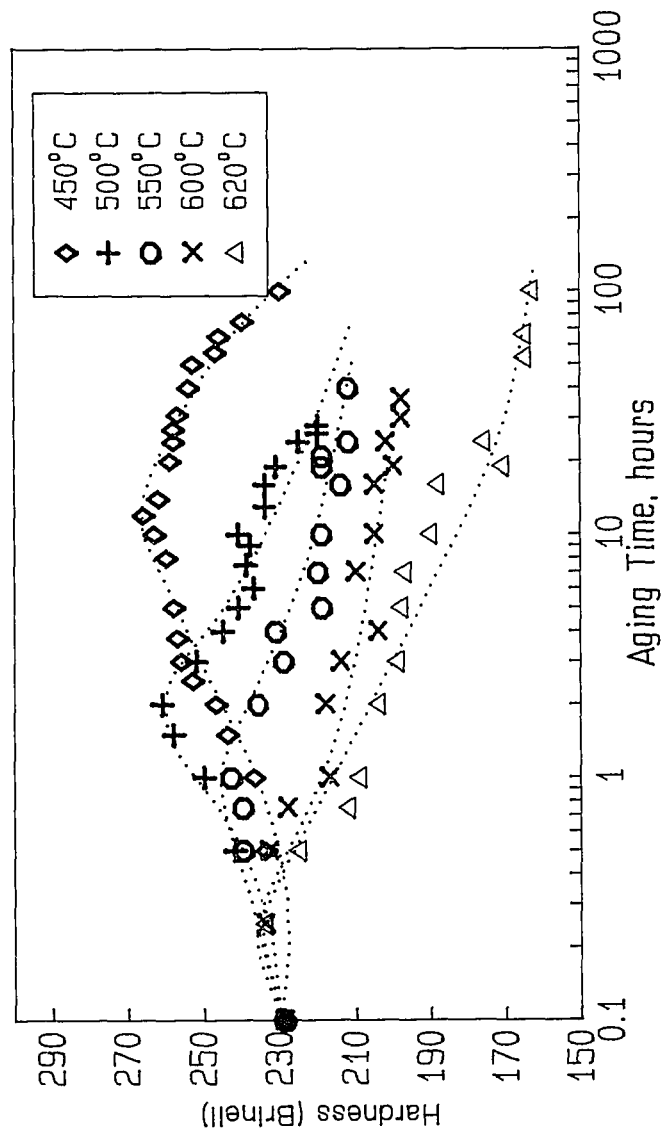


Figure 11: Precipitation hardening of A710 steel

samples aged at temperatures from 455°C to 650°C for various length of time.

Aging hardening occurs when supersaturated alloy elements precipitate from the matrix at the aging temperature and these precipitates raise the strength. The strength increase more than offsets the microstructural softening which occurs simultaneously during aging. Therefore a peak hardness will occur due to the precipitation effect. It can be seen from figure 11 that the hardness vs. aging time curves are typical of a age – hardening alloy. The hardness difference between the as - quenched and peak aged condition are nearly as big as that reported in the literature (Hornbogen, 1960). The most effective precipitation hardening in the steel occurred between 450 ~ 500°C, which was consistent with the results reported by several research groups (Jesseman, 1984, Michael, 1988). When the aging temperature reached to 500°C or higher, the time to reach the maximum hardness was quite short for these steels, usually about 15 ~ 60 minutes. When the precipitates grow to sizes >10 nm in diameter (Michael, 1988), they are relatively ineffective in providing precipitation strengthening and thus the hardness gradually decreases.

4.2 Microhardness

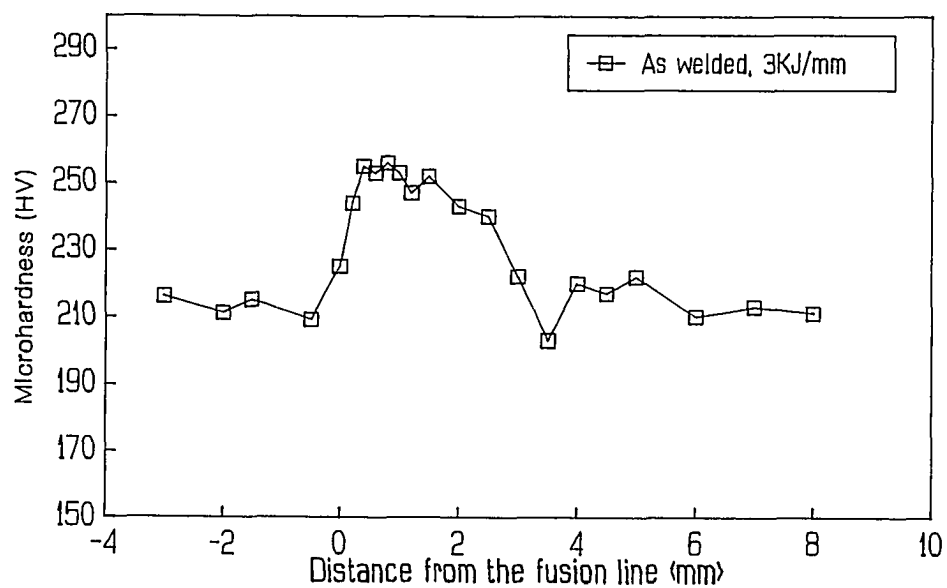
The results of microhardness tests across the HAZs of the welds for both 19 mm and 32 mm thick plates, including as-welded and PWHT conditions, were presented in figures 12 ~ 15. These results have shown that there is a hardness peak just next to the fusion line for all of the welds, which indicated that hard microstructures had been produced in

this region as a result of welding process. For the welds of both thickness plates, the maximum hardness in the as-welded conditions is about 250 VHN for either heat input. A small softened or overtempered zone appears in the HAZ at about 3 mm from the fusion line for lower heat inputs (1.77 KJ/mm and 3 KJ/mm), and about 4.5 mm from the fusion line in the case of higher heat inputs (5 KJ/mm and 4.9 KJ/mm).

The effect of PWHT on the peak hardness of the HAZ for 32 mm welds was not significant in that the profiles showed only small changes. In the case of 19 mm inch plate welds, the effect seemed to be stronger. PWHT for 2 hours raised the peak hardness to about 270 VHN or greater, while 10 hour - treatment caused the peak to decline a little to about 260 ~265 VHN for both heat inputs.

The microhardness testing for the maximum hardness values was also made on the gleeble specimens in order to make a comparison between the real weld HAZ and the simulated conditions. This comparison is given in Table 3.

It was shown that the peak hardness values for the simulated samples were higher compared with those in the weld HAZ for almost all heat treatment conditions. This was probably because harder structures were produced in the simulated conditions due to faster cooling.



(a)

Figure 12: Vickers microhardness traverse on the weld cross section of A710 steel (19 mm plate, 3 KJ/mm)

- (a) as welded,
- (b) PWHT at 620°C for 2 hours,
- (c) PWHT at 620°C for 10 hours.

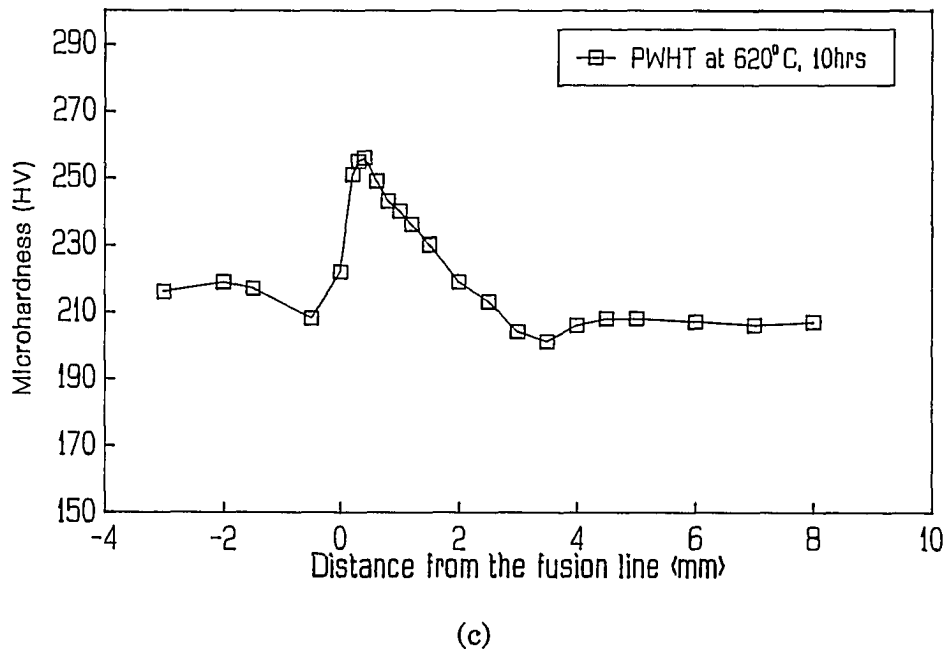
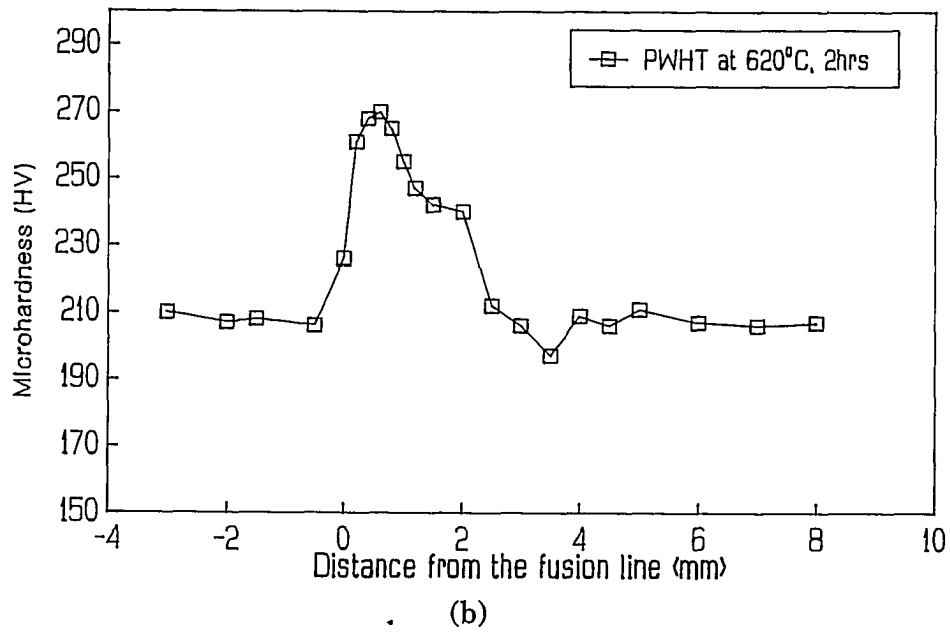
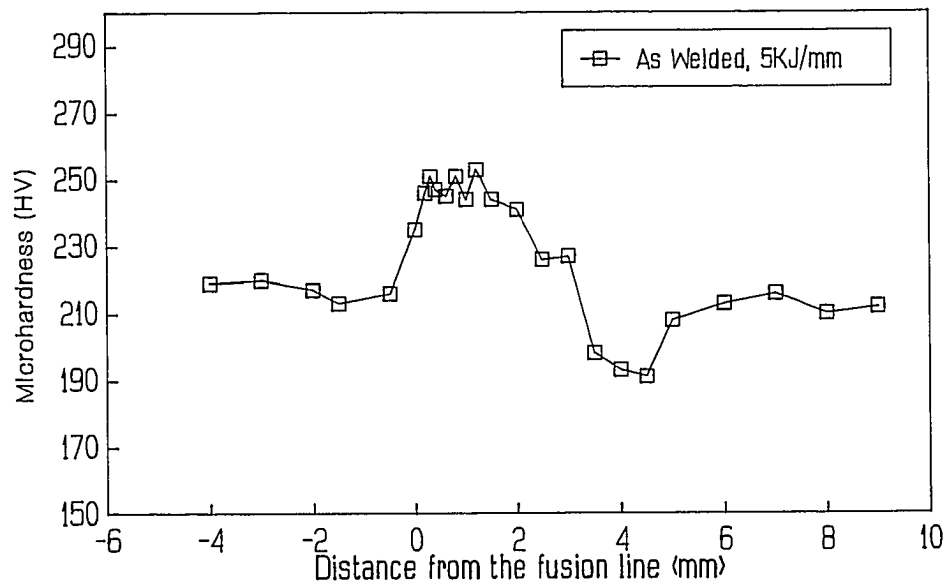


Figure 12: continued



(a)

Figure 13: Vickers microhardness traverse on the weld cross section of A710 steel (19 mm plate, 5 KJ/mm)

- (a) as welded,
- (b) PWHT at 620°C for 2 hours,
- (c) PWHT at 620°C for 10 hours.

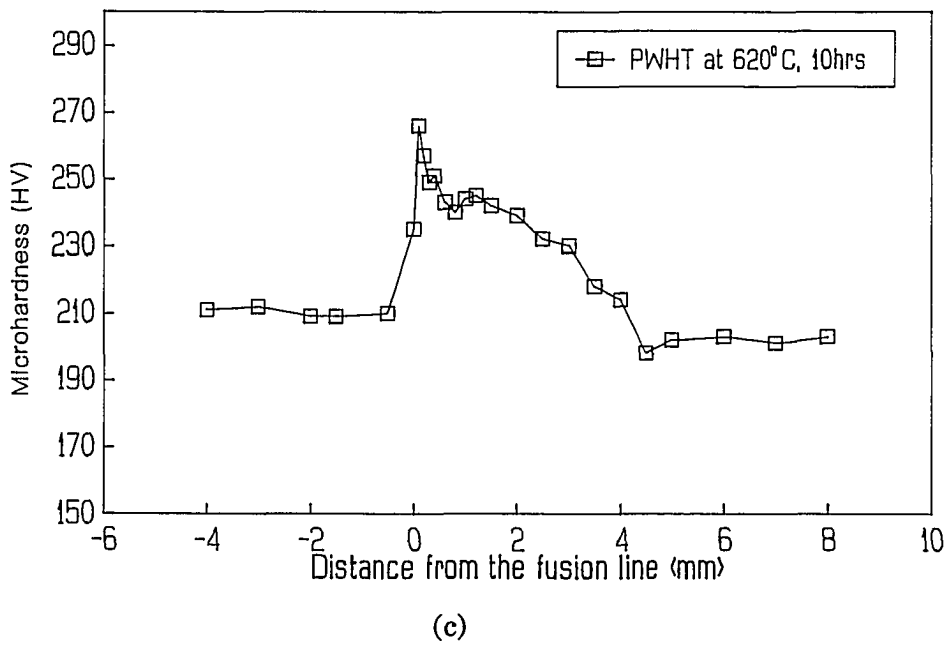
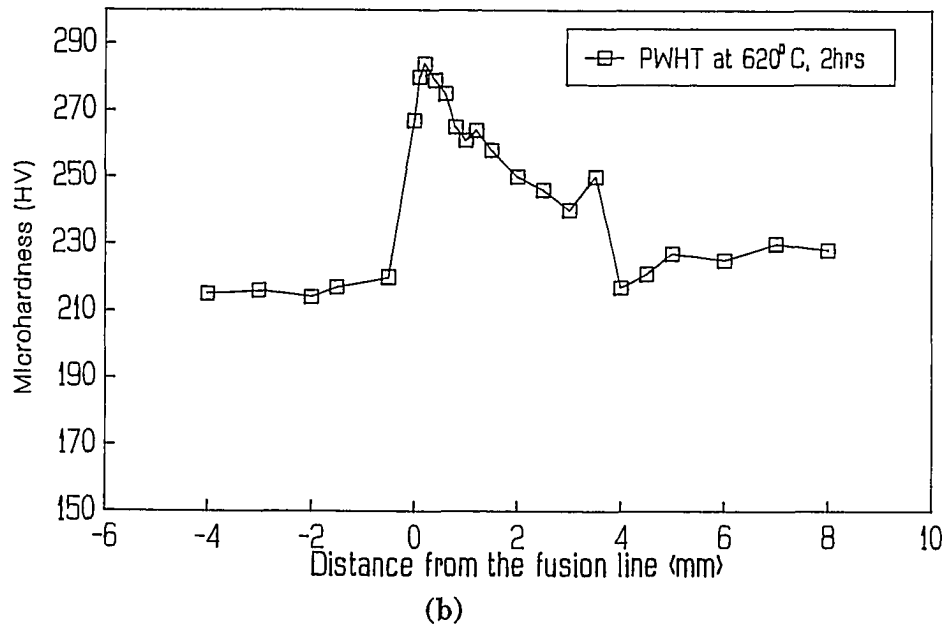
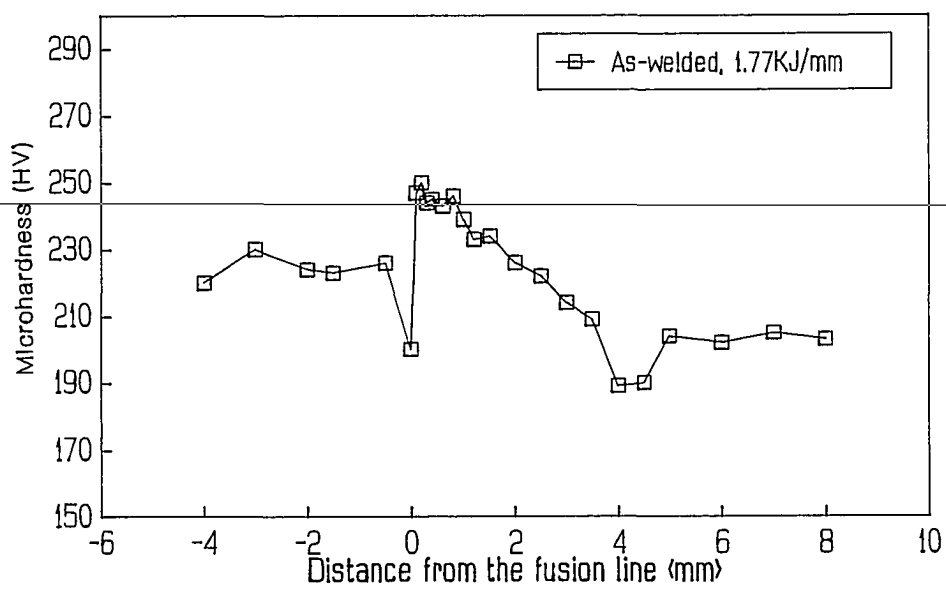


Figure 13: continued



(a)

Figure 14: Vickers microhardness traverse on the weld cross section of A710 steel (32 mm plate, 1.77 KJ/mm)

- (a) as welded,
- (b) PWHT at 620°C for 2 hours,
- (c) PWHT at 620°C for 10 hours.

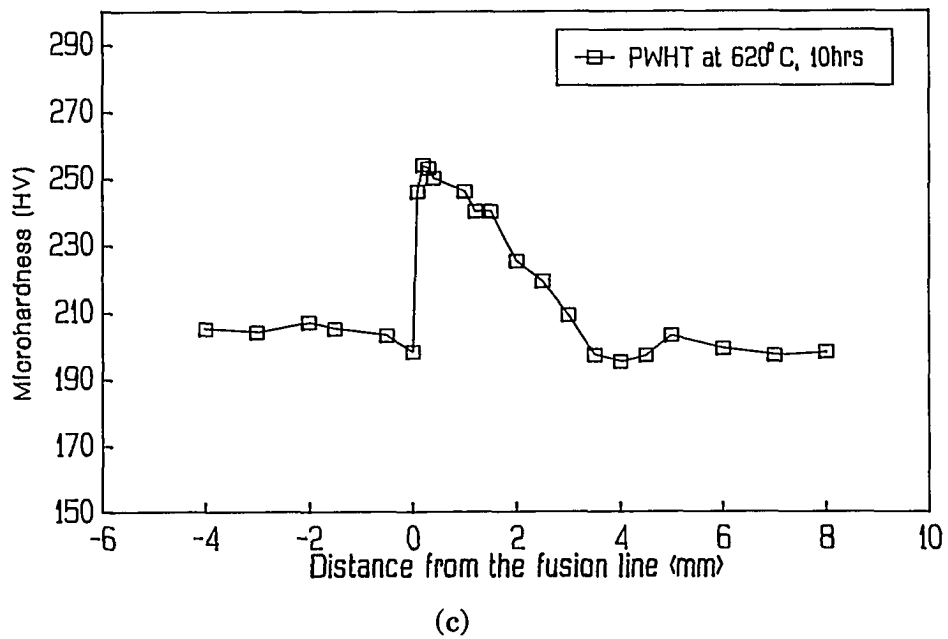
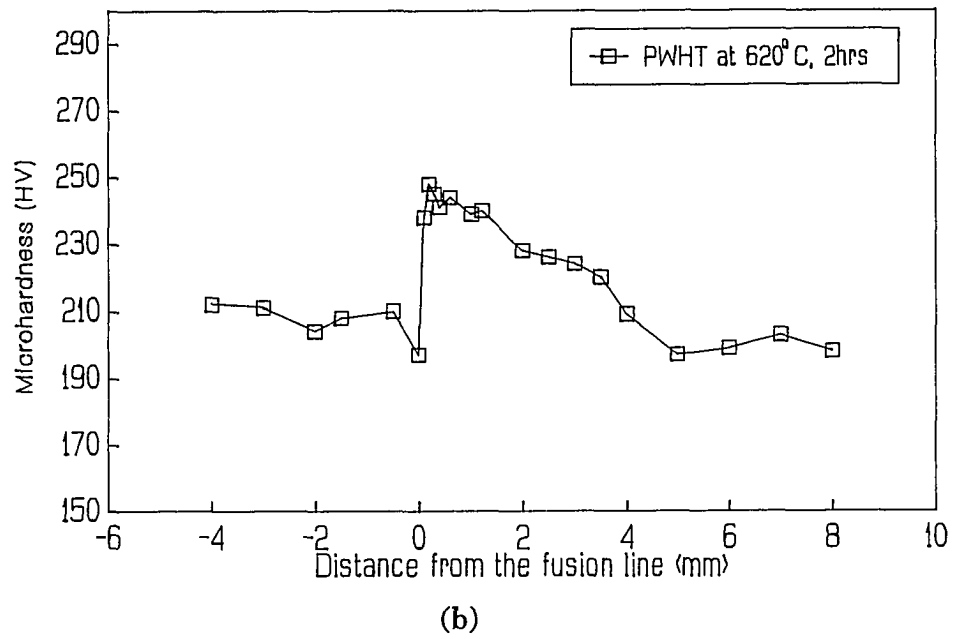
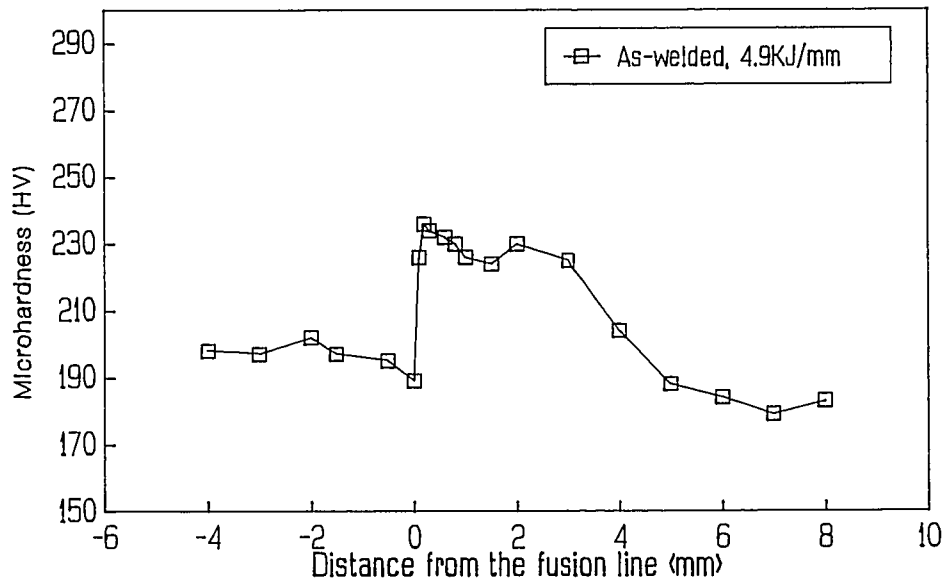


Figure 14: continued



(a)

Figure 15: Vickers microhardness traverse on the weld cross section of A710 steel (32 mm plate, 4.9 KJ/mm)

- (a) as welded,
- (b) PWHT at 620°C for 2 hours,
- (c) PWHT at 620°C for 10 hours.

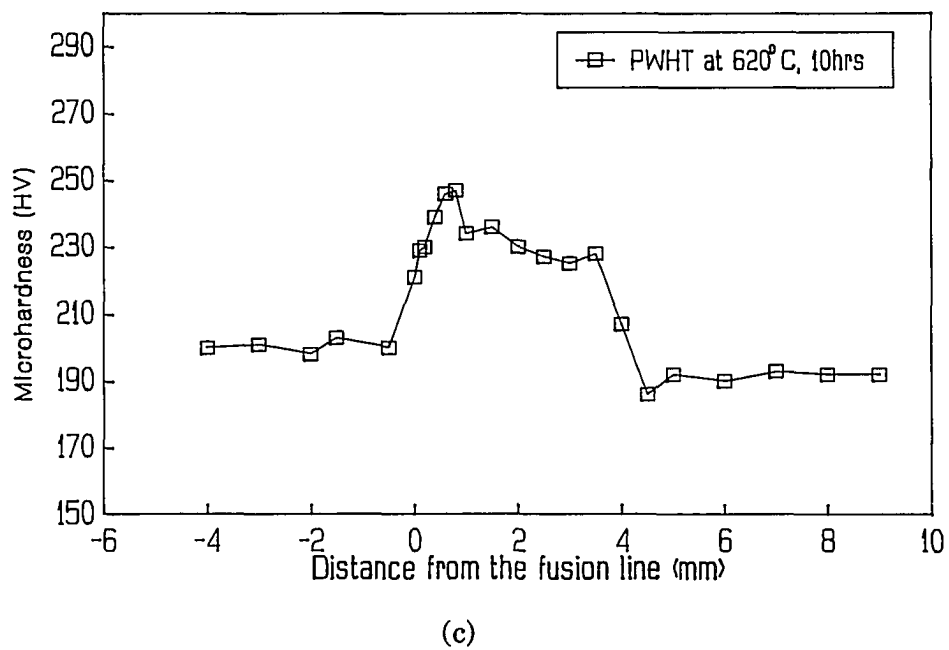
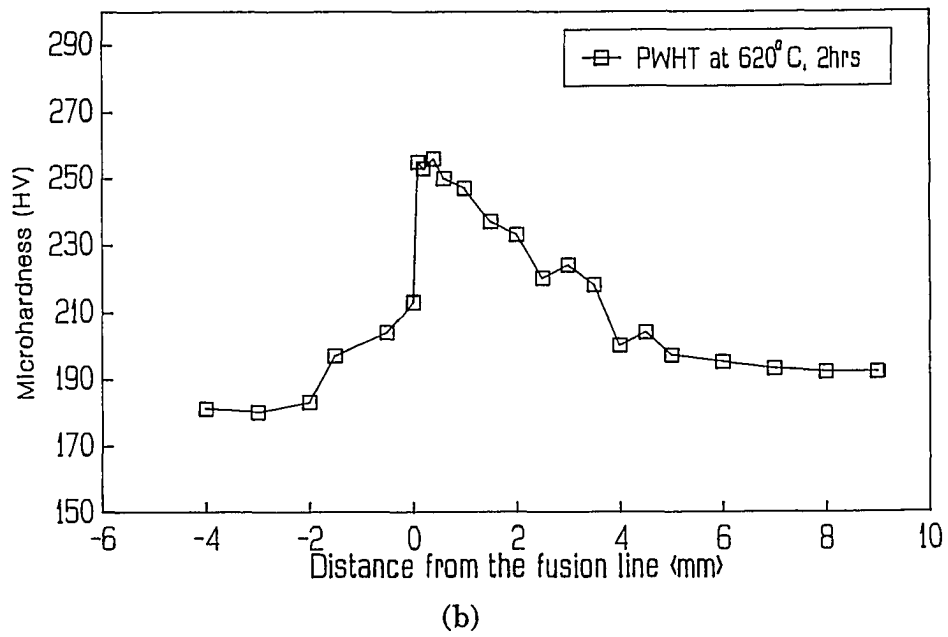


Figure 15: continued

Table 3: Microhardness of Coarse Grain Zones of Gleeble and Welded Specimens (HI = 3 KJ/mm, 19 mm)

<i>PWHT at 620°C</i>	<i>Microhardness (HV)</i>	
	<i>Gleeble Specimen</i>	<i>Weld HAZ (Max.)</i>
as-welded	270	256
2 hours	285	270
10 hours	255	256

4.3 Charpy impact toughness

The impact energy absorption vs. temperature curves for 19 mm base plate, weld metal, HAZ and gleeble specimens are shown in figures 16 ~18, and figures 19 ~20 for 32 mm weldments. A summary of the tested data is provided in Table 4.

For the base materials, it seemed that the 32 mm plate was somewhat tougher than the 19 mm plate, and this was also retained in the HAZs after welding. However, overall it was apparent that the impact toughness of A710 class 3 base plates was excellent. The typical 47J (~35 ft-lbs) transition temperatures were almost down to the range of -135 ~ -145°C, and also the upper shelf energies of both plates were very high, mostly due to the properly controlled inclusions of fine size and spherical shape as shown in figure 21.

The data from the HAZ specimen testing indicated that the coarse grained HAZ resulting from the weld thermal cycle was always low in toughness, and therefore higher in transition temperature, than the base plate material. It was also indicated from these results that higher heat input, which resulted in slower cooling rate and hence larger extent of coarsened microstructures, caused lower toughness in the HAZ. But

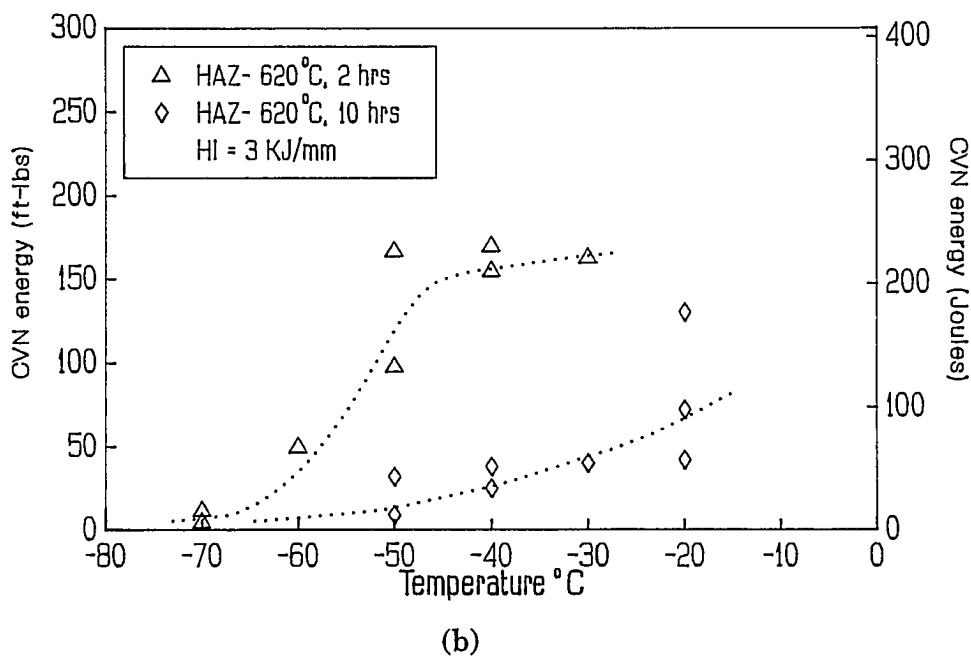
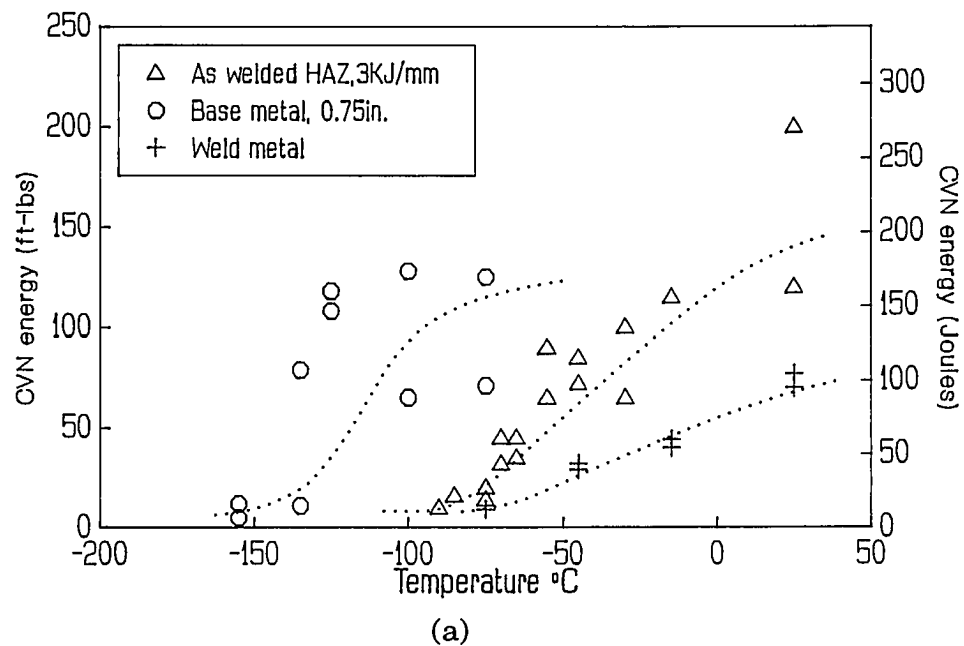


Figure 16: Charpy transition curves of A710 weldments (19 mm plate, 3 KJ/mm)

(a) as welded, (b) HAZ, after PWHT.

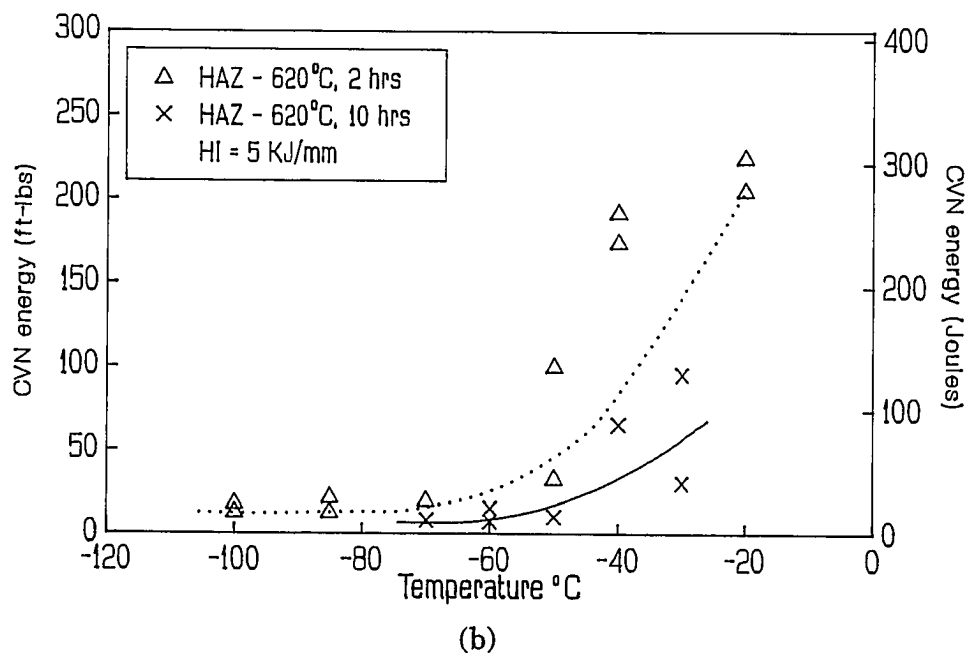
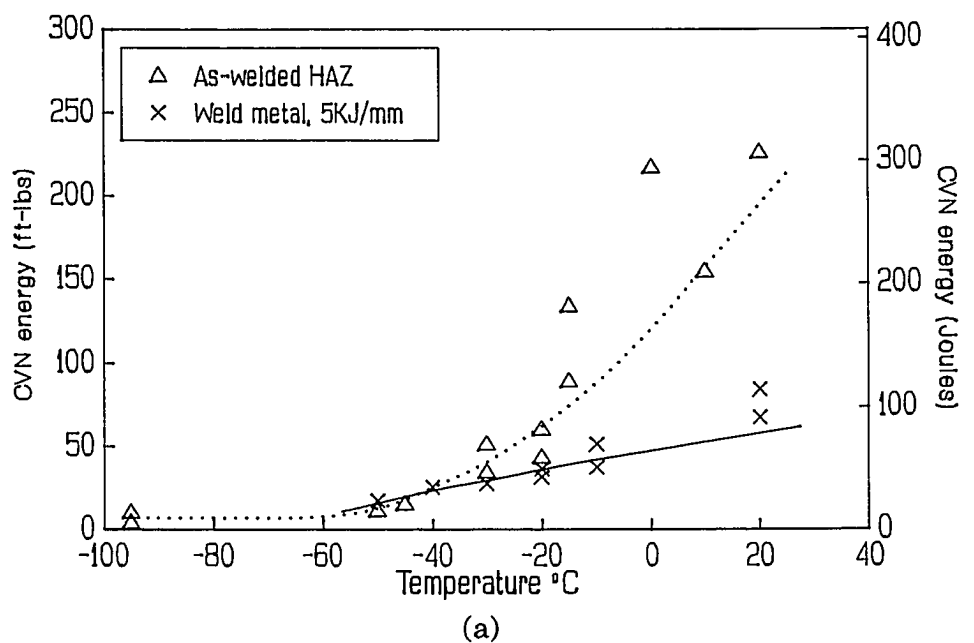


Figure 17: Charpy transition curves of A710 weldments (19 mm plate, 5 KJ/mm)
(a) as welded, (b) HAZ, after PWHT.

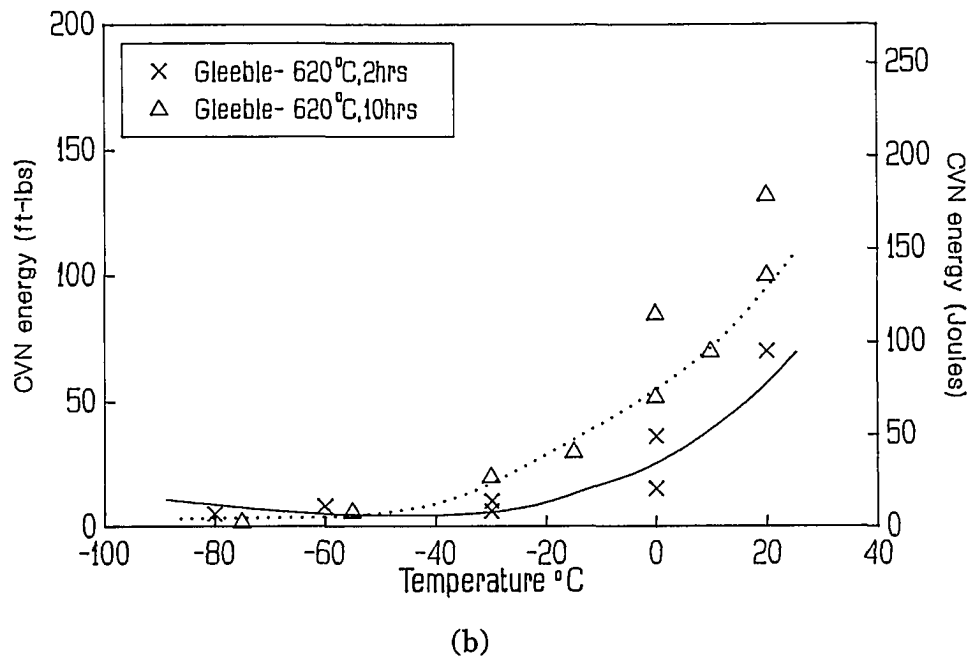
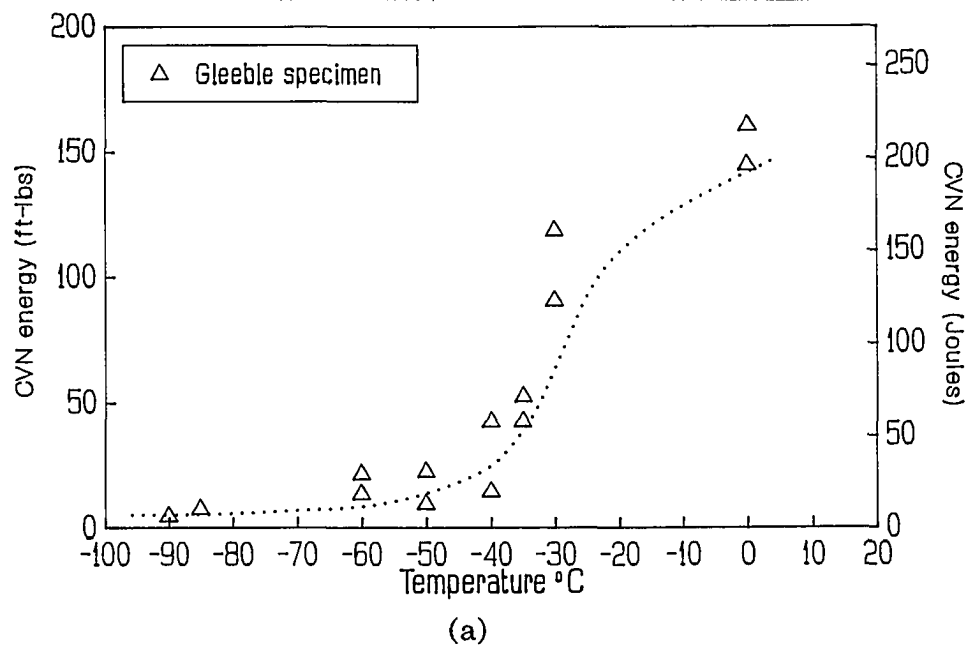
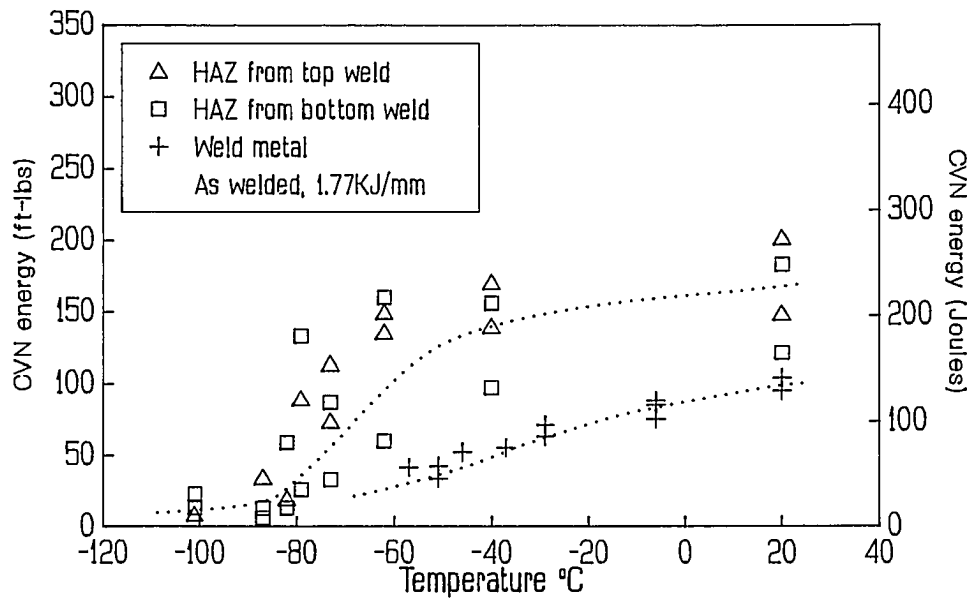


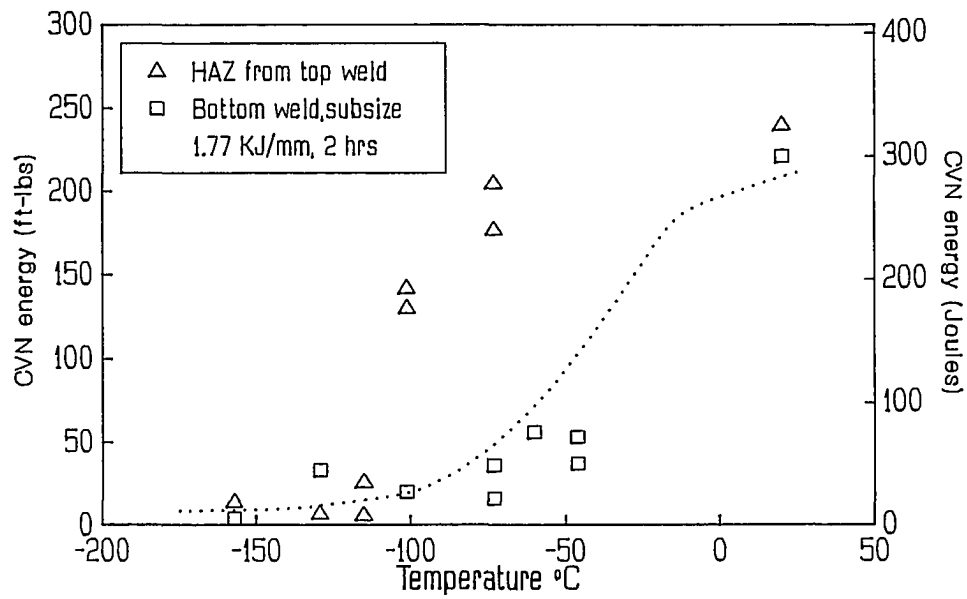
Figure 18: Charpy transition curves of simulated HAZ in A710 steels (19 mm plate, 3 KJ/mm)
 (a) no stress relief, (b) after stress relief.



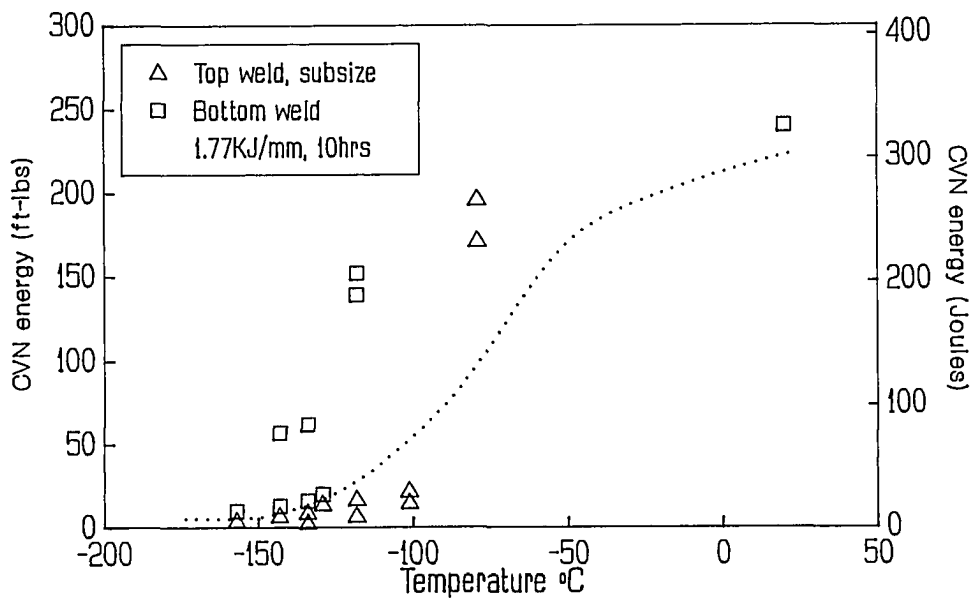
(a)

Figure 19: Charpy transition curves of A710 weldments
 (32 mm plate, 1.77 KJ/mm)

- (a) as welded,
- (b) PWHT at 620°C for 2 hours,
- (c) PWHT at 620°C for 10 hours.



(b)



(c)

Figure 19: continued

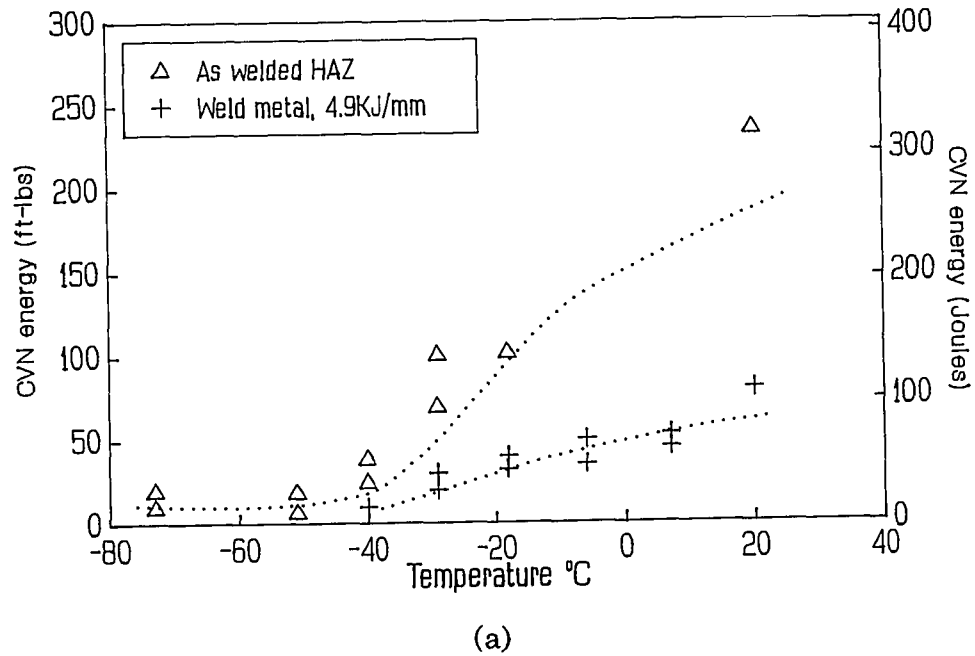


Figure 20: Charpy transition curves of A710 weldments
(32 mm plate, 4.9 KJ/mm)

- (a) as welded,
- (b) PWHT at 620°C for 2 hours,
- (c) PWHT at 620°C for 10 hours.

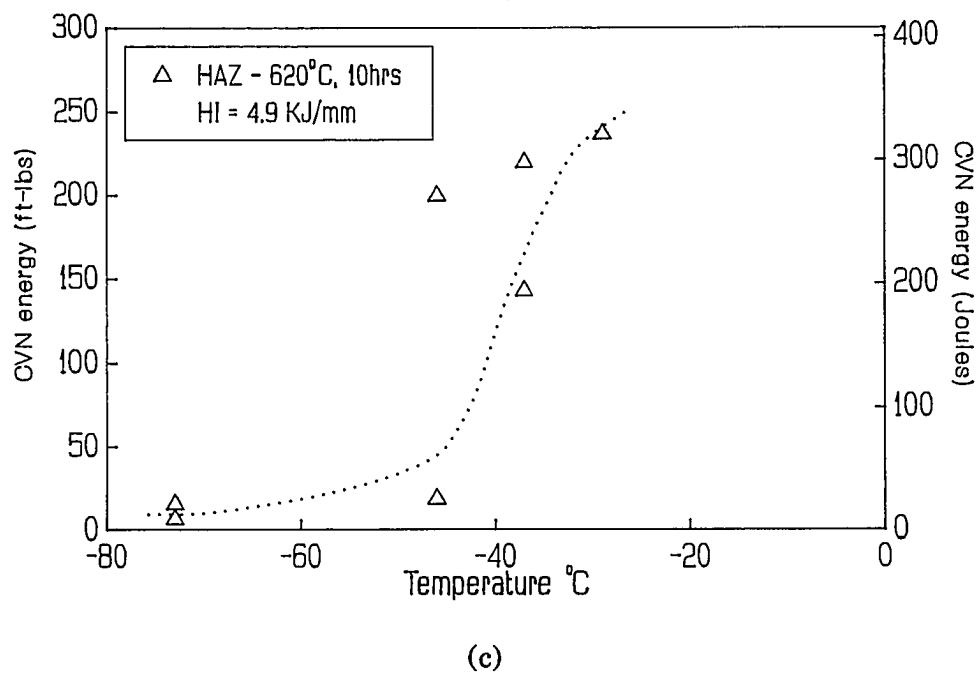
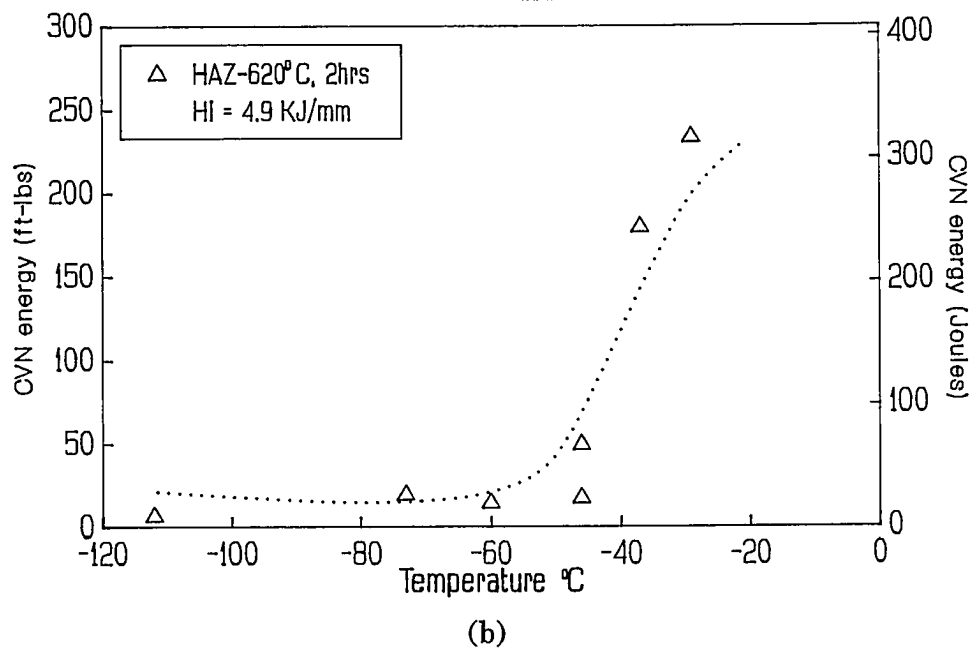


Figure 20: continued

Table 4: Summarized Charpy Impact 47J(35 ft-lb) Transition Temperatures for A710 Class 3 Weldments

<i>Material & Condition</i>	<i>Heat Input KJ/mm</i>	<i>PWHT at 620°C</i>	<i>Transition Temperature</i>	
			<i>°C</i>	<i>(°F)</i>
A. 19 mm Plate				
Base Plate		none	-135	(-211)
Weld Metal	3.0	none	-45	(-49)
	5.0	none	-25	(-13)
HAZ	3.0	none	-70	(-94)
	3.0	2 hours	-61	(-78)
	3.0	10 hours	-30	(-22)
	5.0	none	-30	(-13)
	5.0	2 hours	-45	(-49)
	5.0	10 hours	-38	(-36)
Gleeble Specimen	3.0	none	-45	(-49)
	3.0	2 hours	0	(+32)
	3.0	10 hours	0	(+32)
B. 32 mm Plate				
Base Plate		none*	-145	(-299)
		2 hours*	-147	(-232)
		10 hours*	-148	(-234)
Weld Metal	1.77	none	-58	(-72)
	4.90	none	-18	(0)
HAZ	1.77	none	-80	(-108)
	1.77	2 hours	-60	(-76)
	1.77	10 hours	-87	(-125)
	4.90	none	-33	(-28)
	4.90	2 hours	-40	(-31)
	4.90	10 hours	-44	(-48)

* Data from the work of Bolliger [1986].

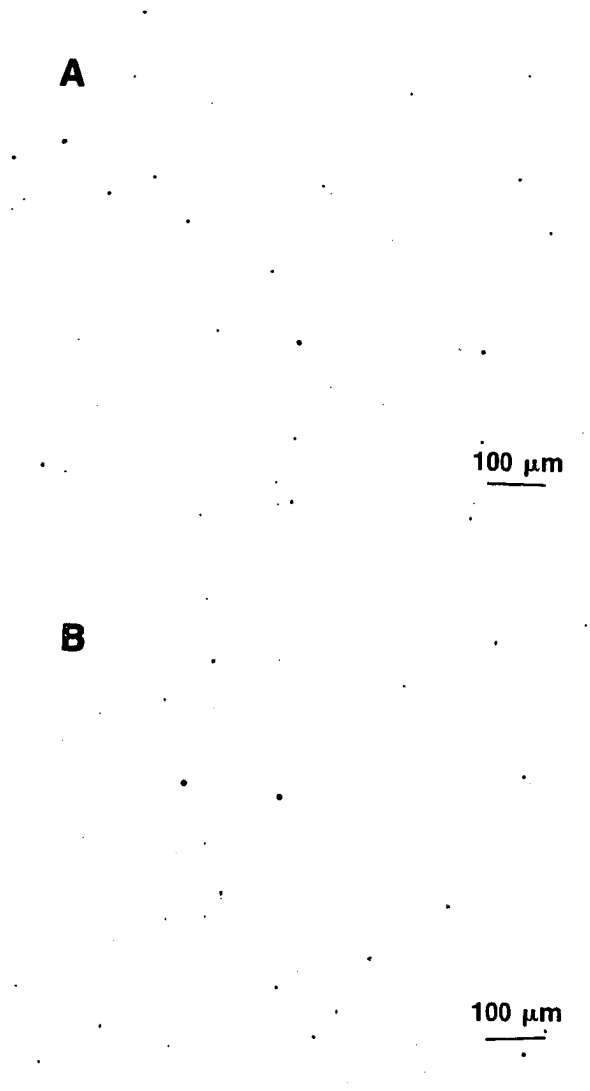


Figure 21: Optical micrographs of inclusions in the as – received A710 base plates
(a) 19 mm plate, (b) 32 mm plate.

thicker plates could tolerate higher heat input than the thinner plates.

The effect of PWHT on base plate toughness was small. For the HAZ toughness in 32 mm plate, the effect was mixed, generally beneficial, but not very significant. PWHT was more helpful for the HAZ toughness from high heat input than for lower heat input weldments. For 19 mm inch plate, PWHT seemed to be detrimental to the HAZ toughness for lower heat input.

The test results from the gleeble specimens indicated that the impact toughness values were much lower than those from the real weldments for the same energy input. The fact that the impact toughness values of real weld and simulated HAZ are different is often observed (Easterling^e, 1983). Generally speaking, the difference is probably due to the following reasons:

1. geometrical effects because of the difficulty in locating a Charpy V-notch in the desired position in the grain growth region of the weld HAZ which is usually very narrow (~ 1 mm);
2. the width of the coarse grain zone in the gleeble specimens (~ 5 mm in this study) is much larger than that in the welded HAZ, hence the toughness in the simulated condition is not affected by the adjacent tougher weld metal or base metal;
3. the gleeble simulation condition might be more or less departed from the actual welding conditions since the calculated thermal cycle was merely based on empirical

equations.

In the case of the present testing results, the specific reason why the toughness level from the gleeble specimens was so unreasonably low compared with that from the weld HAZ, was not clear. Obviously all the factors mentioned above have some effects on the testing data, and it is also possibly due to the fact that gleeble specimens experienced only one thermal cycle in this experiment, while the welds were made by depositing more than one pass.

In the present study, the data scatter problem in the Charpy impact testing still existed as shown in the above figures, which has been observed in the previous study (Bolliger, 1986). The causes of the scatter are still not very clear. At first it was suspected that the scatter came from the inaccuracy of the notch location on the Charpy bar. Thus all the tested Charpy specimens which gave the scattered results had been polished (on one side surface) and etched in order to re-check the notch position under optical microscope. The examination showed that a small amount of specimens did have the notch located further than 0.5mm from the weld fusion line, hence these data were discarded and re-testing was done by newly made specimens. The scatter still existed after re-testing but to a lesser extent. It was also found that some of the specimens had reasonably well positioned notches, but during impact testing, the crack path on the broken specimens did not keep on one surface, instead ran either into the grain refined zone or away from the notch center as shown in figure 22 for just a few examples. This could also lead to scattered toughness data. To make this problem more complicated, the scatter was present even in the data from the base metal [figure 16(a)] which had a uniform microstructure. It seems that the scatter problems are

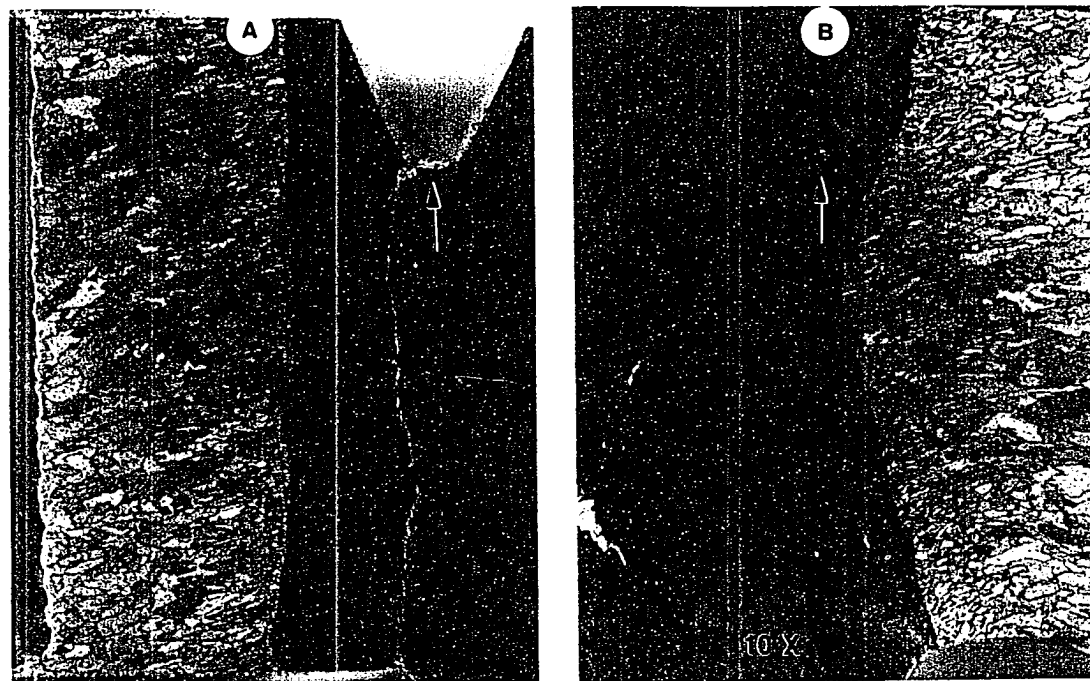


Figure 22: Fracture path examinations on broken Charpy specimen surfaces (arrow indicates the location of V-notch tip)

- (a) crack path towards the weld metal,
- (b) crack path towards the grain refined zone.

characteristic of A710 steels.

4.4 Optical microstructure of A710 weldments

4.4.1 The weld metal

In this study a high toughness filler metal (type T-75) was used. Figure 23 shows the microstructure of the weld metals for both plates. Both of them consisted predominantly of fine acicular ferrite with a small amount of polygonal ferrite. The microstructures of acicular ferrite provided optimum weld metal mechanical properties, from both strength and toughness point of view, by virtue of its small grain size (typically 1 ~ 3 μm) and high angle grain boundaries (Grong, 1986). Therefore, the properties of the weld metal were superior to those with microstructures of upper bainite, ferrite side plates, or grain boundary ferrite which are detrimental to toughness since these structures provide preferential crack propagation paths, especially when continuous films of carbide are present between the ferrite laths or plates.

4.4.2 The coarse - grained HAZ

A. 19 mm plate

Figures 24 ~ 27 were the optical microstructures of the coarse grained region (within 0.5mm from the fusion line) in the HAZ under as-welded and PWHT conditions. In the as-welded HAZ by $H_I = 3 \text{ KJ/mm}$ [figure 24(a)], the prior large austenite grain boundaries were clearly visible. The average grain size was measured to be 46.8 μm in diameter. Within the grain boundaries, the microstructure consisted essentially of bainitic ferrite (there seemed to be a very small amount of acicular ferrite too). There were also some carbides within the grain boundaries.

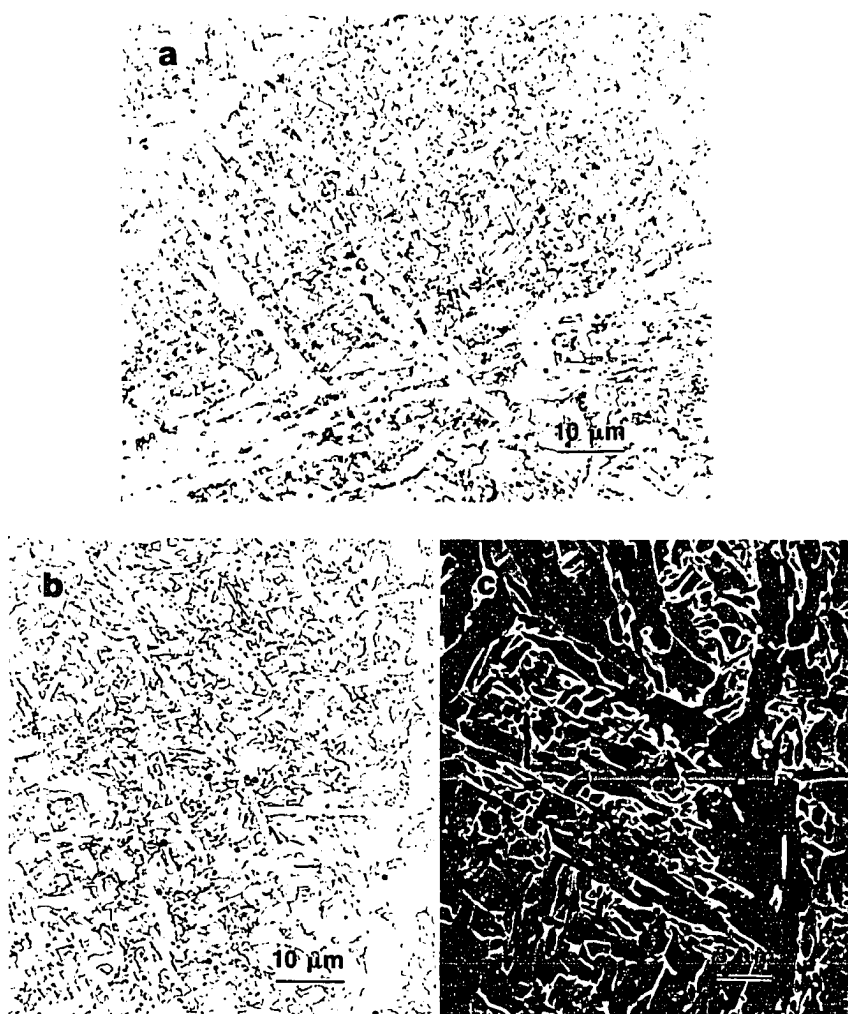


Figure 23: Microstructures of the as welded weld metals

- (a) 32 mm plate (optical),
- (b) 19 mm plate (optical),
- (c) 19 mm plate, SEM.

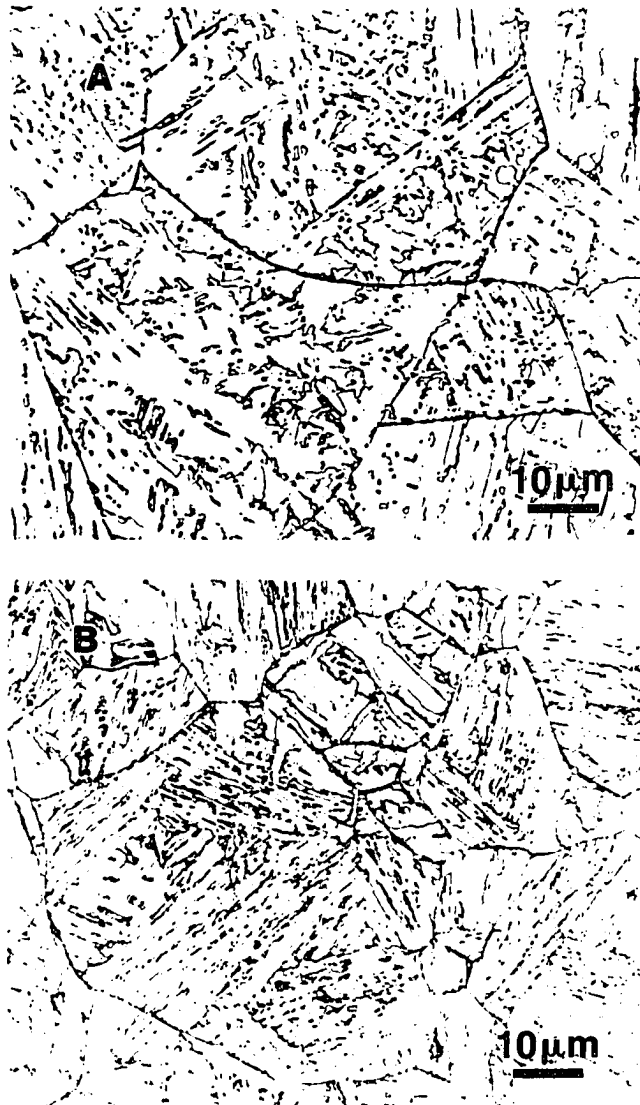


Figure 24: Optical micrographs of the coarse grained HAZ in A710 steels (19 mm plate, 3 KJ/mm, as welded)
(a) weld HAZ, (b) simulated HAZ.

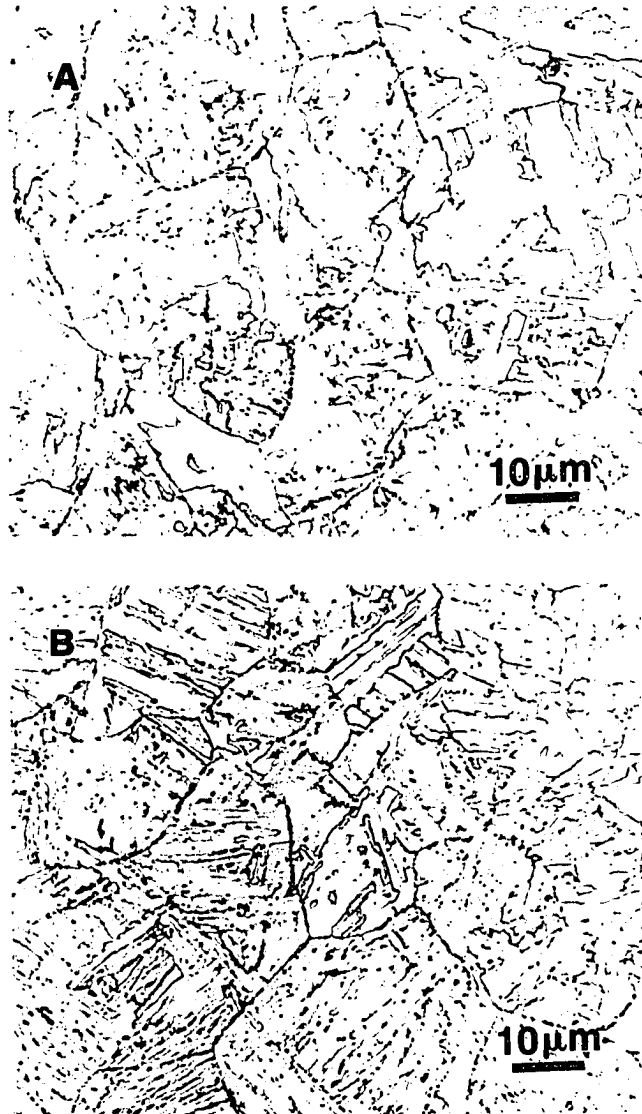


Figure 25: Optical micrographs of the coarse grained HAZ in A710 steels (PWHT at 620°C for 2 hours)
(a) welded HAZ, (b) simulated HAZ.

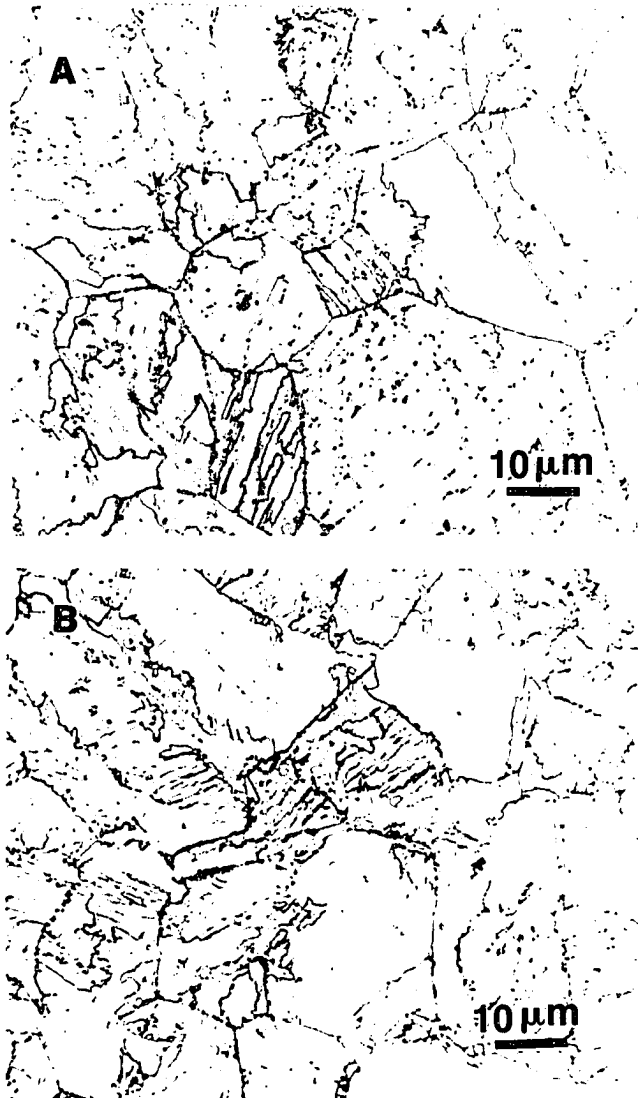


Figure 26: Optical micrographs of the coarse grained HAZ in A710 steels (PWHT at 620°C for 10 hours)
(a) welded HAZ, (b) simulated HAZ.

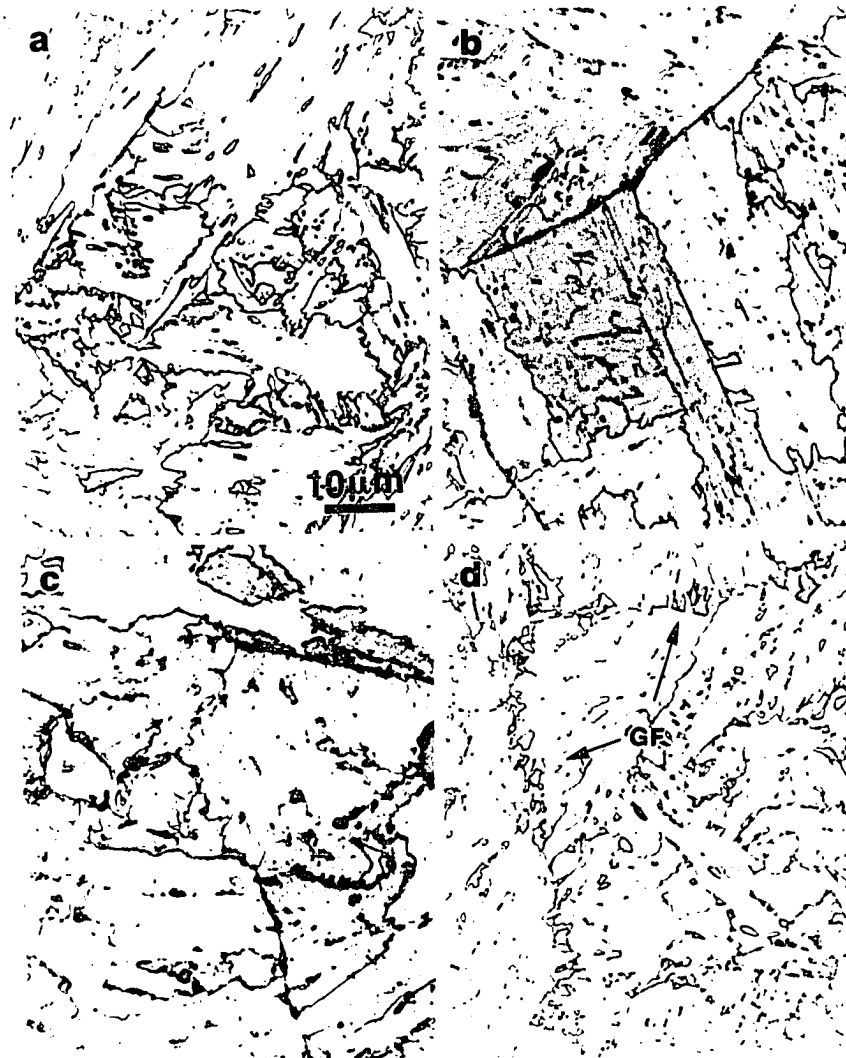


Figure 27: Optical micrographs of the coarse grained HAZ in A710 steels (19 mm plate, 5 KJ/mm)

- (a) as weld HAZ,
- (b) PWHT at 620°C for 2 hours,
- (c) PWHT for 10 hours,
- (d) grain boundary ferrite in as welded HAZ.

During post weld heat treatment, some degree of recovery and recrystallization process took place. The amount of lath type structure gradually decreased and more carbide particles were present within the grain boundaries [figure 25(a) and 26(a)]. However, the coarse grained structure still existed (even after 10-hour tempering), even though some grains seemed to have been grown from recovery.

For comparison, the micrographs of the coarse grained microstructures of the gleeble specimens were also included in the above figures. The width of this coarsened structure on the specimen was about 4 ~ 5 mm. The microstructure as well as the prior austenite grain size seemed to be almost the same as the case in the weld HAZ, except that the lath morphology within the grain boundaries was more distinctive than that in the weld HAZ [comparing figure 24(a) with figure 24(b)].

In the case of higher heat input (5 KJ/mm), the prior austenite grain boundaries were not as clear as those from the low heat input, and the grains were even larger such that the photograph [figure 27(a)] was covered by only one grain. The microstructures within the large grains were similar to those in the HAZ created by low heat input, that is, mostly lath type ferrite, but there were probably more acicular ferrite type structures compared with that by lower heat input. In some regions of the coarse grained zone along the through - thickness direction, grain - boundary proeutectoid ferrite was formed as shown in figure 27(d), presumably as a result of slower cooling rate in the case of high heat input.

B. 32 mm plate

Figures 28 – 29 showed the optical microstructures of the coarse

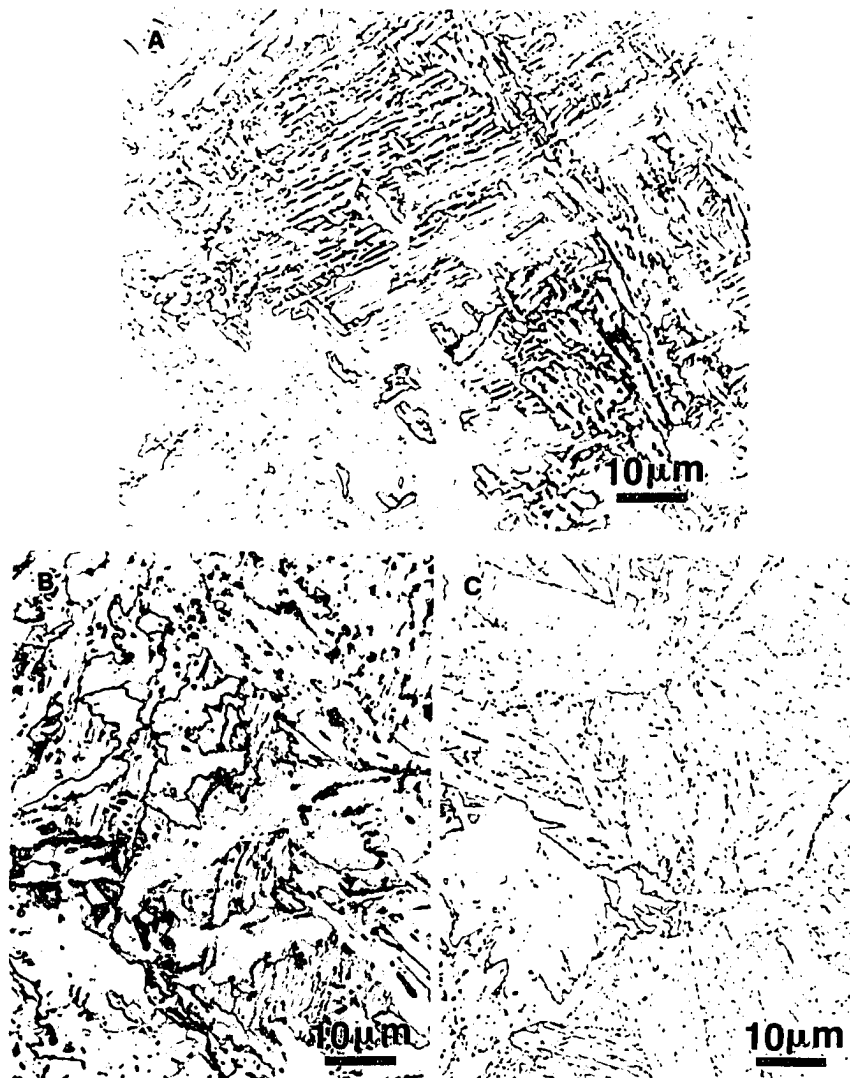


Figure 28: Optical micrographs of the coarse grained HAZ in A710 steels (32 mm plate, 1.77 KJ/mm)

- (a) as welded HAZ,
- (b) PWHT at 620°C for 2 hours,
- (c) PWHT at 620°C for 10 hours.

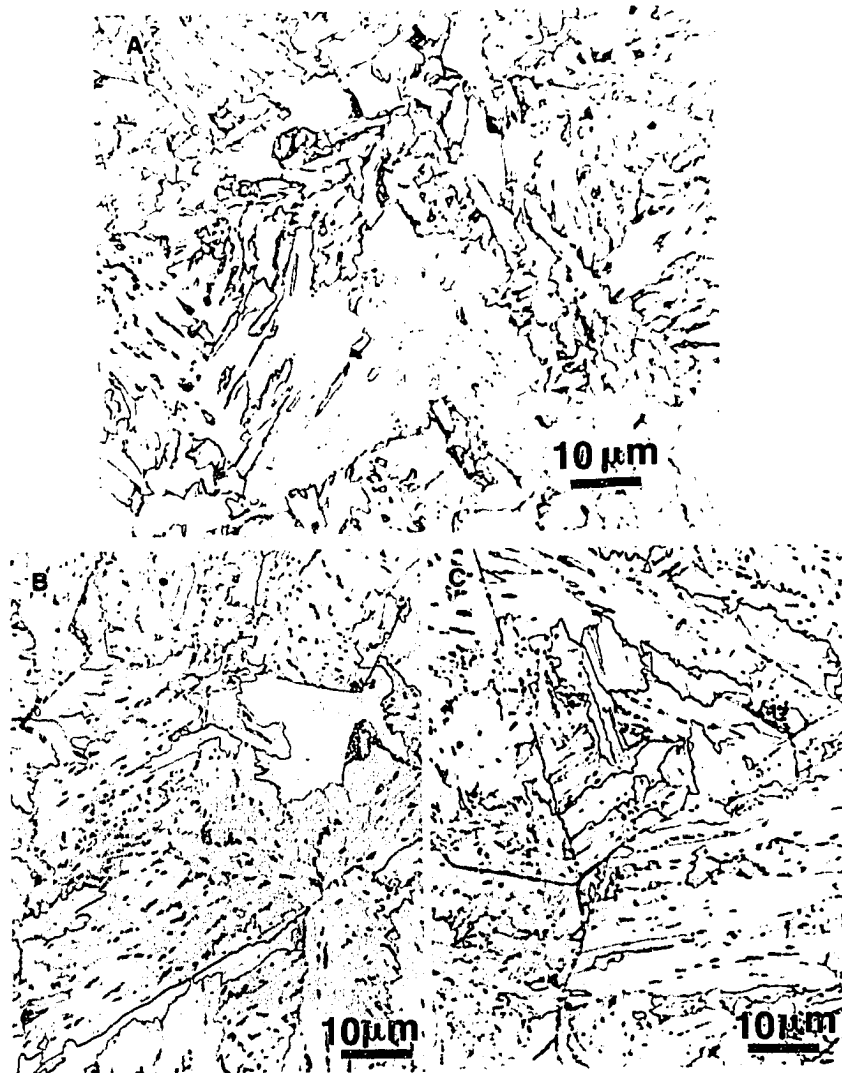


Figure 29: Optical micrographs of the coarse grained HAZ in A710 steels (32 mm plate, 4.9 KJ/mm)

- (a) weld HAZ,
- (b) PWHT at 620°C for 2 hours,
- (c) PWHT at 620°C for 10 hours.

grained zone in 32 mm plate welds. The structure from $HI=1.77$ KJ/mm contains more acicular ferrite than that in the HAZ of 19 mm plate because of faster three dimensional cooling in thicker plates, but grain growth is also very severe. In the coarse grained region the prior austenite grain boundaries were blurred as a result of the reheat effect due to overlap of several welding passes such as 9 passes in the case of heat input = 1.77 KJ/mm [figure 6(a)].

4.5 SEM examination of fracture surfaces

The fracture surfaces of selected broken Charpy specimens for the 19 mm and 32 mm plate weldments were examined using SEM. Representative photographs for 19 mm plate weldments were shown in figures 30 – 32.⁴

In the as – welded condition, the coarse grained regions in both the weld HAZ and simulated specimens showed a mixed ductile and brittle fracture mode (figure 30) instead of a completely brittle fracture mode typical of steels tested at quite low temperatures. In figure 31, the evidence of large bainitic microstructures was very clear since large cleavage facets were present on the fracture surfaces. This will be discussed in more detail later. It was also noted that, in specimens after PWHT for 2 hours, there were many short blunt microcracks generated below the fracture surface, as shown in figure 32, but not as many as cracks of this type were found in the specimens by PWHT for 10 hours (figure 31). It has been suggested (Cutler, 1982) that, under overaged

⁴ The discussion on the fracture surfaces of 32 mm plate welds was omitted here since the SEM features were quite similar to those in the 19 mm plate welds.

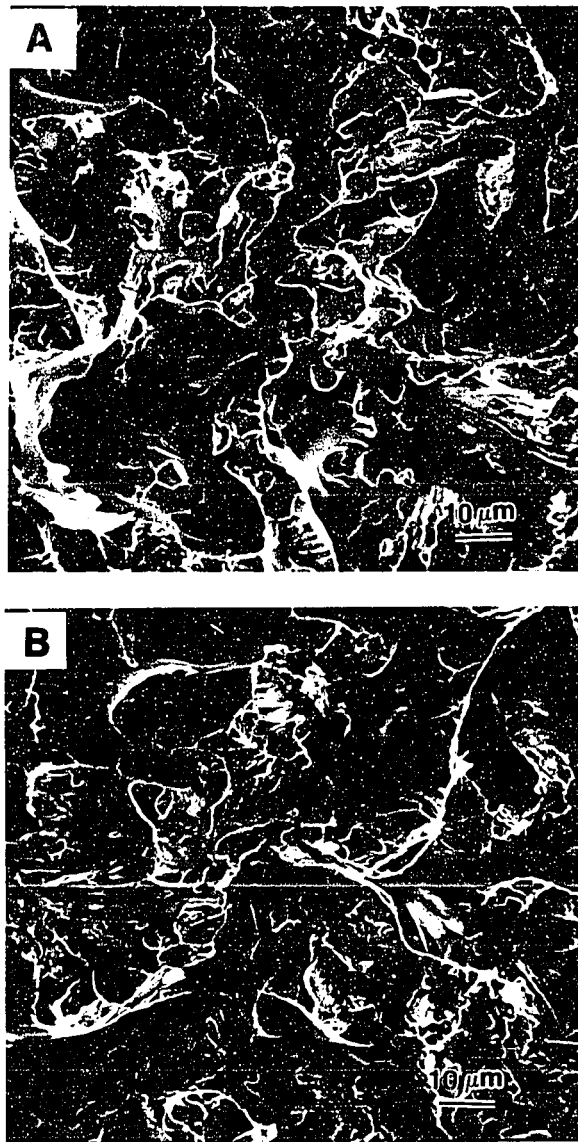


Figure 30: SEM fractographs of broken Charpy specimen surfaces (19 mm A710 plate, 3 KJ/mm)

- (a) as welded HAZ (broken at -55°C),
- (b) gleeble specimen, no SR (broken at -60°C).

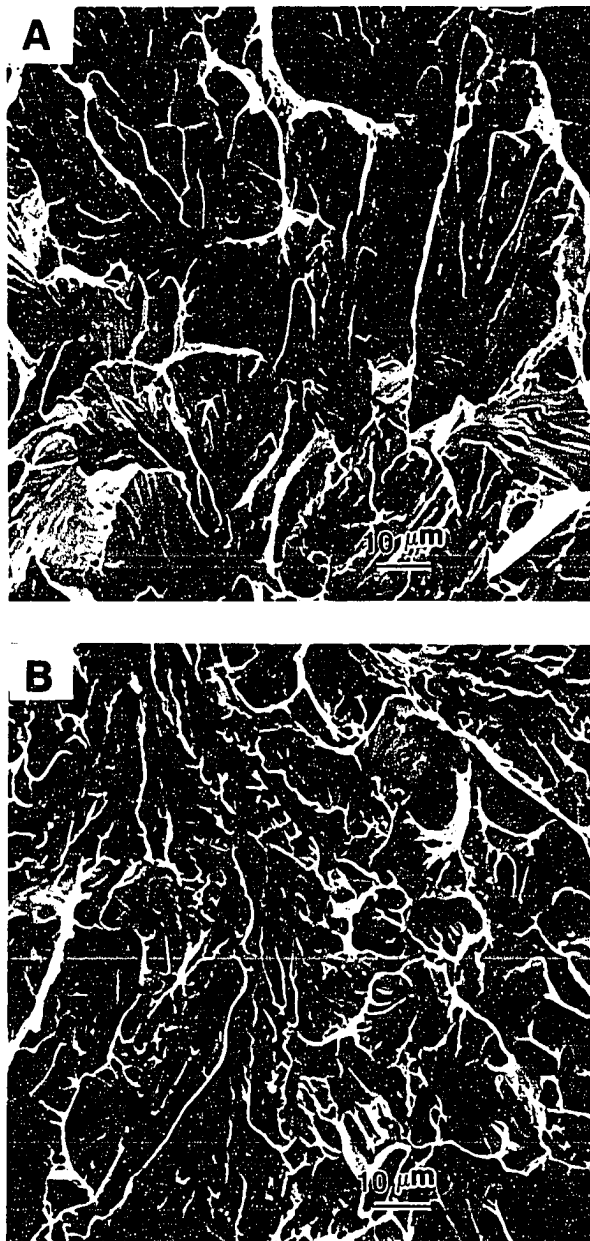


Figure 31: SEM fractographs of broken Charpy specimen surfaces (19 mm plate, 3 KJ/mm, PWHT at 620°C for 10 hours)

- (a) gleeble specimen (broken at -30°C),
- (b) weld HAZ (broken at -40°C).

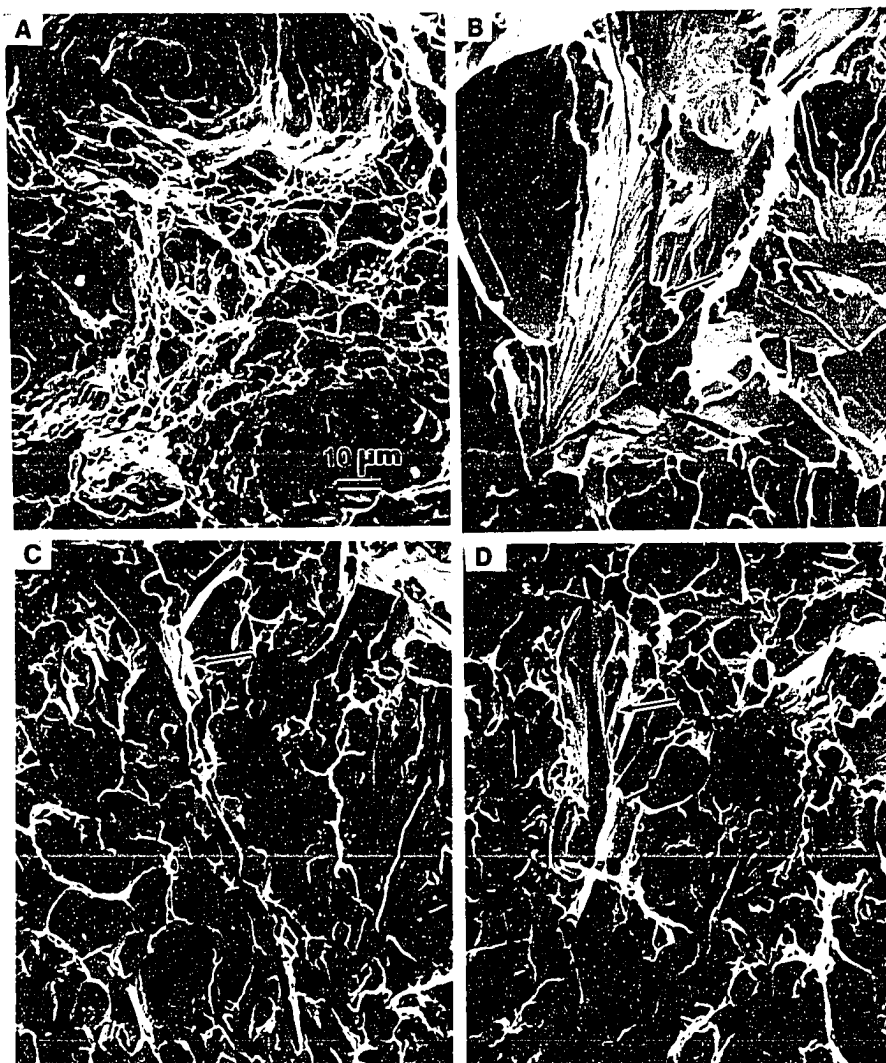


Figure 32: SEM fractographs of broken Charpy specimen surfaces (19 mm plate, 3 KJ/mm, PWHT at 620°C for 2 hours)

- (a) weld HAZ showing inclusions on ductile fractured surfaces (broken at -40°C),
- (b) gleeble specimen (broken at -70°C),
- (c) weld HAZ (broken at -60°C),
- (d) weld HAZ (broken at -60°C).

conditions, these microcracks are quickly blunted, due possibly to the effects of the dispersion of copper precipitates which can deform plastically or can act as a dislocation source. Failure occurs by ductile tearing when the ligaments between these microcracks fail.

4.6 TEM observations

TEM technique was employed primarily for the study of copper precipitation behavior in 19 mm plate of A710 steels (for both heat inputs) including the base materials, the coarse grained zone in the welded HAZ and gleeble specimens. The work which has been done is presented below.

4.6.1 Identification of the precipitates

Prior to the primary study on copper precipitation behavior, the work on precipitation identification was undertaken in order to make certain that the precipitates to be studied were Cu – containing, and more importantly, they were internal to the steel matrix rather than an artifact due to contamination or oxidation. The identification was attempted by selected area diffraction (SAD) and by chemical analysis using EDS on STEM. During SAD pattern analysis for the precipitates, it was found that the diffraction patterns obtained from the thin foil were much more complicated than anticipated since the diffraction pattern was expected to be α -Fe matrix spots plus ϵ -Cu spots (similar to pure copper). One example is shown in figure 33,⁵ which is a SAD pattern of [001] zone from a thin foil specimen (A710 base metal). The pattern consists of three groups of spots: (1) α -Fe matrix spots, (2) an unknown set of spots (indicated by arrows) which were generated by the precipitates to be identified (see figure 34),⁶ and (3) a group of satellite spots (marked “a” in figure 33) around each matrix spot. The satellite

⁵Diffraction pattern indexing in figure 33 (b) will be explained later.

⁶ $(hkl)_p$ will refer to precipitate reflection in the following discussion.

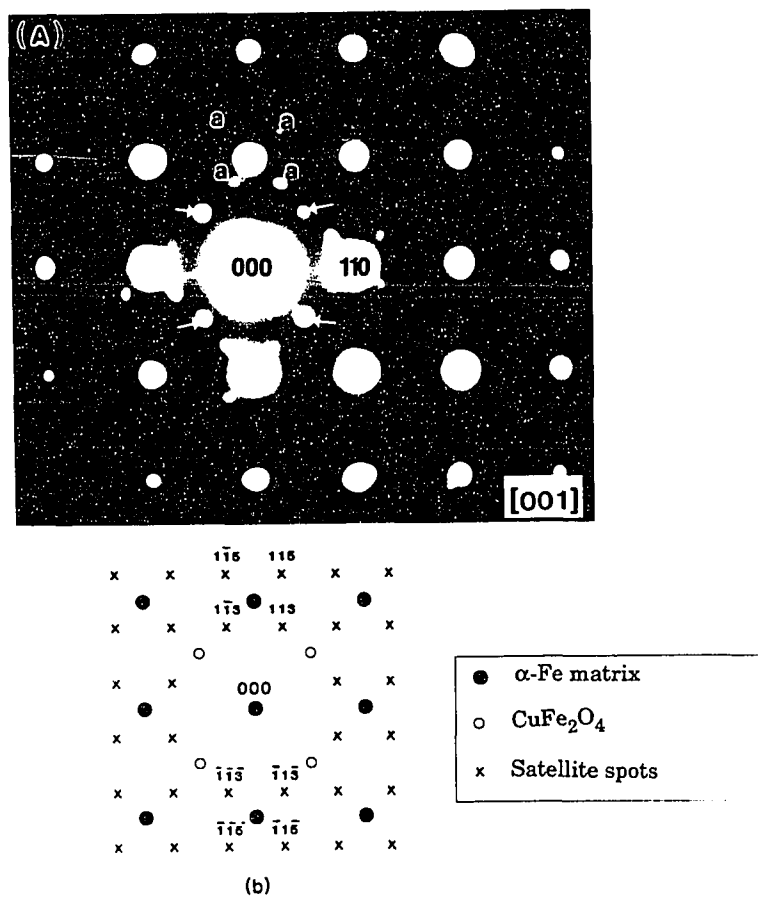


Figure 33: A SADP showing both α -Fe and precipitate spots
 (a) SAD pattern from $[001]_{\alpha\text{-Fe}}$ zone,
 (b) A schematic drawing of the SADP in (a).

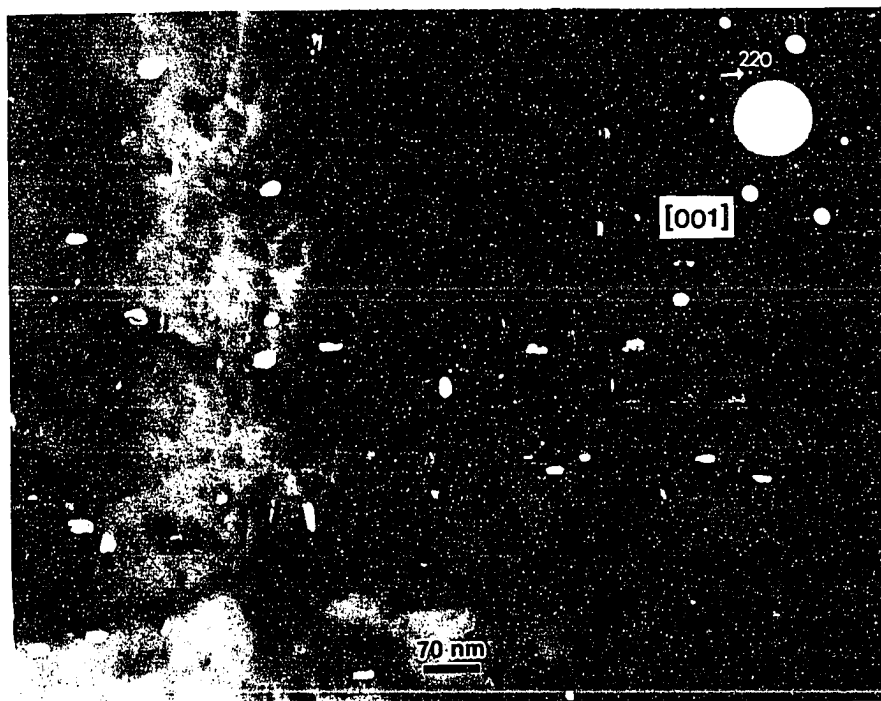


Figure 34: DF image of precipitates from a $(220)_p$ reflection
(A710 HAZ, PWHT at 620°C for 10 hours)

spots appear only when the matrix diffraction pattern is in [001] zone and sometimes they were not seen even when the zone axis was [001] as shown in figure 35, which is why these satellite spots are assumed to be a different set from the unknown set. Figures 36 and 37 are the SAD patterns from other two zone axes.⁷ From these diffraction patterns a series of lattice spacing d values for the precipitate spots (excluding the satellite spots in figure 33) were measured, and the results are listed in Table 5. According to the measured d values, the data for two known substances from the literature with the similar d values are also given in Table 5. Therefore, it seems possible that the precipitates which generated the unknown spots are either the compound CuFe_2O_4 , or an oxide Fe_3O_4 . According to Sheree H. Chen *et al* (1976), a passivating oxide (Fe_3O_4) layer could be formed on the surface of Fe and Fe-Ni alloys exposed to air, thus these oxide particles are not internal to the alloys. To clarify these problems further investigations were undertaken. From the following experimental results and analysis it is evidenced that the precipitates observed in this study are most probably CuFe_2O_4 instead of the oxide:

1. The precipitates contain copper. This can be seen from the chemical analysis by EDS. Figures 38 and 39 showed two examples of the EDS spectra from the precipitates in thin foil specimens. For comparison, the spectrum from the α -Fe matrix was also given in figure 38(3). The Fe peaks associated with the precipitate spectrum in figures 38 (2)

⁷The diffraction pattern indexing for the precipitate spots shown in figures 35 – 37 will be explained later.

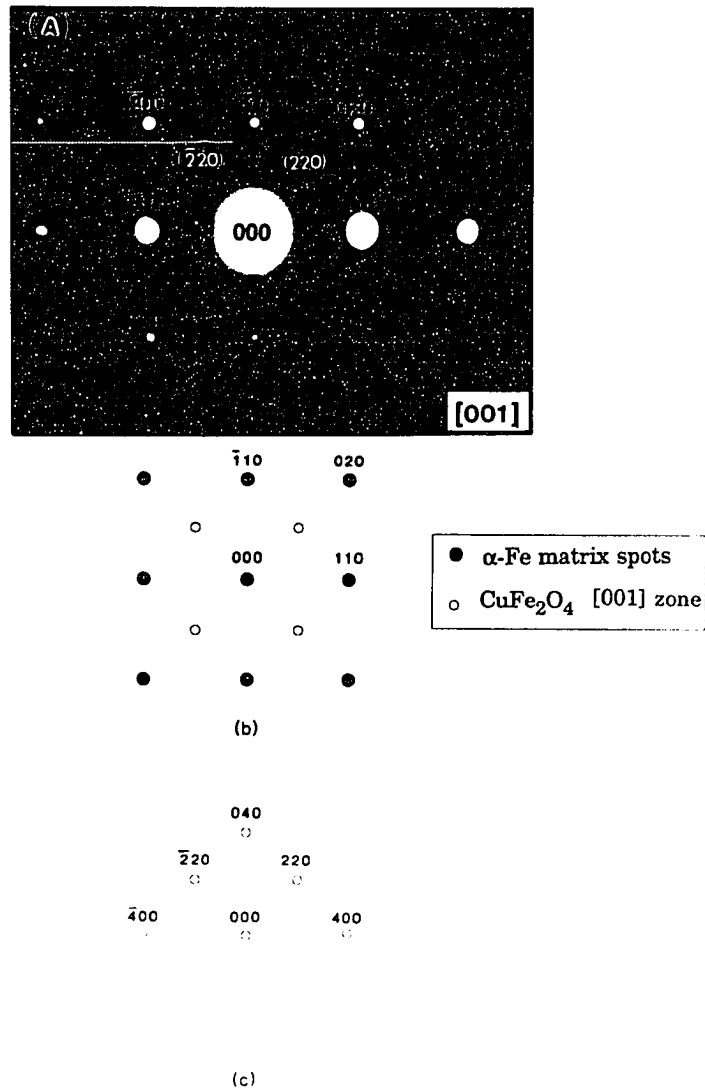


Figure 35: Diffraction pattern indexing for A710 steels

- (a) SADP from as received base metal,
- (b) DP simulation of the SADP in (a),
- (c) Indexing of the precipitate (CuFe_2O_4) spots.

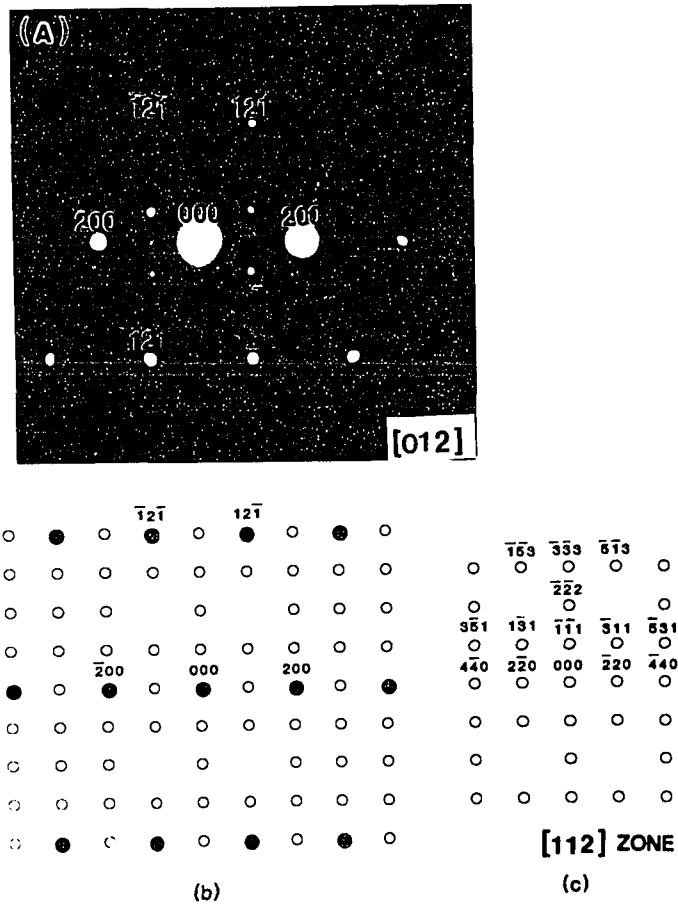


Figure 36: Diffraction pattern indexing for A710 steels

- (a) SADP from as received base metal,
- (b) DP simulation of the SADP in (a),
- (c) Indexing of the precipitate (CuFe_2O_4) spots.

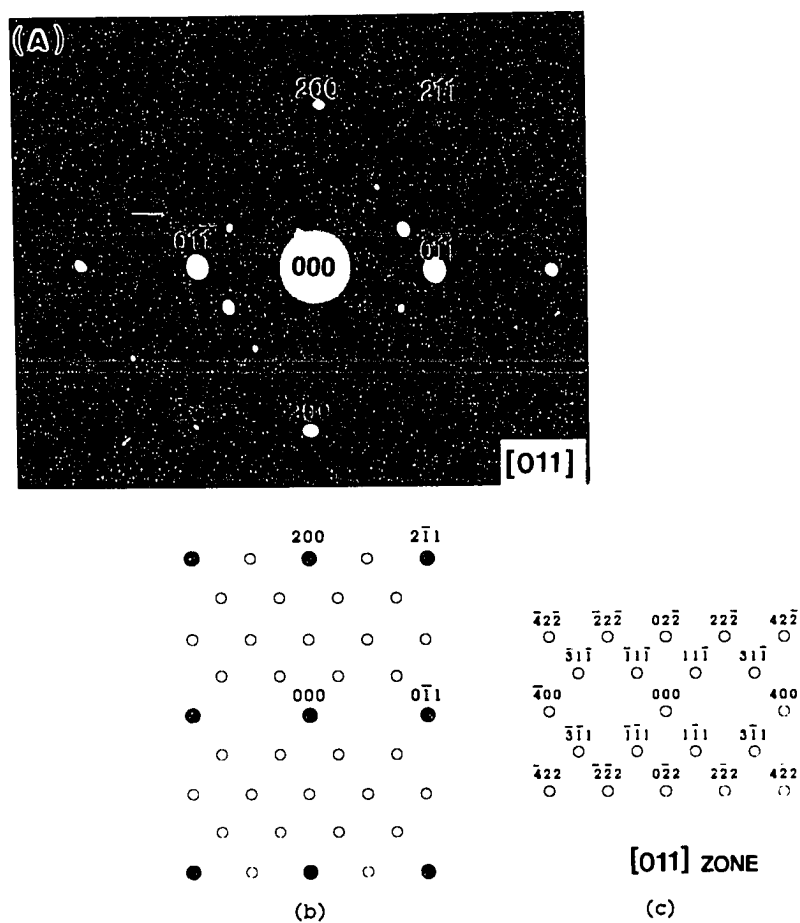


Figure 37: Diffraction pattern indexing for A710 steels

- (a) SADP from as received base metal,
- (b) DP simulation of the SADP in (a),
- (c) Indexing of the precipitate (CuFe_2O_4) spots.

Table 5: Interplanar d Spacings for the Observed
Cu-containing Precipitates (Å)

<i>d values measured from SAD patterns</i>	<i>d values for CuFe_2O_4 from CDF*</i>	<i>d values from simulation for CuFe_2O_4</i>	<i>d values for Fe_3O_4 from CDF</i>	<i>d values for pure Cu</i>
4.84	4.89	4.84	4.85	2.09
2.94	2.99	2.96	2.97	1.81
2.54	2.55	2.55	2.53	1.28
2.43	2.44	2.43	2.42	1.09
2.00	2.12	2.12	2.10	1.04
1.60	1.63	1.63	1.62	0.10

* Crystal Data File from “Elemental and Interplanar Spacing Index”,
NSRDS (1989)

and 39 (2) are mostly from the α -Fe matrix due to electron probe drifting during spectrum collecting, or due to larger probe size (150 ~ 200 Å) than the size of the precipitates.

2. The size and shape of the precipitates are heat treatment dependent, and the growth trend of the precipitates during aging (see figure 40)⁸ is the same as that described for ϵ -Cu particles in the literature (Hornbogen, 1960; Goodman, 1973, Cutler, 1982). However, from the published research by Chen *et al* (1976), it is known that the size and shape of Fe_3O_4 particles on the surface of Fe-Ni alloy never change with heat treatment, since the oxide film was formed on the specimen surface during or after specimen preparation.
3. The observed precipitates in this study always contain “line marks” as shown in figure 41, which is characteristic of the ϵ -Cu particles described by a number of researchers such as Hornbogen (1960) and Miglin (1986). However, there is no evidence of ϵ -Cu found from the diffraction pattern in this study.
4. In Chen’s study, it was found that Fe_3O_4 are always in the form of a layer of extremely fine particles, while in this study the precipitates are well separated and they have certain size and shape (either spherical or rod-like depending on the heat treatment).
5. Interactions between dislocations and precipitates are always observed in this material during TEM study. One example is given in figure 42, and more will be shown in the

⁸The micrographs will be discussed in detail in section 4.6.2.

next section. This indicates that the precipitates are internal to the material.

6. The precipitates appear on specimens prepared either by IBT only (on cold stage) without twin-jet electropolishing, or by electropolishing only without IBT, as shown in figure 43, which indicates that the precipitates are not a result of contamination or oxidation introduced from a certain specimen preparation procedure.⁹

Based on the crystal structure of CuFe_2O_4 as well as its lattice parameters, a computer programming (DF Tools 5.1) for diffraction pattern simulation was employed. The d values obtained from the simulated diffraction patterns are also listed in Table 5, and diffraction pattern indexings for each zone axis according to the simulation are already shown in figures 35 ~ 37. It can be seen that the data from the simulation for CuFe_2O_4 crystal can match the experimental data very well.

There is still another set of diffracted spots, i.e., the satellite spots in figure 33, which do not belong to the CuFe_2O_4 diffraction shown in figures 35 – 37. From dark field imaging examinations it was found that these spots were also from the precipitates, as shown in figure 44. According to Chen (1976),¹⁰ these spots were probably the forbidden first Laue zone diffraction spots which would take place when reciprocal

⁹As the specimens were finally thinned, they were immediately transferred to the vacuum chamber of the electron microscope in order to prevent the specimens from oxidation by air.

¹⁰ Fe_3O_4 and CuFe_2O_4 are both of fcc spinel structure, thus they should have similar diffraction patterns.

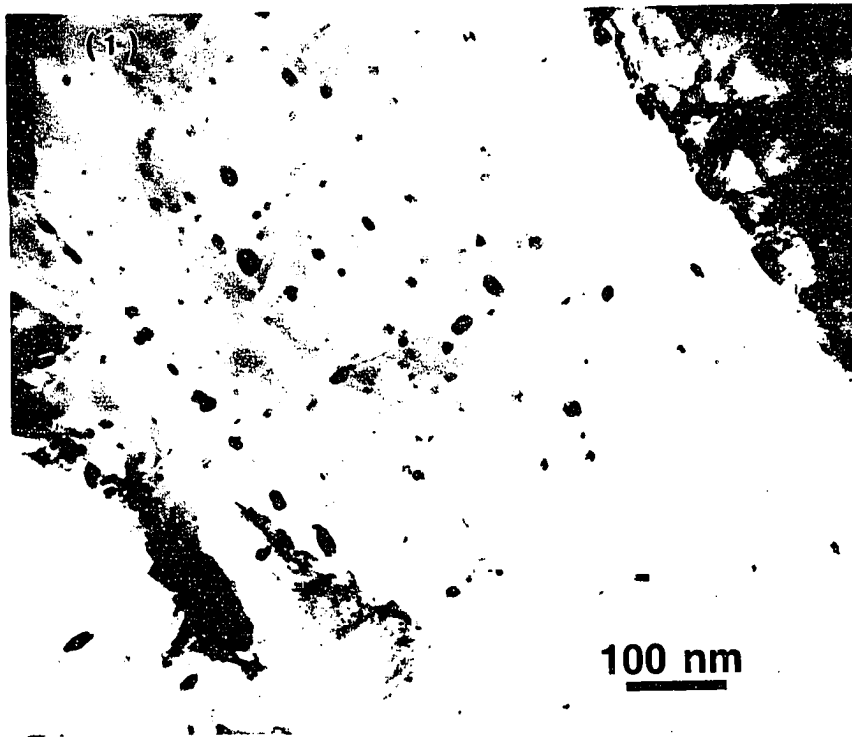
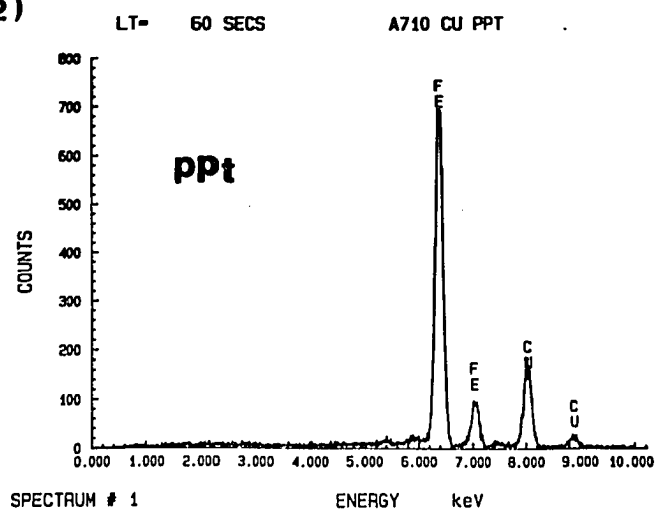


Figure 38: Cu precipitation observations in A710 HAZ
(3 KJ/mm, PWHT at 620°C for 10 hours)

- (1) TEM showing precipitates in the HAZ,
- (2) EDS spectrum from the precipitates in (1),
- (3) EDS spectrum from the matrix in (1).

(2)



(3)

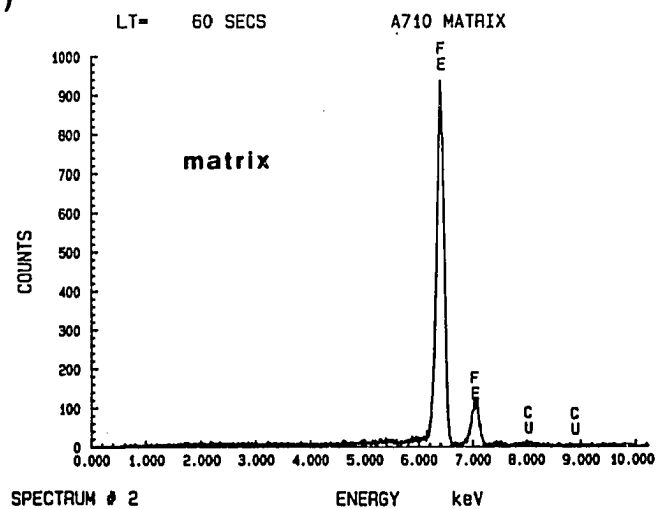


Figure 38: continued

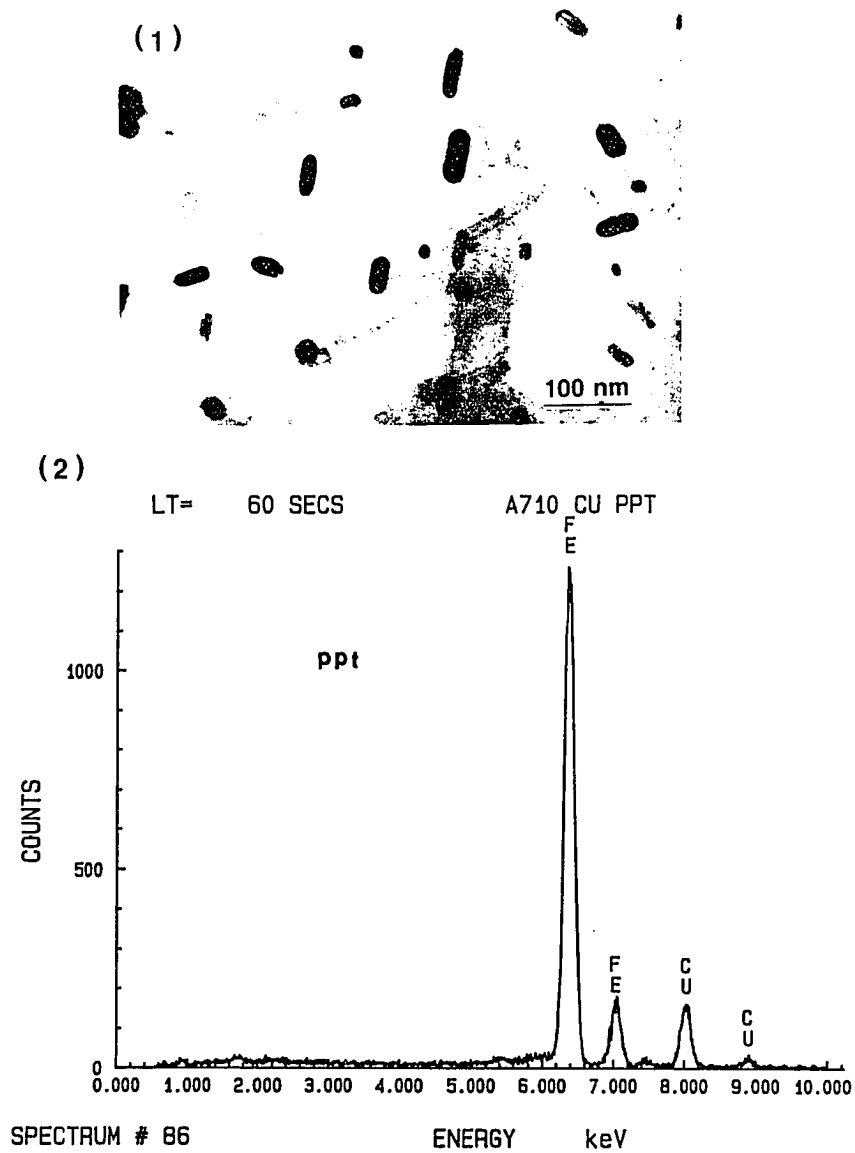


Figure 39: Cu precipitates in A710 base metal (620°C for 100 hours)

- (1) TEM micrograph showing elongated particles,
- (2) EDS spectrum from the particles in (1).

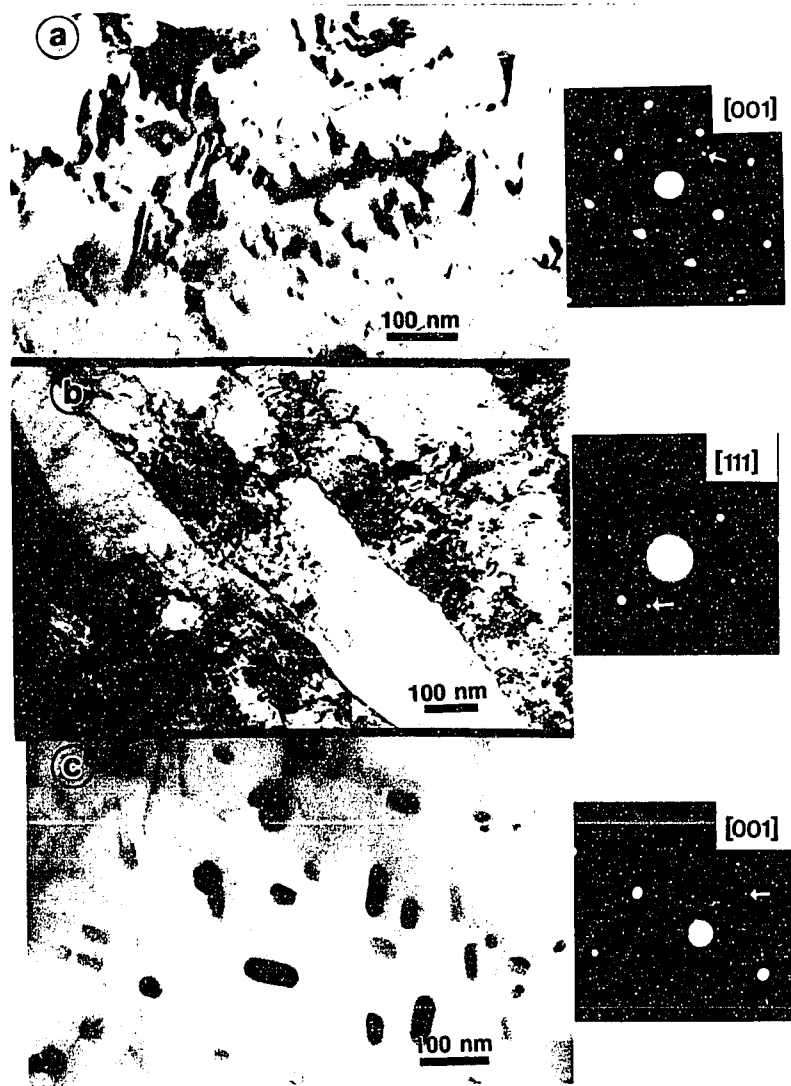


Figure 40: TEM micrographs of aged A710 base metal
 (a) as-quenched condition, precipitates “invisible”,
 (b) 500°C for 2 hours, precipitates “invisible”,
 (c) 620°C for 100 hours, showing large precipitates.

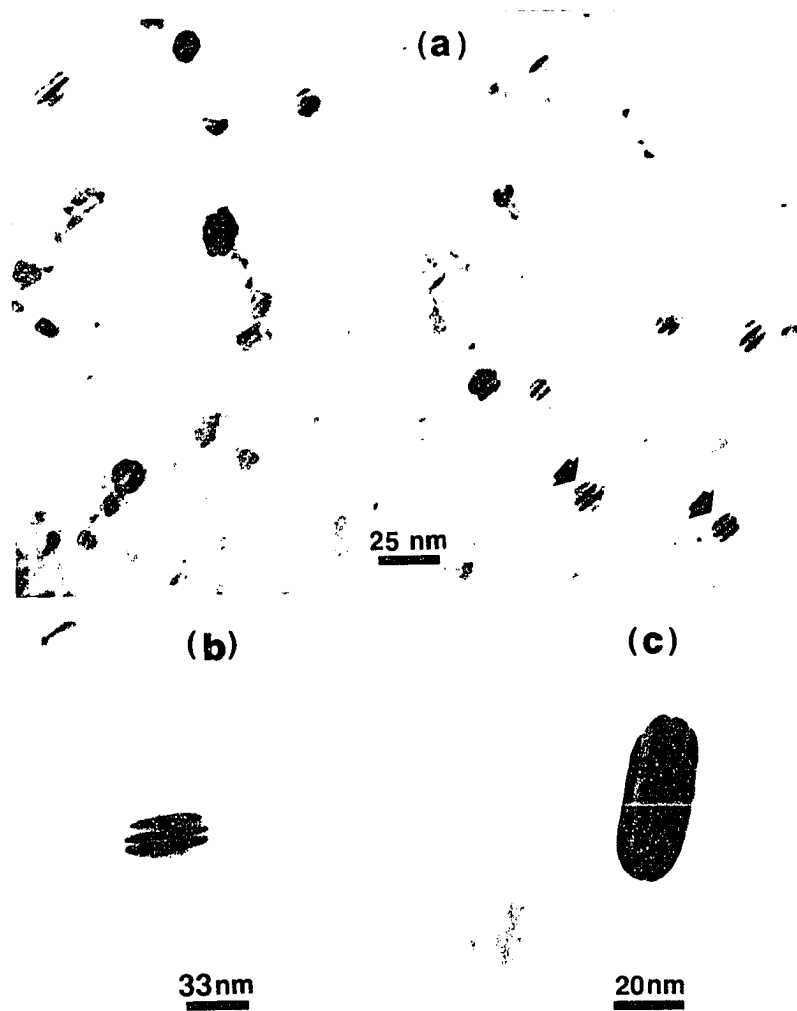


Figure 41: Line marks on Cu-containing precipitates

- (a) two or three lines,
- (b) three lines,
- (c) four lines.

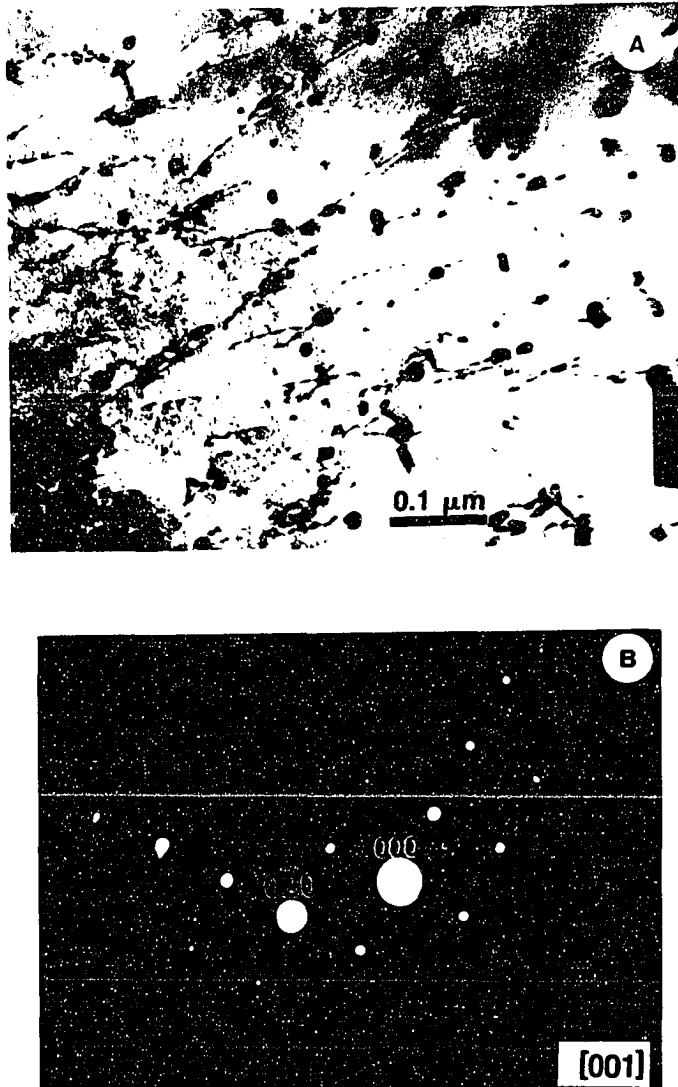


Figure 42: TEM micrographs of A710 HAZ (620°C, 10 hrs)
 (a) interaction between dislocation and precipitates,
 (b) SADP for the BF image in(a).

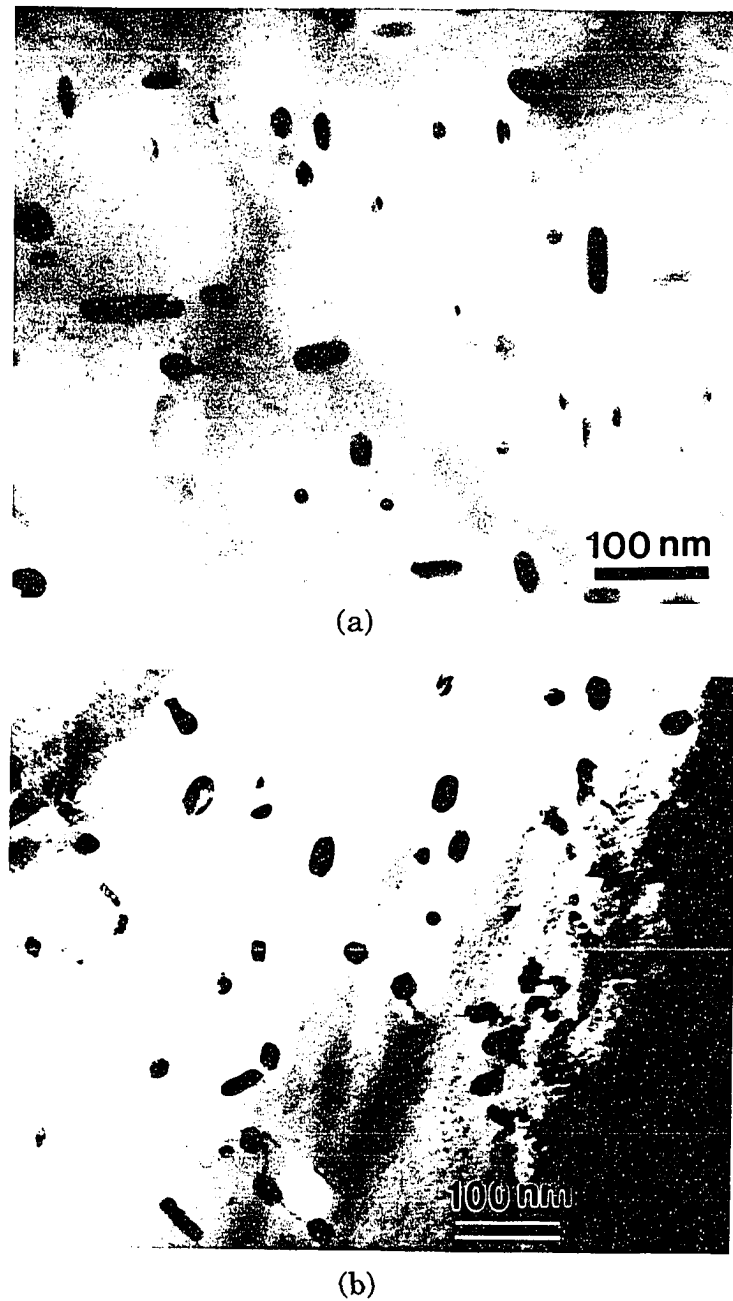


Figure 43: TEM micrographs of A710 base metal (620°C, 100 hrs)
(a) BF image from specimen made by IBT only,
(b) BF image from specimen made by electropolishing.

lattice points are elongated due to thin and small precipitates. These forbidden diffraction spots are $\{113\}_p$ and $\{115\}_p$ types of reflections as indicated in figure 33 (b).

Based on these experimental observations and analysis shown above, it could be concluded that the observed precipitates are internal to A710 steel, and the precipitates do consist of copper, but they seem to be in the form of a compound CuFe_2O_4 , which has a fcc spinel structure, and the orientation relationship between the α -Fe matrix and precipitates is (according to figure 35):

$$\begin{aligned} [001]_{\alpha\text{-Fe}} // [001]_{\text{CuFe}_2\text{O}_4} \\ (011)_{\alpha\text{-Fe}} // (010)_{\text{CuFe}_2\text{O}_4} \end{aligned}$$

However, there is still controversy over these conclusions. The most obvious is the oxygen content required for the formation of CuFe_2O_4 . Based on the calculations for the present results, it is estimated that the maximum amount of oxygen needed for CuFe_2O_4 precipitates range from 0.024 wt.% to 0.165 wt.% depending on the volume fraction of the precipitates. Such amount of oxygen is apparently higher than that A710 steels normally contain (~ 0.02 wt.%). This large difference is probably due to the fact that the calculations were based on the assumption that all the observed precipitates are in the form of CuFe_2O_4 , which might not be true. Therefore, it seems more appropriate to suggest that only a certain amount of Cu-containing precipitates are CuFe_2O_4 and the rest are ϵ -Cu precipitates.

It would be very helpful if the observed precipitates can be extracted out of the α -Fe matrix, so that these precipitates can be

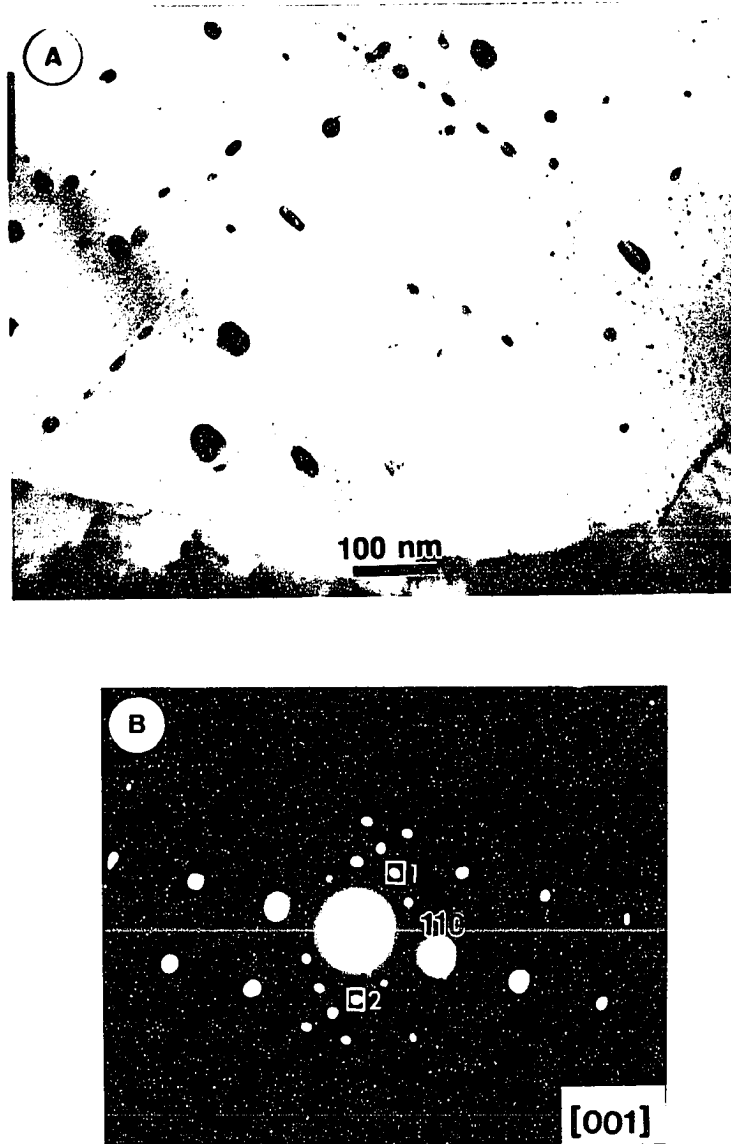


Figure 44: TEM micrographs of the HAZ in A710 steel
(3 KJ/mm, PWHT at 620°C for 10 hours)

(a) BF image, (b) SADP for (a),
(c) DF image from $(113)_p$ reflection (spot 1),
(d) DF image from $(113)_p$ reflection (spot 2).

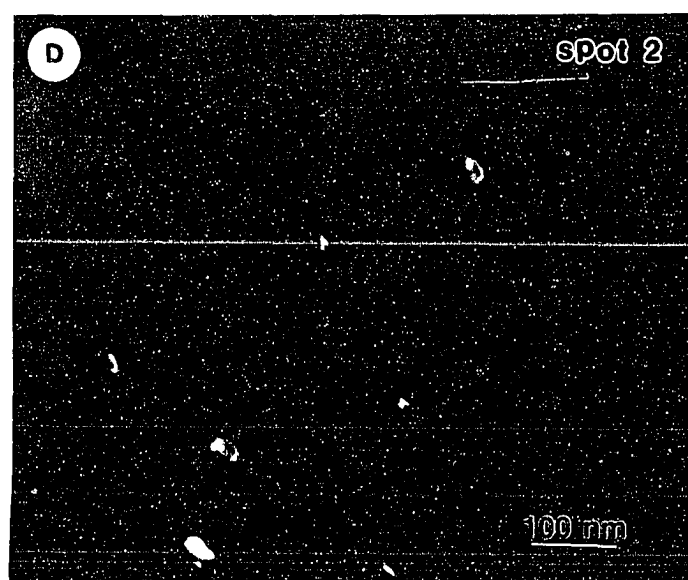
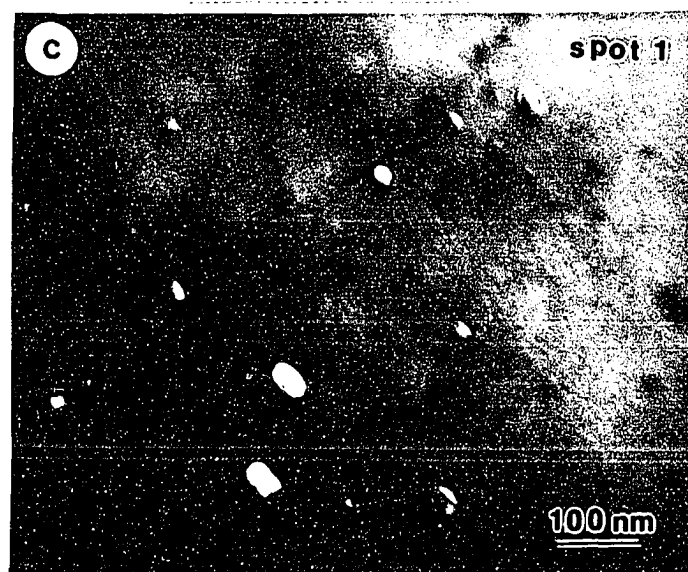


Figure 44: continued

examined without the interference of the matrix. Unfortunately the results obtained from the carbon extraction replica for this study were not helpful probably due to technique problems. On the other hand, it should also be pointed out that even if copper precipitates were found from the extraction replica on TEM, these precipitates might not be from the material matrix. This is because artifacts can be produced very easily when extraction replica was made for Cu precipitates in Fe-Cu alloys (Glenn, 1961). According to Glenn's observations, during the etching of Fe-Cu alloys, Fe may reduce Cu ions to metallic Cu by the reaction: $\text{Fe} + \text{Cu}^{++} \rightarrow \text{Fe}^{++} + \text{Cu}$. Consequently, the observed spherical copper particles are actually artifacts precipitated from the etching solution. Therefore special care is required in interpreting extraction replicas from these alloys.

Due to time limit as well as technique difficulties, additional investigations, such as the detection of oxygen in the precipitates both qualitatively and quantitatively through chemical analysis on AEM, and detection of pure copper (or ϵ -Cu) diffraction by more detailed SAD examinations, have not been conducted. These experimental procedures might contribute more information to further confirm the above conclusions, thus they might be suggestions for further study on this material.

4.6.2 Precipitation hardening during aging

In order to see how the Cu – containing precipitates behaved in A710 base plate during aging, selected specimens which represented the three most critical conditions (see figure 11): as – quenched (under aged), peak aged (at 500°C for 2 hours), and overaged (at 620°C for 100 hours) were examined on TEM. The results had already been shown in figure 40. The initial hardening is probably due to the formation of coherent Fe – Cu clusters which are invisible in TEM [figure 40(a)] due to the lack of a strain field of sufficient intensity. When near the peak hardness, these clusters transform into fine, spherical, coherent or partially coherent precipitates, although the precipitates at this stage were still too small to be detected in TEM as shown in figure 40(b). However, the evidence of their existence could be found on the corresponding diffraction patterns. In fact the existence of copper precipitates at this early stage has been confirmed by other techniques such as by X-ray imaging (Michael, 1988) and by field ion microscopy (Goodman, 1973). It has been reported that at the peak hardness the average diameter of the ϵ -Cu precipitates was only about $24 \text{ \AA}$ (Goodman, 1973). At higher temperatures ($> 600^\circ\text{C}$) or after a long period of time for a given temperature, precipitate coarsening would occur. Overaging [figure 40(c)] produced a coarse but uniformly distributed dispersion of much larger spherical as well as rod - shaped precipitates.

4.6.3 TEM examinations of A710 base plate

From TEM examinations in the as-received base material it was found that, besides polygonal ferrite, small amount of lath type structure (acicular ferrite) was also present as shown in figure 45(a). Under higher magnifications [figure 45(b)], it can be seen that there were many spherical Cu – containing precipitates in the ferritic matrix. Their distribution was quite uniform within the grain, but in some areas the density was different from grain to grain (some grains contained many precipitates and some other grains contained just a few). The sizes of these precipitates ranged from 50~200 Å as shown in figure 46. The precipitates were often found on the dislocations as shown in figure 47. As already illustrated in figure 41, the copper precipitates always contain line marks. Using the technique by McLean and Hale (1962), Hornbogen (1960) suggested that the line marks were twins or stacking faults instead of interference fringes. In his study, the line marks were introduced by plastically deforming the specimen, so that the copper precipitates were also subjected to some degree of deformation which then resulted in twinning or stacking faults in the precipitates. However, in Miglin's study, these line marks were found in the thin foil specimens without deliberate deformation, which is also the case in this study. For the purpose of confirmation, the line marks were also closely checked here. Following the technique used by Hornbogen, specimen tilting was performed and the results were shown in figure 48.¹¹ When the specimen was tilted to any different two - beam conditions, the lines on

¹¹ The photographs only showed two examples of tilting. Actually the specimen was tilted in many directions during the examination, and the lines on the precipitate never moved.

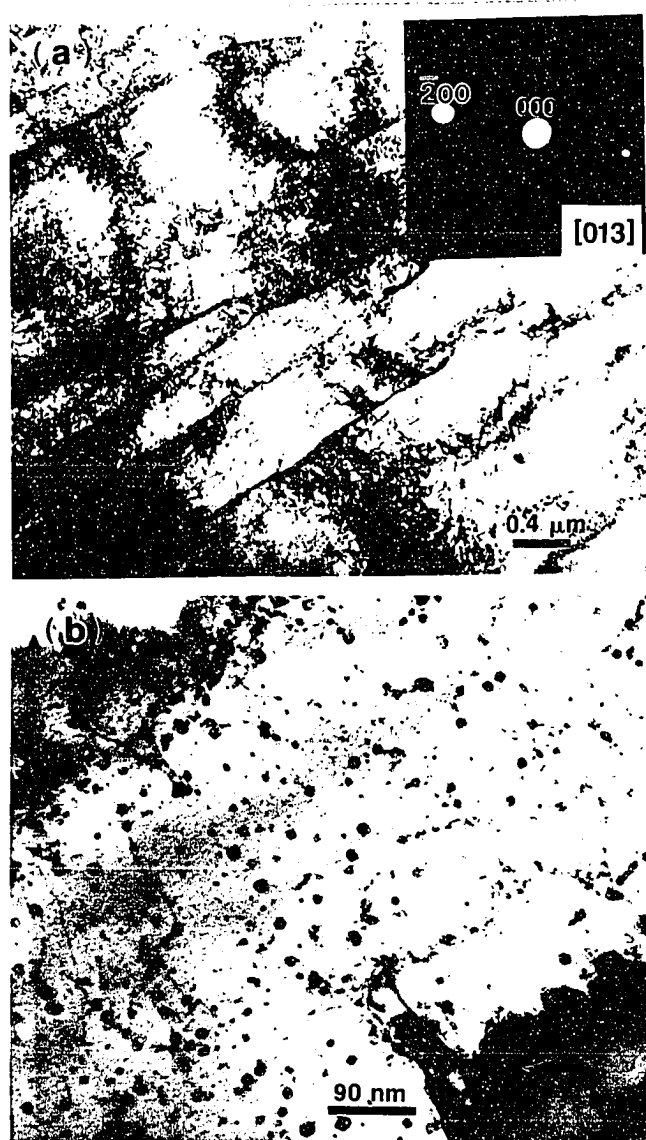


Figure 45: TEM micrographs of as received A710 base metal

- (a) BF image showing lath morphology,
- (b) Cu-containing precipitates in the matrix.

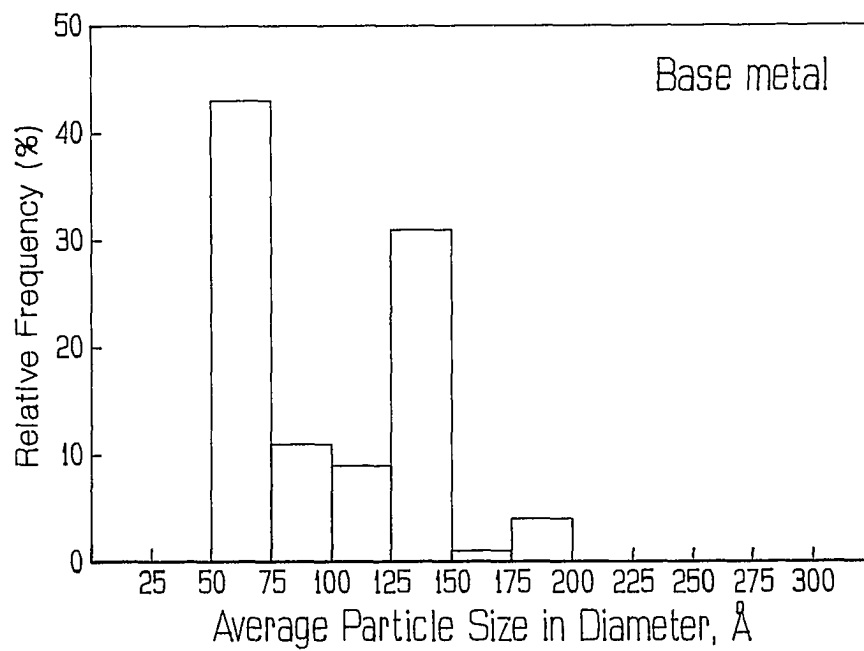


Figure 46: Average size distribution of Cu – containing precipitates in as received A710 base metal

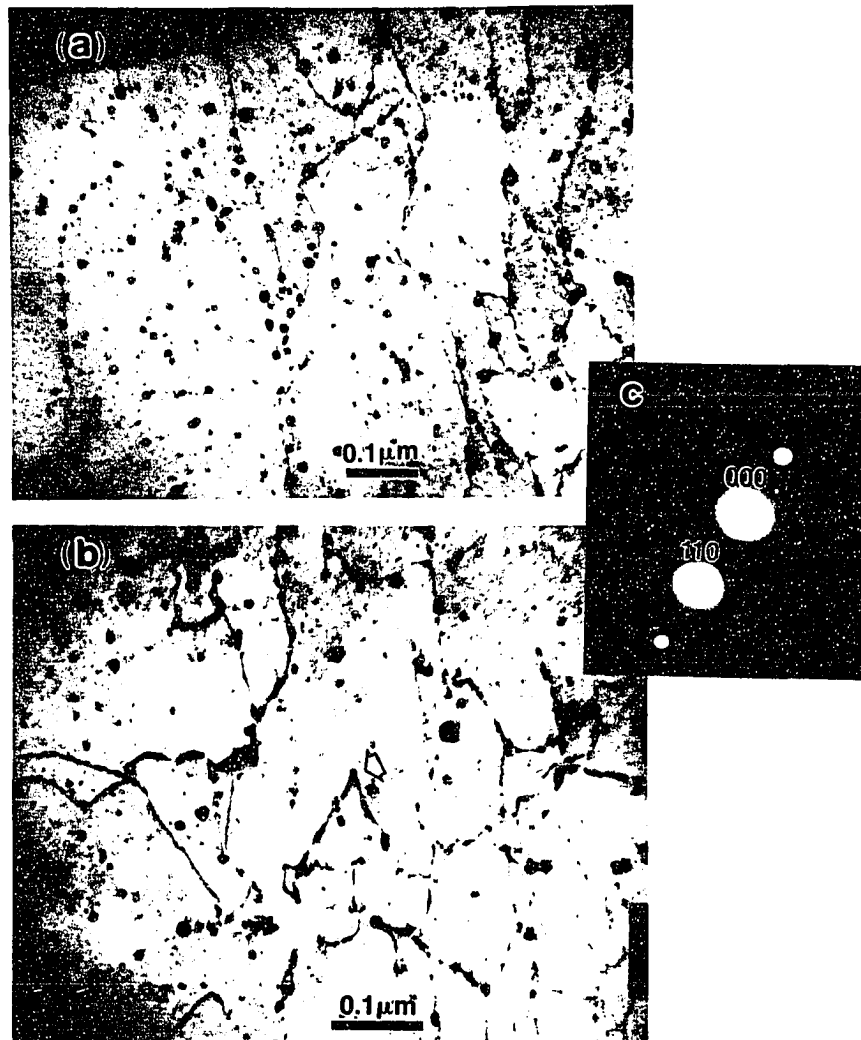


Figure 47: TEM micrographs of Cu precipitates in A710 base metal
 (a) precipitates on dislocations,
 (b) dislocation pinning by precipitates (arrow indicated),
 (c) SADP for (a) and (b).

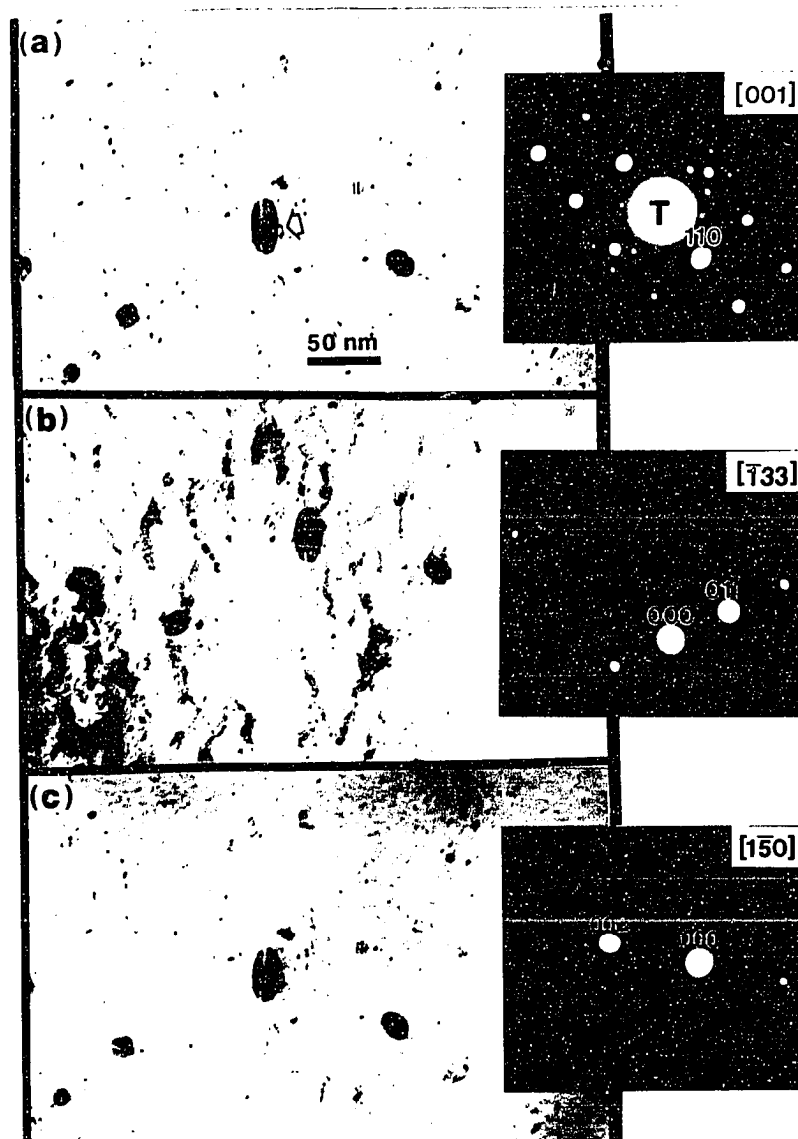


Figure 48: Observations of the line marks on the precipitates during specimen tilting

- (a) before tilting,
- (b) tilted to [133] zone,
- (c) tilted to [150] zone.

the precipitate did not move nor change directions, which indicated that the lines were likely to arise from plates (thin twins or stacking faults) within the particles rather than from dislocation lines [they would be interference fringes if the lines moved during tilting (Hornbogen, 1969)].

4.6.4 Coarse grained HAZ

Figure 49 represented the TEM microstructure of coarse grained zone in the as welded condition (3 KJ/mm). It consists of lath type ferrite with a high density of dislocations which were identified to be $\langle 111 \rangle$ type, typical of BCC crystal dislocations. Precipitates were not visible even at very high magnifications as shown in figure 50(a). This was probably because under as – welded conditions, the precipitates were very fine and still coherent just like the underaged condition during aging, so they were not visible under TEM, but the extra spots on the diffraction pattern in figure 50(b) evidenced the existence of the precipitates.

The effect of PWHT on the precipitates in the coarse grained HAZ was shown in figures 51 ~ 55. After a 2-hour heat treatment, small precipitates appeared on the dislocations [figure 51(c)], which probably were the preferred nucleation sites for the precipitates. The precipitates were relatively homogeneously distributed within the ferrite grains and grain boundary precipitation was hardly found. The shape of the precipitates at this stage was mostly spherical and the size distribution is shown in figure 52. After 10-hour stress relief, the precipitates grew larger and most of them changed from spherical to rod - like shape [figure 53(b)]. The size distribution at this stage is shown in figure 54. It was found that many precipitates were on the boundaries of the lath ferrite as

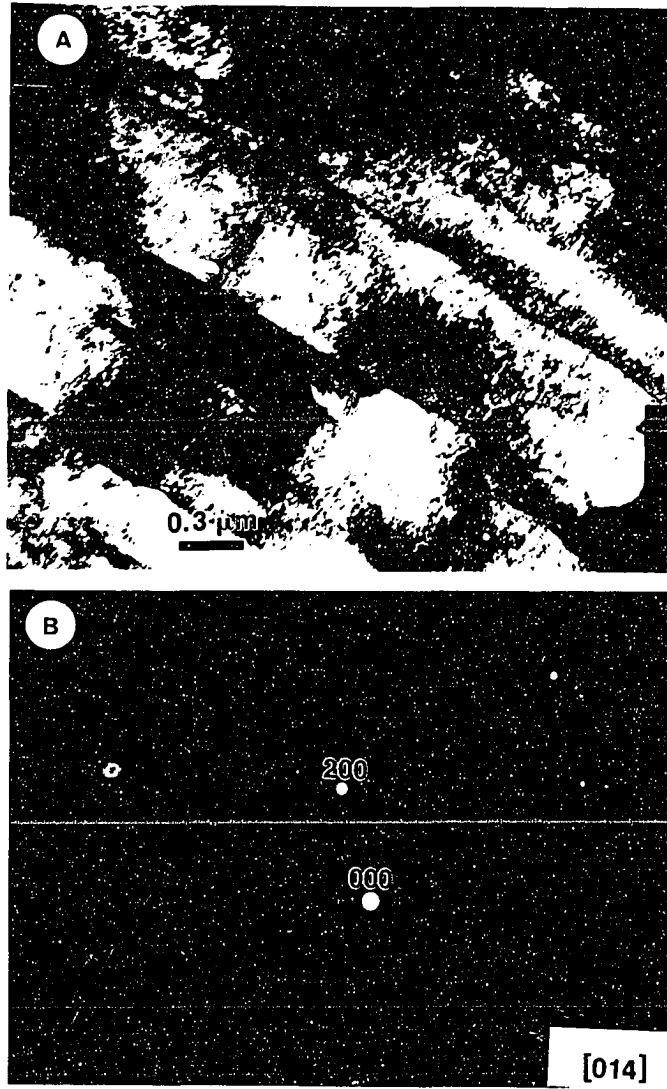


Figure 49: TEM micrographs of the as welded A710 HAZ
 (a) lath structure with high dislocation density,
 (b) SADP for (a).

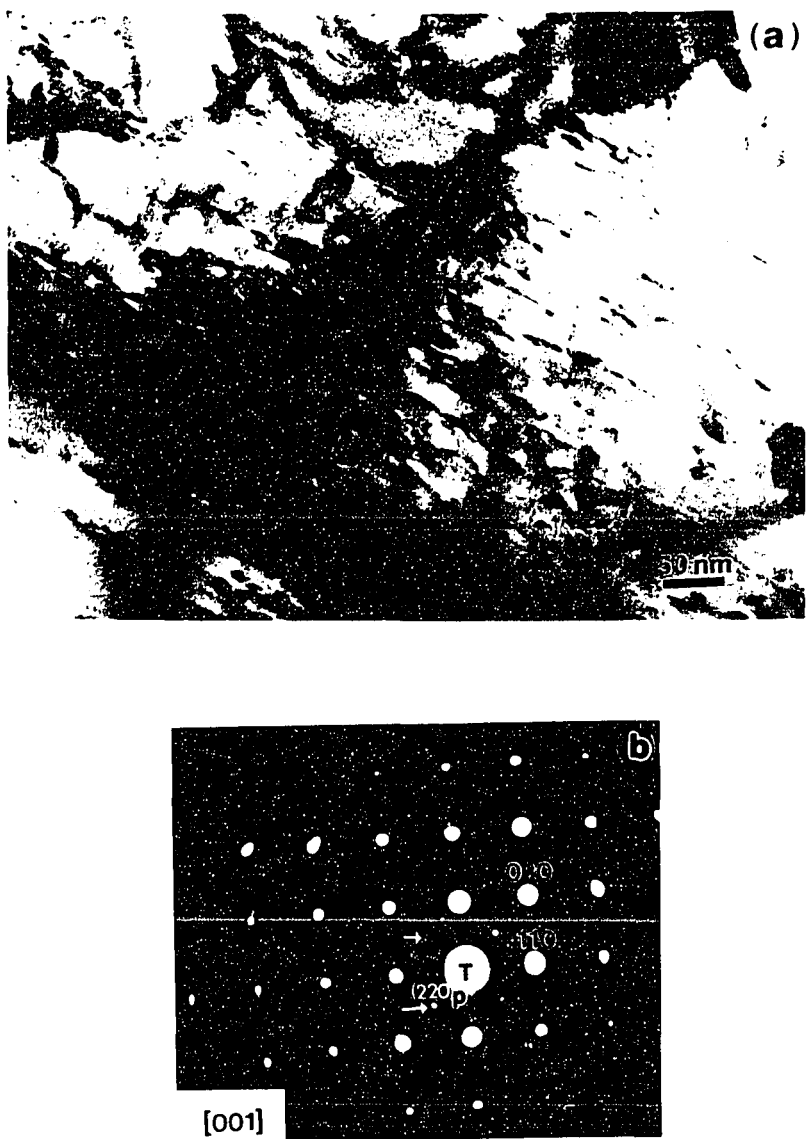


Figure 50: TEM micrographs of the as welded HAZ in A710 steel
 (a) BF image,
 (b) SADP for (a), showing precipitate spots.

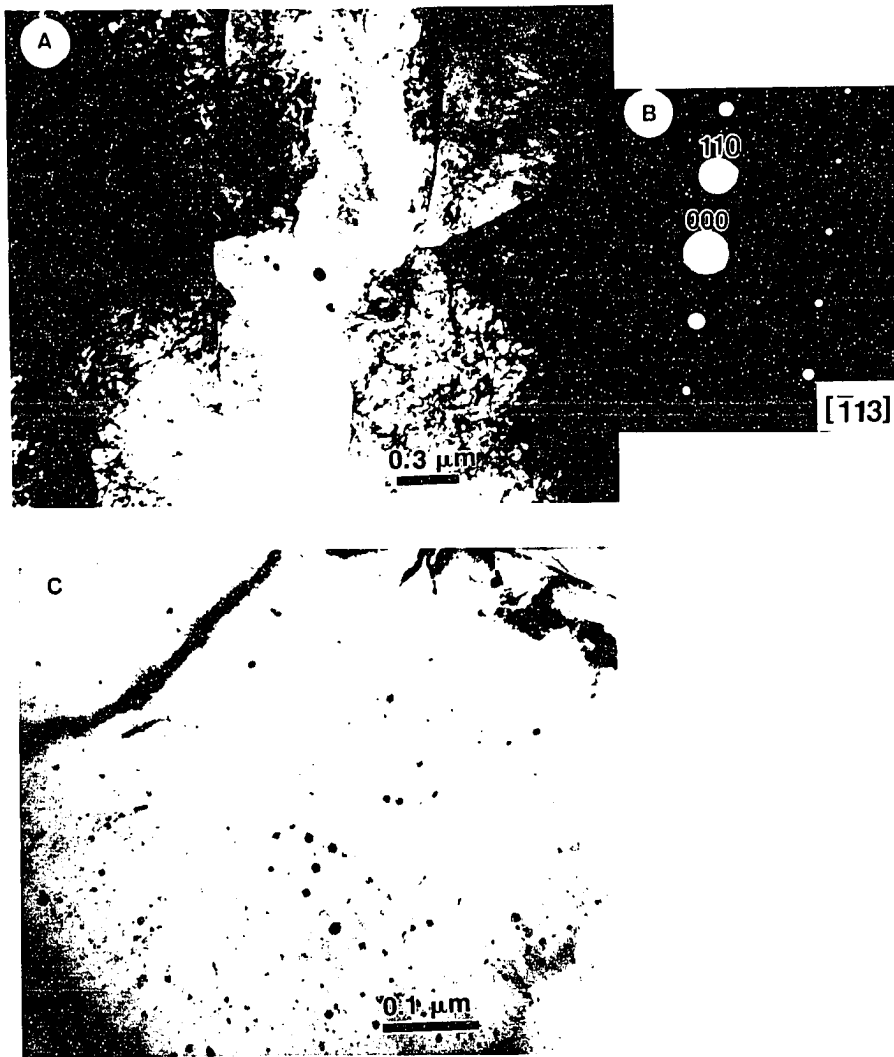


Figure 51: TEM micrographs of the HAZ (620°C for 2 hours)
 (a) ferrite lath and dislocation structure,
 (b) SADP for (a),
 (c) precipitates reappeared.

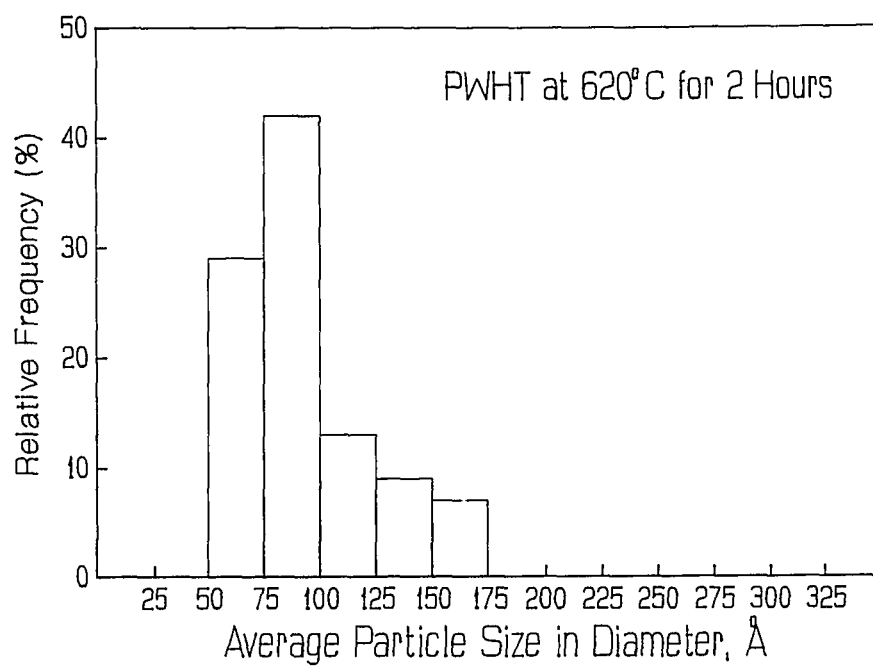


Figure 52: Average size distribution of Cu precipitates in A710 HAZ (PWHT at 620°C for 2 hours)

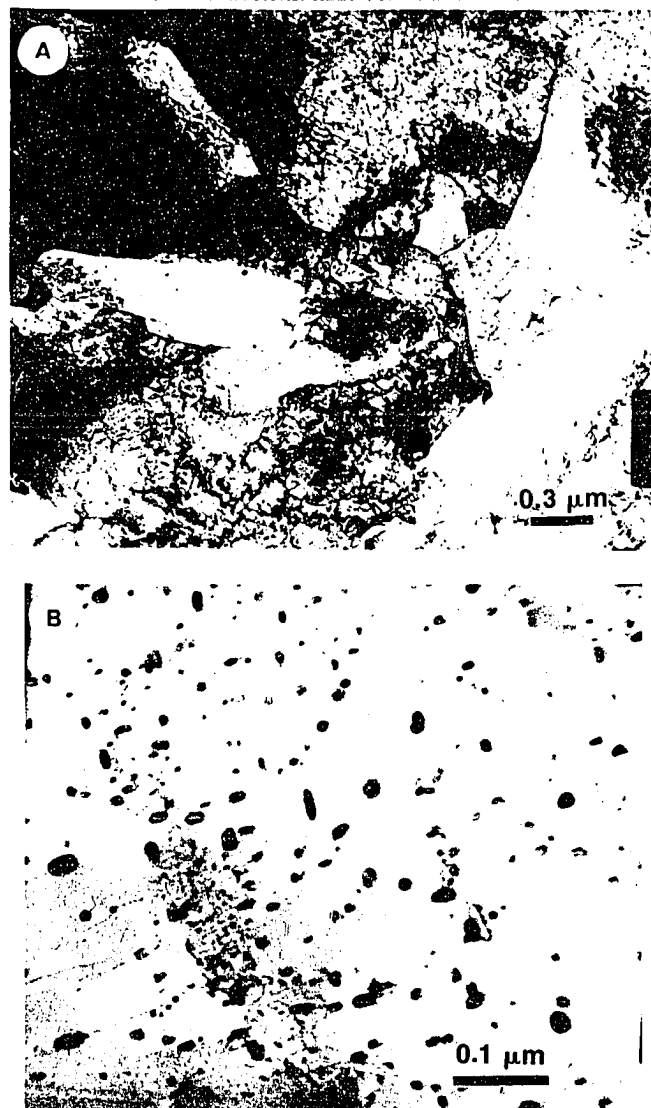


Figure 53: TEM micrographs of the HAZ (620°C for 10 hours)
(a) BF image showing recovery after stress relief,
(b) grown and elongated precipitates.

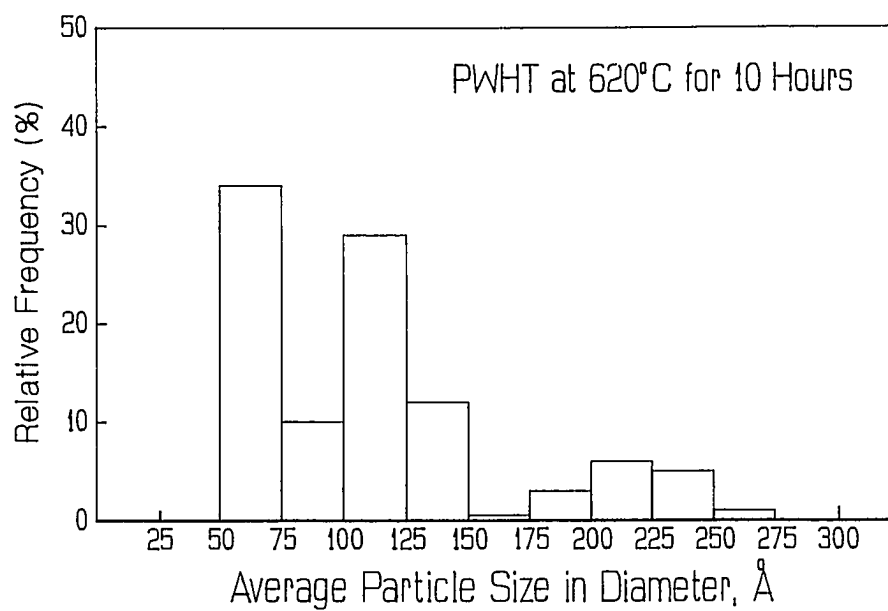


Figure 54: Average size distribution of Cu precipitates in A710 HAZ (PWHT at 620°C for 10 hours)

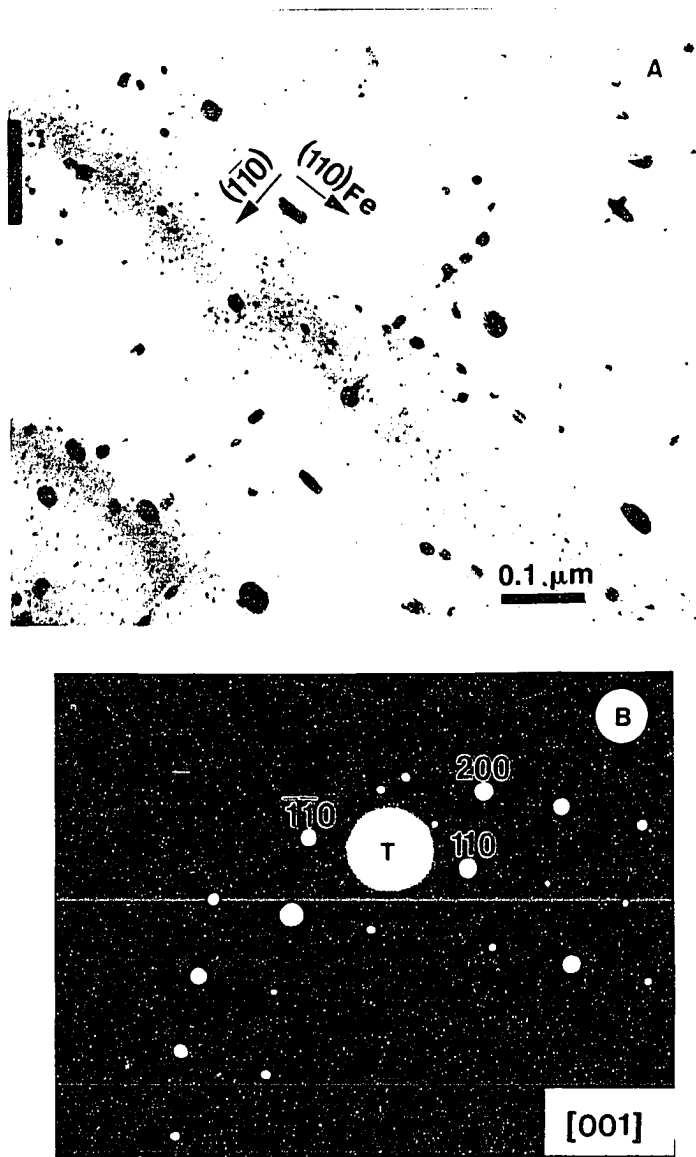


Figure 55: TEM micrographs of elongated precipitates in HAZ
 (a) they are elongated along perpendicular directions,
 (b) SADP for (a).

well as within the ferrite. The long axes of the rod-like precipitates were often aligned along two directions which are perpendicular. One example was shown in figure 55.

For the HAZ welded by 5 KJ/mm, the microstructure and Cu precipitation were also examined. The fine structures as well as the precipitation behavior under various PWHT conditions followed almost the same trend as in the case of the lower heat input just discussed above. Some selected photographs were shown in figures 56 ~ 57.

4.6.5 Coarse grained zone in the gleeble specimens

Results of the TEM study of the coarse grained zone in the gleeble specimens were given in figures 58 ~ 61. In the specimens without stress relief treatment, precipitates were also “absent” [figure 58(a)] as in the case of as-welded HAZ. After 2 or 10 hour stress relief, precipitates re – appeared, and even more precipitates were found on the lath ferrite boundaries than in the welded HAZ for 10-hour stress relief. This was shown in figures 59 ~ 60. Most of these boundary particles were Cu – containing precipitates but there were also some carbides as shown in figure 61.

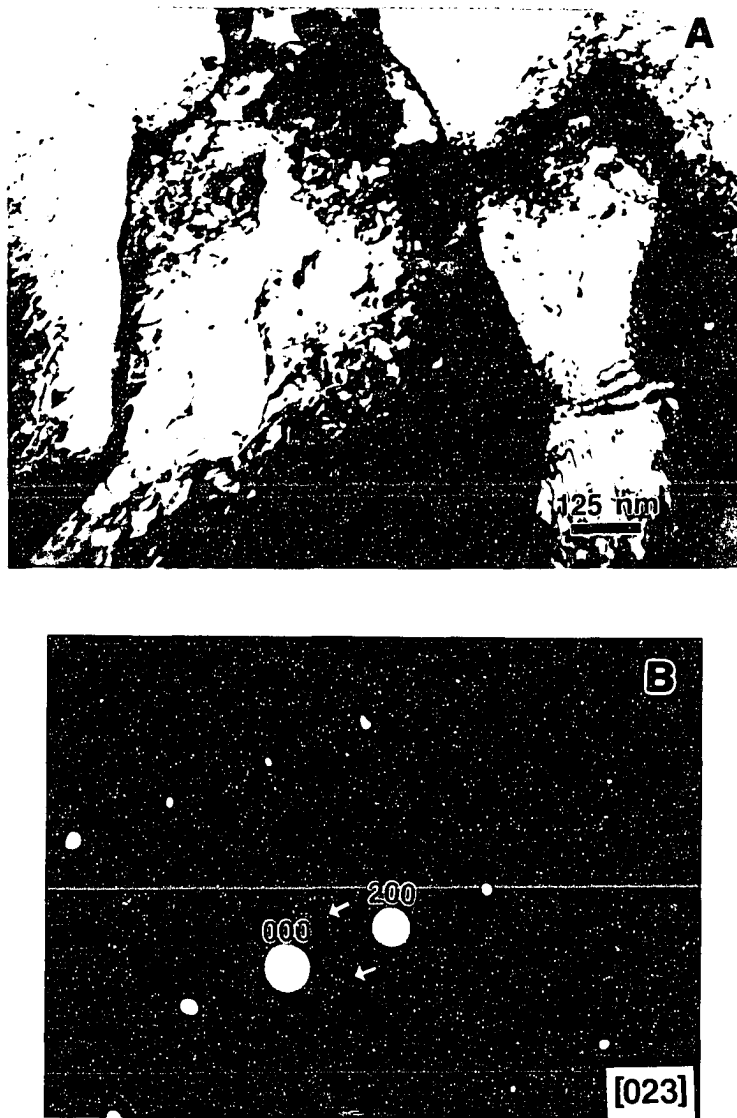


Figure 56: TEM micrographs of A710 HAZ (19 mm, 5 KJ/mm)
 (a) BF image of the as welded HAZ,
 (b) SADP for (a) showing precipitate spots.

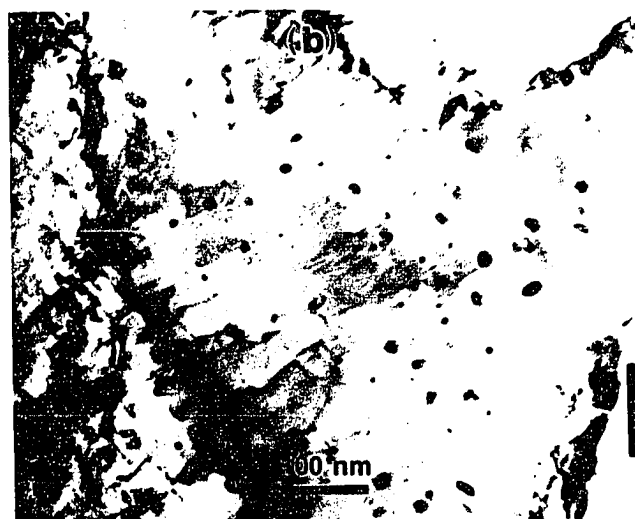
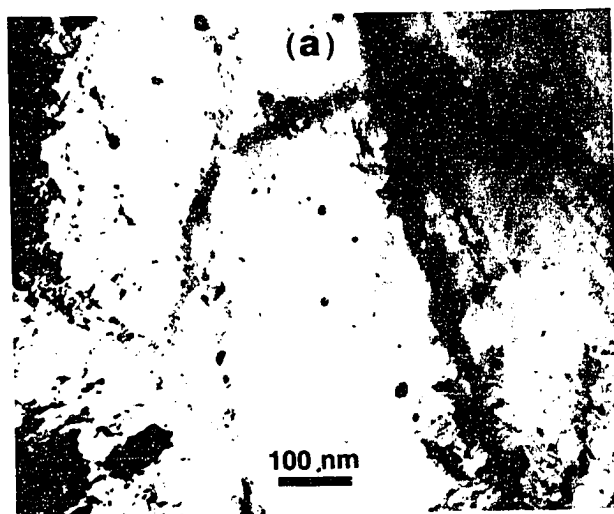


Figure 57: TEM micrographs of A710 HAZ (19 mm, 5 KJ/mm)

(a) BF image of the HAZ (620°C, 2 hours),
 (b) HAZ (620°C for 10 hours).

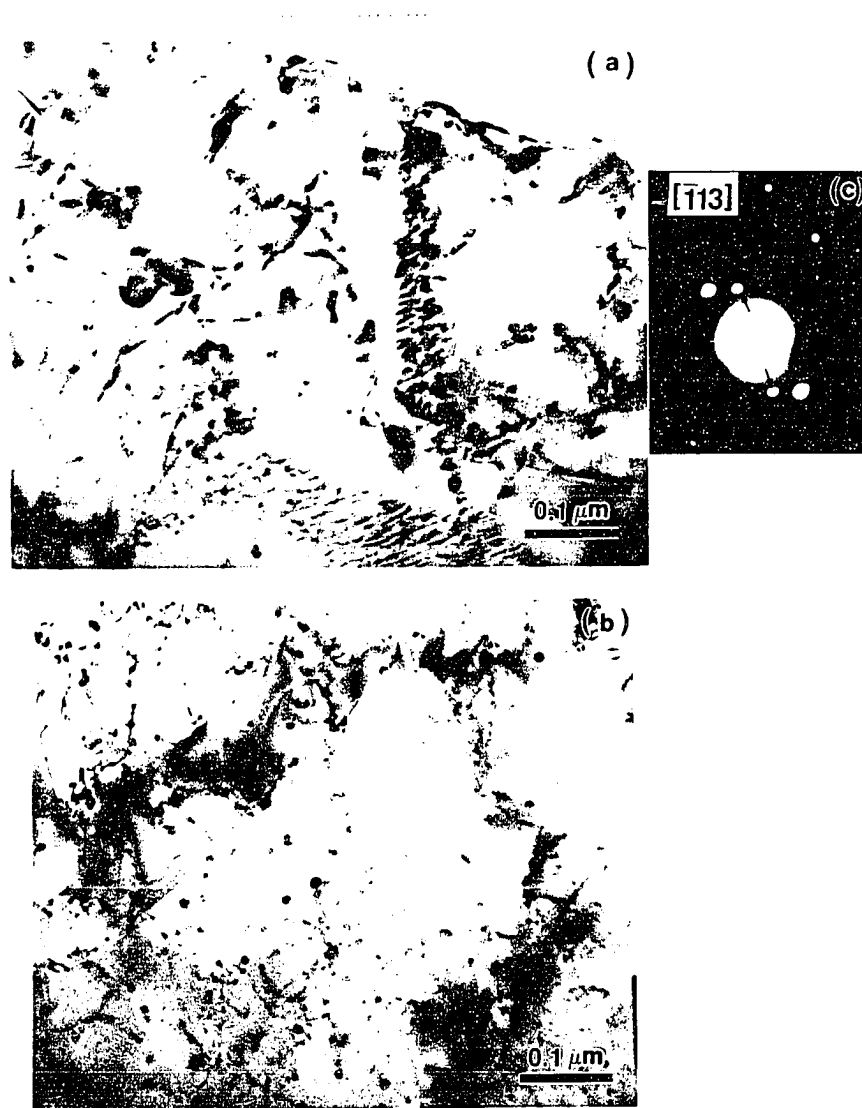


Figure 58: TEM micrographs of the simulated HAZ (3 KJ/mm)

- (a) BF image of ferrite structure (no stress relief),
- (b) BF image of precipitates (2-hr stress relief),
- (c) SAD pattern for (a) showing precipitate spots.

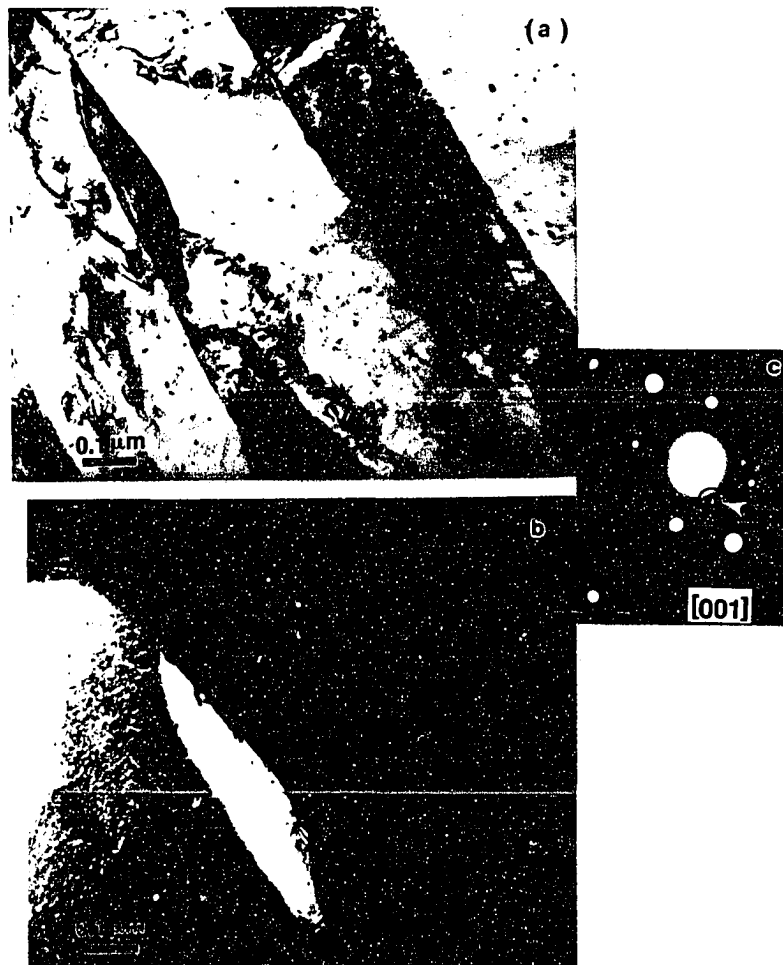


Figure 59: TEM micrographs of the simulated HAZ (10-hr SR)
 (a) BF image showing lath morphology,
 (b) DF image for (a) from a $\{220\}_p$ reflection
 as arrow indicated in (c).



Figure 60: TEM micrographs of the simulated HAZ (10-hr SR)

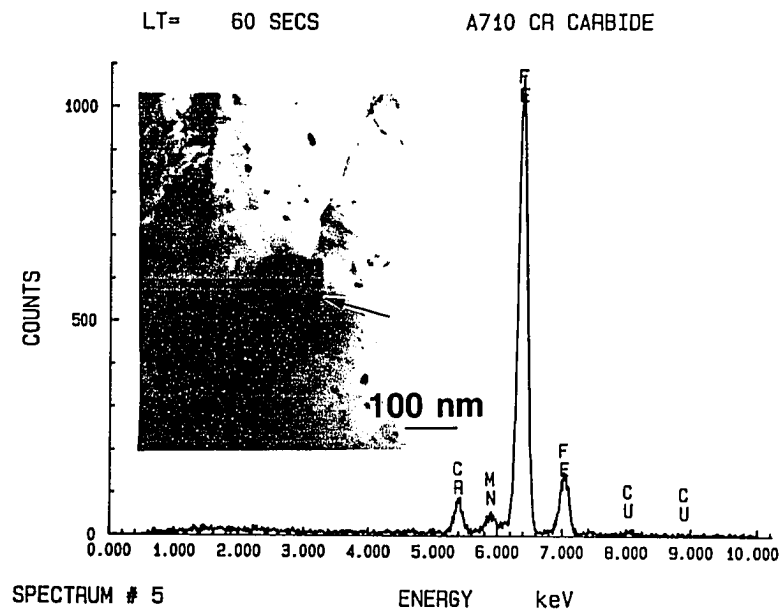


Figure 61: TEM micrographs of the simulated HAZ (620°C, 10 hours) showing grain boundary carbides (EDS spectrum obtained from the particle indicated)

4.7 Discussion on the relationship between the microstructure, strength and toughness

4.7.1 A710 base material

It has been shown that the microstructure of the heat treated A710, grade A, class 3 steel plates used in this study consists of fine grained polygonal ferrite interspersed with small regions of pearlite and acicular ferrite. The small grain size increases the yield strength as predicted by the Hall – Petch relationship (Hall, 1951; Petch, 1953). However, Hornbogen and Staniek (Hornbogen, 1974) found that when the precipitate size exceeded $150 \text{ \AA}$ in Fe – 1%Cu alloy, strengthening was independent of grain size. It seems possible that grain size strengthening has only a minor effect in age hardened A710 steels due to the large distribution and the range of sizes of precipitates. The most important contribution from the fine grain size is the improvement of the fracture toughness of the material through inhibiting crack propagation by the high angle grain boundaries. From the TEM observations it is evident that, besides the contribution of fine ferrite grain size and also fine Nb – carbides (Miglin, 1986), dislocation/precipitation interaction is one of the most important strengthening mechanisms which are dominant in this material. As has been shown in figures 42 and 47, precipitation was apparent on many dislocations, thus these precipitates may be presumed to have a very strong effect on dislocation movement. Thus according to this dislocation/precipitation interaction in this steel, and the nature of hard CuFe_2O_4 particles, it was suggested that the strengthening is caused by an Orowan mechanism (Hornbogen, 1960; Miglin, 1986).

Particle shearing was not observed in this or other studies (Hornbogen, 1974; Miglin, 1986).

The microstructural parameters which affect the strength also play a major role in the resistance of the material to fracture, or the material fracture toughness. The microstructural factors that influence the toughness include the dimensions of the characteristic structural features, e.g., the morphology of prior austenite grains, “co - variant packets” for structures consisting of parallel laths, carbide particles etc., and the basic microstructure, e.g., ferrite - pearlite, bainite, martensite etc. For A710 base material, its high impact toughness results mostly from: (1) fine ferrite grain size (figure 10) which improves the upper shelf energy and lowers ductile - to - brittle transition temperature (DBTT); (2) small amount of pearlite, compared with conventional HSLA steels, which reduces the possibility of cleavage crack propagation, thus lowers the DBTT; and (3) equiaxed and homogeneously distributed inclusions (figure 21), which is the key factor for a high level of upper shelf energy. The effect of Cu – containing precipitation on material strengthening is obvious, but its effect on toughness is more complex and not yet fully understood. Generally speaking, the precipitates provide an increment in strength and thus some loss in toughness, but if the particles are incoherent and finely distributed, they could inhibit localized slip and formation of dislocation pile – ups at grain boundaries, thereby increasing the upper shelf fracture toughness (Miglin, 1986). Fine and uniformly distributed precipitates were often observed in the base material. An example has been shown in figure 45.

Therefore, the properties of A710 steel can be optimized with a microstructure which contains a fine structure of polygonal and acicular

ferrite, precipitation of fine Nb(C,N) which gives a small ferrite grain size, and a mixture of finely distributed coherent and incoherent Cu – containing precipitates.

4.7.2 HAZ microstructure and impact toughness

The microstructure formed in the HAZ is a consequence of the composition of the steel and the thermal cycles resulting from welding. Although in microalloyed steels austenite grain growth can be inhibited by the presence of Nb – carbides which are stable at very high peak temperatures (Easterling^f, 1983), the welding cycle could still “delete” the original microstructures of fine ferrite grains close to the fusion line and replace it with very coarse grains with an unfavorable structure such as bainitic structure in the A710 HAZ. This microstructure has a high dislocation density which imparts high strength, but is detrimental to the impact toughness. A bainitic structure is a packet consisting of a group of parallel ferrite laths, and the lath boundaries are low angle. Hence cleavage cracks can easily propagate across a bainite packet with little resistance encountered at the low angle lath boundaries within a packet. The evidence of large cleavage facets has been demonstrated in the SEM observations discussed previously, which has confirmed the existence of bainitic structure formed in the coarse grained HAZ. It is likely that the HAZ toughness could be improved if the bainitic structure could be replaced by an acicular ferritic structure or a low carbon martensite through proper welding procedures or material composition modifications.

As just mentioned above, the HAZ toughness of A710 steels under this study was worsened mostly by the large grain growth and the

formation of an unfavorable microstructure. Furthermore, the degree of the toughness deterioration increases with increasing heat inputs, that is, the toughness of the HAZ when welded at a HI = 3 KJ/mm is better than that welded at 5 KJ/mm in 19 mm plate, and the toughness of the HAZ by 1.77 KJ/mm is better than that by 4.9 KJ/mm in 32 mm plate. This is because the fracture behavior of a welded structure depends not only on the toughness of the HAZ but also on its size or width. Increasing heat input increases the width of grain growth zone, and this wider coarse grained zone increases the number of possible fracture paths in the HAZ, while a narrow grain growth zone makes it more difficult for a fracture to proceed entirely in the HAZ.

In spite of all these effects, the toughness of the HAZ of A710 steels at heat input of 3 KJ/mm or less remains very satisfactory because of the exceptionally high toughness properties of the base metal and also the narrow width of the HAZ. The transition temperatures between -61°C (as welded) to -70°C (PWHT for 2 hours) for HI = 3 KJ/mm in 19 mm plate, and between -60°C (PWHT for 2 hours) to -97°C (PWHT for 10 hours) for HI = 1.77 KJ/mm in 32 mm plate are all better or approximately as good as the ASTM impact property requirement for the base material (transition temperature at -62°C).

The previous discussions about the copper precipitation effect on the strength and toughness of A710 base material can also be applied to the weld HAZ, i.e., precipitation increases the material strength but at the same time deteriorates the toughness. However, compared with the coarse bainitic structure, copper precipitation hardening plays only a minor role in affecting the HAZ impact toughness under as welded condition.

In this study, the effect of PWHT was contradictory, even though the problem of data scatter was believed to be partially responsible for this uncertainty. At low heat input (3 KJ/mm or less), it seems that PWHT tends to reduce the HAZ impact toughness. From the microstructure observations using TEM as discussed above, it was likely that HAZ hardening due to copper precipitation during stress relief was the major cause for the toughness loss compared with the as-welded condition. Under as-welded conditions, the Cu – containing precipitates in the HAZ were very fine, but probably small in quantity, thus the hardening effect was not very strong. After PWHT for a period of time such as 2 hours, even though the peak – hardening stage had just passed according to figure 11, the precipitates at this stage were numerous and still able to resist the dislocation movement, thus increasing the hardness of the HAZ but decreasing the toughness. For longer period of time such as 10-hour stress relief, the precipitates became quite large and no longer as effective as the smaller precipitates in strengthening, but large amount of grain boundary precipitation were formed, which was responsible for the low toughness.

In the case of higher heat inputs, PWHT seemed to be favorable to the HAZ toughness even though the effect was not very significant. It was probable that under higher heat input, both the extent of grain growth zone and internal stress dominated the toughness change in the HAZ. The toughness loss due to precipitation hardening was possibly offset by the beneficial effect of internal stress relief during tempering which improves the toughness.

These observations suggest that the impact toughness difference between A710 base material and the welded HAZ results from the

formation of coarse bainitic microstructure in the HAZ, and that PWHT is helpful in improving the HAZ toughness of A710 weldments made by high heat inputs ($> 3 \text{ KJ/mm}$), but for lower heat input weldments PWHT is not always necessary. In the welded regions (HAZ), the toughness changes are a result of precipitation hardening during PWHT. However, the overall effects of precipitation hardening on the HAZ toughness are not very serious, and the coarse bainitic microstructure in the grain growth zone is the major cause of HAZ toughness deterioration.

5 Conclusions

The experimental results and discussion lead to the following conclusions:

1. It was confirmed that ASTM A710 grade A class 3 base metal is a material with a combination of high strength and very high toughness based on the comparison between the base plate and weldment properties. The strengthening is mostly through the interaction between the Cu-containing precipitates and dislocations, and its high toughness is the result of fine ferrite grain size, very low carbon content and decreased amount of harmful pearlite.
2. The coarse grained HAZ microstructure is essentially bainitic ferrite. Under as-welded condition, the bainitic microstructure is the major cause for the decreased impact toughness of the weld HAZ.
3. Increased welding energy input (higher than 3 KJ/mm) results in reduced HAZ toughness in both 19 mm and 32 mm plates. However, all welds had a transition temperature below -20°C (for 47J CVN energy) even at heat inputs as high as 5 KJ/mm. For most structural applications, this transition temperature is well below what would be required.
4. The effect of PWHT at 620°C on the HAZ impact toughness is mixed. The test data scatter problem, which seemed to be extensive, prevented a precise prediction of the transition temperature change. The general trend is that stress relief treatment is beneficial for the HAZ welded by higher heat

input, while the effect is negative for the welds by lower heat input. Therefore, from the viewpoint of HAZ toughness improvement, PWHT is not recommended for A710 welded structures, especially for weldments by low heat inputs.

5. There are a certain amount of Cu-containing precipitates which are probably in the form of Fe-Cu compounds instead of ϵ -Cu. Under as welded condition, Cu-containing precipitation hardening only has a minor effect on the HAZ toughness compared with the major toughness loss due to coarse bainitic structure formation. However, under PWHT condition, precipitation hardening is believed to be responsible for further toughness loss in the HAZ compared with that under as welded condition.
6. The precipitate identification in this study is still uncertain. The experimental evidence, such as the confirmation of Cu containing in the precipitates, the heat treatment dependence of the particle size and morphology, and the interactions between dislocations and precipitates, could only confirm that these precipitates are internal to the steel matrix, and they are the right precipitates (containing Cu) that we expected to study. However, there is insufficient evidence to prove that these precipitates are CuFe_2O_4 . In fact, there is some evidence against this identification. The most serious is that the required amount of oxygen seems too high to be realistic. Another problem is that the growth rate of the precipitates during aging in this study is apparently in contradiction with what would be expected for a spinel structure of CuFe_2O_4 . Therefore, a positive conclusion on the nature of the precipitates could not be

drawn, and further study is needed in order to completely resolve these problems.

6 Suggestions for future work

In this dissertation, extensive studies have been conducted on both mechanical properties and microstructures of A710 weldments. Most of the results are well established by the experimentation, but there are also some problems that remain unresolved. Therefore the following work is suggested for continuing investigations on A710 steels:

1. To better understand the welding properties of A710 steels, more mechanical testing on A710 weldments should be made, specifically, Charpy impact testing of HAZ and weld metals on a wider range of heat inputs, and studies on the effect of PWHT on the weld metal toughness under various heat inputs. PWHT temperatures other than 620°C could also be explored.
2. The Gleeble simulation experiment in this study is limited, and the toughness results are not very satisfactory. More studies (various thermal cycles, and multipass welding simulations, etc.) are recommended.
3. In the TEM study, the precipitate identification is still uncertain. It will be necessary to search for a new technique for making thin foil specimens (such as developing a new chemical solution for electropolishing), so that any possible oxidation or contamination will be eliminated.
4. Cu precipitates are believed to have been formed in the coarse grained HAZ under as welded conditions, even though these precipitates were not visible because they were either coherent or too small to be seen using conventional

TEM. Therefore it is suggested that detection of these ultrafine precipitates using more advanced imaging technique should be made so that the effect of precipitation hardening on the HAZ toughness in the as welded conditions can be fully understood.

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