Evaluation of the Response of Armor Alloys to High Temperature Deformation

THESIS

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By

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Abstract

High strength alloys, such as titanium alloys and steels have been widely used for armor applications. However, high strength materials have poor formability at room temperature and are prone to cracking during welding. It is necessary to develop alternative manufacturing methods that can replace conventional welding technologies. The main objectives of this project are: 1) development of a testing procedure for evaluation of the response of high strength alloys to hot induction bending, and 2) development of optimal process control windows for hot induction bending of three high strength materials: alloy Ti-6Al-4V and armor steels Armox 440 and ARL XXX.

A testing procedure has been developed that combines hot ductility testing, high temperature straining using a Gleeble™ thermo-mechanical simulator, high temperature straining followed by room temperature tensile testing, evaluation of response to tempering, phase transformation analysis, thermodynamic simulations, and metallurgical characterization.

Hot ductility testing indicates a gradual increase in ductility of the three tested alloys as temperature increases. Strain rate has no significant effect on hot ductility of alloy Ti-6Al-4V and ARL XXX steel. During hot ductility testing, extensive void formation is observed in alloy Ti-6Al-4V between the starting temperatures of α to β transformation and the recrystallization temperature, and in Armox 440 steel between the A₁ and A₃ temperatures. No voids were found above beta solvus temperature in Ti-6Al-4V and above the A₃ temperature in Armox 440. Limited void formation occurs below the start of α to β
transformation in Ti-6Al-4V and below the A₁ temperature in Armox 440.

High temperature straining tests show that strain-induced porosity in Ti-6Al-4V can be avoided if strain is limited below 24% at 430°C and below 7% at 650°C. In Armox 440 and ARL XXX steels, voids were only observed in samples strained to failure.

High temperature straining followed by room temperature tensile testing shows that in class 2 Ti-6Al-4V alloy highest ultimate tensile strength is obtained after 11% straining at 430°C. For the two armor alloys, straining above the A₃ temperature provides room temperature mechanical properties that are closest to the original properties of two armor steels.

Holloman-Jaffe parameters have been developed for evaluation of the effect of hot induction bending on hardness in Armox 440 and ARL XXX steels.

A continuous cooling transformation diagram has been developed for the coarse grained heat affected zone of ARL XXX steel. The liquidus, solidus, A₁ and A₃ temperatures in this steel have been determined using single sensor differential thermal analysis. The reasons for solidification cracking in fillet welds of ARLXXX steel have been investigated using metallurgical characterization and thermodynamic simulations.

The presented approach allows developing optimized processing windows for hot bending of high strength alloys. Process parameters developed in this research, in terms of optimal bending temperatures and strain ranges, can be applied to avoid defect formation and minimize loss of properties during hot induction bending of the three tested alloys.
This document is dedicated to my parents, who support me whole-heartedly and encourage me to pursue my dream.
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Chapter 1: Introduction

High strength alloys, such as titanium alloys and steels have been widely used for armor and structural applications. Because of their varying microstructures, compositions, treatment and processing methods, they possess different mechanical, ballistic and blast properties. Joining by conventional welding technology can lead to a loss of mechanical properties. The process of making welds in large thick-section components is also expensive and time-consuming.

Titanium alpha-beta alloys, such as Ti-6Al-4V, have good commercial availability and good ductility leading to high degree of ballistic protection. Weldability of this group of alloy is strongly affected by the amount of beta phase present. Alloy systems that are strongly beta-stabilized can be highly susceptible to weld embrittlement and it is impractical to perform post-weld heat treatment to restore the original ductility. Another major issue for welding titanium alloys is the elimination of atmospheric contamination, such as oxygen, nitrogen and hydrogen. Accumulation of these interstitial elements in the weld metal and heat-affected zone (HAZ) will improve strength and hardness but also greatly reduce ductility to the extent that cracking may take place even with moderate restraint [1]. Epitaxial solidifications in multi-pass welds will form large columnar grains that lower the toughness of titanium alloys [2]

Steel materials used in armor application mainly consist of wrought-steel homogeneous and quenched and tempered (Q&T) high-hardness steel. Excellent mechanical properties, such as high hardness, as well as moderate-to-high ductility and toughness provides better
ballistic resistance and multi-hit capability [3]. However, increasing thickness and strength of Q&T steels may also increase cracking susceptibility during welding [4]. Inappropriate welding procedures, such as improper heat input and selection of susceptible welding consumables, may cause cold cracking in the hardened region of HAZ, HAZ softening and solidification cracking in fusion zone, in particular when armor plates are to be welded with high restraint during fabrication and repair [5].

There is a need for the development of alternative manufacturing process for high strength alloys that can replace risk-sensitive welding and maintain mechanical and ballistic properties. Most high strength alloys have poor formability at room temperature. The process of induction heating followed by press-brake bending offers an alternative to welding which can improve formability, and reduce cracking susceptibility and manufacturing cost. The main objective of this project are to: 1) develop a laboratory procedure to determine the response of candidate armor alloys to hot induction bending; and 2) identify optimized procedures for hot induction bending of these alloys. The methodologies used in this investigation for evaluating these materials include: 1) hot ductility testing to determine the optimum forming temperature range; 2) metallurgical characterization to evaluate the material response to elevated temperature exposure; 3) high temperature straining followed by room temperature tensile testing to evaluate the effect on room temperature service properties; and 4) heat treatment response of armor steel on hardness. A new test technique was also developed using the Gleeble™ thermo-mechanical simulator to evaluate the effect of strain parameter on mechanical response.
In addition, a newly developed armor steel, ARL XXX, is examined starting from base metal microstructure and properties, critical phase transformation temperatures, continuous cooling transformation, and response to welding and hot induction bending.

The results of this research can be used as a process development and control tool for the manufacture of armored vehicles. It can be applied to determine failure conditions of high strength materials during bending, as well as to evaluate the effect of strain and temperature on the failure mechanisms in high strength alloys.
Chapter 2: Literature Review

2.1 Titanium

2.1.1 Introduction to Titanium alloys

Titanium, a nonferrous light metal, was first discovered by William Gregor a mineralogist and chemistry scientist in England back in 1791. Four years later, Martin Heinrich Klaproth, a chemist in Berlin separated titanium oxide from a mineral called rutile and named the element after the Titans of the Greek mythology [6]. It was challenging to obtain the metal with high purity because of its tendency to react with oxygen and nitrogen. It took more than a century later in 1910 that Matthew Albert Hunter from Troy, N.Y. extracted the element by heating a mixture of titanium tetrachloride and sodium in a steel bomb. At the start of World War II, Wilhelm Justin Kroll from Luxembourg was able to produce large quantity of titanium in one single process known as Kroll process, which led to his recognition as father of titanium industry and allowed the metal to be used commercially [7]. The invented process isolated titanium in porous spongy form by reducing titanium tetrachloride with magnesium in an inert-gas atmosphere. Since then, titanium alloys became the main material for aerospace industry. As of today, Kroll process is still widely used for batch production of titanium. Its application expands to other industries, such as defense, medicine, power generation, transportation, chemical processing, marine, and structural [6]. In addition, titanium aluminide is a relatively new group of intermetallic compound with high industrial and technological interest.
Titanium and its alloys are a group of materials with attractive mechanical and physical properties. The main advantages are excellent corrosion resistance even in extreme environment, such as seawater, halides and wet halogens, as well as acidic solutions; good elevated temperature properties; and high yield strength with relatively low density that leads to excellent structural efficiency [8]. Figure 2.1 compares strength versus maximum service temperatures of various metal alloys. Ability to resist chemical attack is due to the presence of a thin and chemically stable oxide layer on surface mainly consisting of TiO$_2$, which can heal itself with only traces of oxygen or moisture presence when mechanically damaged. Its driving force is strong affinity of titanium to oxygen and high solid solubility of oxygen of about 14.5% in titanium [6]. Depending on alloy composition, a wide variety of strength and fracture toughness combination is offered, as shown in Figure 2.2 [9]. For Titanium alloy with lower strength, resistance to stress corrosion cracking and corrosion-fatigue in chloride solution is often obtained. They also show good S-N fatigue strength and life regardless of the medium involved. Other appealing properties include superior erosion and cavitation resistance; low thermal expansion coefficient and modulus of elasticity that is about half of steel and nickel alloys; high melting point; high intrinsic shock resistance; excellent ballistic resistance-to-ductility ratio for armor application; biocompatible and good cryogenic properties [10].

However, use of titanium and its alloys into commercial sector, in particular automotive industry, has been limited by the high cost and processing of material. In order to minimize this issue, cost effective manufacturing methods are being developed that can optimize properties to achieve high performance for various usage [8].
Figure 2.1: Specific strength versus maximum service temperature of metal alloys. [9]

Figure 2.2: Plot of fracture toughness versus yield strength of Titanium alloys [8].
2.1.2 Composition and Microstructure of Titanium alloys

Titanium is an allotropic metal and exits in two crystallographic forms. At lower temperatures, it exhibits a hexagonal close-packed (HCP) crystal structure known as alpha (α) phase. Phase change occurs as temperature increases to about 883 °C and transforms to a body-centered cubic (BCC) crystal structure, which is also called beta (β) phase [11]. Alpha-to-beta transformation temperature marks the start of phase change and beta phase is fully transformed at beta-transus temperature. Other secondary phases are omega and alpha-2 that may affect stress corrosion cracking in same case [10]. The transformation temperature of titanium alloys is impacted by purity of the metal, as well as interstitial and substitutional elements. Figure 2.3 displays the unit cell of both phases. c/a ratio of pure α titanium is 1.587. The three most densely packed lattice plans are (0002) basal plan, one of the {1010} prismatic planes and one of the {1011} pyramidal planes. For pure beta titanium, lattice parameter a equals to 0.332 nm at 900 °C [6].

Figure 2.3: Unit cell of a) alpha phase and b) beta phase [6].
By addition of different alloying elements and use of thermo-mechanical processing methods, percentage of phases and properties of alloy will vary. Table 2.1 summarizes the common alloying elements and their effect on microstructure. They are generally classified into two types according to their functions: α and β stabilizers [12]. Figure 2.4 shows the effect of the two stabilizers on the phase diagram of titanium alloy.

![Diagram showing α and β stabilizers](image)

Figure 2.4: Effect of α and β stabilizers on phase transformations of alloy Ti-6Al-4V [6].

Alpha-stabilizers can be used to widen the alpha region in phase diagram and increase α to β transformation temperature, such as aluminum, nitrogen, oxygen, carbon and boron. On the contrary, beta-stabilizers can depress the transformation temperature, and strengthen beta phase with high solubility. They can further divided into beta-isomorphous and beta-eutectoid elements. The former group of elements possess same solubility with beta phase titanium. Examples are molybdenum, vanadium, niobium and tantalum. Latter group of elements have limited solubility in beta titanium, which lead to formation of intermetallic compounds by eutectoid decomposition in beta phase. Examples include iron,
chromium, manganese, which behave more like beta-isomorphous elements due to very slow eutectoid decomposition; copper and silicon, which develop active eutectoid system and help strengthen titanium alloys by intermetallics precipitation [13]. Addition of aluminum, manganese or copper can improve tensile strength of titanium alloy. Vanadium, zirconium, molybdenum or silicon can advance high temperature properties. Better formability can be achieved by adding Tin. Small amount of Boron can also increase hardenability of titanium alloy [14]. Based on phase distribution, titanium alloys are divided into three categories: alpha alloys, beta alloys, and alpha + beta alloys.

Alpha alloys are classified into three types: fully-α alloys, near-α alloys with less than 2 % β stabilizers and age-hardenable alloys, i.e. Ti-Cu alloys. Commercially pure (CP) titanium, is the weakest but most corrosion resistant among various groups of titanium alloy. Yield strength is in range of 170 to 480 MPa depending on interstitial and impurity levels [10]. It is a major material for building chemical and petrochemical processing equipment, heat exchangers and piping facility because of excellent weldability, durability and fabricability [6]. Microstructure of high purity titanium (grade 2) exhibits primary alpha grains in a mixture with small pockets of beta grains, which contain very fine alpha-phase lamellae [15].

Alpha alloys usually contain more α-stabilizers (i.e. aluminum or tin) that are soluble in alpha phase. They have relatively low strength but better creep resistance and elevated temperature properties compared to beta alloys. They are applicable in cryogenic conditions due to lack of ductile-to-brittle transition range. Additional characteristics are relatively good weldability and toughness. However, they have high ease of defect formation during forging [11].
Beta alloys are alloyed with transition metals, such as niobium, molybdenum and vanadium, which promote beta phase formation and lower beta-transus temperature. As beta stabilizer content increases, all-beta structure can be obtained at room temperature. Such microstructure becomes metastable and later decomposes to alpha after holding below beta transus when amount of beta stabilizer is within specific range [16]. They have excellent hardenability, forgeability within wide range of temperatures and prompt response to heat treatment [11]. Commercial thermomechanical treatment can be mainly divided into four stages: working operations, such as hot rolling and forging; solution treatment; quenching; and aging [17].

Alpha + beta alloys contain a mixture of α and β phases. Distribution and types of the two phases will affect properties of the material, which is controlled by heat treatment or thermo-mechanical treatment [11]. This group of alloy has three main types of microstructures: fully equiaxed, fully lamellar, and bimodal. Fully lamellar structure typically has high resistance to creep and fatigue crack propagation, good fracture toughness, and low tensile ductility. It consists of colonies of HCP α-phase lamellae in a matrix of large BCC β grains in diameter of several hundred microns. The β-processing
involves deformation or heat treatment above beta-transus temperature followed by slow cooling [18]. Its mechanical properties can be altered by size of beta grain and colonies of alpha lamellae; thickness of $\alpha$-lamellae and nature of inter-lamellar beta phase [15]. Bimodal structure (duplex) primarily consists of equiaxed $\alpha$ grains and colonies of fine $\alpha$ lamellae inside beta grains of 10-20 $\mu$m in diameter [18]. It is superior in terms of yield and tensile strength, ductility and fatigue resistance [15]. The processing route involves homogenization in beta phase; deformation followed by recrystallization in $\alpha+\beta$ phase region; and final stress relieving and/or aging treatment, in which cooling rate is an important factor after first step [6]. Fully equiaxed microstructure contains globular $\alpha$-phase dispersed in a matrix of $\beta$-phase. It has improved balance of room temperature strength and ductility compared to fully lamellar structure and good fatigue properties [18]. Two processing methods can lead to formation of such structure. The first one follows the route of processing bi-modal microstructure until the recrystallization step. After that, a very slow cooling rate is applied so that only alpha grains will grow and lamellae feature will be hindered. Second route is to perform recrystallization at a very low annealing temperature. This allows equilibrium alpha phase to have high enough volume fraction to develop fully equiaxed structure from lamellar features after deformation procedure and smaller alpha grains will form, which can impact its mechanical properties [19]. Examples of the three distinct microstructure are displayed in Figure 2.5 and 2.6 [20].
Figure 2.5: Titanium alpha+beta alloy a) fully lamellar [20] b) bimodal structure [19].

Figure 2.6: Titanium alpha+beta alloy fully equiaxed microstructure [19].

Ti-6Al-4V alloy is the most popular commercial titanium alloy. It is also called workhorse alloy and is a type of dual phase α+β alloy with superplasticity related to plastic flow stress and critical strain level [21]. N. Poondla et al. studied the microstructure of Ti-6Al-4V alloy in fully annealed condition, which consists of a mixture of near equiaxed alpha and transformed beta phases as bimodal structure. Primary alpha grains are evenly
distributed in a matrix of transformed beta lamellae [15]. Bhattacharyya et al. investigated texture evolution in the alloy to understand the transformation mode of α to β phase and orientation relationship (OR) between the two phases after recrystallization, which can assist in the prediction of mechanical properties of the alloy. The Burgers orientation relationship between α and β phases is lost during deformation or recrystallization, indicating that beta phase develops from pre-existing beta grains instead of nucleation in alpha phase field. When the alloy is heat treated at 800 °C where recrystallization and grain growth take place, the original rolling texture is strengthened in alpha phase [22].

The following sections are focused on the study of alloy Ti-6Al-4V processing and behavior, since this alloy is one of the three high strength alloys evaluated in this project.

2.1.3 Processing of Titanium

The formability of titanium and its alloys can have large difference depending on the composition and microstructure. When they are formed, several issues need to be considered, such as limited workability depending on composition, galling, notch sensitivity, embrittlement caused by gas absorption and overheating, relatively poor shrinking ability and higher degree of springback than ferrous alloys with the same strength level. Therefore, most of the formed titanium parts are made by hot forming or cold forming followed by hot sizing [23].

In terms of alloy Ti-6Al-4V dual phase alloy, it has relatively poor room temperature formability compared to other titanium alloys. Solution treating and aging may improve the strength by 20 to 50% or more based on section size, cooling rate, composition and treatment temperature. As it has low amount of stabilizers, it requires to be quenched
rapidly to achieve noticeable strengthening effect due to poor hardenability. If the section thickness exceeds about 1 inch, watch quench will not be able to show significant hardening. As the amount of β stabilizers increases or thickness of the material decreases, more distortion can be experienced during watch quench.

In general, titanium alloys also suffer from Bauschinger effect in forming, which leads to a reduction of compressive strength after tensile straining that involves stress-strain asymmetry. When a 2% tensile strain is applied, the compressive yield strength of alloy Ti-6Al-4V may deteriorate to less than half of that for common solution-treated materials. Increasing forming temperature will gradually remove the adverse effect. However, it cannot exceed the aging temperature as heating or plastic deformation above that will cause overaging and reduction of mechanical properties for solution-treated Ti-6Al-4V [23].

Various forming processes can affect the microstructure and mechanical properties of the material. As press-brake forming is applied in hot induction bending process, span width of the die and punch radii may also influence formability of bend material. Other examples, such as β extrusion, α+β extrusion, rotary piercing, and flowforming, etc, are used for seamless tube production. Comparing the four processes for Ti-6Al-4V, flowformed material typically has the greatest tensile strength and fatigue strength, as well as good tensile ductility. It develops the finest microstructure with elongated α and intergranular β phase, as shown in Figure 2.7. α+β extruded tubes have similar microstructure but at a coarser scale. Both β-extruded and rotary-pierced tubes exhibit large prior β grain size with α phase present at grain boundaries [24].
On the other hand, fusion welding is a common joining method of titanium alloy and Tungsten inert gas (TIG) welding is the major welding technique. Welding of titanium often has the problem of atmospheric contamination. It may decrease ductility of weld metal and adjacent HAZ significantly that cracking occurs with low level of restraint. The maximum tolerance in weld metal is about 0.3% oxygen, 0.15% nitrogen and 150 ppm hydrogen. Thus, cleaning and degreasing of both parent metal and filler wire is essential prior to welding of titanium alloy and high purity shield gas should be used with an ideal dew point lower than -50 °C. Welding parts are also affected by oxygen and nitrogen absorption when temperature is above 520°C, which can be indicated by surface discoloration. Chill blocks are applied to reduce the heated area. Additional protection makes use of gas shroud in molten weld pool, a trailing gas shield following behind the welding torch, and gas purge at the back of weld. When titanium weld is attacked by contamination, micro-porosity is initially formed inside the dendrite arms during solidification process. They further develop into larger pores that are aligned along centerline of the weld [1].
Application of heat treatment before and after gas tungsten arc welding (GTAW) on Ti-6Al-4V also influences its corrosion behavior, which is affected by distribution of alloying elements and oxide formation in different phases. No effect is observed with use of pre-weld heat treatment on weld metal or in base metal with random, isolated and tangled β-phase particles in α phase matrix and has the best corrosion resistance. Solutionizing and aging post-weld treatment can create uniformity in corrosion potentials of different zones and improve corrosion behavior in fusion zone because of α precipitation from retained β and from β-phase in martensite [25].

Another conventional welding process of Ti alloy is electron beam welding (EBW). The use of vacuum protects molten weld pool from contamination. One weld bead is sufficient enough to form deep penetration joints because of high energy density and relatively low heat input. The effect of two different scanning patterns on EBW of Ti-6Al-4V was studied by S. Wang, et al. Coarser microstructure is formed with circular scanning and mechanical properties will decline compared to linear scanning. The weld metal consists of acicular α’ martensitic structure that greatly impacts mechanical properties of welded joints and base metal contains an equiaxed structure. Therefore, tensile strength of the two regions are almost equivalent, in which weld zone has higher microhardness compared to parent metal [26].

Thermo-mechanical processing (TMP) is another useful approach to optimize microstructure, morphology and size of different phases in titanium alloys. Concurrent deformation at high temperature and phase transformation (Figure 2.8), also known as dynamic strain induced phase transformation is one of the TMP being studied. Different phenomena, such as phase transformation, dynamic coarsening, grain globularization and
recrystallization will be activated. These mechanisms will refine the final microstructure and improve the strength and toughness of alloy material [27].

![Thermo-mechanical processing route](image)

**Figure 2.8:** Thermo-mechanical processing route for strain-enhanced phase transformation of Ti-6Al-4V alloy [27].

Overall, effect of processing on microstructure and properties of dual phase alloy is based mainly on a few basic concepts, including the relationship between cooling rate, slip length and α colony size. Alpha colony size is dependent on cooling rate from beta phase. Increasing its size may improve ductility, yield stress, crack nucleation resistance (HCF strength) and resistance to microcrack propagation. Duplex microstructure offers the most favorable mechanical properties. But a negative alloying element partitioning effect exists resulting in reduction of strength of lamellar feature and creep resistance of the alloy system. It can be prevented by limiting the volume fraction of primary alpha to about 15 vol.% [28].
2.1.4 Titanium properties and behavior

In order to optimize service properties and fatigue design of alpha+beta alloy, it is essential to understand deformation and cracking development. W.J. Evans stated that quasi-cleavage facets in plain geometries and at notches under fatigue loading are formed due to stress redistribution caused by inhomogeneity of slip systems and microstructure. As a result, weaker regions transfer the stress onto stronger features, such as areas in which the basal planes are normal to the loading direction. Development of facets indicates cyclic or time dependent fractures in dual phase titanium. Cold creep and premature failure, as well as low temperature dwell sensitivity will also be promoted [29].

High cycle fatigue (HCF) is the major failure mode in aerospace application. Fatigue crack growth (FCG) behavior and threshold is found to be related to temperature and stress ratio in forged Ti-6Al-4V alloy. At stress ratio greater than 0.7, FCG rate rises as temperature increases to 350°C in near-threshold range, but close to room temperature value in Paris-regime when da/dN is greater than 5x 10⁸m/cycle. J. Ding et al. proposes a FCG equation with respect to two stress intensity parameters that provides good predictions on the effect of temperature and stress ratio [30]. Microstructure also plays a crucial role in crack growth behavior. A study was conducted on dual phase lamellar Ti6242 alloy. The predominant fatigue crack growth behavior is transgranular colony fracture in direction of severely sheared slip bands. Crack tip slip process together with a reduction in loading frequency leads to a decrease in perpendicular-to-lamellae crack paths at lower temperature. The slip at crack tip also loses its density and homogeneity and minimize interactions between adjacent slip planes, resulting in a raise of crack growth rate. At
elevated temperatures, cracking tendency increases along a direction normal to \( \alpha/\beta \) interface [31].

As Ti-6Al-4V alloy is subject to heterogeneous deformation at elevated temperature, microstructural evolution takes place. There are a lot of studies reporting the effect of hot working [32-34]. J. Xiao et al. examined the behavior by conducting uniaxial tensile tests in range of 923-1023K with strain rate of 0.0005-0.05s\(^{-1}\). Deformed samples generally have comparable microstructure as the initial Ti-6Al-4V alloy consisting of equiaxed alpha grains. This is due to dynamic recovery and recrystallization processes occurring at elevated temperatures. Microhardness also decreases in the plane perpendicular to the tensile direction [35].

Similar work is conducted on isothermally compressed Ti-6Al-4V alloy. Different observations are obtained. Flow stress during hot compression increases with increasing strain rate but decreases as temperature increases [32]. A. Momeni et al. found that characteristic strain in dual-phase region is independent to Zenner-Hollomon parameter, which describes the combined effect of temperature and strain rate. But an opposite effect is shown in singe \( \beta \) phase [34]. Increasing deformation temperature will decrease volume fraction of primary \( \alpha \) phase due to \( \alpha \) to \( \beta \) transformation. More lamellar structure will be present as shown in Figure 2.9. On the other hand, strain rate induces effect on grain size and volume fraction of \( \alpha \) phase based on deformation temperature. The grain size of alpha is oscillatory below 1203K but decreases with rising strain rate above 1203K. An opposite trend is discovered in volume fraction of primary alpha. It becomes oscillatory above 1223K but decreases with increasing strain rate below that temperature. Volume fraction of secondary \( \alpha \) phase reduces as strain rate goes up from 0.1 to 10.0s\(^{-1}\). The third factor is
deformation degree that has significant effect on morphology of secondary α phase. As it increases from 20% to 50%, secondary α phase changes from lamellar colony to a mixture of lamellae and equiaxed grains, thus reducing the percentage of α lath structure. Amount of crystal defect also increases, which serves as heterogeneous nucleation sites. Presence of dislocations along shear plane plays a role in transforming the original microstructure to globular form [36].

![Graph](image)

**Figure 2.9:** Effect of deformation temperature on grain size and volume fraction of primary α phase of isothermally compressed Ti-6Al-4V alloy [36].

Besides hot compression, hot rolling process can induce changes in microstructure and tensile properties of Ti-6Al-4V alloy. Ultimate tensile strength, yield strength and elongation decrease from β-phase to dual phase region as rolling temperature increases. Post rolling heat treatment can be applied to break down martensite in β-rolled sample and complete globularization of α phase in two-phase region. Increasing heat treatment temperature results in lower strength for the sample rolled in α+β region but improves the strength for β-rolled Ti-6Al-4V [32]. High globularization efficiency (breakdown of
original lamellar microstructure) can be accomplished when prism and basal slip system work simultaneously in all grains. Changing the primary hot working direction at early stage will assist the breakdown by avoiding deformation direction in presence of hard regions [33].

2.2 High Strength Steel

2.2.1 Introduction to high strength steel

Steel is considered as the most successful and cost-effective of all engineering materials with a long history starting back in prehistoric time. More than a billion tones are consumed every year and it is being applied in every industry. It can possess a wide variety of microstructures and property combinations that are created by a broad range of compositions, solid-state transformations and processing [37]. The main component of steel is iron and its physical metallurgy is fundamentally based on Iron-carbon (Fe-C) binary phase diagram. Iron has three allotropic forms, i.e. \( \alpha \)-ferrite, \( \gamma \)-austenite, and \( \varepsilon \)-hexagonal close packed (HCP). Transformation between the allotropes, addition of carbon and substitutional solutes enrich the variety of microstructures [37]. A lot of different manufacturing techniques has been developed that are relatively cheap for mass production. The performance of steel material is dependent on the distribution, sizes, volume fractions and morphologies of various phases present in the system. A wide range of properties can be achieved with high flexibility. Yield strength may extent from 200 to above 1400 MPa, with fracture toughness increases up to 110 MPa \( \sqrt{m} \) [38].
2.2.2 Composition and Microstructure of high strength steels

Steels have a broad scope of classification systems. They can be based on the composition, manufacturing methods, finishing techniques, product form, deoxidation practice, microstructure at room temperature, required strength level, heat treatment applied and quality descriptors [38]. One type of steel can fall into several categories at the same time. For example, the two armor steels studied in this project are considered as quenched and tempered steel in the heat treatment classification system and advance high strength steel in the strength standard category. Armor steels have wide range of military specifications depending on the application and processing method. For example, wrought armor steel plate for thickness from 0.188 in. to 2.000 in. is classified into two classes. Class 1 refers to wrought armor plate which is liquid quenched and tempered. Wrought armor plate that is air quenched and tempered/autotempered belongs to class 2 [39].

Mechanisms of phase formation in steel can be divided into reconstructive transformation regime and displacive transformation regime. The former regime is in high temperature range in which austenite transform to ferrite crystal lattice by atom movement across the two-phase transformation interface. Reactions are controlled by slow rate substitutional element diffusion and the phases developed are ferrite and pearlite. The latter regime occurs in low temperature range. Austenite crystal structure transforms to ferrite by an invariant plane strain change of shape with a large shearing component and possibly diffusion of interstitial carbon. Reactions are in fast rate and the major phases are Widmanstatten ferrite, bainite and martensite [40].

The first phase formed upon cooling below the upper critical temperature (A$_3$) on prior austenite grain boundaries is allotriomorphic ferrite in form of polygonal grains or veins.
As temperature decreases, idiomorphic ferrite nucleate intragranularly on inclusions as shown in Figure 2.10 [40]. Pearlite transformation is a solid-state eutectoid reaction in between 550-720°C, in which one phase decomposes to two other solid phases. Fast cooling rate will lower the pearlite formation temperature as carbon diffuses from ferrite regions to cementite regions [38]. If it takes place at higher temperature, pearlite will be present as coarse nodules of alternate ferrite and cementite lamellae. When the temperature is reduced, the lamellae structure will be refined [40].

![Figure 2.10: Idiomorphic ferrite /Widmanstätten ferrite formation on large intragranular inclusions [40].](image)

Widmanstätten ferrite formation is a paraequilibrium mechanism as interstitial carbon diffuses rapidly into the remaining austenite during shear transformation. Primary Widmanstätten ferrite grow as colonies of coarse sideplates with aligned microphase from prior austenite grain boundaries [40]. Secondary Widmanstätten ferrite nucleates from allotriomorphic ferrite in the microstructure [37]. Another form of ferrite is called acicular ferrite. It forms inside the grains with the presence of inclusions and consists of a refined
interlocking structure with microphases [40]. Bainite typically forms in temperature range of about 250-550°C. It is also dependent on cooling rate, which is in between that of pearlite and martensite. There are two forms of bainite. Upper bainite at higher temperature contains fine ferrite planes in clusters, known as sheaves. Lower bainite at lower temperature consists of similar structure but with cementite precipitation inside the plates [37]. Martensite transformation is rapid and diffusionless. It exists as lath form with BCC structure at low carbon level and changes to plate form with BCC/BCT structure at high carbon content.

Additional of alloying elements by different proportions can modify the phase equilibria in the basic Fe-C binary system. They are generally divided into two groups: austenite or gamma stabilizers, which are elements that extend the existence range of austenite phase; and ferrite or alpha stabilizers, which reduce the austenite phase and expand the ferrite field [41]. γ- stabilizers can be further classified in two groups. One group suppresses the FCC to BCC transition temperature closer to room temperature before other phase formation. The other group expands the gamma phase field but the existence range is reduced due to compound formation [37]. Examples of γ stabilizers are nickel, manganese, cobalt, ruthenium, rhodium, palladium, osmium, iridium, platinum, carbon, nitrogen, copper, zinc and gold. α- stabilizers can also be sub-divided into two groups. The first group tends to restrict the FCC field into a gamma loop and separate from BCC field. Alpha and delta ferrite phases merge into one region. The second group lowers the delta ferrite transition temperature and increases the alpha ferrite temperature in order to limit the austenite field. Examples of alpha stabilizers are chromium, titanium, vanadium, molybdenum, silicon, aluminum, boron, niobium, zirconium, and tantalum [42]. The gamma stabilizers usually
have FCC structure similar to austenite, whereas alpha stabilizers are in BCC structure like ferrite [41].

Carbon is the major hardening element in all types of steels. Other alloying elements that can improve hardenability are managanese, chromium, molybdenum, nickel and boron [43]. It has moderate tendency to segregation and often lead to formation of carbides, which has significant effect on properties. Low carbon level can lead to excellent ductility, high toughness and good weldability, whereas high carbon content can offer excellent strength properties, hardness, good fatigue resistance, and wear resistance., but poor ductility and weldability [38]. Increasing carbon content can also decrease the martensite start temperature and lower the possibility of auto-tempering [37].

Addition of interstitial elements usually does not influence the formation of major phases. However, they have various interactive precipitation effects that impact the behavior of steels. Sulphur and phosphorus are the major impurities. They have high tendency to segregate in the interdendritic regions during solidification process, which lower the incipient melting temperature and increase hot cracking susceptibility. On the other hand, chromium, molybdenum, and niobium are main carbide formers with high affinity to carbon, which can lead to secondary hardening during martensite tempering [41]. Silicon can improve the stability of martensite by delaying its softening during tempering and reducing chemical activity of carbon [44]. A good combination of strength and toughness can then be obtained in steel.
2.2.3 Processing of high strength steels

The principle technique to improve hardenability of steel is quenching and tempering. The purpose of quenching is to form hard martensite structure and prevent austenite transformation to lamellar structure. The major two factors are carbon content and severity of quenching. Tempering is then conducted to develop structure with good toughness and ductility, which is affected by temperature and time [45]. Hollomon-Jaffe Parameter (HJP) was first developed by J.H Hollomon and L.D. Jaffe. It helped to study tempering of a group of fully quenched steels with difference in carbon content, that were tempered over a range of time at temperatures from room to just below A1. The aim was to establish a method of finding combination of temperature and tempering time that generate the same hardness with minimal amount of experimental work. A more systematic knowledge of the effects of carbon content in steel, temperature and time can be obtained [46].

When high strength steel is subjected to stamping, it is necessary to estimate the press capacity based on its high strain hardening characteristic. High binder pressure is applied to reduce buckling on the binder, which is the holding surfaces that regulate the sheet metal flow into the draw die cavity [47]. High press tonnage is also applied to maintain sheet metal flow in cavity of the die, which can be achieved by a double-action press or hydraulic press cushions. As yield strength of the steel is higher, springback is also higher depending on the deformation mode and tool geometry. Formability decreases at the same time with increasing strength of steel [48]. In general, hot stamping process is divided into two categories: direct and indirect hot stamping, as shown in Figure 2.11. Complete martensitic transformation takes place in high strength steel when subjected to thermal
cycle above austenitization before stamping. It can increase the tensile strength and hardness, but pre-coating is required to prevent oxide scale formation [49].

Bending process of maraging steels is studied by S.H. Khan, et al. It was found that micro-scale inhomogeneity of the material results in stress-induced phase transformation from retained austenite to martensite. A multiphase structure is developed, which is undesirable in terms of properties and use of cutting tools [50]. E. Hemmerich et al. examined the effect of tensile pre-straining and Bauschinger effect in bending of an aged and hot rolled steel, which comes from skin pass rolling, tension levelling or roller levelling before the cold rolling process. Unusual softening occurs when bending mild steel strip after pre-strained in tension, which results in defect formation [51]. Another free forming process used for high strength steel is induction bending of pipes. It is usually conducted above end rolling temperature of the steel to create a homogeneous structure. Composition
and geometry of the parent plate are the key to successful bending. Post bend heat treatment is necessary to retrieve original properties [52].

Welding is the most popular joining process for steel materials. Finding a proper combination of welding parameters and filler wire is essential to obtain the desired microstructure and properties. Cost and time of welding can be reduced. Application of post-weld heat treatment (PWHT) can also be minimized [53]. There are numerous studies on the effect of various welding processes and parameters on quality, service properties and cracking mechanism of steel weld.

C. Akca et al. studied the weldability of class 2 armor steels which are designed to resist explosions using gas tungsten arc welding (GTAW). Steel plates with thickness below 10 mm have been welded successfully with preheat/single pass condition. Heat affected zone (HAZ) has the greatest hardness due to carbide precipitation and formation of martensite [54]. Another HSLA steel plate (ASTM A 517 grade F) is examined, which is welded by gas metal arc welding (GMAW) and a single bead-in groove is deposited. Change in heat input can affect prior austenite grain size and interdendritic spacing, while preheat temperature can determine the coarsening of structure. A good combination of higher strength, lower hydrogen absorption and moderate toughness can be obtained when the steel materials are joined with higher preheat temperature and lower to moderate heat input or lower preheat temperature with intermediate heat input. This is due to the formation of acicular ferrite in the lath martensite weld microstructure [53]. As for quenched and tempered (Q&T) steels, high carbon content and hardness lower their weldability and increase cracking susceptibility, especially with the formation of hard HAZ. Welding consumables need to be selected that can match the strength properties of
parent steel material. PWHT is often necessary to restore properties using a temperature below the tempering temperature of base metal and with controlled heating and cooling rates [4].

Defects in welded joints may involve porosity, slag inclusions or cracks. Cracking is the most catastrophic effect, while the former two have acceptable limits. Types of cracking mainly include solidification cracking and hydrogen assisted cracking [55]. The cracking susceptibility of 10 mm thick high hardness armor (HHA) steel is evaluated using pulsed GMAW in the flat position with high restraints. Use of austenitic consumables can offer greater resistance to underbead cracking. Different electrodes are applied leading to formation of weld metal of different microstructures. The greatest drop of hardness is observed in austenitic stainless steel weld zone and no defect is observed. Hydrogen-induced cracking (HIC) is developed in the hardened HAZ of ferritic fusion zone. On the other hand, duplex fusion zone is prone to solidification cracking. Higher preheat temperature and heat input can reduce sensitivity of cracking [5]. It is also discovered that formation of delta ferrite can reduce weld cracking susceptibility. Weld solidification as primary austenite with eutectic ferrite or primary ferrite provides the best resistance to weld solidification cracking. This is because pinned and irregular $\delta$-$\gamma$ boundaries are formed providing a complex crack path and wetting by liquid films is reduced in the dual-phase boundaries with less surface energies which prevents decohesion of boundaries. Other reasons include higher impurity solubility in ferrite microstructure and partition of minor alloying elements [56].

Use of thermo-mechanical controlled processing (TMCP) wisely can achieve favorable properties. It mainly consists of two procedures: controlled rolling and accelerated cooling
process [57]. TMCP followed by water quenching is applied on a low carbon steel microalloyed with niobium and titanium. High strength and elongation are obtained due to changes in microstructure. Refined and dislocated lath martensite is the predominant structure with fine twins and strain-induced fine precipitates of microalloying carbides/carbon nitrides. Grain refinement takes place in controlled rolling as dynamic recrystallization of austenite occurs during TMCP. The effect of precipitation strengthening is dependent on solubility of precipitates in austenite phase and solubility at various temperatures [58].

Figure 2.12: Microstructural change and control by TMCP with varying temperatures and time [57].

K. Nishioka et al. made a review on the development of TMCP. Besides grain refinement, strength and toughness can be enhanced by the transformation to ferrite and
bainite. Accelerated cooling process can improve productivity compared to rolling in the austenite-ferrite region. Regarding the equipment and technologies of TMCP, different types of accelerated cooling system have been developed to maintain and enhance the uniformity and good control of cooling temperature, as well as plate flatness and productivity of process. Figure 2.12 shows the microstructure transformation in TMCP with a wide range of temperature and processing time [57].

2.2.4 High strength steel properties and behavior

In general, steels can be strengthened by the mechanisms of work hardening, solid solution strengthening by interstitials or substitutionals, grain refinement and dispersion strengthening, such as random dispersed and lamellar microstructures [37]. Understanding the strengthening mechanisms, fatigue properties and response to high temperature deformation of high strength steel is necessary to develop effective new forming or joining process with optimized microstructure and properties. F. Galvez et al. determines the fracture toughness in static and dynamic conditions of Armox 500T, indicating absence of strain rate effect. The studied material is in the same family of high strength armor steel of Armox 440 presented in this project as vehicle armor [59]. The same ultra-hard low alloy steel is studied with Armox 600T by T. Binar et al. in various research, which are often applied in areas with extreme temperature difference. The effect of surrounding temperature is evaluated on the change of failure mode and energy absorption. The failure mode of Armox 500T steel is transcrystalline ductile, while the mechanism in Armox 600T is transcrystalline quasi-cleavage. Assess of notched impact strength shows that significant reduction occurs in both armor steels below 0°C [60]. Based on the above two analysis,
both steels are more likely to undergo the degradation process that leads to a limit state in the material within the temperature range of -80 °C to 0 °C. Armox 500T also exhibits greater ability to absorb the supplied energy than Armox 600T, due to difference in alloying content [60].

P.K. Jena et al. studied the effect of heat treatment on mechanical properties of an ultra-high strength armor steel, which is first austenized at 910°C and then tempered over a range of temperatures. Increasing tempering temperature from 200 to 600°C leads to coarsening of martensitic laths. Both tensile strength and hardness gradually decreases with increasing tempering temperatures. Tempering at 300°C has the highest yield strength and lowest Charpy impact energy[61]. Similar study is performed by P. Zhong on a different type of ultra-high strength steel. Maximum tensile strength is obtained after tempering at 470°C due to the presence of a uniform and fine dispersion of irregular coherent zones or carbides. Better toughness is observed when tempering at 510°C due to reduced fraction of cementite, presence of reverted austenite and fine dispersion of carbides [62]. Figure 2.13 displays the effect of tempering temperature on service properties of the ultra-high strength steel.

Besides the temperature factor, deformation rate also affects the microstructural changes and in turn mechanical properties of high strength steel. A HSLA steel microalloyed with titanium and vanadium is examined under hot compression deformation. Competing mechanisms between dynamic recrystallization (DRX), recovery and grain growth determine the fine structure and prior austenitic grain size. Then, DRX becomes the major phenomenon when the material is above a critical deformation rate [63]. H. Farnoush et al. studied the response of a high-bainite dual phase (HBDP) when subject to
different amount of tensile strains over the temperature range of 150-450°C. When the deformation process is within intermediate temperature range, dynamic strain aging (DSA) is the dominant mechanism as interstitial impurities interact with mobile dislocations. DSA can be affected by strain rate, solute concentration and deformation temperature. The greatest rate of DSA is obtained when HBDP steel is pre-strained at 312°C with 5% strain. As pre-deformation increases, strength properties and fatigue limit also increase significantly [64].

Figure 2.13: Effect of tempering temperature on tensile properties, hardness, impact toughness and fracture toughness [62].

Same parameters are evaluated during multi-axis deformation (MADP) of a low carbon microalloyed steel, which allows application of strain in two alternate axes normal to each other. Reduction of deformation temperature and increase of number of alternating times
result in microstructure refinement and higher hardness. It is because of the formation of uniformly distributed ultra-fine acicular ferrite [65]. Change of cooling rates by different medium can alter the microstructure and service properties as well. Air-cooled material has lower strength than oil-cooled sample because of a coarser microstructure containing martensite, bainite and retained austenite [66].

2.3 Application of High Strength Alloys

2.3.1 Titanium application

There is increasing tendency to utilize titanium armor in U.S Army combat vehicles instead of the common rolled homogeneous armor (RHA) steel. Because it reduces vehicle weight and maintains excellent mechanical and ballistic properties. Some applications of Army systems include the commander’s hatch and top protection armor on M2 Bradley Fighting Vehicle, parts on M1A2 Abrams Main Battle Tank, XM777 155mm VSEL Ultralightweight Field Howitzer and applique armor in M113 armored personnel carrier [67]. Figure 2.14 displays the RHA components that are replaced by titanium in the Abrams Main Battle Tanks. In order to reduce the cost of titanium, single-melt electron beam cold hearth technology and various low-cost thermo-mechanical treatment are utilized for its production [68]. Besides removing the intermediate steps when converting the ingots into armor plate, plasma arc cold hearth melting (PAM) and electron beam cold hearth melting (EBM) can also minimize melt-related defects when manufacturing high quality titanium. Titanium welding processes in high deposition rate has been developed for combat-vehicle production, but they are usually more complex and costly than welding RHA due to complexity of gas shielding. Titanium-encapsulated ceramics are applied in armor to
enhance performance for breaking the projectile and catching the pieces. They are expensive but have high mass efficiencies [68].

Figure 2.14: Titanium replacement of components in M1 Abrams main battle tank [67].

2.3.2 High strength steel application

High strength steel also has widespread applications due to excellent properties. They reaches from energy industry, such as offshore and hydropower constructions to constructional steelworks, such as bridge-building and ship construction.

Steel armor is used extensively in defense application. Homogeneous wrought steel armor was applied for the first tank production in U.S. Army more than 80 years ago. Use of cast steel later became the dominant material for tank, such as the hull and turret, due to cost reduction and increase of production capability. Nowadays, rolled homogeneous armor (RHA) is the major material for heavy combat and recovery vehicles due to manufacture cost. Military specification for RHA is set in MIL-A-12560 [69]. Lightweight armor technology continues to evolve as it can work against armor-piercing projectiles
(AP) and have a wide variety of base material. Besides some low density metals, such as titanium as discussed in previous section, steel alloys are one of the main candidates due to ease of fabrication of armor parts in both military and commercial facility with adequate equipment and personnel. One of the excellent U.S. steel alloys against AP is MIL-A-46100 high hard steel armor with light gage (4.71mm-9.71mm nominal thickness) and a hardness range of 477-534 BHN [70]. It is utilized in structural application of light armored vehicle (LAV). This family of combat vehicles are initially developed for U.S Marine Corps. The armor material is selected in order to reduce vehicle weight and optimize ballistic properties against specified threat (7.62mm M1943 ball, AK47 round). However, its brittleness results in cracking in the welded steel plates of LAV. The processing of high hardness steel armor is typically quenched and tempered below the temper embrittlement range. The final microstructure consists of dispersion of epsilon carbide that can strengthen the material plate [71]. Another high-performing alloy for AP protection is MIL-A-46099C dual hardness armor (DHA), which is processed by roll-bonding a front plate (601-712 BHN) to a back plate (461-534 BHN) but is more challenging to produce [68]. Another high-performing alloy for AP protection is MIL-A-46099C dual hardness armor (DHA), which is processed by roll-bonding a front plate (601-712 BHN) to a back plate (461-534 BHN) but is more challenging to produce [70]. More armor plates are developed to achieve higher ballistic and mechanical properties, with reducing cost and weight.
2.4 Ballistic Properties

Ballistic performance is an important element of armor materials, which consists of three factors in terms of kinetic energy of a ballistic threat: the projectile, striking velocity and obliquity [68]. Department of Defense has defined military specifications of ballistic limit for a variety of metal alloy and ceramics in armor applications, different types of projectiles and testing procedures of determining the ballistic behavior.

2.4.1 Titanium alloy

Army Research Laboratory has conducted multiple research on the ballistic performance of titanium alloys according to their composition and possessing method. T. Jones et al. compares alloy Ti-6Al-4V and Russian alloy for monolithic armor with zero degree obliquity against 0.30-caliber AP M2 projectile. \( V_{50} \) ballistic limit of Ti-6Al-4V is 7% higher than that of Russian titanium alloy normalized for a range of thickness [72]. M. Burkins et al. investigates the performance of Ti-6Al-4V plate manufactured by electron beam single melting with three thicknesses against 20mm fragment simulating projectiles (FSP) ejected from high-explosive artillery rounds and 30mm armor piercing discarding sabot projectiles (APDS), which meets the ballistic requirements in MIL-DTL-46077F. The plate with 1.5 in. thickness has the maximum \( V_{50} \), followed by 1 in. and lastly 2.5 in. thickness [73].

Ballistic and blast properties of metal alloys are also related to dynamic deformation and formation of adiabatic shear bands (ASB). Penetration of projectile in metallic alloy target consists of four stages: 1) deformation; 2) ASB formation; 3) crack formation and propagation; and 4) chipping of target [74]. D-G. Lee et al. studied the effect of equiaxed
and bimodal microstructures of alloy Ti-6Al-4V on its ballistic property with the presence of $\alpha_2$ particles, which is caused by over-aging. Overall, aged bimodal microstructure has the best ballistic property, followed by unaged bimodal, aged and the unaged equiaxed microstructures. Decreasing formation of adiabatic shear bands can lead to improvement of ballistic properties. But aged equiaxed structure has higher possibility of forming ASB because of the distribution of plastic deformation energy. Aged microstructures also tend to have better mechanical properties and dynamic shear properties than the unaged samples that enhance the ballistic performance [75]. F. Martinez et al. investigated the phenomenon on 2.5cm thick Ti-6Al-4V targets impacted by 4340 steel projectiles. Plug formation is observed in between 633 -1027 m/s velocity. ASBs and cracks consisting of dynamic recrystallization structure (DRX) are formed in horizontal and vertical direction with respect to impact projectile axis. The DRX grain features offer solid-state flow mechanism up to fracture strain for the plug, which allow cracks to form during material separation and on free surface. Thickest ASBs are found when testing at highest impact velocity [76].

Another factor is heat treatment and thermo-mechanical processing applied on alloy Ti-6Al-4V. B.B. Singh et al. finds that titanium plates that are against 7.62 mm deformable lead projectiles possess a good combination of strength, ductility and ballistic properties when they are solution-treated below $\beta$-transus temperature and aging. Number of ASBs formed is higher in plates without heat treatment and $\beta$ solution treated followed by aged plates. This is due to lower energy absorption at higher strain rates and less resistance to adiabatic shear localization [77]. M. Burkins et al. studies the effect of rolling and annealing on extra low interstitial (ELI) Ti-6Al-4V against 20mm FSP and 12.7mm AP M2 projectiles. Processing above $\beta$-transus temperature decreases the $V_{50}$ ballistic limit.
But there is no significant effect of additional annealing after rolling on ballistic properties of ELI alloys [78].

Understanding the response of titanium materials can better facilitate their usage on vehicle and body armor against deformable projectiles. Various methods are developed to simulate and evaluate ballistic performance. B. Srivathsa et al. generates ballistic performance map (BPM) of different metallic alloys that are based on ballistic performance index (BPI). Effect of impact velocity, strain-hardening rate and yield strength are taken into account. They aim to determine the armor quality of metallic materials according to their static mechanical properties without conducting costly and time-consuming testing [79]. H. Meyer Jr. et al. characterizes ballistic behavior of alloy Ti-6Al-4V using Zerilli-Armstrong (ZA) strength model, which better represents the actual ballistic performance of titanium alloys in striking velocity range of 1200 to 2000 m/s [80].

2.4.2 High strength steel

Similar to titanium alloys, ballistic and blast performance of high strength steel is affected by the same group of factors. Resistance to ballistic threat of five different high strength steel plates (i.e. Weldox 500E, Weldox 700E, Hardox 400, Domex Protect 500 and Armox 560T) were examined by T. Borvik et al. against 7.62mm Ball ammunition and 7.62mm AP ammunition with different compositions. The former three are structural materials and the latter two are armor steel alloys, which are high in carbon, chromium, nickel and molybdenum. The yield stress of armor material is about three times the yield stress of structural steels. As the target yield stress increases, perforation resistance also
increases linearly for both projectiles. The strength property has stronger influence on small-arms ballistic performance compared to ductility [81].

In addition to the composition, difference in microstructure can impact ballistic properties. P.K. Jena et al. evaluated two low alloy high strength armor steel plates with the same composition and identically processed against deformable lead projectile at an impact velocity of 840m/s (Figure 2.15). Both plates consist of martensite and austenite features. One of them has higher volume fraction of retained austenite that results in lower ballistic resistance. This is due to non-uniform distribution of temperature, cooling rate in
the furnace and formation of oxide layer during heating. As retained austenite regions are softer than other phases, the strength to resist projectile is reduced and eventually leads to failure in those areas of the plate [82].

Ballistic properties of high strength steel are influenced by processing parameters. Deformation with high strain rate is dominated by high strain localization along adiabatic shear bands (ASB). A quenched-and-tempered low alloy steel is investigated by direct impact of a modified Split Hopkinson Pressure Bar (SHPB). Its response to plastic deformation suggests that the critical strain at which ASBs develop is dependent on microstructure and strain rate. Higher tempering temperature at high strain rate results in lower critical strain due to microstructure segregation, which means it is more susceptible to adiabatic shearing. The formed ASBs consist of severely deformed and aligned martensite laths with fine dispersion of carbides [83]. B. Mishra et al. conducts similar research on the effect of heat treatment on mechanical properties and ASB formation on high hardness armor steel plates of 25mm thickness against 7.62mm AP projectile. Same trend is exhibited where higher tempering temperature lowers ballistic impact resistance, due to coarsening of martensite laths, cementite precipitation and highly mobilized dislocations at higher temperature. However, formation of ASBs decreases with rising temperature. Decreasing plate thickness and addition of a 60mm Al-alloy backing leads to petal formation at the back of the plate and reduction of ASB-induced cracks. Inserting regularly spaced holes with 6mm diameter on the armor steel plate with a backing plate leads to non-symmetric damage area and prevents formation of ASBs and ASB-induced crack around the impact area [74].
When quench-and-tempered armor steel plates are welded, the joint may exhibit different ballistic and deformation behavior depending on the welding procedures, as reported by M. Balakrishnan et al. A soft buttering layer of austenitic stainless steel (SS) is deposited in between base metal and hardfaced layer in order to enhance ballistic properties. Weld center line (WCL) possesses the greatest ballistic resistance. Because of the use of SS capping, a fully austenitic weld metal is developed. Together with breakdown of primary carbides and a dispersion of highly distorted δ-ferrite in austenite matrix, more impact energy can be absorbed. This is due to the presence of hard BM and soft SS buttering layers at beveled edges. In addition, the hardfaced interlayer between SS root and capping weld layers contains chromium carbides in the eutectic austenitic matrix offering high hardness and improving ballistic resistance [84].

2.5 Strain-Induced Porosity and Void formation

Void formation is considered as crack initiation site when the material is subjected to deformation, which controls the failure process and defect evolution in metallic alloys. They are hard to detect by non-destructive testing and may deteriorate properties during processing and service [85]. The general mechanism of void formation has been studied extensively. J.R. Rice et al. observed the fracture behavior of ductile solids based on growth and coalescence of microvoids. They developed the first simple model to predict growth rate of a spherical void, which can be affected by the superposition of hydrostatic tension on a field of uniform plastic deformation and stress [86]. F.A. McClintock et al. developed one of the pioneer work on defining the criteria for ductile fracture and characterizing deformation of elliptical holes, which are known as voids, in a ductile material [87]. The
effect of hydrogen on void growth and clustering is later studied using a unit cell model of a general metallic alloy. Mechanism of void coalescence by plastic shear banding near the crack tip is determined by applying a model based on dislocation spacing and strength of obstacles. It is found that hydrogen will cause softening of plastic response and lattice dilation that impact the mechanical and deformation performance [88].

2.5.1 Titanium alloys

In titanium and its alloys, voids are known as strain-induced porosity (SIP). These voids are usually formed predominantly at grain boundaries and two-phase interfaces. Y. Rao et al. examined the room temperature tensile fracture response of a near-α titanium alloy IMI 685 consisting of mainly aluminum, zirconium, molybdenum, and silicon. It is observed that different microstructures result in void nucleation and growth at different regions. The microstructures are obtained by solution treatment in β-field followed by various cooling rates. Void nucleation and growth takes place at α-β interfaces in coarse-aligned α; along α platelets and α-α intersections in basketweave α structure; and at interfaces of α’ lath in martensite α microstructure [89]. Another study on SIP nucleation and coalescence mechanism of Ti-6Al-4V also concentrates on the effect of various transformed β features. In colony type microstructure, SIP initiation occurs at low strain at low subtransus temperatures, which increases rapidly as β-phase increases in Widmanstatten plates. The relationship between SIP kinetics and temperature leads to a dip in ductility at lower temperatures. In martensite α structure, void growth kinetics are within low-moderate range over the working temperatures. Therefore, no reduction of ductility is observed [85].
Another type of titanium alloy, ELI grade Ti-6Al-4V alloy consisting of transformed β develops two types of SIP (shear cracking and void nucleation) during α-β cogging, which is affected by the cogging speed and temperature. Cogging is an open-die forging process between two flat dies using slow speed machines. Large grained superplasticity (LGSP) of β causes void nucleation along prior-β grain boundaries due to incomplete grain boundary sliding. Intragranular voids are also formed near the β-transus temperature at strain rate lower than 0.1 s\(^{-1}\) due to slow diffusional flow. Number of voids increases rapidly in repeated cogging and resoaking cycles. Figure 2.17 is a microstructure mechanism map in terms of strain rate and temperature for the titanium alloy during cogging and resoaking [90].

![Microstructure of Ti-6Al-4V alloy](image)

Figure 2.16: Shearing of laths in ELI Ti-64 deformed 800°C/0.01s\(^{-1}\) [91].

T. Seshacharyulu et al. also explores the same Ti-6Al-4V alloy with a Widmanstatten structure during high temperature deformation. Both of the papers address the issue of LGSP and flow instabilities. Void nucleation and shear cracking (Figure 2.16) takes place...
at similar range of temperature and strain rate, while tensile ductility reduces [90, 91]. On the other hand, as temperature increases and strain rate decreases, flow localization bands, also known as adiabatic shear bands, are formed because of the adiabatic condition during deformation and low thermal conductivity of Ti-6Al-4V alloy, which affect the deformation and ballistic behavior [91].

Figure 2.17: Microstructural mechanism map of ELI alloy Ti-6Al-4V during cogging. [90]

When dual phase titanium alloy is subjected to compressive stress deformation, void formation takes place at the alpha-beta interfaces through a debonding process, which is mainly dependent on the stress state. SIPs are not found in regions under uniaxial compression, but in areas where stress state greater than -1. Decreasing volume fraction of
primary alpha phase may bring beneficial effect on SIP nucleation, but other factors, such as grain shape, bond strength and particle distribution also need to be considered [92].

2.5.2 High strength steel

There is very limited research that focus on the void nucleation phenomenon of high strength armor steel. Most study target on either the modelling of void nucleation mechanism of general metal alloys or ductile failure mechanisms of high strength steels [86, 93]. Two mechanisms are established: 1) Void coalescence phenomenon in which equiaxed voids develop to impingement under high triaxiality loading. 2) Void sheet mechanism in which microvoid shear localization leads to final failure under low triaxiality loading. E. Maire, et al. made use of X-ray tomography to illustrate the progression of void distribution, location and growth after tensile testing, as shown in Figure 2.19 [94]. C. McVeigh et al. studied the latter void nucleation process of an air melted high strength steel by a finite element cell model. The interaction between microvoid nucleation and decohesion of TiC secondary particles is simulated under pure shear-loading condition. As degree of deformation and shear strain rate increases, secondary carbides move towards adjacent particles and open the voids. Microvoids are elongated and forced to coalesce in the shear plane, as shown in Figure 2.18. Rise of interfacial strength of TiC can setback the accumulation of instability strain that initiates the debonding and coalescence process [93].
Figure 2.18: Modelling of the micro-void shear localization mechanism [93].

Figure 2.19: X-ray tomography scans of a DP600 steel: a) before tensile testing and b) before fracture [94].
2.6 Adiabatic Shear Bands

As discussed in previous sections, adiabatic shear bands (ASBs) are narrow zones of plastic shear strain within a homogeneous deformation region, affecting ballistic and deformation behavior of titanium and steels. They can be further classified into “transformed” or “deformed” shear bands, depending on how the deformation divide between two shear zones. Metallic alloys with low thermal diffusivity and low resistance to adiabatic shear localization favor the formation of “transformed” shear bands, which is the advance stage of adiabatic shear localization [95]. X. Teng et al models the development of ASBs to fracture in ABAQUS, which discovers hot temperature spots present in growing shear bands are the cracking initiation sites. When the metallic material is under compression, formation and propagation of cracks are caused by unloading stress waves [96]. Figure 2.20 displays ASB features in dual phase microstructure of a hot rolled titanium alloy.

Figure 2.20: Adiabatic shear bands in hot-rolled titanium alloy [95].
A.R. Shahan et al conducts a critical review on ASBs in titanium alloys, which are considered as the most susceptible group of material to ASB formation. Two phase α+β alloys are more prone to ASBs compared to CP titanium, which usually exist as shear zones with secondary fine bands. There are mainly two microstructures in the bands: highly deformed α grains with β phase grain boundary and without a main β grain boundary. Two types of voids are developed within the bands in circular shape and elliptical shape with sharp edges. Some of them are linked and further coalesce, which may bring detrimental effect on the mechanical properties [97].

In terms of high strength armor steel with Ni-Cr-Mn alloy system, it is discovered that microstructure transformation occurred within ASB is different from the outside regions of the shear bands, which contains a refined equiaxed structure of delta ferrite with iron carbides and thin martensite laths [98]. Temperature usually increases at least 200 °C when plastic strain is converted to thermal energy. Thus, temperature gradient is developed around ASBs, together with the presence of defects or inhomogeneity, ASBs are formed as there are changes in material properties. After that, a competition between workhardening and thermal softening takes place within the shear bands and eventually the latter one prevails leading to growth of ASBs [98, 99]. As ASBs are hard and brittle, this creates adverse effect on the steel material, heat treatment is applied to replace these white-etching bands. A study conducted by A.G. Odeshi et al. finds that annealing at 650 °C for 20 minutes can decrease hardness and also risk of cracking of ASBs due to carbides coarsening. Increasing the heat treatment time to 2 hours further reduces the hardness back to base metal level and the deformed bands become more ductile [100].
The above literature review provides a basic overview of the major properties, processing techniques, applications and deformation behavior of the two high strength alloys investigated in this project. It is essential to understand the relationship between microstructure, properties, and processing in order to develop optimized procedure of hot induction bending. However, there is lack of research that focuses on the effect of hot induction bending of high strength alloys in terms of armor applications. This work can help to establish a database of the parameters applied in hot induction bending and provide testing procedures for studying the response of other high strength alloys to high temperature straining.
Chapter 3: Objective

The major objectives of this research project are: 1) Development of testing procedure for evaluation of the response of high strength alloys to hot induction bending. 2) Development of optimal process control windows of hot induction bending for particular armor alloys. In order to achieve the purpose, various effects of hot induction bending on materials’ microstructure and properties are evaluated. Three types of materials are studied, including Ti-6Al-4V alloy, Armoq 440 and ARL XXX high strength armor steels. The specific goals of this work include:

1. Evaluation of the effect of temperature, strain, and extension rate experienced by high strength alloys during hot induction bending through hot ductility testing and high temperature straining testing using the Gleeble™.

2. Determination of defect-free conditions and development of optimal range of parameters for hot bending process of high strength alloys. Formation of voids/strain-induced porosity is the major indication of sample failure.

3. Study of the effect of induction bending on room temperature service properties of high strength alloys by straining samples at high temperature followed by room temperature tensile testing.

4. Evaluation of the response to tempering in Armoq and ARL armor steels.

5. Metallurgical characterization and phase transformation analysis on ARL XXX steel welds.
Chapter 4 : Materials and Procedures

4.1 Materials

Titanium alloys and high strength steels were the two main types of armor material being studied in this research project.

4.1.1 Titanium 6-4

There were 4 different grades of Titanium alloy Ti-6Al-4V, including grade 5 plate (heat # MJL272), two grade 23 plates (heat # MJL 271 and MJL 03AP) and MIL-STD-46077 class 2 plate (heat # MJL 268), which were from Titanium Industries, Inc. All Ti-6Al-4V alloys were α/β processed. The chemical composition and mechanical properties at room temperature of these materials are shown in Table 4.1 and 4.2.

Table 4.1: Ti-6Al-4V alloys material composition

<table>
<thead>
<tr>
<th>Material</th>
<th>Mil Class/ Form</th>
<th>Heat # (MJL)</th>
<th>Al</th>
<th>V</th>
<th>O</th>
<th>Fe</th>
<th>C</th>
<th>H</th>
<th>N</th>
<th>Ti</th>
</tr>
</thead>
<tbody>
<tr>
<td>ASTM B265 grade 5</td>
<td>2/Plate</td>
<td>N15K</td>
<td>6.29</td>
<td>4</td>
<td>0.17</td>
<td>0.17</td>
<td>0.011</td>
<td>0.0055</td>
<td>0.0056</td>
<td>Bal.</td>
</tr>
<tr>
<td>ASTM B348 grade 23</td>
<td>1/Plate</td>
<td>271</td>
<td>6.03</td>
<td>4</td>
<td>0.11</td>
<td>0.13</td>
<td>0.01</td>
<td>0.0077</td>
<td>0.003</td>
<td>Bal.</td>
</tr>
<tr>
<td>ASTM B265 grade 23</td>
<td>1/Plate</td>
<td>03AP</td>
<td>6.14</td>
<td>4</td>
<td>0.09</td>
<td>0.18</td>
<td>0.019</td>
<td>0.0044</td>
<td>0.009</td>
<td>Bal.</td>
</tr>
<tr>
<td>MIL STD 46077</td>
<td>2/Plate</td>
<td>268</td>
<td>6.3</td>
<td>3.8</td>
<td>0.2</td>
<td>0.15</td>
<td>0.016</td>
<td>0.006</td>
<td>0.013</td>
<td>Bal.</td>
</tr>
</tbody>
</table>
Table 4.2: Ti-6Al-4V alloys mechanical properties at room temperature

<table>
<thead>
<tr>
<th>Material</th>
<th>Military Class/Form</th>
<th>Heat # (MJL)</th>
<th>UTS (ksi)</th>
<th>0.2% proof YS (ksi)</th>
<th>Elongation (%)</th>
<th>Reduction-in-area (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>ASTM B265 grade 5</td>
<td>2/Plate</td>
<td>N15K</td>
<td>148.8</td>
<td>137</td>
<td>18</td>
<td>36</td>
</tr>
<tr>
<td>ASTM B348 grade 23</td>
<td>1/Plate</td>
<td>271</td>
<td>136</td>
<td>127</td>
<td>16</td>
<td>39</td>
</tr>
<tr>
<td>ASTM B265 grade 23</td>
<td>1/Plate</td>
<td>03AP</td>
<td>143.5</td>
<td>134.2</td>
<td>20.3</td>
<td>41.3</td>
</tr>
<tr>
<td>MIL-STD-46077</td>
<td>2/Plate</td>
<td>268</td>
<td>147</td>
<td>141</td>
<td>20</td>
<td>22</td>
</tr>
</tbody>
</table>

4.1.2 Armor Steels

Two types of armor steel plates were examined in project. They were identified as Armox 440 (heat # MJL 342) and ARL XXX (heat # MJL 461), which were provided by our sponsor AEM. The materials composition, as well as mechanical properties at room temperature are shown in Table 4.3 and 4.4 below.

Table 4.3: Armor steel material composition

<table>
<thead>
<tr>
<th>Material</th>
<th>Form</th>
<th>Heat #</th>
<th>C</th>
<th>Mo</th>
<th>Si</th>
<th>Cr</th>
<th>Mn</th>
<th>Ni</th>
<th>S</th>
<th>P</th>
</tr>
</thead>
<tbody>
<tr>
<td>Armox 440</td>
<td>Plate</td>
<td>MJL 342</td>
<td>0.19</td>
<td>0.58</td>
<td>0.20</td>
<td>0.56</td>
<td>0.80</td>
<td>1.85</td>
<td>0.002</td>
<td>0.008</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>B</td>
<td>Cu</td>
<td>V</td>
<td>Ti</td>
<td>Al</td>
<td>Pb</td>
<td>Sn</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>0.0023</td>
<td>Bal</td>
<td>0.014</td>
<td>0.023</td>
<td>0.006</td>
<td>0.064</td>
<td>0.007</td>
</tr>
<tr>
<td>ARL XXX</td>
<td>Plate</td>
<td>MJL 461</td>
<td>0.22</td>
<td>0.29</td>
<td>0.40</td>
<td>1.74</td>
<td>3.64</td>
<td>0.001</td>
<td>0.013</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Al</td>
<td>Cu</td>
<td>Nb</td>
<td>V</td>
<td>Ti</td>
<td>N</td>
<td>Co</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>0.05</td>
<td>0.06</td>
<td>0.01</td>
<td>0.01</td>
<td>0.01</td>
<td>0.006</td>
<td>0.01</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>B</td>
<td>Fe</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
4.2 Temperature Distribution Testing

Temperature distribution tests were performed in order to determine temperature uniformity of samples when subjected to heating in the Gleeble™. Two types of materials (i.e. Ti-6Al-4V grade 23 MJL03AP and Armox 440 MJL342) were examined using the Gleeble™ 3800 thermo-mechanical simulator at various temperatures. Samples of three different dimensions were machined from each type of material for testing as shown in Figure 4.1, 4.2 and 4.3.

**Table 4.4: Armor steels mechanical properties at room temperature**

<table>
<thead>
<tr>
<th>Material</th>
<th>Form</th>
<th>Heat #</th>
<th>UTS (ksi)</th>
<th>0.2% proof YS (ksi)</th>
<th>Elongation (%)</th>
<th>Hardness (HRc)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Armox 440</td>
<td>Plate</td>
<td>MJL 342</td>
<td>213</td>
<td>172</td>
<td>12</td>
<td>47.6</td>
</tr>
<tr>
<td>ARL XXX</td>
<td>Plate</td>
<td>MJL 461</td>
<td>229</td>
<td>173</td>
<td>14</td>
<td>46</td>
</tr>
</tbody>
</table>

Figure 4.1: Gleeble™ ¼” hot ductility test sample dimension
Figure 4.2: Gleeble™ 3/8” tensile test sample dimension.

Figure 4.3: Gleeble™ 3/8” rod sample dimension.

Figure 4.4: Gleeble™ ¼” dumbbell sample.
Machined samples were first prepared by polishing with 320-grit SiC abrasive grinding paper and then washing with alcohol. After that, thermocouples were welded to the center point of sample using DSI thermocouple welder machine. Type-K thermocouples were applied in all testing. The second thermocouples were welded 1/6” away at the right of center point of sample. Third thermocouples welded on sample were located at 1/3” away at the left of center point, followed by the fourth one (optional), which was welded ½” away from the center on the right. All thermocouples should be aligned at the same level.

Figure 4.5: Gleeble™ set-up inside test chamber during temperature distribution test.

After placing thermocouples, the sample was gripped at two ends by stainless steel jaws and placed inside the Gleeble™ test chamber. Thermocouples at different positions were connected to four temperature sensors and the one welded at center must be connected to
first sensor, as shown in Figure 4.5. The atmosphere inside test chamber was under low
vacuum state, which was below 8.1x10^-2 torr.

Samples were heated by resistance heating under a closed loop process control at a rate
of 10 °C/sec. Two temperatures at 430 °C and 650 °C were tested for titanium samples for
each of the three sample dimensions, based on β- transus temperature. For armor steel
samples, they were tested at temperatures 650 °C and 880 °C, which are below A₁ and
above A₃ temperature respectively. After reaching desired temperature, samples were held
at the same temperature for 10 minutes in order to stabilize. The same sample was reused
for testing different temperatures and a total of 12 tests were conducted.

4.3 Hot Ductility Testing

Hot ductility tests of Ti-6Al-4V alloy and Armox 440 were performed in the Gleeble™
3800 Thermo-mechanical simulator by Nicholas Kullman. In the current thesis, these
samples were characterized by cross-section optical metallography and SEM fractography.
Eighteen additional tests were conducted on ARL XXX high strength steel in current work.
Cylindrical samples were machined as shown in figure 4.1. Dumbbell samples, as shown
in Figure 4.4, were used for Gleeble testing of high strength steel below 500 °C. The
preparation process was very similar to that of temperature distribution test.

Samples were first polished using 320- grit SiC abrasive grinding paper followed by
rinsing with alcohol. Type-K thermocouples were welded to the center of sample. Digital
caliper was used to record diameter of sample at the center, so that reduction in cross-
section area could be measured after testing.
The sample was gripped by copper jaws at both ends in previous testing and it was replaced by stainless steel jaws in current work. The sample was then placed into the Gleeble™ test chamber under low-vacuum atmosphere as shown in Figure 4.6. Samples were then heated at a rate of 10 °C/sec to various temperatures and held for 6 minutes in order to stabilize the temperature. The testing temperature range for titanium samples was from 427°C to 982 °C plus one sample at room temperature. For Armox 440, the temperature range was from 300 °C to 1080 °C and ARL XXX was tested in range of 400 °C to 800 °C, based on A1 and A3 temperatures. Afterwards, titanium samples were strained at extension rates of 1 mm/sec and 10 mm/sec to 40 mm stroke until the sample failed. Two extension rates were chosen for comparison because the press brake ram travel speed was 8.9 mm/sec during induction bending process. Armox 440 samples were pulled at a rate of 10 mm/sec. ARL XXX steels were tested at extension rate of 1 mm/sec and 10 mm/sec. Testing conditions for hot ductility test were listed in Table 4.5.

Figure 4.6: Gleeble™ set-up during hot ductility test.
Table 4.5: Testing conditions for hot ductility testing

<table>
<thead>
<tr>
<th>Materials</th>
<th>Extension</th>
<th>Testing Temperatures (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ti-6Al-4V</td>
<td>1 &amp; 10</td>
<td>38 427 538 649 760 871 982 982</td>
</tr>
<tr>
<td>Armox 440</td>
<td>10</td>
<td>800 840 880 920 960 1000 1040 1080</td>
</tr>
<tr>
<td>ARL XXX</td>
<td>1 &amp; 10</td>
<td>400 450 500 550 600 650 700 750 800</td>
</tr>
</tbody>
</table>

After testing, the final diameter of sample where fracture occurred was measured using digital calipers. Reduction in cross section area was calculated and ductility of sample was determined.

4.4 High Temperature Strain-Controlled Testing

4.4.1 Gleeble™ Tests

High temperature strain-controlled tests were also performed in the Gleeble™ 3800 thermo-mechanical simulator. Samples used were machined as shown in figure 4.1. The procedures were similar to that of hot ductility test, except that the changing variable is strokes and extensometer was inserted for recording strain data. Testing materials were Ti-6Al-4V grade 23 plate, Armox 440 and ARL XXX.

Samples were first polished using 320- grit SiC abrasive grinding paper followed by rinsing with alcohol. Type-K thermocouples were welded to the center of sample using the thermocouple welder. Two thermocouple wires (about 1 cm long) were then welded on each side about 5 mm away from center thermocouples. These wires were used to fix the placement of high temperature extensometer and avoid the measuring rods from moving towards each other. After that, digital micrometer was applied to measure diameter of
sample at the center and digital caliper was used to record original length. The sample was gripped by stainless steel jaws and inserted into the Gleeble™ test chamber with low-vacuum atmosphere. High temperature extensometer was attached onto the sample using ceramic fiber cords as shown in Figure 4.7.

Samples were heated at a rate of 10 C°/sec to various temperatures and held for 6 minutes. The sample was subjected to 5-10 kg constant force within the 6 min. temperature holding period. High temperature tensile test at 1 mm/s extension was first conducted. The amount of strain corresponding to yield strength (YS), ultimate tensile strength (UTS), between YS and UTS and above UTS were obtained from the plot of engineering stress versus strain at specific temperature. After that, samples were stained to various strain levels at 1 mm/s extension rate until they reached yield strength (YS), between YS and UTS, ultimate tensile strength (UTS), and above UTS of the material respectively. Table 4.6 shows testing parameters for Gleeble™ testing at various strains.

Figure 4.7: Gleeble™ set-up inside test chamber during hot ductility test and high temperature strain-controlled test.
After testing, the final cross section area of sample was measured using digital calipers and reduction in cross sectional area was calculated. The final length was also measured to determine total engineering strain.

### 4.4.2 DIC Measurements at Oak Ridge National Laboratory

True stress-strain data for Armox 440 was obtained at Oak Ridge National Laboratory (ORNL) using Gleeble\textsuperscript{TM} 3500 thermo-mechanical simulator and Digital Image Correlation system (DIC). High temperature strain controlled tests were conducted by Dr. Dongxiao Qiao using the same procedure in section 4.1.1. A total of four tests were performed.
recorded. The set-up was shown in Figure 4.8 and the test conditions were listed in Table 4.7. Temperature was controlled at the center of sample. Two additional thermocouples were attached to monitor temperature distribution at 5mm and 15mm away from center.

Figure 4.8: Gleeble™ set-up at ORNL with DIC camera system.

Lagrange strain data was recorded from DIC system with respect to distance from center, as well as versus time. True stress-strain curve at center of sample and 5 mm from center was plotted by synchronizing data from Gleeble™ files and DIC data file manually. True strain data was calculated using equations (1). True stress data was converted from engineering stress data of Gleeble™ using equation (2).

\[
\varepsilon_T = \sqrt{1 - 2 \varepsilon_L} - 1 \quad (1)
\]

\[
\tau = \sigma * (1 + \varepsilon_e) \quad (2)
\]
Samples were sent back to Ohio State for metallographic analysis. Void locations were identified using optical microscope. Correlation between true strain and void formation was analyzed. Engineering stress-strain data obtained from Gleeble testing at Ohio State were also converted to true stress-strain values until ultimate tensile strength only using Law of Ramberg-Osgood modified by Raumussen based on the following equations (3) and (4):

\[
\varepsilon = \frac{\sigma}{E} + 0.002 \left(\frac{\sigma}{\sigma_{0.2}}\right)^n \quad \text{for } \sigma \leq \sigma_{0.2} \quad (3)
\]

\[
\varepsilon = \frac{\sigma - \sigma_{0.2}}{E_{0.2}} + \varepsilon_u \left(\frac{\sigma - \sigma_{0.2}}{\sigma_u - \sigma_{0.2}}\right)^m + \varepsilon_{0.2} \quad \text{for } \sigma \geq \sigma_{0.2} \quad (4)
\]

where \(\varepsilon\) is true strain, \(\sigma\) is true stress, \(E\) is Young’s modulus, \(\sigma_{0.2}\) and \(\varepsilon_{0.2}\) are 0.2% yield stress and strain, \(n\) is a dimensionless constant that is calculated using 0.01% and 0.2% yield stress. \(E_{0.2}\) is Young’s modulus at 0.2% proof stress, \(\varepsilon_u\) and \(\sigma_u\) are ultimate tensile strain and strength, \(m\) is a constant calculated by \(\sigma_{0.2}\) and \(\sigma_u\). The fitted curves were then compared with the true stress-strain curves obtained from DIC measurements of Oak Ridge National Laboratory.

Table 4.7: Test conditions of Armox 440 high temperature strain-controlled test at ORNL

<table>
<thead>
<tr>
<th>Sample #</th>
<th>Temperature (°C)</th>
<th>Strokes applied (mm)</th>
<th>Sample condition</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>800</td>
<td>18.5</td>
<td>Failure</td>
</tr>
<tr>
<td>3</td>
<td>650</td>
<td>10.5</td>
<td>Failure</td>
</tr>
<tr>
<td>6</td>
<td>650</td>
<td>3.5</td>
<td>Necking</td>
</tr>
<tr>
<td>8</td>
<td>800</td>
<td>8.5</td>
<td>Necking</td>
</tr>
</tbody>
</table>
4.5 Metallurgical Characterization

4.5.1 Metallographic Sample Preparation

Metallographic samples were prepared according to the following procedures. First, samples were sectioned into shorter length using Allied TechCut 5 or Leco CM-15 cut-off machine from larger samples. Then, sectioned materials were mounted as 1.25 inch or 1.5 inch diameter samples using the Leco PR-32 mounting press machine and were held fix in position using sample holding clips if they were cylindrical in shape. Samples that were characterized by light optical microscope were mounted using Leco Bakelite thermoset powder and Buehler ConductoMet conductive mounting resin for analysis under scanning electron microscope (SEM). After that, the top surface layer of mounted samples was sectioned using Allied TechCut 5 so that cross section of samples could be revealed.

The initial polishing steps were the same for both titanium and steel samples. Leco Spectrum System 100 polisher was used with SiC abrasive grinding papers, which started from 180, 240, 320, 400, 600 and down to 800 grit. The samples were first polished in one predominant direction until all scratches appeared to be in same direction, then in direction perpendicular to previous one to eliminate all scratches made from pervious step. Water and ethanol were used to remove debris in between each stage. After polishing with 800 grit SiC paper, samples were cleaned with ethanol thoroughly by putting them in an ultrasonic bath for about 5 minutes. For steel samples, no running water should be used during polishing as corrosion would take place.

At the final stage, Ti-6Al-4V samples were polished automatically on the Buehler Vibromet 2 using a 0.05 micron non-crystallizing colloidal silica suspension for about 16 hours (overnight) until samples were shiny and smooth. Then, samples were rinsed with
warm water for at least one minute and cleaned with detergent before placing in the ultrasonic ethanol bath for another 5 minutes.

Steel samples were polished by hands continuously with 9, 6, 3, and 1 micron diamond paste on polishing cloth with oil lubricant after 800 grit SiC paper. They were cleaned with water between each polishing stage. After that, samples were polished with 0.5 micron colloidal silica and then placed on Buehler Vibromet 2 for about 3-4 hours in order to remove all scratches and create completely flat surfaces.

After the polishing procedures, samples were subjected to etching so that microstructure could be observed. For titanium samples, they were etched using Kroll’s Reagent (10 mL HF, 5 mL HNO3 and 85 mL H2O). Sample surface was immersed in etchant for 5-10 seconds until the microstructure stood out. They were rinsed with detergent and water immediately. After that, they were placed in ultrasonic ethanol bath for 5 minutes and dried with hot air.

Steel samples were etched with 2% Nital solution (2 mL concentrated Nitric acid and 98 mL ethanol) or Le Pera’s solution (50 mL 1% sodium metabisulfite and 50 mL 4% piric acid). Samples were swabbed with etching solution using cottonball until microstructure stood out. They were then cleaned with detergent and water.

4.5.2 Light Optical Microscopy (LOM)

Light optical microscopy was performed using an Olympus GX51 microscope, which was used for taking low magnification optical images and Differential Interference Contrast (DIC) images of samples that required metallurgical characterization. The
magnification range was between x12.5 and the highest was x1000. Images were captured using a digital image acquisition system which connected the microscope to computer. Necessary measurements on optical images could also be made using the image software.

4.5.3 Scanning Electron Microscopy (SEM)

Scanning electron microscopy was performed using mainly two scanning electron microscopes at the Campus Electron Optics Facility (CEOF) of the Materials Science and Engineering Department at Ohio State University. Quanta 200 general purpose tungsten source SEM was mainly applicable for analysis on fracture surface and performing Energy Dispersive Spectroscopy (EDS). The system was operated in a high vacuum environment. Secondary electron (SE) detector and Backscattered Electron detector (BSE) were available for taking high resolution and well defined images and detecting contrast between regions with different chemical composition respectively. For higher resolution images at higher magnification, images were taken using Philips XL-30 ESEM microscopes.

4.5.4 Fractography

Metallurgical characterization of sample fracture surface was performed using Quanta 200 general-purpose tungsten source SEM at CEOF. Only secondary electron detector was used for capturing images. Samples were rinsed in ethanol ultrasonic bath for about 5 minutes before analysis. They were attached to the sample holder using conductive graphite paint.
4.6 Mechanical Testing

In order to study effect of strain during induction bending on mechanical properties, high temperature tensile tests and high temperature straining followed by room temperature tensile tests were conducted. Part of the tests on Ti6-4 alloys and Armox 440 steel took place at Miami Valley Materials Testing Center (MVMTC) and National Institute of Standards and Technology (NIST) as shown in Figure 4.11. All tests on ARL XXX were conducted at NIST with assistance of Dr. Jeff Sowards.

Some tests were also done at the Materials Science and Engineering department at Ohio State. The set up was shown in Figure 4.10. Cylindrical tensile samples (Figure 4.9) were placed in high temperature extension rods and high temperature extensometer was inserted against the reduced section of sample with ceramic fiber cord. Samples were then connected to the Instron Model 1322 Tensile test frame. A MTS 653 Furnace was placed around the reduced section of sample and each was heated to desired temperature and held for about 10 minutes until the temperature was stabilized. They were then strained at rate of 1 mm/s until reaching pre-determined amount of strain, which was controlled by extensometer and determined by high temperature tensile test. The straining rate of tensile test frame was selected to replicate the actual bending process. When high temperature straining was completed, samples were cooled down to room temperature and room temperature tensile test was performed according to ASTM E8 standard. Summary of mechanical testing parameters are listed in table 4.8 and 4.9.

At NIST, similar experimental procedures were performed using MTS uniaxial load frame. Samples were heated in ATS Series 3210 furnace chamber first at a rate of 10 °C/s up to 80 °C, then at a rate of 20 °C/s to 50 °C below the testing temperature, and finally at
a rate of 5 °C/s to testing temperature in order to avoid temperature overshoot. After that, sample was held at elevated temperature for 20 minutes so that uniform temperature could be obtained along the reduced section. For samples tested at 750 °C or above, temperature holding time was reduced to 3 minutes as oxidation occurred rapidly, which would lead to slippage of extensometer. Two sets of thermocouples were welded to the sample for furnace control and data acquisition. Testing samples were under constant load of 5 lbf before straining to prevent unwanted compression or pulling. After straining at high temperature, samples were under constant load of 30 lbf and cooled down to room temperature with a fan, followed by standard room temperature tensile testing. The set up is shown in Figure 4.11. Test commands were programmed into computer software, which is shown in Figure 4.12. Three ARL XXX samples tested at NIST were then subjected to cross-section optical metallography and SEM fractography at OSU. These samples were 450 °C strained at 7%, 600 °C strained at 11% and 750 °C strained at 11%.

![Diagram](image_url)

Figure 4.9: High temperature straining/room temperature tensile test sample dimensions.
Figures 4.10: Tensile test set up at the Materials Science & Engineering Department facility of OSU.

Figures 4.11: Left image showed the sample within furnace chamber attached to high temperature extensometer. Right image showed entire set up at NIST.
Table 4.8: Testing parameters of high temperature straining/room temperature tensile test

<table>
<thead>
<tr>
<th>Material</th>
<th>Temperature (°C) &amp; Strain %</th>
<th>Strain rate (mm/s) at HT</th>
<th>Gauge Length (mm)</th>
<th>Note</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ti6-4 Gr 5</td>
<td>250 / 5</td>
<td>1 mm/s</td>
<td>25.4</td>
<td>MSE</td>
</tr>
<tr>
<td>Ti6-4 Gr 5</td>
<td>430 / 10</td>
<td></td>
<td>25.4</td>
<td>MSE</td>
</tr>
<tr>
<td>Ti6-4 Cls 2</td>
<td>430 / 11</td>
<td></td>
<td>30</td>
<td>NIST</td>
</tr>
<tr>
<td>Ti6-4 Gr 5</td>
<td>650 / 10</td>
<td></td>
<td>25.4</td>
<td>MSE</td>
</tr>
<tr>
<td>Armox 440</td>
<td>25 (RT) / 4</td>
<td></td>
<td>30</td>
<td>MVMT</td>
</tr>
<tr>
<td></td>
<td>25 (RT) / 5</td>
<td></td>
<td>30</td>
<td>MVMT</td>
</tr>
<tr>
<td></td>
<td>650 / 11</td>
<td></td>
<td>30</td>
<td>NIST</td>
</tr>
<tr>
<td></td>
<td>880 / 11</td>
<td></td>
<td>30</td>
<td>NIST</td>
</tr>
<tr>
<td></td>
<td>500/ 11 #1</td>
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<td>NIST</td>
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<tr>
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<td>600/ 11 #3</td>
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</tr>
<tr>
<td>ARL XXX</td>
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<td></td>
</tr>
<tr>
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<td>450/7 #2</td>
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</tr>
<tr>
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<td>600/11 #1</td>
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<tr>
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<td></td>
<td>750/11 #2</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Figure 4.12: Software program controlling tensile test commands and data recording at NIST.
Table 4.9: Testing parameters of high temperature tensile test

<table>
<thead>
<tr>
<th>Material</th>
<th>Temperature (°C)</th>
<th>Strain rate at HT (mm/s)</th>
<th>Gauge Length (mm)</th>
<th>Note</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ti6-4 Gr 23</td>
<td>25 (RT)</td>
<td>1</td>
<td>25.4</td>
<td>MSE</td>
</tr>
<tr>
<td>Ti6-4 Gr 23</td>
<td>25 (RT)</td>
<td>0.45 mm/min</td>
<td>30</td>
<td>MSE</td>
</tr>
<tr>
<td>Ti6-4 Gr 5</td>
<td>250</td>
<td>1</td>
<td>25.4</td>
<td>MSE</td>
</tr>
<tr>
<td>Ti6-4 Gr 5</td>
<td>430</td>
<td>1</td>
<td>25.4</td>
<td>MSE</td>
</tr>
<tr>
<td>Ti6-4 Gr 23</td>
<td>430</td>
<td>0.45 mm/min</td>
<td>25.4</td>
<td>MSE</td>
</tr>
<tr>
<td>Ti6-4 Cls 2</td>
<td>430</td>
<td>1</td>
<td>30</td>
<td>NIST</td>
</tr>
<tr>
<td>Ti6-4 Gr 23</td>
<td>650</td>
<td>1</td>
<td>25.4</td>
<td>MSE</td>
</tr>
<tr>
<td>Armox 440</td>
<td>25 (RT)</td>
<td>0.45 mm/min</td>
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<td>MSE</td>
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<td>1</td>
<td>25.4</td>
<td>NIST</td>
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<td>500 #2</td>
<td>1</td>
<td>25.4</td>
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</tr>
<tr>
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<tr>
<td></td>
<td>650 #2</td>
<td>1</td>
<td>30</td>
<td></td>
</tr>
<tr>
<td></td>
<td>650 #3</td>
<td>1</td>
<td>30</td>
<td></td>
</tr>
<tr>
<td></td>
<td>650</td>
<td>0.45 mm/min</td>
<td>25.4</td>
<td>MSE</td>
</tr>
<tr>
<td></td>
<td>880</td>
<td>1</td>
<td>30</td>
<td>NIST</td>
</tr>
<tr>
<td>ARL XXX</td>
<td>450 #1</td>
<td>1</td>
<td>25.4</td>
<td>NIST</td>
</tr>
<tr>
<td></td>
<td>450 #2</td>
<td>1</td>
<td>25.4</td>
<td>NIST</td>
</tr>
<tr>
<td></td>
<td>600 #1</td>
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</tr>
<tr>
<td></td>
<td>750 #2</td>
<td>1</td>
<td>25.4</td>
<td>NIST</td>
</tr>
</tbody>
</table>

4.7 Determination of Tempering Response (Hollomon-Jaffe parameter)

In order to study tempering response of Armox 440 and ARL XXX steel and determine the relationship of Hollomon-Jaffe parameter and hardness data, small disc samples with 4 mm thickness and 0.25 inch of diameter, were heated in a ULVAC-RIKO MILA-5000 light radiation furnace (LRF), shown in Figure 4.13, at a rate of 12 °C/s and held for different times as shown in Table 4.10. A typical heating and cooling curve is
shown in Figure 4.14. A hole was drilled down to the center of sample. Thermocouples were welded inside the hole and it was connected to the control channel of furnace, so that sample and furnace had identical temperature. After heat treatment, samples were allowed to cool down freely to room temperature. Hardness tests were then conducted using Leco M-400-H1 hardness testing machine by making 3-4 random indentations on sample surface and taking average hardness value. Hollomon-Jaffe parameters were obtained using the following equation (5):

$$HJ\mathcal{P} = T \ast (c + \log t) \quad (5)$$

where $T$ is absolute temperature in K, $c$ is a constant determined by carbon content of sample, and $t$ refers to holding time in seconds or hours. Constant $c$ is calculated using equation (6) or (7) below:

$$c = 17.7 - 5.8 \ast \text{carbon}\% \quad \text{for time in seconds} \quad (6)$$

$$c = 21.3 - 5.8 \ast \text{carbon}\% \quad \text{for time in hours} \quad (7)$$

Armox 440 had carbon content of 0.19%, $c$ was equal to 16.6, for time in seconds. ARL XXX contained 0.22% carbon and had $c$ constant of 16.42. A plot of hardness data versus Hollomon-Jaffe parameters (i.e. $T \ast (c+\log t)$) was generated.

Table 4.10: Testing parameters of heat treatment of Armox 440

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Holding time (min)</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Armox 440</strong></td>
<td></td>
</tr>
<tr>
<td>500</td>
<td>1 5 10 20 60 99.59</td>
</tr>
<tr>
<td>600</td>
<td>1 5 10 20 60 99.59</td>
</tr>
<tr>
<td>660</td>
<td>1 5 10 20 60 99.59</td>
</tr>
<tr>
<td><strong>ARL XXX</strong></td>
<td></td>
</tr>
<tr>
<td>450</td>
<td>1 5 10 20 60 99.59</td>
</tr>
<tr>
<td>750</td>
<td>1 5 10 20 60 99.59</td>
</tr>
</tbody>
</table>
Five samples were chosen to conduct metallographic characterization under light optical microscope. The study was for comparing microstructure, understanding large variation of hardness on particular sample and sharp drop of hardness. These samples include Armox 440 base metal, heat treatment at 500 °C and 660 °C holding for 1 hour; ARL XXX heat treatment at 750 °C holding for 60 and 99.59 minutes respectively. Temperature versus time data of the two ARL XXX samples subjected to metallography were also analyzed with single sensor differential thermal analysis (SS-DTA) to study possible phase transformations.

Figure 4.13: Light Radiation Furnace (LRF)
4.8 Phase Transformation Analysis

4.8.1 JMatPro™ and ThermoCalc™ Simulations

For the purpose of studying ARL XXX phase transformation at various conditions, two simulation software were used.

JMatPro™ predicts a wide variety of material properties for multi-components alloys, such as mechanical properties, solidification behavior and phase transformations. It consists of a Java-based user interface and C/C++ calculation modules [101].

ThermoCalc™ is another prediction software that simulate thermodynamic and diffusion controlled processes for different materials, which is based on some thermodynamic experimental databases that has been evaluated with the CALPHAD method [102]. Phase fraction plots under equilibrium condition and enthalpy-temperature plots were generated for both ARL XXX and Armox 440 steels in order to analyze carbides.

Figure 4.14: Heating and cooling curve of ARL XXX heated at 750 °C held for 1 hour.
formation, austenite-ferrite transformation and other phase changes. Scheil-Gulliver solidification model was also utilized for simulations of non-equilibrium solidification paths and study of possible segregation.

4.8.2 Single-Sensor Differential Thermal Analysis (SS DTA)

In order to verify simulation results from ThermoCalc/JMatPro and have more thorough study on ARL XXX, single-sensor differential thermal analysis was applied. SS DTA is a technique used for studying whole range of solid-state phase transformations, solidification process and structural changes in materials under welding or other thermal-mechanical processes. There is no limit in temperature range, as well as heating and cooling rates. It is developed based on the concept of classic Differential Thermal Analysis (DTA), which evaluates the difference of thermal histories during continuous heating and cooling between tested and reference samples. The uniqueness of SS DTA is that it does not make use of an actual reference sample. Instead, a reference thermal curve is calculated from heat flow modeling equations and thermal history measured by specially developed computer software [103]. Only one temperature sensor is used to record temperature changes at any specific point in tested sample. Phase transformation temperatures are determined by enthalpy difference between measured data and reference thermal histories.

4.8.3 Determination of Solidification range

Liquidus-solidus phase transformation of ARL XXX was investigated using SS DTA. One 20 g and one 1g ARL XXX steel buttons were first made using a button-melter. The 1 g button was then melted onto 20 g button using gas-tungsten arc welding (GTAW). A
hole was drilled through the center of larger button in order to place the small button on top and thermocouples within the button, so that it was attached to 1 g button when being melted. The set up was connected to Instrunet software and all thermal data could be recorded during melting and solidifying process. Each sample was tested three times. Miller welding machine was set to control sidebeam with 10 seconds preflow and 50 seconds postflow of shielding gas. First run typically melted the small button only and not much useful data was obtained. Current was set at 250 Amp with 4 seconds arc time and 0.05 inches start gap. Second and third run had identical parameters with 300 Amp current, 7 seconds arc time and 0.08 inches start gap. The sample was allowed to cool down to about 40 °C before running next test, so that sufficient amount of cooling data was collected.

During melting process of ARL XXX buttons, maximum temperature can be reached up to 1700 °C. Thermal data recorded was then processed in SS DTA program. Heating curve was usually compared with 2nd polynomial model and cooling curve was compared with Rosenthal reference curve. A plot of temperature versus degree of deviation between measured and reference curve δT, would be generated after selecting a temperature range for analysis. The start and finish of phase transformation would be indicated by sharp peaks deviated from zero degree.

4.8.4 Phase Transformation study in Light Radiation Furnace

In order to determine critical transformation temperatures and other possible phase transformations, light radiation furnace, as shown in figure 4.13, was utilized to heat ARL XXX samples at very slow heating rate. Small disc samples with 4 mm thickness and 0.25
inch diameter were used. Three tests were performed. In the first test, sample was allowed
to heat up to 1000 °C at a rate of 600 °C/hr, followed by free cooling down to room
temperature. Schematic of first test thermal cycle is shown in Figure 4.15. The purpose was
to obtain an estimated range of temperature where most phase transformations took place,
in particular the ferrite-austenite phase change. Second test was an overnight test, which
took about 15 hours. ARL XXX was first heated to 400 °C at a rate of 10 °C/min, then
heating rate was greatly reduced to 28 °C/hr and heated up to 800 °C, so that all enthalpy
change details could be collected. After that, sample was subjected to free cooling back to
room temperature. For the third test, similar thermal cycle was applied as the second test,
except temperature range subjected to slowest heating rate (28 °C/hr ) was widen from 300
°C to 800 °C. Schematic of third test thermal history is shown in Figure 4.16. The heating
and cooling curves collected through Instrunet software were then analyzed by SS DTA.
Phase transformations occurred at different temperature range could be determined.

Figure 4.15: Schematic of first test thermal cycle for ARL XXX phase transformation
analysis in Light Radiation Furnace (LRF).
4.9 Continuous Cooling Transformation Diagram

The Continuous Cooling Transformation (CCT) diagram of ARL XXX was generated in order to study phase transformations under different cooling rates and their effects on microstructure. The Gleeble™ was used to perform a series of dilatometry experiments. Type K thermocouples were welded to center of sample for temperature control and acquisition. A quartz dilatometer was attached to the sample where thermocouples were welded, for purpose of measuring changes of sample diameter in transverse direction during heating and cooling cycle. Typically, diameter of sample changes linearly as temperature increases or decreases. Data collected were then used to generate a plot of dilation versus temperature. Set-up is shown in Figure 4.18. Different cooling rates were simulated by adjusting the free span of samples in between copper grips. Stainless steel grips was used in one test to simulate the slowest cooling rate. Cylindrical samples, shown

Figure 4.16: Schematic of third test thermal cycle for ARL XXX phase transformation analysis in Light Radiation Furnace (LRF).
in Figure 4.1 and 4.17, were cut to exact length according to specific free span so that no sample parts were displayed outside of grips.

ARL XXX sample was heated to 1300 °C at a rate of 100 °C/sec and held for one second, followed by free cooling to room temperature. Test program, as shown in Figure 4.19, was allowed to run at least 7 more minutes after heating was completed, so that most cooling data could be collected. For testing with longer free span and slower cooling rate, more time should be required. A plot of dilation versus temperature was created in order to measure austenite-ferrite and martensitic transformation temperatures, as well as to investigate other possible phase transformations. A summary table of free span, corresponding $\Delta t_{8/5}$ and total length of CCT tests is shown in Table 4.11. Two samples tested with fastest and slowest cooling rates (Free span: 10 mm and 70 mm with stainless steel grips) were subjected to microstructural characterization using optical microscope and hardness measurements under 1 kg load.
Figure 4.18: GleebleTM dilatometry set-up for ARLXXX formation of CCT diagram.

Figure 4.19: GleebleTM test program in QuikSim2 for ARLXXX CCT testing.
Table 4.11: Sample free span, T8/5 time and Total length (* SS stands for Stainless Steel. † Total length = free span distance + total length of both grips (~61mm).)

<table>
<thead>
<tr>
<th>Free Span (mm)</th>
<th>T8/5 (sec)</th>
<th>Total Length (mm)†</th>
</tr>
</thead>
<tbody>
<tr>
<td>10</td>
<td>3.1</td>
<td>71</td>
</tr>
<tr>
<td>15</td>
<td>6.43</td>
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<td>121</td>
</tr>
<tr>
<td>70</td>
<td>41.9</td>
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</tr>
<tr>
<td>70 (SS grips)*</td>
<td>46.76</td>
<td>131</td>
</tr>
</tbody>
</table>

4.10 ARL XXX weld characterization

Four ARL XXX welds were made by the sponsor company, American Engineering and Manufacturing, Inc. (AEM). Two 20 mm thick ARL XXX plates were set up in T-configuration and welded with ER70S-6 filler wire of 0.045” in diameter using gas metal arc welding (GMAW) process and 90%Ar/10%CO₂ shielding gas. The wire feed speed was 350 ipm; voltage used was 26 volts and travel speed was 13 ipm. All welds have 9” weld length. The only difference between the welds is preheat temperature. Weld MA10 was preheated to 17°C before welding: MA04 was preheated to 66°C; MA06 to 149°C and
MA08 to 260°C. The macro sections of these four welds were then cut to show the face near middle of weld.

The weld sections were sent to OSU and subjected to sample preparation. Optical metallography was performed on all samples using Le Pera’s etchant. Samples with weld defects, i.e. MA04, MA06 and MA08, were further investigated using SEM for fractography.

In addition, MA04 was chosen to perform more analysis as it contains the largest weld defects. Chemical analysis was performed on parent metal, filler wire and three regions within the molten weld pool obtained by drilling into the weld (Figure 7.19) using an Agilent 710 inductively coupled plasma spectrometer at Miami Valley Materials Testing Center (MVMTC). Carbon and sulfur analysis was carried out using LECO CS200 carbon/sulfur analyzer. In comparison, energy dispersive spectroscopy (EDS) of SEM was also utilized at OSU center of electron microscopy and analysis (CEMAS) for studying compositional changes at regions around weld cracks and without present of defects. In addition, composition of diluted weld was calculated based on dilution ratio. These data were then input to ThermoCalc™ software to perform thermodynamic simulations, including generation of equilibrium phase fraction plots and Scheil simulation, in order to understand the reasons of forming weld defects after applying the preheat temperature.
Chapter 5: Results and discussion of Ti-6Al-4V armor alloys

This chapter presents result analysis and discussion on the testing of Ti-6Al-4V alloy. Testing conducted for Titanium alloy materials include temperature distribution testing at Gleeble™ using samples with different dimensions. This test was conducted in order to find out the temperature difference at various locations along the sample, which can lead to non-uniform distribution of strain. The sample with dimension that had the most uniform temperature distribution was used for high-temperature straining tests at Gleeble™. High temperature straining tests were developed as a methodology to study the effect of strain and temperature on formation of strain-induced porosity and failure mechanism during plate bending in this alloy. Mechanical data obtained can be applied to develop optimal process window for induction bending process of Ti-6Al-4V and possibly other high strength alloys. These samples were subjected to cross-section metallographic analysis to study microstructural changes and void formation after high temperature straining. Some of the samples subjected to hot ductility tests by Nick Kullman [3] were investigated under light optical microscope and scanning electron microscope (SEM) to understand failure mechanism during Gleeble™ testing and void formation. High temperature straining, followed by room temperature tensile tests were performed for studying the effect of induction bending on room temperature mechanical properties of titanium plates. Amount of strain applied was determined by conducting high temperature tensile test on testing materials. Some of the tensile tested samples were then subjected to metallographic analysis.
5.1 Temperature Distribution Testing

Gleeble™ 3800 is a physical simulation and thermal-mechanical testing system. Its thermal system is operated by direct resistance, which can heat samples at a very fast rate or hold at steady equilibrium temperature by adjusting the current input required to pass through the sample. Sample grips and water-cooled jaws are used to link the system to sample for heat transfer and electrical contact points [104]. Because of such heating mechanism, isothermal temperature planes will be produced through the sample diameter. [105] However, thermal energy experienced by the sample will be different along longitudinal direction and lead to temperature gradient along the sample.

Non-uniform temperature distribution will induce various levels of thermal expansion, as well as other temperature dependent flow properties along the specimen. This results in changes to the response of loading and different amount of deformation at different parts of the sample. Lower temperature at one point can lead to lower strain level compared to a point closer to the center with higher temperature. One of the ideal temperature profile of Gleeble™ sample determined by S.D. Norris, et al, is setting the mid region to achieve test temperature, followed by a sharp drop to room temperature at both sides [105]. This allows all load and strain to concentrate in the center zone without the present of spurious strains in other sections, so that mechanical data collected are more applicable to real situations.

It is important to know the temperature distribution of a sample when subjected to heating in the machine. The aim of using the Gleeble™ is to simulate effects of different parameters, such as strains and temperature, on different materials during heat treatment and various welding processes. If temperature difference between each point along sample is too large, strain-stress measurements obtained after testing, as well as the sample
microstructure might have huge deviation from what is expected to see in the test conditions. As a result only the center point of sample with control thermocouples is subjected to exact temperature and strain level that is preset at start of experiment. The data collected will not be accurate enough to apply to the optimal process window for real manufacturing environment.

Stainless steel jaws instead of common copper jaws are used in experiments for lower thermal conductivity in order to reduce heat extraction from testing sample, which helps to achieve more uniform temperature distribution. Free span between contact points of sample to grips can affect the length of isothermal zone. A shorter distance will lead to smaller section of uniform temperature distribution. In addition, materials of testing samples can also influence temperature distribution as they have different structures and conductivity properties.

In this set of experiments, three samples with different dimension, shown in Figure 5.1, were tested at two temperatures (i.e. 430 °C and 650 °C) respectively. They are cylindrical samples with 0.25” diameter and threads on both ends, standard tensile samples with 0.375” diameter shoulders and 1.25” in length of 0.25”-diameter reduced section, as well as 0.375” diameter cylindrical rods. These temperatures are chosen according to α to β transformation temperature and β transus temperature of Ti-6Al-4V alloys. Based on SS-DTA analysis performed by Nick Kullman, β-transus temperature of Ti-6Al-4V alloy is about 989 °C and α to β start temperature is 795 °C [3]. The samples are heated at the rate of 10 °C/s. Three to four thermocouples are attached to sample at different distance away from center. Control thermocouples are attached to the center of sample, which have the highest and applied temperature. Plots of temperature versus time are shown in the
following pages, together with a sample diagram at top right corner of each plot. A table (Table 5.1) is also created to show degree of temperature difference between different points of samples with different dimensions. The goal of testing is to investigate the possible effect of sample dimension and geometry on temperature distribution after heating. The one that fits a relatively uniform thermal profile along longitudinal direction will be the best sample applied for Gleeble™ testing that involves heating and loading.

Figure 5.1: Gleeble™ samples for Temperature distribution testing.
Figure 5.2: Plot of Temperature vs Time of Gr 23 Ti6-4 HDT sample at 430°C.

Figure 5.3: Plot of Temperature vs Time of Gr 23 Ti6-4 standard tensile sample at 430°C.
Figure 5.4: Plot of Temperature vs Time of Gr 23 Ti6-4 3/8" rod sample at 430°C.

Figure 5.5: Plot of Temperature vs Time of Gr 23 Ti6-4 HDT sample at 650°C.
Figure 5.6: Plot of Temperature vs Time of Gr 23 Ti6-4 standard tensile sample at 650°C.

Figure 5.7: Plot of Temperature vs Time of Gr 23 Ti6-4 3/8” rod sample at 650°C.
Table 5.1: Temperature difference between center and other thermocouples located along the sample

<table>
<thead>
<tr>
<th>Sample dimensions/ Temperature difference</th>
<th>Temperature (°C)</th>
<th>TC3 1/6” from center</th>
<th>TC2 1/3” from center</th>
<th>TC4 1/2” from center</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>430</td>
<td>2</td>
<td>12</td>
<td>14</td>
</tr>
<tr>
<td></td>
<td>650</td>
<td>1</td>
<td>15</td>
<td>19</td>
</tr>
<tr>
<td></td>
<td>430</td>
<td>14</td>
<td>33</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td>650</td>
<td>27</td>
<td>44</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td>430</td>
<td>0</td>
<td>8</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td>650</td>
<td>-8</td>
<td>0</td>
<td>-</td>
</tr>
</tbody>
</table>

Thermocouples are located at the center (TC1), 1/6” (TC3), 1/3” (TC2) and half inch (TC4) away from center. Results show that at 430°C · HDT samples with ¼” diameter and threads at both ends, have only 2°C temperature difference between center and the point at 1/6” from center, followed by a larger difference of 12°C with the point at 1/3” from center and 14°C temperature difference between center and half inch from mid-point. For tensile samples with 1.25” in length of reduced section, they have a temperature difference of 14°C between center and the closest thermocouple location, which is equivalent to the difference between center and TC2 for HDT samples. On the other hand, temperature difference of TC1 and TC2 for tensile samples is 33°C, more than two times of center and TC3 temperature difference. Rod sample in 3/8” diameter does not show temperature
difference when comparing thermocouples at center and 1/6” inch from center. Between TC1 and TC2, there is only 8 °C difference.

For testing heated to 650°C, HDT samples show 1°C temperature difference between center and TC3, followed by a difference of 15°C between TC1 and TC2 and 19°C temperature difference between center and ½” from center. Tensile samples exhibited 27°C difference between TC1 and TC3. Comparing the center and 1/3” from center, there is 44°C temperature difference. For rod samples in 3/8” diameter, temperature at 1/6” from center shoot up to 658°C and there is no temperature difference between thermocouples at center and 1/3” from center.

According to data above, all samples show higher degree of non-uniform temperature distribution at higher temperature regardless of difference in sample dimensions. Both HDT samples and 3/8” rod samples have relatively uniform temperature distribution. 3/8” rod samples have longer isothermal region (i.e. more than 1/3”) compared to HDT samples. However, the highest temperature in sample is not at center when heated up to 650°C, as it is 8°C higher than set temperature at 1/6” from center, which is due to larger cross-section area allowing more current to pass through and becoming harder to control current input. For HDT samples, isothermal zone is within 2/3” at center, followed by larger drop of temperature between TC3 and TC2. From 1/3” to ½” away from center, temperature drop is smaller in range of 2-4°C. Such temperature distribution allows all load and strain to concentrate in central region and provides mechanical data with higher accuracy. Standard tensile samples have the most non-uniform temperature distribution. Although the function of reduced section is to allow strain concentration, difference in cross sectional area between center region (¼” diameter) and two shoulders (3/8” diameter) leads to a large
temperature drop even at short distances from the center when subjected to direct resistance heating in the Gleeble™. This will cause great variation in strains even at central zone and loading data collected will have error. Therefore, standard tensile samples with reduced section should not be used for Gleeble™ high temperature testing. HDT samples with ¼” diameter are chosen as Gleeble™ testing samples, given that they have uniform temperature distribution zone in center, and more samples can be machined from same amount of materials compared to 3/8” rod samples. Because of the minimal temperature difference between center and the closest thermocouples that are 1/6” from center, a gauge length of 10 mm (~1/3”) is selected for high temperature straining tests as extensometer is used for strain measurements.

The temperature gradient exhibited by sample in the Gleeble™ thermal system is analogous to that found in the full size induction heating system. In the full size system a plate will typically be placed on top of an induction coil and heated to the desired temperature. Although only a band of material is subjected to heating, non-uniform temperature distribution can be formed depending on plate thickness, materials, transportation time from heating to press-brake machine, etc. One way to improve this issue in actual bending process is to combine the heating and bending facilities together.

5.2 Hot Ductility Testing

A series of hot ductility tests were conducted on five different grades of Ti-6Al-4V alloys in forms of plate or bar by Nick Kullman in the Gleeble™ as described in section 4.3. Several parameters were investigated by varying temperatures, extension rate, forms of Ti-64 alloys and testing environment. The goal of these tests is to determine the
formability of titanium alloys at high temperatures, which helps to develop the optimal process control windows for hot induction bending process. Figure 5.8 and 5.9 shows plots of reduction-in-area versus temperature of five types of Ti-6Al-4V alloys in two different extension rates: 1mm/s and 10mm/s, which are chosen according to ram speed of press brake machine used for bending. Testing chamber is under rough vacuum atmosphere at around 2.8- 3.2 x10^{-2}torr. MIL-STD 46077 Class 2 plate is the material with lowest ductility, as it exhibits the lowest reduction-in-area data over a range of elevated temperatures tested. Therefore, MIL-STD 46077 Class 2 plate is further investigated by longitudinal-section metallurgical characterization under light optical microscope (LOM) and fractography using scanning electron microscope (SEM) after Gleeble™ testing in current work.

Figure 5.8: Plot of reduction-in-area (RA) vs temperature for Ti alloys at 1mm/s extension rate.
5.2.1 Metallurgical Characterization

Results of hot ductility test show that as temperature increases, reduction-in-area of Ti6-4 Class 2 material also increases, indicating increasing ductility. This is because increasing temperature leads to faster atom vibrations at greater amplitude, which increases the ease of atom slippage to new crystallographic plane under stress [106]. Phase transformations at different temperatures are also another important factor. In-depth analysis of optical micrographs enables us to observe the relationship between ductility and temperature, as well as the relationship between phase transformation temperatures and formation of strain-induced porosity. Ten samples, circled in Figure 5.10 and 5.11, were chosen from two groups of HDT tests with different extension rates. Figures 5.12 to 5.16 present cross section images of Ti6-4 HDT samples tested with extension rate of 1 mm/s at 982 °C, 871 °C, 649 °C, 538 °C, and 429 °C respectively. Figure 5.17-5.22 show
micrographs of HDT samples tested with 10 mm/s extension rate at 982 °C, 871 °C, 649 °C, 429 °C, 316°C and 37 °C. The left image is at lower magnification of x5 or x10 and right picture is at higher magnification of x100. All samples were etched with Kroll’s solution, which is a general etchant for titanium alloys. Black regions usually represent beta phase and light regions present alpha phase.

Figure 5.10: Plot of Reduction-in-Area (RA) vs Temperature for Ti-6Al-4V Class 2 plate at 1mm/s extension rate.
Base metal microstructure of MIL-STD-46077 class 2 Ti6-4 plate, which is a type of α-β alloy, consists of equiaxed alpha and transformed beta grains, with banding features observed along the rolling direction. As stated in previous section, β-transus temperature is about 989°C and α to β start temperature is 795°C [3]. Recrystallization temperature is approximately 890 °C (0.6 T_m when T_m is melting point in degree Celsius) [107].

The cross-section of sample tested at 982 °C at 1mm/s extension rate, as shown in Figure 5.12. This testing temperature is very close to β-transus temperature and above the recrystallization temperature. It has the highest reduction-in-area (94.41%) and also the greatest degree of necking as seen from cross section compared to other examined samples, indicating that it fails in a ductile fracture mode. No strain-induced porosity is observed in the sample, which means no crack initiation site is formed. This is due to dynamic recrystallization and almost complete formation of β phase. It consists of recrystallized, equiaxed grains of alpha with intergranular beta.

Figure 5.11: Plot of RA vs Temperature for Class 2 plate at 10mm/s extension rate.
Figure 5.13 shows optical images of the sample tested at 871 °C with 1 mm/s extension rate. This sample is tested above α to β start temperature and below the estimated recrystallization temperature. Reduction-in-area (RA) is about 80.00%. The sample contains elongated grains of alpha along extension direction with intergranular beta. It forms the largest amount of strain-induced porosity among the examined samples. These voids are elongated along the extension direction and have tendency of coalesce. Most of them locate within 1 mm below fracture surface and along prior β grain boundaries.

The sample tested at 649 °C, as shown in Figure 5.14, is below α to β start temperature. RA is approximately 62.25%. Its microstructure consists of alpha grains with beta phase in intergranular regions. Grains are elongated due to extension force. The amount of strain-induced porosity formed is less compared to previous sample. Size of voids is approximately 20 µm and they are mostly circular in shape.

Figure 5.15 shows that sample tested at 538 °C, which is below α to β start temperature. RA is about 59.55%. It has the same microstructure as sample tested at 649 °C, but with fewer amount of voids formed, which are round in shape. These strain-induced porosity mainly appear in the central section of fracture surface, as well as in between the edge and bottom of cup fracture surface, where a crack opening has already started to propagate.

The last sample examined at 1mm/s extension rate is tested at 427 °C, which is also below α to β start temperature. RA is about 47.93%. It contains elongated alpha grains and less amount of intergranular beta, as shown in Figure 5.16. The amount and shape of strain-induced porosity formed is similar to the sample tested at 538 °C, but size are generally less than 20 µm.
Figure 5.12: Microstructure of HDT sample of Class 2 plate tested at 982°C with 1mm/s extension rate.

Figure 5.13: Microstructure of HDT sample of Class 2 plate tested at 871°C with 1mm/s extension rate.

Figure 5.14: Microstructure of HDT sample of Class 2 plate tested at 649°C with 1mm/s extension rate.
Six hot ductility tested samples at 10 mm/s extension rate were analyzed by cross section metallography. The first sample, as shown in Figure 5.17, was tested at 982 °C. It is very close to β-transus temperature and above the estimated recrystallization temperature. The necking region is greater than the one tested with 1 mm/s extension at same temperature. Reduction-in-area is also higher at 96.64%. Its microstructure consists of equiaxed alpha due to recrystallization and intergranular beta. No strain-induced porosity is observed.
Sample shown in Figure 5.18, was tested at 871 °C, which is between α to β start temperature and recrystallization temperature. RA is about 78.31%. It has the greatest amount of strain-induced porosity among samples strained at a rate of 10 mm/s, but contains fewer voids when compared to the sample tested at same temperature at 1 mm/s extension rate. Most of them are found within 500 μm below fracture surface and at alpha-beta phase boundaries. Both the voids and microstructure of sample are elongated along the extension direction, which consists of alpha grains (light region) with beta phase in intergranular regions (dark region).

Figure 5.19 shows the sample with 10 mm/s extension rate tested at 649 °C, which is below α to β start temperature. RA is about 58.72%. It comprised of the same microstructure as previously described sample and grains are elongated due to stress applied in longitudinal direction. The majority of voids formed are observed at the middle section of fracture surface in beta phase regions and are spherical in shape. Both number and size of strain-induced pores are smaller than that tested at 871 °C. Sample tested at 427 °C is shown in Figure 5.20 with RA of 52.58%. It is also tested below α to β start temperature. It consists of similar microstructure and amount of voids compared to sample at 649 °C, except that the voids are smaller in size of approximately 10 μm. For sample tested at 316 °C, as shown in Figure 5.21, elongated alpha grains and intergranular beta phase is observed. RA is approximately 40.26%. Only a few voids are found near the edge of cup fracture surface. Figure 5.22 shows the Ti-6Al-4V sample tested at 38 °C, which is far below α to β temperature and close to room temperature, microstructure of elongated alpha grains and beta phase along intergranular bounaries is observed. RA is the lowest among examined samples at about 23.42%. There are only 2-3 strain-induced porosity
found 500μm below fracture surface. One of them is irregular in shape, as it is developed through coalescence of voids. It is also observed that samples heated at lower temperature consist of lower percentage of intergranular beta.

Figure 5.17: Microstructure of HDT sample of Class 2 plate tested at 982°C with 10mm/s extension rate.

Figure 5.18: Microstructure of HDT sample of Class 2 plate tested at 871°C with 10mm/s extension rate.
Figure 5.19: Microstructure of HDT sample of Class 2 plate tested at 649°C with 10mm/s extension rate.

Figure 5.20: Microstructure of HDT sample of Class 2 plate tested at 427°C with 10mm/s extension rate.

Figure 5.21: Microstructure of HDT sample of Class 2 plate tested at 316°C with 10mm/s extension rate.
5.2.2 Fractography

Fracture surface of hot ductility tested samples were also analyzed under SEM. The following five samples were tested at 1 mm/s extension rate. Figure 5.23 reveals fractography images of sample tested at 982 °C. It has very small fracture surface area and fracture features cannot be detected clearly. The entire area is covered with a melt layer. This is due to arcing when sample was heated at such elevated temperature in Gleeble™.

Sample tested at 871 °C is shown in Figure 5.24. Fracture surface exhibits elliptical shape. There are local melting regions at two sides of fracture surface, similar to the sample at 982 °C. At middle section, dimples of various sizes can be observed, which indicates ductile fracture mechanism. Small deep dimples are found at the edge or inside large shallow dimples. White features observed within dimples might be local oxidation at fracture.

Figure 5.25 shows the sample tested at 649 °C. Its fracture surface has an elliptical shape and displays 100% dimple rupture. The center region reveals equiaxed dimples with uniform size due to uniaxial tensile load. There are small dimples located inside and around

Figure 5.22: Microstructure of HDT sample of Class 2 plate tested at 38°C with 10mm/s extension rate.
rim of larger dimples (see arrow). Around the edge of fracture surface are shallow elongated dimples that are open at one end and point toward fracture origin, which result from mode I tear loading condition.

Sample tested at 538 °C is shown in Figure 5.26. Fracture features are similar to previous sample. Ductile dimples exhibited in central area maintain uniformity in size with conical shape but are relatively shallow compared to the sample tested at 649 °C.

Figure 5.27 displays hot ductility sample tested at 427 °C. It reveals elliptical fracture surface and ductile dimples are all over surface due to tension loading. These equiaxed dimples are relatively shallow, conical in shape and mostly identical in size, which are similar to those observed in sample tested at 538 °C. However, more fine dimples can be seen around the surface, especially along edges of coarser dimples. The size of dimples may be controlled by the amount and distribution of nucleated microvoids [108].

Figure 5.23: Fractography of HDT sample of Class 2 plate tested at 982°C with 1mm/s extension rate.
Figure 5.24: Fractography of HDT sample of Class 2 plate tested at 871°C with 1mm/s extension rate.

Figure 5.25: Fractography of HDT sample of Class 2 plate tested at 649°C with 1mm/s extension rate.

Figure 5.26: Fractography of HDT sample of Class 2 plate tested at 538°C with 1mm/s extension rate.
The following four hot ductility samples were tested at 10 mm/s extension rate. Figure 5.28 shows the sample tested at 982 °C. It has even smaller fracture surface area compared to the sample tested at same temperature but with a slower extension rate. The surface is covered by melt layer due to arcing when tested in the Gleeble™. No distinct fracture features can be observed.

Sample tested at 871 °C is shown in Figure 5.29. It contains an elliptical shape fracture surface covered with ductile dimples. Local melting can be found at some edges of surface. Equiaxed Dimples are deep and fine, and relatively uniform in size due to uniaxial tension load.

Figure 5.30 shows the sample tested at 649 °C, with a fracture surface in elliptical shape. It fails as 100% dimple rupture. Central region consists of coarse dimples and numerous small dimples within larger ones. At two sides of fracture surface, dimples are relatively shallow and conical in shape, which may be resulted from microvoid coalescence by shear along slip bands [108]. A lot of fine dimples are located at the rim of larger dimples.
Sample tested at 38 °C is shown in Figure 5.31. Fracture surface is in spherical shape. Not much necking took place before fracture occurred. Fracture mode is considered as microvoid coalescence subjected to tensile tear. Equiaxed dimples are present all over the surface. Around the edge of surface are fine shallow dimples. Those that are located closer to the edges are more elongated and opened at one end. Tear ridges can also be observed at intergranular regions.

Figure 5.28: Fractography of HDT sample of Class 2 plate tested at 982°C with 10mm/s extension rate.

Figure 5.29: Fractography of HDT sample of Class 2 plate tested at 871°C with 10mm/s extension rate.
Figure 5.30: Fractography of HDT sample of Class 2 plate tested at 649°C with 10mm/s extension rate.

Figure 5.31: Fractography of HDT sample of Class 2 plate tested at 38°C with 10mm/s extension rate.
5.2.3 Summary of Hot Ductility Testing

The ductility of MIL-STD 46077 Class 2 plate increases with increasing temperature. Metallographic characterization shows that formation of strain-induced porosity (SIP) is dependent on the test temperature as it leads to change of phases and microstructure. Development of SIP is a serious issue, which cannot be detected by non-destructive testing. It can only be fixed by costly and time-consuming methods such as hot-isostatic pressing (HIP). SIP usually appears in the form of wedge cracks or pores at the prior β grain boundaries. As local tensile stress is applied to sample during extension, voids start to grow and eventually develop into cracks [90]. The maximum amount of voids formed in between 795 °C and 890 °C. This is related to the effect of α to β transformation and recrystallization are taking place at this temperature range. No voids were found in samples tested above recrystallization temperature and close to β-transus temperature. Samples that were tested below α to β start temperature have lower amount of voids.

The amount of strain-induced voids formed in samples with two different extension rates is relatively similar at respective temperatures. Therefore, in the tested titanium Class 2 plate samples, the extension rate does not affect the formation and growth of strain-induced voids.
5.3 Gleeble™ High Temperature Straining Testing

In order to study the effect of strain at elevated temperature on Ti-6Al-4V alloys during hot induction bending process, Gleeble™ was utilized to simulate the effects and to study changes of microstructure, formation of strain-induced porosity and mechanical properties. Two series of high temperature straining tests were conducted using ASTM B265 grade 23 plate materials (MJL 271/03AP) at 430 °C and 650 °C respectively below α to β start temperature with 1 mm/s extension rate. All samples were first heated at a rate of 10 mm/s and held at elevated temperature for about six minutes. High temperature extensometer was attached to the center of sample for measuring change of sample length and hence the engineering strain in longitudinal direction. A total of five tests were performed at each temperature. The first test was a high temperature tensile test, which applied tension force until the sample failed. An engineering stress-strain curve was generated for each of the two test temperatures as shown in Figure 5.32. By analyzing the tensile curve, the corresponding strain and stroke applied by Gleeble™ was identified at: a) The yield strength (YS); b) In between YS and ultimate tensile strength (UTS); c) At UTS, and d) Above the UTS (circled in Figure 5.32). The applied stroke was then adjusted in remaining four tests, so that the strain applied to the four samples were: a) At YS; b) In between YS and UTS; c) At UTS, and d) Above UTS respectively. These samples were then cross-sectioned and investigated under optical microscope, as shown in Figure 5. 35- 5.39 for samples tested at 430 °C and Figure 5.40 – 5.44 for 650 °C. Engineering stress-strain plots for each test are presented in Figure 5.45-5.54. Figure 5.33 shows the test cycle for high temperature tensile test at 430 °C in terms of temperature, strokes, force, and change of length measured by extensometer with respect to time in second.
Figure 5.32: Ti-6Al-4V grade 23 high temperature tensile tests at 430 °C and 650 °C.

Figure 5.33: Ti-6Al-4V Gleeble™ testing cycle at 430 °C pulled to failure.
5.3.1 Cross-section Metallurgical Characterization

Base metal microstructure of Ti-6Al-4V grade 23 plate consists of equiaxed alpha and transformed beta with acicular alpha due to faster cooling rate when processing (Figure 5.34). When sample was strained until it reached the state of yield strength (YS) at 1 mm/s extension rate at 430 °C, as shown in Figure 5.35, equiaxed alpha and intergranular beta can be observed. But amount of transformed beta phase containing acicular alpha has reduced greatly due to heating below α to β start temperature. Evidence of banding is found at top of image, as shown by some elongated alpha grains. The cross-section area of sample reduces uniformly by 1.2%. The amount of strain applied does not have significant effect on microstructure and grain morphology.

Figure 5.36 and Figure 5.37 display the sample strained to the state between YS and ultimate tensile strength (UTS) with 3.2% RA and to the state at UTS with 6.3% RA respectively. Both tested samples have similar microstructure. They contain equiaxed alpha grains as presented by light regions. The sample at UTS has grains that start to elongate and in slightly larger size, when compared to the one in between YS and UTS. Intergranular beta and transformed beta phase with acicular alpha are also revealed and continue to decrease. Changes in grain morphology, such as size and shape, are also observed as sample reached UTS state.

In Figure 5.38, the sample heated to 430 °C is strained to above UTS. Necking can be observed in the middle of sample as strain increases rapidly and cross-section area decreases non-uniformly. RA is about 38.1%. It consists of elongated alpha grains. The amount of intergranular beta is similar to the sample that reached UTS state. Only a small
amount of transformed beta with acicular alpha is left in microstructure. They are elongated along direction of extension force.

Figure 5.39 shows Ti-6Al-4V grade 23 plate sample heated to 430 °C and extended until failure at a rate of 1 mm/s. RA is approximately 58.06%. It contains alpha grains and intergranula beta phase elongated to a greater extent. All acicular alpha has dissolved. Strain-induced porosity is observed as stress concentration regions exceed maximum stress withstand. Most of them were located within 500 μm under fracture surface and along alpha-beta phase boundaries. These voids are spherical in shape and about 10 μm diameter or smaller. The amount of strain introduced at sample fracture significantly affects grain morphology and formation of strain-induced porosity, which is the initiation site of cracking.

Figure 5.34: Base metal microstructure of titanium grade 23 plate.
Figure 5.35: Ti-6Al-4V Gr 23 strained to yield strength (YS) level at 430°C.

Figure 5.36: Ti-6Al-4V Gr 23 strained to between YS and ultimate tensile strength (UTS) level at 430°C.

Figure 5.37: Ti-6Al-4V Gr 23 strained to UTS level at 430°C.
The following five samples were tested at 650 °C in the Gleeble™, which is also below but closer to the estimated α to β start temperature. Figure 5.40 and Figure 5.41 exhibit samples strained in longitudinal direction at 650 °C to the state in between YS and UTS with 4.5% and 7.8% RA respectively. More strain is applied to the sample shown in Figure 5.41. They both contain alpha grains and intergranular beta phase. More transformed beta with acicular alpha are present compared to samples tested at 430 °C. Grain morphology has already been affected and most grains are elongated along extension direction.
The sample extended to the state of UTS at temperature of 650 °C is shown in Figure 5.42. RA is about 37.7%. There is no significant change in microstructure and grain morphology compared to samples strained to the state between YS and UTS. It contains elongated alpha grains, small amount of intergranular beta, and transformed beta with acicular alpha. However, reduction-in-area is more than four times the percentage of sample in Figure 5.41.

Figure 5.43 displays the sample strained to above UTS at 650 °C. Necking occurs as cross-section starts to decrease non-uniformly. RA is approximately 75.4%. Its microstructure consists of alpha and intergranular beta phase elongated in longitudinal direction at much greater extent compared to samples with less strains. Grain morphology is severely affected and most grains have lost their shape. Strain-induced porosity is formed mainly at the necking region. In center necking area, voids are in larger size of about 20 μm. They tend to be more irregular in shape and form by coalescence of smaller voids. Further away from center voids in smaller size are found. There are voids in ellipsoidal shape elongated along extension direction, as well as spherical ones of approximately 10 μm. Most of them are located at intergranular regions.

The sample strained until fracture at 650 °C is shown in Figure 5.44 with 71.79% RA. Its microstructure is similar to the sample strained to above UTS and the one tested to failure at 430 °C, which consists of elongated alpha grains with intergranular beta phase. Strain induced pores continue to grow and coalescence as more strain is introduced. The voids formed are larger in average, which is about 30 μm in diameter. They are located within 600 μm under fracture surface. Most of the voids are relatively spherical, but slightly elongated along extension direction, as shown in right image of Figure 5.40. There are also
a small amount of ellipsoidal voids due to greater extent of elongation at intergranular boundaries.

Figure 5.40: Ti-6Al-4V Gr 23 strained to between YS and UTS level at 650°C #1.

Figure 5.41: Ti-6Al-4V Gr 23 strained to between YS and UTS level at 650°C #2.
Figure 5.42: Ti-6Al-4V Gr 23 strained to UTS level at 650°C.

Figure 5.43: Ti-6Al-4V Gr 23 strained to above UTS level at 650°C.

Figure 5.44: Ti-6Al-4V Gr 23 strained to failure level at 650°C.
5.3.2 Comparison of Mechanical Properties

Besides investigating the microstructure of tested samples, mechanical data obtained from Gleeble™ at elevated temperatures are also studied. Figure 5.45 to 5.49 show stress-strain curve of Ti-6Al-4V tested at 430 °C with increasing amount of strains until failure. Figure 5.50–5.54 display same series of curves tested at 650 °C. Table 5.2 and 5.3 show the mechanical data after analyzing the curves, including strokes applied, engineering stress, engineering strain, elongation and reduction-in-area (RA). It was found that because of increasing amount of strain, reduction-in-area gradually increases. Higher temperature leads to higher RA, indicating higher ductility. At 430 °C, yield strength of Ti-6Al-4V is about 722.9 MPa with 0.63% elongation, and ultimate tensile strength is approximately 807.6 MPa with 5.91% elongation. Total elongation at failure is 30.67%. At 650 °C, the sample tested close to yield strength consists of 367.9 MPa engineering stress with 1.44% elongation. Its ultimate tensile strength is 416.6 MPa with 7.32% elongation. It is observed that below α to β start temperature, lower temperature has almost double engineering stress regardless of the strain applied. On the other hand, elongation of samples are slightly higher at 650 °C compared to that at 430 °C. This might be due to degree of softening and difference in microstructure. At 650 °C (Figure 5.40), the amount of transformed beta with acicular alpha present in samples is more than that at 430 °C (Figure 5.35), together with greater extent of softening. Therefore, flow stress experienced by the material reduces significantly.

In addition, some inconsistency of high temperature straining tests in Gleeble™ can be found in the data. Table 5.3 shows that at 650 °C, amount of stroke applied to fail the sample is less than that applied to cause necking of sample. Failure to conduct the test that
reaches yield strength at 650 °C is due to inability to simulate same effect using the stroke determined from existing data. This is because it is difficult for Gleeble™ to apply accurate stroke control when its magnitude is to the nearest tenth. It leads to discrepancy between programmed and actual strokes, so that results shown might not correspond to the effect of programmed strain. One possible way to improve the problem is regulating the application of strain using extensometer, which measures strains simultaneously as the sample is being extended.

Table 5.2: Mechanical properties of grade 23 Ti-6Al-4V alloy tested at 430 °C with various strain levels

<table>
<thead>
<tr>
<th>430 °C</th>
<th>Stroke (mm)</th>
<th>Engr. Stress (MPa)</th>
<th>Engr. Strain (mm/mm)</th>
<th>Elongation (%)</th>
<th>RA (%)</th>
<th>Voids (Y/N)</th>
</tr>
</thead>
<tbody>
<tr>
<td>YS</td>
<td>3.4</td>
<td>722.9</td>
<td>0.0063</td>
<td>0.63</td>
<td>1.20</td>
<td>N</td>
</tr>
<tr>
<td>Between YS and UTS</td>
<td>5.64</td>
<td>783.9</td>
<td>0.0379</td>
<td>3.79</td>
<td>3.20</td>
<td>N</td>
</tr>
<tr>
<td>UTS</td>
<td>6.5</td>
<td>807.6</td>
<td>0.0591</td>
<td>5.91</td>
<td>6.30</td>
<td>N</td>
</tr>
<tr>
<td>Above UTS</td>
<td>7.75</td>
<td>559.1</td>
<td>0.24</td>
<td>24.0</td>
<td>38.10</td>
<td>N</td>
</tr>
<tr>
<td>Failure</td>
<td>11</td>
<td>420.4</td>
<td>0.3067</td>
<td>30.67</td>
<td>58.06</td>
<td>Y</td>
</tr>
</tbody>
</table>
Table 5.3: Mechanical properties of grade 23 Ti-6Al-4V alloys tested at 650 °C with various strain levels.

<table>
<thead>
<tr>
<th>650 °C</th>
<th>Stroke (mm)</th>
<th>Engr. Stress (MPa)</th>
<th>Engr. Strain (mm/mm)</th>
<th>Elongation (%)</th>
<th>RA (%)</th>
<th>Voids (Y/N)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Between YS &amp; UTS #1</td>
<td>1.7</td>
<td>367.9</td>
<td>0.0144</td>
<td>1.44</td>
<td>4.5</td>
<td>N</td>
</tr>
<tr>
<td>Between YS &amp; UTS #2</td>
<td>3</td>
<td>402.8</td>
<td>0.0431</td>
<td>4.31</td>
<td>7.8</td>
<td>N</td>
</tr>
<tr>
<td>UTS</td>
<td>4.5</td>
<td>416.6</td>
<td>0.0732</td>
<td>7.32</td>
<td>37.7</td>
<td>N</td>
</tr>
<tr>
<td>Above UTS</td>
<td>10</td>
<td>158.0</td>
<td>0.191</td>
<td>19.1</td>
<td>75.4</td>
<td>Y</td>
</tr>
<tr>
<td>Failure</td>
<td>8</td>
<td>168.9</td>
<td>0.4529</td>
<td>45.29</td>
<td>71.8</td>
<td>Y</td>
</tr>
</tbody>
</table>

Figure 5.45: Stress-strain curve of Ti-6Al-4V 430°C strained to YS level.
Figure 5.46: Stress-strain curve of Ti-6Al-4V 430°C strained to between YS & UTS level.

Figure 5.47: Stress-strain curve of Ti-6Al-4V 430°C strained to UTS level.
Figure 5.48: Stress-strain curve of Ti-6Al-4V 430°C strained to above UTS level.

Figure 5.49: Stress-strain curve of Ti-6Al-4V 430°C strained to failure level.
Figure 5.50: Stress-strain curve of Ti-6Al-4V 650°C strained to between YS & UTS. #1

Figure 5.51: Stress-strain curve of Ti-6Al-4V 650°C strained to between YS & UTS. #2
Figure 5.52: Stress-strain curve of Ti-6Al-4V 650°C strained to UTS level.

Figure 5.53: Stress-strain curve of Ti-6Al-4V 650°C strained to above UTS level.
5.3.3 Summary of High Temperature Straining Testing

Based on metallographic characterization and analysis of mechanical data, it is found that both temperature and strain levels can affect microstructure, high temperature mechanical behavior and cracking susceptibility of Ti-6Al-4V. At 650 °C, strain-induced porosity begins to form in sample strained to above ultimate tensile strength, which greatly increases availability of crack initiation sites. Therefore, induction bending at the same temperature must be under 19.1 % engineering strain to avoid adverse effect. At 430 °C, strain-induced porosity is only found in the sample that tested to failure. There is no void observed in samples with strain level up to 24.0 %. Thus, the amount of strain applied has less restriction at 430 °C. Hot induction bending at lower temperature below α to β transformation has lower risk of crack formation and propagation. High temperature mechanical data can be used for optimal bending procedure, such as the strain and load required to bend the material at specific temperature.

Figure 5.54: Stress-strain curve of Ti-6Al-4V 650°C strained to failure.
5.4 Tensile Testing

Besides understanding effect of hot induction bending parameters on high temperature properties and cracking susceptibility, it is essential to know their effects on room temperature (RT) mechanical properties, which greatly influence quality and applications of final product. Three types of Ti-6Al-4V alloy plates were studied, which are grade 5, grade 23, and Mil-Std 46077 class 2 plate materials. High temperature (HT) tensile tests (TT) were first performed at 1mm/s, to determine the amount of strain needed to reach yield strength, tensile strength and complete failure. Afterwards, test samples were strained with the same extension rate to predetermined strain levels at high temperature to simulate the effect of hot induction bending, followed by standard RT tensile tests. Three temperatures were investigated in the testing, i.e. 250 °C, 430 °C and 650 °C below α to β transformation. Part of the tests is done at Ohio State MSE department facilities using grade 5 and grade 23 plate materials and some are completed at National Institute of Standards and Technology (NIST) by Dr. Jeff Sowards using Mil-Std 46077 class 2 plate samples. Figure 5.55 displays the combined stress-strain plot of Ti6-4 grade 5 tensile test results. Grade 5 material shows a sharp drop of both UTS and total elongation at RT to 185.5 MPa and 1.6% respectively after 10% straining at 430°C compared to the sample without pre-straining. It might be due to higher percentage of interstitial elements, such as oxygen, carbon and nitrogen compared to Ti-6Al-4V grade 23. High temperature pre-straining at 650 °C with 10 % strain also leads to reduction of UTS and total elongation to 359.0 MPa and 3.9 % respectively at room temperature, but at a smaller extent compared to the sample strained at 430 °C. In addition, 5% straining at 250 °C leads to decrease of RT UTS to
187.0 MPa. A significant decrease of total elongation is also noticed at 3.5% and is similar to the value tested at 650 °C with 10% pre-strains.

Figure 5.56 shows the combination plot of engineering stress-strain curves for Ti6-4 grade 23 samples. Room temperature tensile test without pre-straining was conducted. It has UTS of 965MPa and total elongation at 21.6%. HT tensile test at 650 °C was performed to determine the amount of strain applied to Ti-6Al-4V HT pre-straining, followed by RT tensile tests.

Figure 5.57 shows the combined stress-strain curve of Ti-6Al-4V class 2 tensile test results. For class 2 materials, 11% straining at 430 °C leads to significant decrease of RT total elongation from 21.6% to 9.7% compared to Ti6-4 grade 23 sample without pre-straining. But there is an increase of ultimate tensile strength (UTS) to 1353 MPa due to strain hardening.

Summary of mechanical properties for all tensile tests of Ti6-4 is listed in Table 5.4. Some data error can be seen in Figure 5.55 and 5.56, and they should be used with caution. For example, slippage of extensometer is observed in the sample pre-strained at 250 °C and 430 °C. The actual elongation at failure is not recorded for HT tensile tests at 250 °C and 650 °C due to exceeding the measurement limit of extensometer after incorrect set-up.
Table 5.4: Summary of mechanical properties of Ti-6Al-4V alloys high temperature (HT) tensile tests (TT) and HT straining, followed by RT

<table>
<thead>
<tr>
<th>Sample</th>
<th>Ultimate TS (MPa)</th>
<th>Yield Strength (MPa)</th>
<th>Elongation %</th>
</tr>
</thead>
<tbody>
<tr>
<td>MSE Grade 23 RT TT</td>
<td>965</td>
<td>897.1</td>
<td>21.6</td>
</tr>
<tr>
<td>MSE Grade 23 650°C HT TT</td>
<td>304</td>
<td>277</td>
<td>&gt;13.4</td>
</tr>
<tr>
<td>MSE Gr 5 250°C HT TT</td>
<td>900.3</td>
<td>817.5</td>
<td>&gt;10.4</td>
</tr>
<tr>
<td>MSE Gr 5 5% str 250°C, RT TT</td>
<td>187.0</td>
<td>187.0</td>
<td>3.5</td>
</tr>
<tr>
<td>MSE Gr 5, 430°C HT TT</td>
<td>683.1</td>
<td>648.7</td>
<td>13.4</td>
</tr>
<tr>
<td>MSE Gr 5 10% str 430°C, RT TT</td>
<td>185.5</td>
<td>185.5</td>
<td>1.6</td>
</tr>
<tr>
<td>MSE Gr 5 10% str 650°C, RT TT</td>
<td>359.0</td>
<td>328.3</td>
<td>3.9</td>
</tr>
<tr>
<td>NIST Cls 2 430 °C HT TT</td>
<td>789</td>
<td>675</td>
<td>16.6</td>
</tr>
<tr>
<td>NIST Cls 2 11% str 430°C, RT TT</td>
<td>1353</td>
<td>1335</td>
<td>9.7</td>
</tr>
</tbody>
</table>
Figure 5.55: Ti-6Al-4V alloys grade 5 tensile testing results.
Figure 5.56: Ti-6Al-4V alloys grade 23 tensile testing results.
Figure 5.57: Ti-6Al-4V Class 2 tensile testing results.
5.5 Summary of Ti-6Al-4V alloys

Based on the preliminary work conducted by Nick Kullman, additional testing and analysis were done to commence the study on hot induction bending of Ti-6Al-4V alloys. New methodology of evaluating the effect of bending process parameters was also developed with the use of Gleeble™ thermo-mechanical simulator.

Temperature distribution tests were performed in Gleeble™ to evaluate the effect of resistant heating on temperature gradient along the sample, as it will affect strain levels experienced across the material. Two temperatures and three samples with different dimensions were utilized. Standard tensile test samples exhibit the widest temperature gradient. Around 14-27°C difference is found at 1/6” away from center. Hot ductility samples with diameter ¼” are determined to be the best for Gleeble™ testing with only 1-2 °C difference at 1/6” from center, followed by 12-15°C difference at 1/3” from center and 14-19°C in between ½” and center of sample. A gauge length of 10 mm is selected for straining tests that use extensometer.

Metallographic characterization on longitudinal sections using light microscope and fractography analysis with SEM were conducted on some hot ductility test samples to investigate the effect of temperature and extension rate on microstructure and formation of strain induced porosity. It shows that void formation is dependent on temperature and strain applied but not on extension rate. The maximum amount of strain-induced pores is formed in between the α to β transformation starting temperature and the recrystallization temperature. Decreasing number of void is observed in samples below the α to β transformation temperature. No void is found in Ti-64 sample tested at about β-transus
temperature at 989°C. Therefore, hot induction bending of Ti6-4 alloys in range of 795 - 890 °C is not practical as it has high risk of cracking.

High temperature straining test at Gleeble™ was newly developed as a technique to study the effect of strain applied to the material’s microstructure, formation of crack initiation sites and high temperature mechanical properties. At 650 °C, strain-induced porosity begins to form above ultimate tensile strength (UTS) as alpha grains deform and lose their original morphology. But no void is found at 430 °C until failure. Amount of strain has stronger effect at higher temperature below α to β transformation temperature. Thus, bending at 430 °C for Ti-64 alloys is recommended.

Lastly, additional high temperature straining followed by room temperature (RT) tensile tests were conducted to evaluate the effect of induction bending on the material’s RT mechanical behavior. High temperature tensile tests were first performed to determine amount of pre-straining: 5% at 250 °C; 11% at 430 °C and 10% at 650°C. It shows that straining at various high temperatures leads to decrease of RT total elongation compared to base metal sample without pre-straining., Effect of hot induction bending at 430 °C results in significant increase of UTS for Ti6-4 class 2 sample, but a decrease of strength for grade 5 material. For testing at 650 °C and 250 °C, the latter temperature leads to reduction of UTS at a greater extent compared to the higher testing temperature. In conclusion, hot induction bending of Ti6-4 might cause adverse effect on RT mechanical properties that limit its applicability and productivity.
Chapter 6: Results and discussion of Armox 440 armor steels

This chapter presents experimental results and analysis discussion of Armox 440 armor steels. Testing conducted on Armox 440 steels include temperature distribution testing in Gleeble™ with samples of different dimensions. The purpose was finding temperature difference between center and various locations along the sample, which can affect strain distribution.

Additional hot ductility tests were performed in order to broaden the range of data recorded initially by Nick Kullman [3]. Similar to Ti-6Al-4V alloys, high temperature straining tests were performed in the Gleeble™ for Armox 440 to evaluate the effect of strain and temperature on void formation, microstructure, and mechanical properties. Several tests were conducted at Oak Ridge National Laboratory for measurement of true strain data using a digital image correlation system (DIC). The objective was to generate local strain history along the sample due to non-uniform temperature distribution and correlate mechanical data with void formation at specific locations. All Gleeble™ tested samples were characterized by optical microscopy along longitudinal section and some by fractography by SEM.

High temperature straining, followed by room temperature tensile tests were conducted in order to study the effect of induction bending on room temperature mechanical behavior of Armox 440 steels. Amount of strain applied at elevated temperatures was determined by
performing high temperature tensile tests. Some of these tensile tested samples were then characterized in cross section by optical microscope and SEM for fracture surface analysis.

Tempering response of Armox 440 was investigated by developing relationship between Hollomon-Jaffe parameters and hardness of heat-treated samples at various temperatures and holding time in light radiation furnace. Some samples were subjected to metallographic characterization under optical microscope.

6.1 Temperature Distribution Testing

As mentioned in chapter 5.1, the resistance heating mechanism of Gleeble™ can lead to temperature gradient along longitudinal direction of a sample. Mid-point of sample will be at highest temperature, where control thermocouples are welded. Temperature then gradually decreases on both sides as it is further away from center. During HT straining, difference in temperature can lead to various strain levels along the sample. This would affect analysis of microstructure, void formation and mechanical behavior as they might not correspond to the tested temperature and strains applied.

Similar set of experiments applied to Ti-6Al-4V is tested for Armox 440 steel. Three samples with different dimension, shown in Figure 5.1, were tested at two temperatures (i.e. 650°C and 880°C) respectively. These samples were 1/4” diameter in cylindrical shape with threads on both ends, 3/8” diameter of cylindrical rod, as well as standard tensile samples, i.e. 1 1/4” in length reduced section of 1/4”-diameter at center with 3/8” diameter shoulders. These temperatures were chosen according to $A_1$ and $A_3$ temperature of Armox 440. The samples were heated at a rate of 10 °C/s. Three to four thermocouples (TC) were attached to sample at different distance away from center. Plots of temperature versus time
were shown in the following pages, together with a schematic diagram of sample at top right corner of each plot. A table (Table 6.1) was also generated to show degree of temperature difference between each point and center of samples with different dimensions. The objective of temperature distribution testing is to investigate the possible effect of sample dimension and geometry on temperature gradient along longitudinal direction after heating. The one that maintains relatively uniform thermal profile will be the best sample to apply for Gleeble™ testing that involves heating and loading cycles.

Figure 6.1: Plot of temperature vs time of Armox 440 HDT sample at 650 °C.
Figure 6.2: Plot of temperature vs time of Armox 440 HDT sample at 880 °C.

Figure 6.3: Plot of temperature vs time of Armox 440 standard tensile sample at 650 °C.
Figure 6.4: Plot of Temperature vs Time of Armox 440 standard tensile sample at 880 °C.

Figure 6.5: Plot of Temperature vs Time of Armox 440 3/8” rod sample at 650 °C.
Table 6.1: Temperature difference between center and other thermocouples located along the sample

<table>
<thead>
<tr>
<th>Sample dimensions/ Temperature difference</th>
<th>Temperature (°C)</th>
<th>TC3 1/6” from center</th>
<th>TC2 1/3” from center</th>
<th>TC4 1/2” from center</th>
</tr>
</thead>
<tbody>
<tr>
<td>650</td>
<td>1</td>
<td>26</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>880</td>
<td>6</td>
<td>27</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>650</td>
<td>14</td>
<td>40</td>
<td>93</td>
<td></td>
</tr>
<tr>
<td>880</td>
<td>16</td>
<td>55</td>
<td>136</td>
<td></td>
</tr>
<tr>
<td>650</td>
<td>11</td>
<td>22</td>
<td>31</td>
<td></td>
</tr>
<tr>
<td>880</td>
<td>10</td>
<td>11</td>
<td>39</td>
<td></td>
</tr>
</tbody>
</table>

Figure 6.6: Plot of Temperature vs Time of Armox 440 3/8” rod sample at 880 °C.
Thermocouples were located at the center (TC1), 1/6” (TC3), 1/3” (TC2) and half inch (TC4) away from center. Experimental results revealed that Gleeble™ heating at 650°C, HDT samples with ¼” diameter and threads at both ends, has only 1°C temperature difference between center and the point at 1/6” from center. At point of 1/3” from center, temperature difference increases to 26°C compared to mid-point. For standard tensile samples with 3/8” shoulders and 1.25” in length of ¼” diameter reduced section, they have temperature gradient of 14°C between center and the nearest thermocouples location. In addition, temperature difference of center and the point at 1/3” from center for tensile samples increases significantly to 40 °C, almost three times the different between center and TC3. Then, temperature further drops with a degree of 93 °C as the thermocouples are located at ½” from center, which is more than two times the difference between TC1 and TC2. Rod samples in 3/8” diameter exhibits smaller degree of temperature drop of 11 °C from center to the point of 1/6” from center. Between TC1 and TC2, there is 22°C temperature difference and is two times of that between center and the closest thermocouples. Between TC1 and TC4, temperature difference is 31 °C.

As heating temperature goes up to 880°C, HDT samples show 6 °C temperature difference between center and TC3, followed by a difference of 27 °C between center and the point at 1/3” from center. Standard tensile samples display 16 °C difference between center and the closest thermocouples location. Further away at 1/3” from center, temperature decreases rapidly with an extent of 55 °C, which is more than three times the temperature difference between TC1 and TC3. Comparing temperature at center and ½” from center, it has the biggest difference of 136 °C. For rod samples in 3/8” diameter, temperature at 1/6” from center is 10 °C lower than the tested temperature at center.
Thermocouples at center and 1/3” from center show temperature difference of 11 °C. At ½” away from center, there is a 39 °C temperature drop compared to center, which is more than three times the temperature difference between TC1 and TC2.

According to the results discussed above, in general all samples show higher degree of non-uniform temperature distribution at higher temperature (i.e. 880 °C) independent of sample dimensions, which is similar to the results of Ti-6Al-4V alloys. Comparing three dimensions, ¼” diameter HDT samples have relatively most uniform temperature distribution. The width of isothermal zone is about 2/3” at center region, as temperature drops within 6 °C or less, which allows us to investigate sample microstructure at desire tested temperature and strains applied. It is followed by more rapid decline of temperature between TC3 and TC2, which allows most load and strain to concentrate in central region and provides mechanical behavior with higher accuracy. Such pattern can also be observed in Ti-6Al-4V HDT samples. Compared to HDT samples, 3/8” rod samples do not have an isothermal region. Temperature drops steadily by a degree of about 10 °C from center to each point of thermocouples, except the one half inch away from center when heated up to 880 °C. This might be due to larger cross-sectional area. Standard tensile samples of Armox 440 have the most non-uniform temperature distribution. Because of the presence of reduced section, difference in cross sectional area between center region and two shoulders lead to significant temperature drop of 40 – 55 °C even for the nearest thermocouples. Huge difference in temperature leads to greater variation in strain levels experienced by various points along sample. Mechanical data and microstructural change collected might not correspond to the preset temperature and strains applied. Therefore, standard tensile samples with reduced section are not recommended for use in Gleeble™ high temperature
testing. Comparing overall results of Armox 440, it is found that resistant heating mechanism of Gleeble™ has more adverse effect on high strength steels samples than Ti-6Al-4V alloys. Temperature difference between center and other points along sample is greater for Armox 440 regardless of sample dimensions. This is due to difference in heat transfer coefficient of the two materials. As testing temperatures for Armox 440 are generally in higher range than Ti alloys, it is necessary to be aware of the effect of temperature gradient present. HDT samples with ¼” diameter are selected as Gleeble™ Armox 440 testing samples and a gauge length of 10mm is chosen for high temperature straining tests, as they are the only samples with uniform temperature distribution region in center.

6.2 Hot Ductility Testing

Hot ductility tests were performed on Armox 440 plate materials by Nick Kullman in the Gleeble™ thermo-mechanical simulator in temperature range of 500-1080 °C at an extension rate of 10 mm/s with copper grips. The results were compared with standard HHA steel and RHA steel. Additional tests were conducted in this research project in temperature range of 300-450 °C in order to obtain a wider range of data. Two tests at 300°C were conducted by Dr. Ivan Mendoza. The goal of these tests is to determine the ductility property of Armox 440 at elevated temperatures, which is one of the important factors for developing the optimal process control windows for hot induction bending. Figure 6.7 displays the plot of reduction-in-area against temperature of Armox 440 at 10 mm/s extension rate. Samples were heated at a rate of 10 °C/s and held for 6 minutes. Stainless steel grips was used in this work for more uniform temperature distribution along
sample. Test chamber was under rough vacuum atmosphere at around 2.8- 3.2 x 10⁻² torr. Cross-section area was measured before and after test for calculating reduction-in-area as an indication of material ductility. Table 6.2 exhibits a summary of hot ductility test temperatures and reduction-in-area data of Armox 440 tested in this project.

At range of 300- 450 °C, reduction-in-area (RA) increases gradually, which indicates increasing ductility. Some of the hot ductility Armox 440 samples (i.e. circled in Figure 6.7) were characterized by fractography using SEM and hardness testing by Nick Kullman. Five samples were further investigated by longitudinal sectioning and metallographic characterization under light optical microscope (LOM) to focus on microstructural changes close to fracture surface and void formation.

Table 6.2: Summary of Armox 440 hot ductility test results

<table>
<thead>
<tr>
<th>Testing Temperature (°C)</th>
<th>Reduction-in-area (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>300</td>
<td>57.49</td>
</tr>
<tr>
<td>300</td>
<td>59.40</td>
</tr>
<tr>
<td>350</td>
<td>78.19</td>
</tr>
<tr>
<td>400</td>
<td>80.11</td>
</tr>
<tr>
<td>450</td>
<td>81.18</td>
</tr>
</tbody>
</table>
6.2.1 Metallurgical Characterization

Micrographs of longitudinal sections in five chosen samples, circled in Figure 6.7, are presented in Figure 6.8- 6.12. These are Armox 440 hot ductility samples tested at 1080 °C, 880 °C, 800 °C, 650 °C, and 500 °C respectively at 10 mm/s extension in rough vacuum. The left image is taken at magnification of x500 and right picture is taken at same magnification using differential interference contrast (DIC). All samples were etched with 2% Nital etchant, which is a general etchant sensitive to crystal orientation for revealing martensite, ferrite in martensite matrix and bringing up ferrite grain boundaries [109]. Ferrite phase and retained austenite are usually in white. Martensite is in brown color.
Base metal microstructure of Armox 440 was characterized by Nick Kullman, which consists of a tempered martensitic structure after quench and temper processing. Based on SS-DTA, $A_1$ temperature of Armox 440 steel is determined as 700 °C and $A_3$ temperature is at 858 °C.

The longitudinal section (LS) of HDT sample tested at 1080 °C at 10 mm/s extension rate is shown in Figure 6.8, which is far above the $A_3$ temperature when austenite phase is completely transformed from ferrite. It has the greatest reduction-in-area (98.54%). It consists of fine needles in brown, which are newly formed martensite structure upon cooling. No void is observed in sample, indicating lower risk of crack formation. Figure 6.9 displays LS of Armox 440 sample tested at 880 °C, which is higher than the estimated $A_3$ temperature. Reduction-in-area (RA) is about 94.53%. It is found that prior austenite grain size has decreased significantly due to recrystallization. Same as previous sample, no void is formed when straining to failure at this temperature.

LS micrographs of Armox 440 tested at 800 °C is shown in Figure 6.10, which is in between $A_1$ and $A_3$ temperature. RA is approximately 89.96%. New austenite starts to form but transformation is not completed yet. Therefore, it consists of a mix of constituents, with fresh martensite and tempered martensite. Grains are refined and slightly elongated along the straining direction. Voids are found close to fracture surface due to local stress concentration. This sample consists the highest number and relatively larger voids among observed ones. Most of them are slightly elongated to form tear shape along longitudinal direction and some are irregular. Some voids have coalesced to form larger defects with diameter up to 100 μm.
Figure 6.11 reveals the cross-section of Armox 440 sample tested at 650 °C, which is below A₁ temperature and no austenite is formed upon heating. This is equivalent to tempering treatment, which heats and holds at a temperature below A₁. Therefore, its microstructure consists of over-tempered martensite. Due to reheating of tempered martensite in base metal, it further softens. RA is about 83.09%. Grain morphology changes greatly and the microstructure is elongated along the tensile stress direction. Voids can be observed in this sample but in fewer amount compared to the sample heated above A₁. Most of them are located right beneath the fracture surface and have an average size of 20 μm. Coalescence of microvoids can be observed in center region. Shape of larger voids is affected by straining and elongated along pulling direction.

LS images of Armox 440 tested at 500 °C are shown in Figure 6.12, which is further below A₁ temperature. It has the lowest RA at about 78.01%). Similar to previous sample, its microstructure mainly consists of over-tempered martensite, with grains elongated along straining direction and lost their original shape. The amount of voids present is slight less than that at 650 °C. Most of them have an average size of 20 μm that are relatively round or tear shaped. There are some particular huge and irregular voids formed by coalescence, which are located in central region or in between edge and bottom of cup fracture surface with highest stress concentration.

One issue of Armox steel sample preparation and metallography analysis is pitting corrosion. This steel material is very vulnerable to corrosion attack when samples are polished with running water and cleaning with ethanol. Even samples are etched with mild solution, pitting regions are first attacked and easily over-etched before other phases are revealed. These over-etched regions then lead to difficulty of phase’s characterization and
identification of voids caused by high temperature straining to failure. In order to distinguish the difference between voids and pits, size, etching effect and shape of these features are important factors. In general, pits are small in size less than 10 μm in diameter, round in shape and over-etched surrounding with brown stains. On the other hand, voids formed are usually larger in size, lower risk of being over-etched and contain a variety of shapes depending on testing parameters.

Figure 6.8: Microstructure of HDT sample of Armox 440 plate tested at 1080°C with 10mm/s extension rate.

Figure 6.9: Microstructure of HDT sample of Armox 440 plate tested at 880°C with 10mm/s extension rate.
Figure 6.10: Microstructure of HDT sample of Armox 440 plate tested at 800°C with 10mm/s extension rate.

Figure 6.11: Microstructure of HDT sample of Armox 440 plate tested at 650°C with 10mm/s extension rate.

Figure 6.12: Microstructure of HDT sample of Armox 440 plate tested at 500°C with 10mm/s extension rate.
6.2.2 Summary of Hot Ductility Testing

Hot ductility testing of Armox 440 at 10 mm/s extension rate shows a sharp increase of ductility from 300 °C to 350 °C. After that, ductility of Armox 440 continues to go up until reaching 1080 °C with reduction-in-area very close to 100%.

Metallographic characterization shows that void formation is dependent on temperature applied on samples. The maximum amount of voids is found in sample tested at 800 °C, which is formed in between A1 and A3 temperature (i.e. 700 °C and 858 °C). This is due to the effect of austenite phase transformation that is not yet completed. There is possible void nucleation at austenite/martensite phase boundaries. Therefore, hot induction bending is not recommended within the temperature range as a mix of phases is developed. There is no void formation for samples tested above A3 temperature, when austenite is fully formed. Samples that are tested below A1 temperature also lead to void formation, but possess fewer amount compared to the sample within A1 and A3.

6.3 Gleeble™ High Temperature Straining Testing

Similar to study of Ti-6Al-4V alloys, Gleeble™ high temperature straining tests were conducted on Armox 440 to study the effect of strain at elevated temperatures on changes of microstructure, void formation and mechanical properties. Four temperatures were selected to perform the tests, i.e. 500 °C, 600 °C, 650 °C, and 880 °C. The former two were chosen based on the tempering response study, which will be discussed in later section. The latter two were based on A1 and A3 temperature. Test cycle is similar to the one shown in Figure 5.31 for Ti-6Al-4V. All samples were first heated at a rate of 10 mm/s and held at tested temperature for about six minutes under rough vacuum. A total of five tests were
conducted at each temperature. The first test is high temperature tensile test, in which the sample was subjected to failure. Engineering stress-strain curve is generated at four temperatures as shown in Figure 6.13. By analyzing the complete tensile test curve, amount of strain experienced at yield strength (YS), in between YS and ultimate tensile strength (UTS), at UTS and above UTS can be identified with corresponding strokes applied, which are circled points in Figure 6.13. Changing the strokes for each test can change the strain applied on samples. The LS of these samples were then observed under optical microscope, as shown in Figure 6.14- 6.17 for 500°C, Figure 6.18- 6.21 for 600 °C, Figure 6.22-6.26 for 650 °C and Figure 6.27-6.31 for 880 °C. Subsection 6.3.2 discusses the mechanical behavior during high temperature straining tests in Gleeble™ and compares the results at two testing temperatures.

Figure 6.13: High temperature tensile tests of Armox 440 at 500/600/650/880 °C.
6.3.1 Cross-section Metallurgical Characterization

Armox 440 originally possesses tempered martensite structure as it is quenched and tempered. When it is heated to 500 °C below the lower critical temperature and is subjected to amount of strain that reaches between YS and UTS at 1 mm/s extension rate, the sample shown in Figure 6.14 displays spheroidized carbides due to effect of tempering and refined ferrite grains in average size of approximately 10 µm. The cross-section area of sample reduces uniformly by only about 6%. The amount of strain does not have significant effect on microstructure and grain morphology. As more strains is applied to the sample and reaches UTS, similar but slight coarser microstructure is observed in Figure 6.15. Figure 6.16 shows the sample strained to above UTS, in which necking occurs and reduction-in-area increases significantly to about 42%. Grains are elongated along the tension force in longitudinal direction. As Armox 440 is strained to failure at 500 °C in Figure 6.17, martensite structure is completely deformed. All globular features have disappeared and only a few voids are formed right under fracture surface with about 10-12 µm in diameter. These voids are irregular in shape and are developed due to local stress concentration.

When Armox 440 is subjected to straining in between YS and UTS at 600 °C below A₁ temperature, as shown in Figure 6.18, similar microstructure can be observed in the sample tested at 500 °C as the tempering effect also applies. A slightly higher reduction-in-area of roughly 10 % is also recorded. As the amount of strain increases to reach UTS as shown in Figure 6.19, no significant change in grain morphology can be seen compared to previous sample. The sample tested until necking occurs consists of deformed grains elongated in the straining direction. For the failure sample as shown in Figure 6.21, grains are more
deformed compared to the sample tested at 500 °C due to degree of softening. Only a few voids are found within 10 µm in diameter and microvoid coalescence can be observed.

At 650 °C below A1 temperature, similar microstructure can be found in samples strain to YS and between YS and UTS, as shown in Figure 6.22 and Figure 6.23. They consist of spheroidized carbides and refined ferrite grains, but coarser structure compared to the samples at lower temperatures. When sample reaches UTS, grain morphology starts to be effected by the amount of strain. Some grains are deformed and elongated in longitudinal direction, as shown in Figure 6.24. As necking occurs, more are deformed to greater extent. When sample reaches failure in Figure 6.26, the grains lose their morphology and more voids are formed because of stress accumulation. Some of these voids are irregular or elongated in straining direction with a length greater than 20 µm.

When Armox 440 is tested at 880 °C above the upper critical temperature, fresh martensite is formed on cooling to room temperature. As the sample reaches YS in Figure 6.27, grains are refined with average size of less than 20 µm and are even more globular compared to those found in samples tested at 500 °C because of recrystallization. For samples tested between YS and UTS and strained to UTS, similar microstructure is observed as the sample in Figure 6.27. As Armox 440 reaches above UTS and starts necking in Figure 6.30, grains are more refined compared to the samples experienced lower strain. No deformation can be seen that leads to grain elongation. When sample is tested to failure, as shown in Figure 6.31, the spheroidized grains completely lose their morphology and no distinct grain features is found. In addition, there is no void found in the five samples tested at 880 °C.
Figure 6.14: Armox 440 strained to between YS and ultimate tensile strength (UTS) level at 500°C.

Figure 6.15: Armox 440 strained to UTS level at 500°C.

Figure 6.16: Armox 440 strained to above UTS level at 500°C.
Figure 6.17: Armox 440 strained to failure at 500 °C.

Figure 6.18: Armox 440 strained to between YS and UTS level at 600°C.

Figure 6.19: Armox 440 strained to UTS level at 600°C.
Figure 6.20: Armox 440 strained to above UTS level at 600°C.

Figure 6.21: Armox 440 strained to failure at 600°C.

Figure 6.22: Armox 440 strained to YS level at 650°C.
Figure 6.23: Armox 440 strained to between YS and UTS level at 650°C.

Figure 6.24: Armox 440 strained to UTS level at 650°C.

Figure 6.25: Armox 440 strained to above UTS level at 650°C.
Figure 6.26: Armox 440 strained to failure at 650°C.

Figure 6.27: Armox 440 strained to YS level at 880°C.

Figure 6.28: Armox 440 strained to between YS and UTS level at 880°C.
Figure 6.29: Armox 440 strained to UTS level at 880°C.

Figure 6.30: Armox 440 strained to above UTS level at 880°C.

Figure 6.31: Armox 440 strained to failure at 880°C.
6.3.2 Comparison of Mechanical Properties

Mechanical data of Armox 440 high temperature straining tests at Gleeble™ were also analyzed. They can be used as the parameters in hot bending process, such as the strain and flow stress applied at specific temperatures without void formation. Table 6.3 to 6.6 show summaries of mechanical properties, including stroke applied, engineering stress, engineering strain, elongation, reduction-in-area (RA) and true strength after analyzing the stress-strain curves of Gleeble™ tests at various strain at four different temperatures. Samples that display voids are also indicated in the summary table. A series of five engineering stress-strain curves with increasing amount of strain until failure that were tested at 500 °C, 600 °C, 650 °C and 880 °C are combined in one plot and displayed in Figure 6.32 to 6.35 respectively.

In terms of RA, it is observed that increasing temperature leads to increasing percentage of area reduction as the amount of strain increases. The range of RA at 500 °C starts from 5.56% for sample reaching YS to 79.86% for sample strained to failure. The range of RA is slightly higher at 600 °C, ranging from 9.17% to 85.48%. RA of the three samples with fewer strains at 650 °C are roughly the same at about 1%. Then, it increases rapidly to 89.34% as the sample fails. Testing at 880 °C has a wide range of RA, which is from 0.60% for the sample at YS up to 96.32% for the sample tested to failure.

As for total elongation, testing conducted below A₁ temperature are within a narrow range of values. At 500 °C and 600 °C, elongation at failure is 35.90% and 34.72 % respectively, which is approximately the same. It increases slightly to 39.62% as testing temperature increases to 650 °C.
In terms of yield strength, testing performed below lower critical temperature have decreasing magnitude as temperature increases from 500 °C to 650 °C, which decreases from 676.7MPa down to 381.5MPa. Same trend can be observed in tensile strength properties. The ultimate tensile strength at 500 °C is 851.78MPa, reducing to 646.38MPa at 600 °C and further decreasing to 518.9MPa at 650 °C. This might be due to degree of softening as temperature increases, which reduce the strength but increase ductility of Armox 440 at elevated temperatures.

At 880 °C above A₃ temperature, original tempered martensite structure is fully transformed to austenite phase with high carbon content. This significantly reduces strength of Armox 440 steel, which results in the lowest values compared to samples tested below A₁ temperature. Yield strength of 78.66 MPa is recorded and the ultimate tensile strength is 134.4 MPa.

Table 6.3: Mechanical properties of Armox 440 tested at 500 °C with various strain levels

<table>
<thead>
<tr>
<th>500 °C</th>
<th>Stroke (mm)</th>
<th>Engr. Stress (MPa)</th>
<th>Engr. Strain (mm/mm)</th>
<th>Elongation (%)</th>
<th>RA (%)</th>
<th>True Strength (MPa)</th>
<th>Voids (Y/N)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Yield Strength</td>
<td>2.61</td>
<td>676.67</td>
<td>0.004943</td>
<td>0.4943</td>
<td>5.561</td>
<td>716.51</td>
<td>N</td>
</tr>
<tr>
<td>Between YS and TS</td>
<td>3.33</td>
<td>808.22</td>
<td>0.01092</td>
<td>1.092</td>
<td>5.836</td>
<td>858.31</td>
<td>N</td>
</tr>
<tr>
<td>Tensile Strength</td>
<td>3.8</td>
<td>851.78</td>
<td>0.01503</td>
<td>1.503</td>
<td>8.556</td>
<td>931.47</td>
<td>N</td>
</tr>
<tr>
<td>Above Tensile Failure</td>
<td>4.93</td>
<td>612.90</td>
<td>0.1750</td>
<td>17.50</td>
<td>42.29</td>
<td>1061.97</td>
<td>N</td>
</tr>
<tr>
<td>Failure</td>
<td>6.86</td>
<td>336.34</td>
<td>0.3590</td>
<td>35.90</td>
<td>79.86</td>
<td>1670.06</td>
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Table 6.4: Mechanical properties of Armox 440 tested at 600 °C with various strain levels

<table>
<thead>
<tr>
<th>600 °C</th>
<th>Stroke (mm)</th>
<th>Engr. Stress (MPa)</th>
<th>Engr. Strain (mm/mm)</th>
<th>Elongation (%)</th>
<th>RA (%)</th>
<th>True Strength (MPa)</th>
<th>Voids (Y/N)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Yield Strength</td>
<td>2.04</td>
<td>508.82</td>
<td>0.004103</td>
<td>0.4103</td>
<td>9.173</td>
<td>560.21</td>
<td>N</td>
</tr>
<tr>
<td>Between YS and TS</td>
<td>2.50</td>
<td>580.68</td>
<td>0.004750</td>
<td>0.4750</td>
<td>9.675</td>
<td>642.88</td>
<td>N</td>
</tr>
<tr>
<td>Tensile Strength</td>
<td>2.80</td>
<td>646.38</td>
<td>0.01412</td>
<td>1.412</td>
<td>10.85</td>
<td>725.06</td>
<td>N</td>
</tr>
<tr>
<td>Above Tensile</td>
<td>4.50</td>
<td>436.41</td>
<td>0.1499</td>
<td>14.99</td>
<td>46.35</td>
<td>813.49</td>
<td>N</td>
</tr>
<tr>
<td>Failure</td>
<td>6.41</td>
<td>204.43</td>
<td>0.3472</td>
<td>34.72</td>
<td>85.48</td>
<td>1408.12</td>
<td>Y</td>
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Table 6.5: Mechanical properties of Armox 440 tested at 650 °C with various strain levels

<table>
<thead>
<tr>
<th>650 °C</th>
<th>Stroke (mm)</th>
<th>Engr. Stress (MPa)</th>
<th>Engr. Strain (mm/mm)</th>
<th>Elongation (%)</th>
<th>RA (%)</th>
<th>True Strength (MPa)</th>
<th>Voids (Y/N)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Yield Strength</td>
<td>1.5</td>
<td>381.5</td>
<td>0.003759</td>
<td>0.3759</td>
<td>1.166</td>
<td>386.00</td>
<td>N</td>
</tr>
<tr>
<td>Between YS and TS</td>
<td>1.7</td>
<td>474.5</td>
<td>0.004523</td>
<td>0.4523</td>
<td>1.000</td>
<td>479.26</td>
<td>N</td>
</tr>
<tr>
<td>Tensile Strength</td>
<td>1.9</td>
<td>518.9</td>
<td>0.006699</td>
<td>0.6699</td>
<td>1.398</td>
<td>526.30</td>
<td>N</td>
</tr>
<tr>
<td>Above Tensile</td>
<td>2.9</td>
<td>486.4</td>
<td>0.06460</td>
<td>6.460</td>
<td>15.18</td>
<td>573.41</td>
<td>N</td>
</tr>
<tr>
<td>Failure</td>
<td>6</td>
<td>102.50</td>
<td>0.3962</td>
<td>39.62</td>
<td>89.34</td>
<td>961.39</td>
<td>Y</td>
</tr>
</tbody>
</table>
Table 6.6: Mechanical properties of Armox 440 tested at 880 °C with various strain levels

<table>
<thead>
<tr>
<th>Temperature</th>
<th>Stroke (mm)</th>
<th>Engr. Stress (MPa)</th>
<th>Engr. Strain (mm/mm)</th>
<th>Elongation (%)</th>
<th>RA (%)</th>
<th>True Strength (MPa)</th>
<th>Voids (Y/N)</th>
</tr>
</thead>
<tbody>
<tr>
<td>880 °C</td>
<td>0.35</td>
<td>78.66</td>
<td>0.001794</td>
<td>0.1794</td>
<td>0.600</td>
<td>79.14</td>
<td>N</td>
</tr>
<tr>
<td>Yield</td>
<td>3</td>
<td>125.1</td>
<td>0.08739</td>
<td>8.739</td>
<td>8.056</td>
<td>136.05</td>
<td>N</td>
</tr>
<tr>
<td>Between YS</td>
<td>4.7</td>
<td>134.4</td>
<td>0.1388</td>
<td>13.88</td>
<td>13.35</td>
<td>155.09</td>
<td>N</td>
</tr>
<tr>
<td>and TS</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Tensile</td>
<td>10</td>
<td>126.0</td>
<td>0.2532</td>
<td>25.32</td>
<td>39.44</td>
<td>208.53</td>
<td>N</td>
</tr>
<tr>
<td>Strength</td>
<td>14.5</td>
<td>41.84</td>
<td>0.5011</td>
<td>50.11</td>
<td>96.32</td>
<td>1137.6</td>
<td>N</td>
</tr>
<tr>
<td>Above</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Failure</td>
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<td></td>
<td></td>
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</tr>
</tbody>
</table>

Figure 6.32: Armox 440 engineering stress strain curves at 500 °C.
Figure 6.33: Armox 440 engineering stress strain curves at 600 °C

Figure 6.34: Armox 440 engineering stress strain curves at 650°C
6.3.3 Digital Image Correlation (DIC) analysis

As discussed in section 6.1, a non-uniform temperature distribution existed across the sample when it is treated in Gleeble™ through resistance heating. The maximum temperature is at the center where control thermocouples are located, and then gradually decreases towards either end. Different degree of softening caused by temperature gradient leads to a range of yield strength and strains experienced along the specimen, which results in non-uniform deformation [104]. In addition, as the sample is strained in axial direction until necking occurs, it deforms disproportionately. Extensometer can only measure strain between two points along one direction, which is just part of the total strain. Several HDT samples were sent to Oak Ridge National Laboratory (ORNL) to conduct high temperature straining tests at Gleeble™ with the aid of digital image correlation (DIC) system [110] by
Dr. Dongxiao Qiao. This is to better understand the effect of true strain on Armox 440 and develop relationship between void formation and strain field of sample. These tested samples were then sent back to OSU for metallographic characterization etching with 5% Nital’s solution. DIC technique can be used to trace the strain distribution in longitudinal direction of sample and obtain local strain history at any specific point. True stress vs. true strain curve can then be generated. The working mechanism of DIC is first taking consecutive digital images of the painted specimen surface by two synchronized cameras as the sample is tested in the Gleeble™ chamber. Then, the image correlation algorithm analyzes the deformation history of speckle pattern through image order and measures the surface strain field in terms of Lagrange strain [104]. Details of experimental set-up are discussed in section 4.4.2.

Four testing at ORNL include straining at 1mm/s extension rate at 650 °C and 800°C until necking occurs and to failure. Figure 6.36 displays the non-uniform strain distribution on the necking sample heat-treated at 650 °C and the right plot is true strain along axial direction versus distance of the red line in left image. The center of necking region has the maximum strain of 0.27 and decreases down to about 0.08 at 5mm from center at both sides. Figure 6.38 a) compares the engineering stress-strain curves from OSU and ORNL. Total elongation of ORNL data is about 29%, which is about four times of that tested by OSU Gleeble™. This is because the latter test measures average strain over the gauge length of 10 mm, but ORNL measures the local strain at center of gauge section. The engineering strain corresponds to UTS is 0.036 from ORNL and 0.017 for OSU data. Figure 6.37 b) displays the true stress-strain curve with total strains of 0.26. The number of data recorded for this test is half of the other samples, which might not be sufficient to
generate results with high accuracy. Figure 6.38 are LS images at the center region of corresponding sample in Figure 6.36 and 6.37. Some grains within necking area change their orientation towards straining direction. No void is found in the sample.

Figure 6.36: Strain field of Armox 440 sample necking at 650 °C.

Figure 6.37: a) Comparison of engineering stress-strain curves between ORNL (local strain) and OSU (gauge length=10mm) Gleeble™ data. b) True stress-strain curve for Armox 440 necking sample at 650°C.
Figure 6.39 reveals the strain distribution of the sample strained to failure at 650 °C. The plot at right indicates the maximum true strain at point of fracture is about 1.68. However, the actual value is not measurable and should be greater because the strain gradient at center is very large. A comparison between engineering stress-strain curves obtained from ORNL and OSU is shown in Figure 6.40. Total strain from the ORNL curve is about 2.3, which is more than 5 times of the magnitude from OSU. The strain corresponding to UTS is about 0.19 from DIC results, and 0.011 for OSU data. Figure 6.40 b) displays the true stress-strain curve with increasing true stress due to changing cross-section area. LS images near fracture surface are exhibited in Figure 6.41. A total of 6 voids that have a diameter less than 10 microns are found at the edge of failure point, which has the maximum strain.
Figure 6.39: Strain field of Armox 440 tested at 650 °C to failure.

Figure 6.40: a) Comparison of stress-strain curves between ORNL DIC (local strain) and OSU Gleeble™ (average strain) data. b) True stress-strain curve for Armox 440 sample tested at 650 °C to failure.

Figure 6.41: Armox 440 sample tested at 650°C to failure at ORNL.
The third test was straining at 800 °C in between A₁ and A₃ temperatures until necking at 1 mm/s extension rate. Its strain distribution across sample is revealed in Figure 6.42. The maximum true strain is approximately 0.71 at center of necking region. Figure 6.43 shows the engineering and true stress-strain curves generated from ORNL DIC data. The engineering strain at failure is 0.92 and true strain is about 0.64. Similar to the necking sample at 650 °C, no void is observed in Figure 6.44. The morphology of microstructure at center is not affected by the strains applied.

Figure 6.42: Local strain history of Armox 440 necking sample at 800 °C.

Figure 6.43: Engineering vs. true stress-strain curves of Armox 440 necking sample at 800 °C using ORNL DIC technique.
Figure 6.44: Armox 440 necking sample at 800 °C tested at ORNL using DIC technique.

Figure 6.45 shows the non-uniform strain distribution of Armox 440 sample strained to failure at 800 °C. The maximum true strain marked in the plot is around 2.13, similar to the failure sample at 650 °C, actual final strain is not available due to the large strain gradient at center of fracture. Engineering and true stress-train curves based on DIC data are displayed in Figure 6.46. Local engineering strain at fracture is approximately 1.13 and true strain at failure is about 0.71. As shown in Figure 6.47, the sample has a very ductile fracture with no void formation. Microstructure morphology can still be seen after complete deformation. Grain refinement is observed due to recrystallization process. No void is found in this temperature, which might be due to slower extension rate of 1mm/s was applied.
Figure 6.45: Local strian history of Armox 440 tested at 800 °C to failure.

Figure 6.46: Engineering vs. true stress-strain curves of Armox 440 tested at 800 °C to failure.

Figure 6.47: Armox 440 tested at 800 °C to failure at ORNL using DIC technique.
A relationship between local strain and void formation is also developed for the sample tested to failure at 650 °C using digital image correlation technique (DIC). Six voids are observed near the fracture center with maximum true strain of about 1.68. The local strain data obtained by DIC can also be compared with the data of average strain over 10mm gauge length from the Gleeble™ for testing at 650°C. As Armox 440 is strained to above UTS level at 650°C, the maximum local strain at center is about 0.27, while the average strain is approximately 0.064. When the sample is strained to failure at the same temperature, the local strain at fracture measured by DIC is 2.3. It is more than five times the average strain measured by the Gleeble™ (i.e. 0.40). As there is a temperature distribution along the sample, the local strain obtained by DIC at ORNL can provide more precise strain data at the specific testing temperature.
6.4 Tensile Testing

A set of high temperature (HT) straining, followed by room temperature (RT) tensile testing was performed to evaluate Armox440 response to hot induction bending in terms of RT mechanical behavior. Similar to the testing procedures for Ti-6Al-4V alloys, high temperature tensile tests at a rate of 1mm/s were utilized first to determine the amount of strain applied to the sample. The results of Gleeble™ high temperature straining testing were also considered to avoid application of excessive strain that might result in void and cracking formation. Four temperatures were examined in the study, i.e. 650 °C below A1 temperature, 880 °C above A3 temperature, 500 °C and 600 °C, which were added later based on tempering response study. All samples were cooled down by fan before RT tensile testing. Majority of testing were completed at National Institute of Standards and Technology (NIST) with the assistance of Dr. Jeff Sowards. Two tensile tests were done at Ohio State Material Science & Engineering department facility. The rest of RT tensile testing were performed at Miami Valley Materials Testing Center (MVMTC).

Figure 6.48 shows the combination plot of Armox 440 pre-strain data at elevated temperatures. It indicates the amount of pre-strain applied as total elongation at each temperature. For room temperature pre-straining, 4-5 % of strain was applied. For the remaining tests of pre-straining at high temperatures, about 11% of strain was applied. It was based on the result of high temperature straining tests at Gleeble™, indicating no void formation as Armox 440 is strained up to above UTS level.

Figure 6.49 displays the combined stress versus elongation curves for RT tensile tests of RT pre-strained and not pre-strained samples. At RT without pre-straining, Armox 440 has ultimate tensile strength (UTS) in range of 1446- 1489 MPa and yield strength (YS)
ranges from 1131 -1193 MPa. Total elongation at failure is about 21.7%. These data are then compared with other RT tensile test results after HT straining. The RT tensile graph with multiple point of slippage might consist of experimental error and should be used with caution. Two RT pre-strain samples result in an increase of UTS to 1593- 1628 MPa but reduction of total elongation from about 21.7 to 15.0 %, which might be due to strain-hardening.

Results of HT tensile testing and HT 11% straining followed by RT tensile testing at 500 °C are exhibited in Figure 6.50. Each test was repeated once. It shows that the UTS of HT tensile test and RT tensile test after HT pre-straining have little difference. Yield point phenomenon can be observed in HT pre-strained samples, which represents a localized and heterogeneous transition to plastic deformation from elastic region [111]. The upper yield strength is in between 867- 869 MPa and lower yield strength is in range of 854-857 MPa. There is a sharp drop of UTS to about 869 MPa compared to RT sample without pre-straining and the yield/tensile ratio is almost equal to 1. Total elongation at fracture has the maximum decrease among HT pre-strained samples at various temperatures, which is reduced to 6.94 -7.18%.

Figure 6.51 displays the combined results of tensile testing at 600 °C (below A1 temperature). HT pre-straining, followed by RT tensile testing was repeated twice because the second test (green curve) resulted in different mechanical behavior change. Since the reason is not known, this test is not used for analysis. However, the first test has repeatable result as in the third test. Similar to testing at 500 °C, yield point phenomenon is also found at 600 °C, which is caused by the presence of interstitial impurities pinning the increasing population of dislocations during plastic deformation [111]. Lower yield strength is in
range of 643.1 to 742.9 MPa and upper yield strength is about 749 MPa. UTS at RT has
the greatest decrease from 1489 MPa without pre-straining down to 790.3 -797.5 MPa,
which might be due to over-temperering and coarsening of microstructure.

Figure 6.52 shows the results of tensile testing at 650 °C conducted by Dr. Jeff Sowards
at NIST. The green curve represents HT tensile testing performed at OSU at a different
straining rate. Total elongation was not measurable due to lack of equipment and improper
set up of extensometer. Yield point phenomenon is observed in RT tensile test after
strianing below A1 temperature. Comparing the RT mechanical properties of Armox 440
without pre-strains and with 11% pre-straining at 650 °C, UTS reduces to around 1016
MPa due to over-temperering, but it is higher than the pre-strained samples at 500 °C and
600 °C. Similar trend is also observed in elongation at failure. It decreases to 9.47 % and
is greater than that of pre-strained Armox 440 at lower temperatures.

Tensile testing results at 880°C are revealed in Figure 6.53. After pre-straining above
A3 temperature, it exhibits the closest UTS and total elongation at RT compared to the
sample without straining. The two properties slightly reduce to 1334 MPa and 14.2%
respectively and are at maximum compared to other pre-strained samples at lower
temperatures. This is due to complete austenite transformation at elevated temperature that
leads to some martensite formation upon cooling. A summary of mechanical properties of
HT tensile testing and HT straining followed by RT tensile testing of Armox 440 are
displayed in Table 6.7 and 6.8.
Figure 6.48: Armox 440 tensile testing prestrain data.
Figure 6.49: Armox 440 room temperature tensile tests.
Figure 6.50: Armox 440 500°C tensile tests.
Figure 6.51: Armox 440 600 °C tensile tests.
Figure 6.52: Armox 440 650°C tensile tests.
Figure 6.53: Armox 440 880°C tensile tests.
Table 6.7: Summary of mechanical properties of Armox 440 high temperature tensile tests

<table>
<thead>
<tr>
<th>Temperature</th>
<th>Yield Strength (MPa)</th>
<th>Elong. at yield (%)</th>
<th>Tensile Strength (Mpa)</th>
<th>Elong. at Tensile (%)</th>
<th>Elong. at Fracture (%)</th>
<th>RA (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>RT TT #1</td>
<td>1193</td>
<td>1489</td>
<td>21.7</td>
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<td></td>
</tr>
<tr>
<td>RT TT #2</td>
<td>1131</td>
<td>1446</td>
<td>21.6</td>
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<tr>
<td>500°C #1</td>
<td>799.30</td>
<td>0.70</td>
<td>897.56</td>
<td>2.47</td>
<td>16.56</td>
<td>77.21</td>
</tr>
<tr>
<td>500°C #2</td>
<td>797.35</td>
<td>0.70</td>
<td>898.73</td>
<td>2.31</td>
<td>16.82</td>
<td>76.08</td>
</tr>
<tr>
<td>600°C #1</td>
<td>643.13</td>
<td>0.65</td>
<td>707.89</td>
<td>1.74</td>
<td>14.29</td>
<td>79.43</td>
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<td>600°C #2</td>
<td>634.78</td>
<td>0.67</td>
<td>703.58</td>
<td>1.84</td>
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<tr>
<td>650°C #2</td>
<td>505</td>
<td>539</td>
<td>17.7</td>
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<tr>
<td>650°C #4</td>
<td>512</td>
<td>549</td>
<td>19.8</td>
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<tr>
<td>880°C #3</td>
<td>58</td>
<td>116</td>
<td>28.4</td>
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Table 6.8: Summary of mechanical properties of Armox 440 high temperature straining, followed by room temperature tensile tests

<table>
<thead>
<tr>
<th>Temperature</th>
<th>Lower YS (MPa)</th>
<th>Elong at LYS (%)</th>
<th>Upper YS (Mpa)</th>
<th>Elong. at UYS (%)</th>
<th>UTS (MPa)</th>
<th>Elong. at UTS (%)</th>
<th>Elong. at Fracture (%)</th>
<th>RA (%)</th>
</tr>
</thead>
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<td>RT 4%</td>
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<td></td>
<td></td>
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<td>RT 5%</td>
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<tr>
<td>500C 11% #1</td>
<td>857.62</td>
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<td>869.34</td>
<td>0.58</td>
<td>869.34</td>
<td>2.47</td>
<td>6.94</td>
<td>66.03</td>
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<tr>
<td>500C 11% #2</td>
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<td>0.71</td>
<td>867.05</td>
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<td>869.90</td>
<td>1.87</td>
<td>7.18</td>
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<td>600C 11% #1</td>
<td>643.13</td>
<td>0.65</td>
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<td>0.58</td>
<td>797.54</td>
<td>2.47</td>
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<td>600C 11% #2</td>
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<td>867.09</td>
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<td>600C 11% #3</td>
<td>742.94</td>
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<td>749.49</td>
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<td>790.30</td>
<td>2.31</td>
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<td></td>
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<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>880C 11%</td>
<td>1051</td>
<td>1334</td>
<td>14.2</td>
<td></td>
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</table>
6.5 Study of Tempering Response

6.5.1 Development of Hollomon-Jaffe Parameters

In order to evaluate the effect of induction bending on properties of Armox 440, it is necessary to study the tempering response in the steel through development of Hollomon-Jaffe Parameter (HJP).

Armox 440 has a carbon content of 0.19% and its HJP is developed using the equation \( T^*(16.6 + \log t) \), where \( T \) is absolute temperature in K and \( t \) is the tempering time. Small disc samples were heat treated in light radiation furnace (LRF) below lower critical temperature at 500 °C, 600 °C and 660 °C with tempering time in range of 1 to 100 minutes. After that, several hardness indents were made on sample surface with 1 kg load in order to calculate average hardness for each sample. A plot of Vickers hardness versus HJP for Armox 440 is shown in Figure 6.54.

The hardness of base metal is about 478 HV. Tempering at all tested temperatures causes significant drop of hardness compared to base metal (at least ~90HV), which can lead to loss of other mechanical properties. Further study is needed to obtain the desired balance between toughness and strength.

Tempering at 500°C leads to gradual decrease of hardness between holding time of 1 min to 100 min (drop of ~15 HV). Induction bending at this temperature is practical, since dwell time does not have significant effect on hardness.

However, tempering at 600°C has significant effect on Armox 440 after 20 minutes holding time, which leads to great loss of hardness. If induction bending is applied at this temperature, tempering time up to 20 minutes is recommended.
There is a significant drop of hardness when tempering at 660 °C for more than 1 minute (~150 HV). Average hardness of sample after 1 minute dwell time is equivalent to that tempered at 600 °C with 20 minutes holding time. Sample holding for 5 minutes also has very similar hardness compared to the one tempering at 600 °C for 100 minutes. Induction bending at this temperature is not recommended. On the other hand, the Armox 440 sample held for 1 hour at this highest testing temperature has great reduction in hardness data. Metallographic characterization was performed by comparing samples tested at different temperatures and to find out the reasons behind.

Regression analysis was carried out in three ranges of data that were tested at 500 °C, 600 °C and 660 °C. Prediction formulae of Vickers hardness (HV) with respect to Hollomon-Jaffe parameters are generated. This allows us to find out the hardness of material by simply plugging in the HJP value corresponding to certain tempering temperature and time that lies within the data range. No additional practical work is necessary. Figure 6.43 displays the regression analysis of Armox 440 tempering study data involving three temperatures. The hardness formula is predicted as following:

$$HV = -5 \times 10^{-9}(HJP)^3 + 0.0003(HJP)^2 - 4.105(HJP) + 22135 \quad (8)$$

R-squared value is 0.94, which is a relatively high goodness value for trendline fitting. The equation is valid within the range of HJP of 14200 to 19000. If the HJP range is further narrowed down by eliminating two data points with lowest hardness and highest HJP, a much simpler linear equation can be defined as following:

$$HV = -0.0163(HJP) + 623.69 \quad (9)$$

R-square value for equation 9 is 0.92, which is slightly lower than that of equation 8. The validity of equation is within HJP range of 14200 to 18300.
Figure 6.54: Plot of Vickers hardness versus Hollomon-Jaffe parameters of Armox 440.

Figure 6.55: Regression analysis of Armox 440 HJP range between 14200 and 19000.

*Prediction Formula:

\[ HV = -0.0163(HJP) + 623.69 \]

\[ R^2 = 0.92009 \]
6.5.2 Metallurgical Characterization

Metallography analysis was performed using optical microscope for two heat treated samples and compared them with Armox 440 base metal. The objective is to observe the effects of tempering at certain temperature and time, as well as to understand the reasons of unusual hardness data in one sample. The sample tempered at 660 °C for 1 hour contains a huge variation of hardness. More indents were made on this specific sample and the hardness values range from 160.77 HV up to 227.39 HV, which has a difference of 67 HV and standard deviation of 19.93. Figure 6.57 a) shows the sample without any heat treatment. Part b) is the sample after tempering at 500 °C for an hour and c) exhibits the sample tempering with same amount of time at 660 °C.

*Prediction Formula:*

\[
HV = -5E-09(HJP)^3 + 0.0003(HJP)^2 - 4.105(HJP) + 22135
\]

\[R^2 = 0.93808\]

Figure 6.56: Regression analysis of Armox 440 HJP range between 14200 and 18300.
It is observed that Armox 440 base metal consists of tempered martensite structure. As the material is heat treated at 500 °C for an hour, some of the original lath-shaped tempered martensite have disappeared. More grains become spheroidized and refined, together with more carbides being precipitated. As the tempering temperature goes up to 660 °C closer to A₁ temperature, all lath-shaped martensite have dissolved. Only dispersion of globular carbides is found in the matrix of ferrite. Changes in microstructure and grain morphology lead to decrease in hardness as tempering temperature increases. This phenomenon indicates overtempering. ThermoCalc™ simulation also reveals the possible effect. The equilibrium phase fraction plot in range of 500- 800°C of Armox 440 is shown in Figure 6.58. The red curve represents the austenite phase. Its formation begins at about 630°C, which is lower than the A₁ temperature measured by SS-DTA. The orange and blue curves indicate the formation of M7C3 and M23C6 carbides near 660°C. M7C3 reaches the maximum at about 580°C and dissolves at 640°C, while M23C6 is at maximum at about 660°C and dissolves at approximately 725°C. Therefore, these carbides might lead to coarsening and even dissolution after a long dwell time (i.e. 1 hour).

For the hardness variation of Armox 440 at 660°C in one hour, it might be due to inhomogeneity of microstructure and composition variations caused by the steel making process and previous thermo-mechanical processing.
Figure 6.57: Armox 440 a) Base metal b) Tempering at 500°C 1 hour c) Tempering at 660°C 1 hour.
6.6 Summary of Armor 440

As a continuation of research work started by Nick Kullman [3], similar set of analysis on Ti-6Al-4V alloys were also performed on Armox 440 armor steel. In order to develop the optimal process control window for hot induction bending of Armox 440, Gleeble\textsuperscript{TM} was used to conduct temperature distribution testing, hot ductility testing, and high temperature testing at various strain levels. All the tested samples were then subjected to longitudinal sectioning and metallurgical characterization using light optical microscope (LOM). High temperature (HT) straining followed by room temperature (RT) tensile testing was conducted to study the effect of HT bending on RT service properties. Tempering response of the armor steel was also evaluated by establishing a relationship between material hardness and Hollomon-Jaffe parameters.
In order to understand the effect of Gleeble™ heating on temperature distribution along the sample, three different designs of Gleeble™ samples were tested at 650 °C and 800 °C. Higher temperature leads to wider temperature gradient compared to the same testing on Ti-6Al-4V alloys. However, both high strength alloys show that standard tensile test samples with reduced gauge section have the largest temperature difference between center and other points along the sample. ¼” diameter HDT samples also have the most uniform temperature distribution region within 10 mm at the center, which were then used for all Gleeble™ testing of Armox 440.

Hot ductility testing at 10 mm/s extension rate was conducted to study the effect of temperature on changes in material ductility. Five samples were selected for hardness testing and fractography using scanning electron microscope (SEM). Three additional tests were performed at lower temperatures of 300 to 350 °C to provide a more comprehensive range of data. Longitudinal sectioning and metallography shows that the greatest amount of voids, which are the initiation site of cracking, is formed in between 700 °C and 858 °C, the measured A₁ and A₃ temperatures. Small amount of voids are found in Armox 440 tested below 700 °C and there is no void formation when tested above the upper critical A₃ temperature. Therefore, hot induction bending between A₁ and A₃ temperatures is not practical.

High temperature straining using the Gleeble™ was utilized to investigate the effect of strains on microstructure, void formation and high temperature mechanical properties. Armox 440 samples were strained at 1 mm/s to yield strength (YS), in between YS and ultimate tensile strength (UTS), UTS, above UTS and at failure at four elevated temperatures, i.e. 500 °C, 600 °C, 650°C and 880 °C. Samples tested at 880 °C (above the
A3 temperature) have no void formation even when strained to fracture with 0.5 of strain. Testing at lower temperatures under A1 leads to formation of voids only at failure, which consist of 0.36 strain at 500 °C; 0.35 strain at 600 °C; and about 0.40 strain at 650 °C. Lower strain levels only cause some changes in morphology of the microstructure. In hot induction bending below the A1 temperature, the localized strain should be kept below 0.3 in order to avoid local void formation and failure. Hot induction bending above A3 temperature is most recommended in terms of material ductility.

In addition, digital image correlation (DIC) technique was applied to 4 HT straining test at ORNL. It provides the local strain distribution across Armox 440 samples caused by the temperature gradient and allows to generate true stress-strain data that will be experienced by the material during actual bending process.

For examination of hot induction bending effect on RT mechanical properties, a series of HT straining followed by RT tensile testing were utilized. Four temperatures tested in Gleeble™ HT straining tests were applied in this study with same amount of strain at 11%. The results show that after HT straining, there is a drop of both ultimate tensile strength and total elongation at failure regardless of the pre-strained temperature. However, pre-straining above A3 at 880 °C restores most of the RT mechanical properties. This is due to full transformation of austenite phase at elevated temperature and formation of martensite on cooling. Below A1 temperature, testing at 650 °C offers the best UTS and elongation compared to RT mechanical properties without pre-straining, followed by pre-straining at 500 °C and lastly at 600 °C. Thus, hot induction bending above A3 temperature is also preferred in terms of RT mechanical properties.
Lastly, tempering response of Armox 440 was analyzed in order to study the effect of hot induction bending on hardness of material. Small samples were heated in light radiation furnace (LRF) at three temperatures holding from 1 to 100 minutes. A relationship of Vickers hardness versus Hollomon-Jaffe parameters (HJP) was established. Regression analysis was conducted for generating a prediction formula of hardness with respect to HJP. This allows to produce the same hardness of material with optimized combination of temperature and tempering time. The results indicate that tempering at 500°C, 600°C and 650°C result in a decrease of at least 90 HV compared to the hardness of base metal. Hot induction bending at 500°C, and at 600 °C within 20 minutes is practical because the hardness is not seriously affected by the holding time. However, bending at 650 °C is not practical since significant reduction on hardness will occurs if the hot induction bending process takes longer than 5 minutes.
Chapter 7: Results and discussion of ARL XXX armor steels

This chapter covers the experimental results and discussion of ARL XXX armor steels. Since it is a new material, analysis started with base metal metallographic characterization. Critical phase transformation temperatures, such as $A_1$, $A_3$, liquidus and solidus, were determined using multiple methods, including use of computer simulation software, single-sensor differential thermal analysis (SS-DTA), and Gleeble™ dilatometry testing. Continuous cooling transformation diagram was generated for the simulated coarse-grain heat affected zone (CGHAZ) of ARL XXX to study possible phase changes at various cooling rates. Some tested welds of ARL XXX made by AEM, the sponsor company, were sent to Ohio State for metallography analysis using optical microscope and fractography using SEM. Gas metal arc welding (GMAW) was applied in these welds with different preheat temperatures. The objective of defining the solidification range and weld characterization is to evaluate effects of welding on the new material and compare with hot induction bending process. Similar to other materials in previous chapters, in order to develop the optimal process control window for hot induction bending of ARL XXX, Gleeble™ testing was performed. These included hot ductility testing for studying effects of temperature and extension rate, and high temperature straining tests at two temperatures for studying the effect of strain on material microstructure, void formation and properties. Samples of the latter tests were then longitudinal-sectioned (LS) and characterized using optical microscope. High temperature straining, followed by room temperature tensile tests
were conducted at NIST to evaluate effect of hot induction bending on room temperature mechanical properties. Some of the tensile tested samples were subjected to optical metallography and SEM factography. In addition, tempering response of ARL XXX was studied by developing Hollomon-Jaffe parameters and its relationship with hardness of samples at various heat-treating temperature and time in light radiation furnace.

7.1 Base Metal Characterization

Base metal microstructure of ARL XXX armor steel is shown in Figure 7.1 and 7.2. The samples were prepared from a 1-inch thick plate and were etched with two solutions, which allows analysis of more different phases. Figure 7.1 a) and 7.2 were etched using 5% Nital solution, which is a grain boundary etching. The former image in magnification of x50 shows the rolling direction of steel plate, which is in horizontal direction. The latter figures are in higher magnification of x1000 and 7.2 b) is taken with DIC technique. Similar to Armox 440 steel, it consists of tempered martensite structure, as a result of the quenched and tempered process. Figure 7.1 b) was etched with Le Perla’s solution, which follows the anodic surface layer etching principle. It reveals tempered martensite as brown structured features. The application of the second etchant assists in identifying ferrite and fresh martensite separately [112], but with a limitation of distinguishing austenite from martensite clearly [113]. The average hardness of ARL XXX base material is 453.0 HV under 1 kg load.
Figure 7.1: ARL XXX base metal microstructure a) etched by 5% Nital b) etched by Le Pera's solution.

Figure 7.2: ARL XXX base metal microstructure etched with 5% Nital solution. Part b) was taken with DIC.

7.2 Determination of Phase Transformation Temperatures

Multiple methods were applied to determine phase transformation temperatures of ARL XXX steel. Phase transformation temperatures are important points of change in microstructure and properties that affects application of hot induction bending and welding processes on the material. First, computer simulations were performed using both JMatPro™ and ThermoCalc™ to generate equilibrium phase fraction plots, which is shown in Figure 7.3 -7.5 and Scheil simulation, as shown in Figure 7.6. The results are then
compared with that of Armox 440 steels, as shown in Figure 7.7. As shown in Figure 7.3 generated by JMatPro software, the plot reveals phase formation at equilibrium state within a range of temperature. Blue curve represents the liquid phase, which completely forms at about 1493.36 °C and is called the liquidus temperature. The gray curve represents austenite as the first solid forming phase. Solidus temperature is approximately 1440.0 °C. $A_3$ temperature is 730.13 °C, which is the point when ferrite represented by yellow curve, begins to transform from austenite phase. On the other hand, retained austenite is maintained even at lower temperature. Therefore, there is no 100% ferrite before $A_1$. The lower critical temperature is estimated at about 500 °C, when amount of austenite begins to increase.

Fe-0.05Al-1.74Cr-0.06Cu-0.01Co-1.0Mn-0.29Mo-0.01Nb-3.64Ni-0.4Si-0.01Ti-0.01V-0.

Figure 7.3: JMatPro equilibrium phase fraction plot of ARL XXX.
Figure 7.4 and 7.5 are the same plot showing phase changes at complete mixing state using ThermoCalc™ software, which is in terms of weight fraction of phase versus temperature. Blue curve is the liquid phase and green curve is face-center-cubic (FCC) austenite phase. Light orange curve represents body-face-cubic (BCC) ferrite phase. A₃ temperature, also regarded as the upper critical temperature, is the point when austenite reaches phase fraction of 1.0 at about 733 °C. A₁ temperature, which is called lower critical temperature, is at about 500 °C. Similar to prediction in JMatPro, small amount of austenite phase remains in the material below 400 °C. Figure 7.5 emphasize other phase formations in between 400 °C and 800 °C. Purple curve represents M7C3 carbides, in which M consists mainly of iron and chromium. It reaches maximum in between A₁ and A₃ temperature. Chromium-rich M23C6 carbides, shown as yellowish green curve, are present at lower temperature and slowly dissolve as austenite starts to form.

Figure 7.4: ThermoCalc™ simulation of ARL XXX equilibrium phase fraction plot.
Figure 7.5: ARL XXX equilibrium phase fraction plot in range of 400 - 800 °C.

Figure 7.6: ThermoCalc™ Scheil simulation of ARL XXX.
Figure 7.6 and 7.7 are Scheil-Gulliver simulation plots of ARL XXX and Armox 440 respectively, which predicts the solidification process. The assumptions of this model are no back diffusion is allowed in solidified phases unless specified in program command; liquid phase has uniform composition due to rapid diffusion; and phase equilibrium exists in between liquid-solid phase interface [102]. The interstitial elements present in alloy system are stated as fast diffusing components, including carbon, sulphur and nitrogen, so that diffusion of these elements is allowed in solid phase during calculation. For ARL XXX, liquidus temperature is approximately 1493 °C and 99.4% solidus temperature is at 1370 °C. The solidification temperature range (STR) is 123 °C. As shown in diagram, only one phase is formed during solidifying process, which is austenite FCC phase. For Armox

Figure 7.7: ThermoCalc™ Scheil simulation of Armox 440.
440, liquidus temperature is found at 1505 °C and the 99% solidus temperature is at approximately 1375 °C. Therefore, STR is about 130 °C. Figure 7.7 shows that two phases are formed during solidification. The BCC delta ferrite phase is first formed up to 50% solid phase from liquid. Then, a second FCC phase starts to transform and later completely solidifies as austenite.

7.2.1 Single Sensor-Differential Thermal Analysis

Several tests were run in light radiation furnace (LRF) in order to determine the upper and lower critical temperatures, as well as other possible phase transformations by heating ARL XXX samples at very slow rate of 28 °C/hr within the predicted A₁ and A₃ temperature range, following ASTM A1033-10 standard. The SS-DTA button melting test was utilized for finding STR. Temperature data versus time were recorded throughout these tests and analyzed using single sensor differential thermal analysis (SS-DTA). The second test increased the temperature range with slow heating rate from 300 °C to 800 °C. Figure 7.8 shows SS-DTA result of its heating curve portion, indicating A₁ temperature as 465°C and A₃ temperature to be at around 712°C.
Figure 7.8: ARL XXX LRF 28°C/ hr heating test for determining A1 and A3 temperature.

Free cooling portion of LRF tests are also analyzed using SS-DTA. Figure 7.9 and 7.10 display the SS-DTA results of button melting test. The former graph indicates the temperature of complete solid phase formation from liquid at about 1267 °C. Liquidus temperature is about 1401 °C, which is marked in Figure 7.9 and is more difficult to identify, as it is near the maximum temperature reached during the test, where a lot of noise was also recorded. Solidification temperature range is therefore 134 °C.

Figure 7.11 displays the temperature range of martensite transformation. Figure 7.12 shows the martensite start temperature $M_s$, which is approximately 378°C. It is usually the most distinct sharp peak seen at low temperature. Martensite finish temperature $M_f$ can be
seen in Figure 7.13 at about 251°C. Another phase transformation is also observed after martensite transformation between 148- 200°C, which is probably solute segregation during solidification process.

Optical metallographic characterization was also performed on the cross-section of ARL XXX sample subjected to button melting using Le Pera’s etchant. The left image of Figure 7.14 is taken where the small button was melted into large button and above the hole where thermocouples was attached. Right image is located at the right side of drilled hole in large button. Solidification grains and their growth direction are observed clearly in both images, where the solidification sub-grain boundaries are in white color. Inside these grains, a lot of needle-like structures can be seen, which should be the martensite formation on cooling from austenite state. The interdendritic constituent that etches in white is most probably austenite stabilized to room temperature due to partitioning of austenite stabilizing elements during solidification.
Figure 7.9: ARL XXX liquidus temperature.

Figure 7.10: ARL XXX solidus temperature.
Figure 7.11: ARL XXX martensite phase transformation.

Figure 7.12: ARL XXX martensite start temperature.
Figure 7.13: ARL XXX martensite finish temperature.

Figure 7.14: Optical micrographs of ARL XXX samples after button melting test.
7.2.3 Summary of Phase Transformation Temperatures

Table 7.1 provides a summary of phase transformation temperature values obtained by different methods. Based on the conclusion of phase transformation study, two testing temperatures are selected for performing most of the following analysis. They are 450 °C below $A_1$ temperature and 750 °C above $A_3$ temperature.

On the other hand, solidification process of Armox 440 and ARL XXX steels are compared using ThermoCalc Scheil simulation. They both possess a wide STR (i.e. exceed 100 °C), which may be related to higher susceptibility to solidification cracking. But for Armox 440, about 50% of delta ferrite is formed before complete austenite solidification. This can reduce solidification cracking susceptibility, mainly due to increased solubility of impurities leading to decrease of low melting constituents [56].

Table 7.1: Summary of major phase transformation temperatures determined by various techniques

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<th>Temperature (°C)</th>
<th>ThermoCalc</th>
<th>SS-DTA</th>
</tr>
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<tbody>
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<td>1401</td>
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<td>Solidus</td>
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<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>ThermoCalc</th>
<th>JMatPro</th>
<th>SS-DTA #2</th>
</tr>
</thead>
<tbody>
<tr>
<td>$A_1$</td>
<td>488</td>
<td>500</td>
<td>465.4</td>
</tr>
<tr>
<td>$A_3$</td>
<td>733</td>
<td>730.1</td>
<td>712.3</td>
</tr>
</tbody>
</table>
7.3 Continuous Cooling Transformation Diagram

A continuous cooling transformation diagram (CCT) for ARL XXX was generated by a series of Gleeble™ dilatometry testing. In order to vary the cooling rate, free spans between cooper jaws was varied. Stainless steel jaws were utilized with longest free span in one test so that the slowest cooling rate can be achieved. Table 7.2 lists the parameters applied for Gleeble CCT testing with dilatometer, including the free span ranging from 10 to 70 mm, corresponding t_{8/5}, martensite start and finish temperatures, as well as \(A_c_1\) and \(A_c_3\) temperatures in heating curves. \(t_{8/5}\) is the time required for the sample to cool from 800 down to 500 °C. Shorter free span leading to faster cooling rate will result in shorter \(t_{8/5}\) time, which is in between 3.1 and 46.76s. The \(A_c_1\) temperature varies between 655 and 672°C. The \(A_c_3\) temperature is in between 785 and 797°C. Figure 7.15 shows the developed CCT diagram with cooling curves at various rates. \(M_s\) and \(M_f\) temperatures are also marked for each plot. The \(M_s\) temperature is roughly the same over a range of cooling rates tested, which is in between 330.9 to 338.6 °C. The finish temperature gradually increases as cooling rate becomes slower from 121.9 to 155.2 °C. Among the CCT tests performed at different cooling rates, martensite transformation is the only phase change observed with dilatometer and SS-DTA technique in cooling session. However, the simulated CCT diagram of ARL XXX generated by JMatPro in Figure 7.16 shows that small amount of bainite might form in the slowest cooling rate at 0.1 °C/s. Estimated \(M_s\) temperature is lower but \(M_f\) is higher compared to the experimentally developed CCT diagram.

Two samples were selected to perform cross-section metallography etched with 5% Nital solution using light optical microscope and hardness measurement. Figure 7.17
displays two images of the CCT sample with fastest cooling rate. The free span applied is 10 mm and \( t_{8/5} \) time is 3.1 sec. Average hardness under 1 kg load is 510.02 HV. The CCT sample with slowest cooling rate is shown in Figure 7.18. The free span is 70 mm and the \( t_{8/5} \) time is 46.76 sec. Average hardness is 484.11 HV, which is lower than that of the first sample. Comparing microstructure of the two samples, both consist of martensite lath-shape features as expected, since its transformation is shown in the dilatometry curves. The lower magnification image reveals that the grain size of sample with shortest free span is smaller than that of the CCT sample with slowest cooling rate, which might explain the softness of the latter sample.

Table 7.2: Summary of CCT testing parameters and corresponding phase transformation temperatures (*S indicates stainless steel jaws applied).

<table>
<thead>
<tr>
<th>Free Span (mm)</th>
<th>( t_{8/5} ) (s)</th>
<th>( M_s ) (°C)</th>
<th>( M_f ) (°C)</th>
<th>( A_3 ) (°C)</th>
<th>( A_1 ) (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>10</td>
<td>3.1</td>
<td>334.9</td>
<td>121.9</td>
<td>793.7</td>
<td>666.7</td>
</tr>
<tr>
<td>15</td>
<td>6.43</td>
<td>338.6</td>
<td>125.1</td>
<td>789.1</td>
<td>665.5</td>
</tr>
<tr>
<td>20</td>
<td>6.81</td>
<td>337.7</td>
<td>126.8</td>
<td>785.9</td>
<td>667.0</td>
</tr>
<tr>
<td>25</td>
<td>9.36</td>
<td>334.9</td>
<td>130.4</td>
<td>792.8</td>
<td>672.4</td>
</tr>
<tr>
<td>30</td>
<td>12.66</td>
<td>334.7</td>
<td>136.9</td>
<td>788.5</td>
<td>661.7</td>
</tr>
<tr>
<td>35</td>
<td>17.05</td>
<td>336.5</td>
<td>137.5</td>
<td>791.1</td>
<td>665.4</td>
</tr>
<tr>
<td>40</td>
<td>18.6</td>
<td>332.4</td>
<td>138.5</td>
<td>791.1</td>
<td>664.1</td>
</tr>
<tr>
<td>45</td>
<td>23.6</td>
<td>334.5</td>
<td>141.9</td>
<td>792.7</td>
<td>658.5</td>
</tr>
<tr>
<td>50</td>
<td>27.63</td>
<td>332.2</td>
<td>144.3</td>
<td>784.5</td>
<td>668.5</td>
</tr>
<tr>
<td>60</td>
<td>34.24</td>
<td>330.9</td>
<td>146.6</td>
<td>791.7</td>
<td>654.7</td>
</tr>
<tr>
<td>70</td>
<td>41.9</td>
<td>336</td>
<td>151.7</td>
<td>792.2</td>
<td>660.4</td>
</tr>
<tr>
<td>70 S*</td>
<td>46.76</td>
<td>334.8</td>
<td>155.2</td>
<td>797.1</td>
<td>666.2</td>
</tr>
</tbody>
</table>
Figure 7.15: Continuous cooling transformation diagram (CCT) of ARL XXX steel.
Figure 7.16: Computer simulated CCT diagram of ARL XXX using JMatPro™.

Figure 7.17: ARL XXX CCT sample tested with copper jaws. Free span 10 mm, ts/5 3.1 sec, HV₁= 510.
7.4 Metallographic characterization of ARL XXX welds

Four T-joint welds of 20 mm thick ARL XXX plates were made with ER70S-6 filler wire using gas metal arc welding (GMAW) in 90% Ar/10CO₂ shielding gas. All of them have a weld length of 9” and macro-sections were cut in the middle of weld. Different preheat temperatures were applied on four respective welds: 17°C for weld #MA10; 66°C for weld #MA04; 149°C for weld #MA06 and 260°C for weld #MA08. All weld samples were subjected to metallurgical characterization using LOM & SEM. Extensive analysis was performed on MA04 as the defects in this sample had the largest size. Chemical analysis was conducted at Miami Valley Materials Testing Center on ARL XXX base metal, ER70S-6 filler wire, and three chip samples extracted from weld metal of MA04, which are the circled regions in Figure 7.19. MA04-C1 is along the centerline close to weld root; MA04-C2 is also located at center of weld but near the weld face and MA04-C3 is near the location of cracks. Their respective compositions were input to ThermoCalc™ in order to simulate the solidification process and determine the cause of cracking.
Figure 7.20 displays optical micrographs of cracks within fusion zone of MA04. Le Pera’s solution was applied as etchant for all optical microscopy analysis. Microstructure of weld metal consists of mainly lath martensite and white etching interdendritic constituent that may contain retained austenite. Dendritic structures and columnar grains are observed throughout weld metal due to weld solidification. Direction of solidification grain growth can also be recognized. Two large cracks and one small crack are located at one side of fusion zone near the horizontal plate. These cracks are all formed and propagated along white etching constituent along the solidification grain boundaries. Figure 7.21 reveals the crack tip and edge in SEM that exhibit smooth surface of solidification liquid. This provides an evidence that these defects should be identified as solidification cracks.

The results of chemical analysis are listed in Table 7.3. An estimated composition of diluted weld metal was based on the dilution D. The dilution was determined as the ratio of the cross-section area of deposited weld metal over the total weld area using image processing software. Diluted weight percent of each alloying element is then calculated using the following equation [114]:

\[
\% \text{ element } E \text{ in diluted weld} = (D \times \% E \text{ in BM}) + [(1 - D) \times \% E \text{ in FM}]
\]

Due to dilution of the filler metal by base metal, the weld metal content of carbon, nickel and chromium significantly decreases. Figure 7.22 and 7.23 reveal Scheil solidification predictions based on calculated weld dilution and on the measured composition in location MA04-C1 at weld root respectively. Other Scheil simulation plots that correspond to ARL XXX and other local chemical compositions in weld MA04 (Table 7.3) are shown in Appendix B. Table 7.4 summarizes the results of ThermoCalc Scheil prediction, including
solidus and liquidus temperatures, solidification temperature range (STR) and solidification mode. These thermodynamic simulations show that an undiluted weld in ARL steel will solidify as austenite and is expected to be potentially susceptible to solidification cracking. Upon solidification, filler metal ER70S-6 diluted with ARL steel will form 30/50 ferrite followed by solidification as austenite.

Unit of crack susceptibility (UCS) is an index that predicts cracking susceptibility in autogenous welds. It is based on the following formula that correlates with the weld metal composition in weight % [115]:

$$UCS = 230C + 190S + 75P + 45Nb - 12.3Si - 5.4Mn - 1$$

This equation is valid for composition that contains carbon in 0.03- 0.23%; sulphur in 0.01 to 0.05%; phosphorus in 0.01 to 0.045%; silicon in 0.15 to 0.65%; manganese in 0.45-1.6% and niobium in range of 0 to 0.07%. The calculated diluted composition of MA04 is found to have UCS value of 17.12 and ARL XXX base metal 40.90. UCS less than 10 usually means high resistance to cracking and above 30 as high risk of cracking. Based on the UCS calculations, autogenous welds in ARL steel can be highly susceptible to solidification cracking. Welds in ARL steel with E70S-6 filler wire can be ranked as moderately susceptible to solidification cracking. It should be noted that some alloying elements with low weight % in MA04 are out of the valid composition range developed for UCS. Thus, resulting values for the weld sample should be used with caution.
Figure 7.19: Macro image of ARL XXX weld with 66°C preheat (MA04).

Figure 7.20: Optical images of MA04 weld zone.
Figure 7.21: SEM fractography images of MA04 cracks.

Table 7.3: Summary of chemical composition of ARL XXX base metal, filler metal and MA04 fusion zone

<table>
<thead>
<tr>
<th>Element/ Wt%</th>
<th>Parent Metal</th>
<th>Filler Metal</th>
<th>Calculated Diluted weld</th>
<th>MA04-C1</th>
<th>MA04-C2</th>
<th>MA04-C3</th>
</tr>
</thead>
<tbody>
<tr>
<td>C</td>
<td>0.22</td>
<td>0.08</td>
<td>0.144</td>
<td>0.19</td>
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<td>0.16</td>
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<tr>
<td>Mn</td>
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<td>1.27</td>
<td>1.20</td>
<td>1.22</td>
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<tr>
<td>P</td>
<td>0.013</td>
<td>0.009</td>
<td>0.011</td>
<td>0.025</td>
<td>0.018</td>
<td>0.002</td>
</tr>
<tr>
<td>S</td>
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<td>0.005</td>
<td>0.003</td>
<td>0.003</td>
</tr>
<tr>
<td>Si</td>
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<td>Cr</td>
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<tr>
<td>Ni</td>
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<td>Al</td>
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<tr>
<td>V</td>
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<td>0.009</td>
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</tr>
<tr>
<td>Ti</td>
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<td>B</td>
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<td>-</td>
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<td>Dilution (%)</td>
<td>46</td>
<td>51</td>
<td>62</td>
<td>64</td>
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<td></td>
</tr>
</tbody>
</table>
Table 7.4: Summary of ThermoCalc Scheil simulation of ARL XXX base metal, applied filler metal and MA04 weld zone

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>BM</th>
<th>FM</th>
<th>Calculated diluted weld</th>
<th>MA04-C1</th>
<th>MA04-C2</th>
<th>MA04-C3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Liquid</td>
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<td>1510</td>
<td>1505</td>
<td>1495</td>
<td>1497</td>
<td>1495</td>
</tr>
<tr>
<td>Solidus</td>
<td>1370</td>
<td>1365</td>
<td>1380</td>
<td>1365</td>
<td>1380</td>
<td>1385</td>
</tr>
<tr>
<td>STR</td>
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<td>145</td>
<td>125</td>
<td>130</td>
<td>117</td>
<td>110</td>
</tr>
<tr>
<td>Solid. Mode</td>
<td>100% A</td>
<td>Initial ~80% F Finish 100% A</td>
<td>Initial ~50% F Finish 100% A</td>
<td>Initial ~30% F Finish 100% A</td>
<td>Initial ~30% F Finish 100% A</td>
<td></td>
</tr>
</tbody>
</table>

Figure 7.22: ThermoCalc™ Scheil simulation of the solidification in weld MA04 calculated based on weld dilution.
Figure 7.23: ThermoCalc™ Scheil simulation based on the composition in location MA04-C1 of weld MA04.

Macro-image of weld MA06 is shown in Figure 7.24, with arrows pointing the position of defects in the fusion zone. Figure 7.25 reveals similar microstructure as weld MA04 at higher magnification around the cracking region. Three cracks are found in this weld. These cracks are formed along solidification grain boundaries. The solidification grains where cracking occurs are in smaller size and shorter in length compared to those observed in weld MA04. SEM fractography images in Figure 7.26 clearly display features at the edge and depth of solidification cracks, which consist of smooth solidified surface layer.
Figure 7.24: Macro image of ARL XXX weld with 149°C preheat temperature (MA06).

Figure 7.25: Optical images of MA06 weld zone.

Figure 7.26: SEM fractography images of MA06 cracks.
Figure 7.27-29 correspond to optical metallography and SEM characterization of weld MA10 with the lowest tested preheat temperature. Besides two small solidification cracks located along solidification grain boundaries, intergranular hydrogen-induced cracking (HIC) can be observed, which propagate from weld root across heat affected zone (HAZ) in martensitic structure. The HIC is probably related to insufficient preheating that leads to higher cooling rate and results in harder microstructure and insufficient time at high temperature for hydrogen effusion from the weld [55].

Figure 7.27: Macro image of ARL XXX weld with 17°C preheat temperature (MA10).

Figure 7.28: Optical images of MA10 weld zone and heat affected zone (HAZ)
Figure 7.29: SEM fractography images of MA10 HIC cracking at HAZ.

Figure 7.30: Macro image of ARL XXX weld with 260°C preheat temperature (MA08).

Figure 7.31: Optical images of MA08 fusion zone.
Optical images of MA08 weld sample are shown in Figure 7.30 and 7.31. It was tested with the highest preheat temperature. No cracking or other defect are observed in this sample. Dendritic features can be observed in solidification grains. But the grain boundaries are not distinctive compared to previous weld samples. Dark structured bainite is formed all over the weld metal, which is likely due to slower cooling rate caused by preheat treatment.

In conclusion, metallographic analysis shows that most defects observed are weld solidification cracks, which are formed along solidification grain boundaries inside weld zone. Fracture surface usually exhibits of solidified liquid films with smooth surface. Their corresponding weld bead geometry might be one of the factors resulting in high thermal contraction stress. The weld width-to-depth ratio is close to 1.25 and the formation of concave weld surface next to the vertical leg can result in local stress concentration and cracking.

Solidification behavior predictions using ThermoCalc reveal that ARL XXX base material has wide solidification range (123 °C) and primary austenite solidification mode. Weld metal of ER70S-6 diluted with ARLXXX leads to primary ferrite solidification followed by solidification as austenite, with a wide solidification range (up to 130 °C). Based on UCS, diluted weld has moderate risk of cracking and base metal has high risk of cracking under autogenous welding condition.
7.5 Hot Ductility Testing

In order to study the effect of temperature and extension rate on ARL XXX steel materials, hot ductility testing was performed using the Gleeble™. A total of nine tests with different temperatures were performed for each of the two extension rates at 1 mm/s and 10 mm/s. The temperature range is based on A1 and A3 temperatures of ARL XXX determined by ThermoCalc™ simulation as discussed in section 7.2. The lower critical temperature is around 500 °C and upper critical is about 733 °C. Each sample was heated at a rate of 10 °C/s and held at tested temperature for about 6 minutes. Rough vacuum was applied in testing chamber and reduction-in-area was measured by digital calipers.

Figure 7.32 shows the plot of reduction in area versus temperature of ARL XXX. Red line represents samples tested at 10 mm/s and blue curve shows samples with 1 mm/s extension rate. Both of them establish a gradual increase of reduction in area over the temperature range from 400 to 800 °C. There is only at most 5 % difference in hot ductility at corresponding temperatures between the two curves. Thus, change in extension rate does not lead to significant effect on ductility at high temperatures. Data of ARL XXX hot ductility testing is also listed in Table 7.5.

In addition, results of ARL XXX are compared with three other armor steel materials, including Armox 440, standard high hard armor steel (HHA 279 Hard) and rolled homogenous armor steel (RHA 280F). Gleeble testing on these material were completed by Nick Kullman [3]. All hot ductility test results are presented in Figure 7.33. Vertical lines represent A1 and A3 temperatures of the material marked with same color. Unlike RHA and HHA steels that have a drop of hot ductility between their respective A1 and A3 temperatures, both Armox 440 and ARL XXX exhibit a steady increase of reduction-in-
area. At lower temperatures, the hot ductility of ARL XXX is significantly lower than Armox 440 but rises at a faster rate compared to Armox 440. Above 750 °C, reduction in area of the two materials is similar.

Figure 7.32: Plot of reduction in area versus temperature of ARL XXX with two extension rates.
Table 7.5: Summary of hot ductility test results of ARL XXX

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Reduction-in-area at 1 mm/s extension rate (%)</th>
<th>Reduction-in-area at 10 mm/s extension rate (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>400</td>
<td>42.62</td>
<td>40.63</td>
</tr>
<tr>
<td>450</td>
<td>45.40</td>
<td>50.74</td>
</tr>
<tr>
<td>500</td>
<td>58.79</td>
<td>60.58</td>
</tr>
<tr>
<td>(A_1)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>550</td>
<td>67.56</td>
<td>63.39</td>
</tr>
<tr>
<td>600</td>
<td>75.70</td>
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<tr>
<td>650</td>
<td>84.24</td>
<td>79.60</td>
</tr>
<tr>
<td>(A_3)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>700</td>
<td>86.41</td>
<td>81.36</td>
</tr>
<tr>
<td>750</td>
<td>89.46</td>
<td>85.95</td>
</tr>
<tr>
<td>800</td>
<td>95.94</td>
<td>89.81</td>
</tr>
</tbody>
</table>

Figure 7.33: Comparison of hot ductility analysis on four armor steel materials.
7.6 Gleeble™ High Temperature Straining Testing

In order to evaluate the effect of strain at high temperatures, Gleeble high temperature straining tests were performed on ARL XXX steels. Two temperatures were chosen for the tests according to \( A_1 \) and \( A_3 \) temperatures determined by SS-DTA results, which are 450 °C and 750 °C. Similar to the testing cycle and plan described in section 6.3, samples were heated to predetermined temperature at 10 °C/s and held for 6 minutes. Then, they were strained at 1 mm/s with various levels of strain so that they reached yield strength (YS), between yield strength and ultimate tensile strength (UTS), at UTS, above UTS and at failure. Figure 7.34 displays the complete engineering stress-strain curves at two testing temperatures, which are used to determine the strain applied in other tests. Metallographic characterization was performed on the longitudinal-section (LS) of all tested samples using light optical microscope. Figure 7.35- 7.39 shows the samples tested at 450 °C and Figure 7.40 -7.44 reveals ARL XXX LS tested at 750 °C. Summary of test results during high temperature straining at 450 and 750 °C is shown in Table 7.6 and 7.7. Figure 7.45 consists of engineering stress-strain graphs at 450 °C with different strain and Figure 7.46 displays similar set of curves tested at 750 °C.
7.6.1 Cross-section Metallurgical Characterization

As discussed in section 7.1, ARL XXX base material consists of tempered martensite structure. When it is subjected to heating at 450 °C below estimated A₁ temperature and straining to its YS, as shown in Figure 7.35, tempered martensitic microstructure can be observed. Morphology of microstructure is not affected and no void formation is found. At straining between YS and UTS and at UTS (Figure 7.36 and 7.37), similar microstructure is found compared with the previous sample, which means there is no effect of amount of strain applied. When ARL XXX sample is strained to above UTS and necking occurs (Figure 7.38), tempered martensite is still observed. However, orientation towards the straining direction is observed. Figure 7.39 shows the sample tested to failure, in which the deformed microstructure is elongated in the straining direction. Small voids in average size of 20 microns or less are observed next to the fracture surface.
ARL XXX is then tested at 750 °C above the austenite finish temperature. All optical images display a microstructure with much refined grains regardless of the strain applied, when compared to those tested at 450 °C. The refinement of microstructure is due to the complete transformation to austenite, and possibly dynamic recrystallization. Small fresh martensite needles can be observed after cooling from full austenite phase at elevated temperature. Similar microstructure can be observed from Figure 7.40 to 7.43, which are ARL XXX samples strained to above UTS as necking occurs. Figure 7.44 shows the sample strained to failure. The martensite needles found in previous samples have disappeared after deformation and the grains are refined to a greater extent with an average size of 5 micron diameter. A small amount of voids are formed and they are relatively large in size compared to those found in the failure sample at 450 °C. Size of some voids are greater than 20 microns along the straining direction.

Figure 7.35: ARL XXX strained to yield strength (YS) level at 450°C.
Figure 7.36: ARL XXX strained to between YS and UTS level at 450°C.

Figure 7.37: ARL XXX strained to UTS level at 450°C.

Figure 7.38: ARL XXX strained to above UTS level at 450°C.
Figure 7.39: ARL XXX strained to failure at 450°C.

Figure 7.40: ARL XXX strained to YS level at 750°C.

Figure 7.41: ARL XXX strained to between YS and UTS level at 750°C.
Figure 7.42: ARL XXX strained to UTS level at 750°C.

Figure 7.43: ARL XXX strained to above UTS level at 750°C.

Figure 7.44: ARL XXX strained to failure at 750°C.
7.6.2 Comparison of Mechanical Properties

Mechanical behavior of ARL XXX during Gleeble™ high temperature straining tests have been determined. The following two tables contain the stroke applied, engineering stress and strain, elongation, reduction in area (RA) and true strength for each test at 450 °C and 750 °C respectively. Five engineering stress strain curves at same temperature with increasing level of strain are combined in one plot, as shown in Figure 7.45 and 7.46.

Comparing testing at two temperatures, those at 450 °C below A1 temperature have very high yield strength and ultimate tensile strength in the range above 1000 MPa, which is about five times the magnitude of strength properties at 750 °C when microstructure becomes fully austenitic. However, in terms of elongation and RA, higher temperature has higher values than that at 450 °C, indicating better ductility at elevated temperatures. True strength is calculated based on final cross section area, therefore it continues to increase as strain level increases.

Comparing the analysis with Armox 440 in section 6.3.2, ARL XXX exhibit better strength in both range of temperatures below A1 and above A3. Total elongation is also higher compared to the Armox 440 sample tested in same temperature range above A3.
Table 7.6: Mechanical properties of ARL XXX tested at 450 °C with various strain levels

<table>
<thead>
<tr>
<th>450 °C</th>
<th>Stroke (mm)</th>
<th>Engr. Stress (MPa)</th>
<th>Engr. Strain (mm/mm)</th>
<th>Elongation (%)</th>
<th>RA (%)</th>
<th>True Strength (MPa)</th>
<th>Voids (Y/N)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Yield Strength</td>
<td>3.3</td>
<td>1070.60</td>
<td>0.01104</td>
<td>1.104</td>
<td>3.90</td>
<td>1114.08</td>
<td>N</td>
</tr>
<tr>
<td>Between YS and TS</td>
<td>3.9</td>
<td>1168.41</td>
<td>0.02102</td>
<td>2.102</td>
<td>5.30</td>
<td>1233.79</td>
<td>N</td>
</tr>
<tr>
<td>Tensile Strength</td>
<td>4.4</td>
<td>1196.99</td>
<td>0.03842</td>
<td>3.842</td>
<td>6.11</td>
<td>1274.93</td>
<td>N</td>
</tr>
<tr>
<td>Above Tensile</td>
<td>5.5</td>
<td>1067.14</td>
<td>0.1248</td>
<td>12.48</td>
<td>22.71</td>
<td>1380.67</td>
<td>N</td>
</tr>
<tr>
<td>Failure</td>
<td>6.5</td>
<td>735.30</td>
<td>0.2014</td>
<td>20.14</td>
<td>45.40</td>
<td>1346.77</td>
<td>Y</td>
</tr>
</tbody>
</table>

Table 7.7: Mechanical properties of ARL XXX tested at 750 °C with various strain levels

<table>
<thead>
<tr>
<th>750 °C</th>
<th>Stroke (mm)</th>
<th>Engr. Stress (MPa)</th>
<th>Engr. Strain (mm/mm)</th>
<th>Elongation (%)</th>
<th>RA (%)</th>
<th>True Strength (MPa)</th>
<th>Voids (Y/N)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Yield Strength</td>
<td>0.45</td>
<td>124.43</td>
<td>0.002409</td>
<td>0.2409</td>
<td>5.68</td>
<td>131.92</td>
<td>N</td>
</tr>
<tr>
<td>Between YS and TS</td>
<td>7.50</td>
<td>209.98</td>
<td>0.09378</td>
<td>9.378</td>
<td>16.49</td>
<td>251.44</td>
<td>N</td>
</tr>
<tr>
<td>Tensile Strength</td>
<td>4.50</td>
<td>207.09</td>
<td>0.2030</td>
<td>20.30</td>
<td>23.39</td>
<td>283.38</td>
<td>N</td>
</tr>
<tr>
<td>Above Tensile</td>
<td>4.50</td>
<td>206.79</td>
<td>0.4162</td>
<td>41.62</td>
<td>40.71</td>
<td>348.78</td>
<td>N</td>
</tr>
<tr>
<td>Failure</td>
<td>11.49</td>
<td>79.95</td>
<td>0.7020</td>
<td>70.20</td>
<td>89.46</td>
<td>758.57</td>
<td>Y</td>
</tr>
</tbody>
</table>

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Figure 7.45: ARL XXX engineering stress strain curves at 450 °C

Figure 7.46: ARL XXX engineering stress strain curves at 750 °C
7.6.3 Summary of High Temperature Straining Testing

As strain is introduced into the plate during hot induction bending, it is important to study its effect on microstructure, void formation and high temperature mechanical behavior of ARL XXX. Metallography examination of longitudinal sections shows that strain has no effect on microstructure of samples strained at or below UTS at 450 °C. Some grains in the necking sample change orientation towards the straining direction and only the failure sample with 20.14 % strain contains small voids that can lead to crack formation. In the samples tested above A3 temperature, no void formation is found in samples strained up to 41.62 %. A few larger size voids are formed in the sample strained to failure at 70.20 %. Therefore, in terms of void formation the strain level does not have strong influence on ARL XXX steel during hot bending at these temperatures. However, at 450 °C ARL XXX will fail at lower strain compared to straining at 750 °C. It is recommended to perform hot induction bending of ARL XXX armor steel above A3 temperature according to the study on the effect of strain on void formation.
7.5 Tensile Testing

Tensile testing was conducted at National Institute of Standards and Technology (NIST), Boulder CO, for the purpose of studying effect of induction bending process on room temperature mechanical properties of ARL XXX steels. Two types of mechanical tests were performed using MTS uniaxial load frame together with ATS furnace chamber. Six ARL XXX samples were used for high temperature tensile testing at 450 °C, 600 °C and 750 °C at a rate of 1 mm/s. The aim is to understand stress-strain behavior of ARL XXX steels at elevated temperatures and to determine amount of strain that the material may experience during bending process. Each test was conducted twice for getting accurate and repeatable data. Six other steel samples were utilized for high temperature straining, followed by room temperature tensile testing. All samples were heated and held at testing temperature for 20 minutes, except the one tested at 750 °C for 3 minutes. They were then strained to simulate the effect of hot induction bending. Samples were cooled with assistance of fan down to about 25 °C, and standard tensile tests were performed to evaluate the mechanical behavior at room temperature similar to after bending. A summary of tensile properties for high temperature tensile testing and high temperature straining followed by room temperature tensile testing is shown in Table 7.8 and 7.9 respectively. Results of high temperature tensile tests are shown in Figure 7.47. Figure 7.48 is the combined plot of engineering stress versus strain when ARL XXX samples were pre-strained at high temperatures. High temperature pre-strained, followed by room temperature tensile tests are represented as a combined graph in Figure 7.49.
Table 7.8: Mechanical properties of ARL XXX high temperature tensile tests

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>YS (MPa)</th>
<th>Elong. at yield (%)</th>
<th>UTS (MPa)</th>
<th>Elong. at UTS (%)</th>
<th>Elong. at Fracture (%)</th>
<th>RA (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>450C #1</td>
<td>946.55</td>
<td>0.79</td>
<td>1147.78</td>
<td>2.80</td>
<td>10.84</td>
<td>48.34</td>
</tr>
<tr>
<td>450C #2</td>
<td>933.21</td>
<td>0.78</td>
<td>1154.85</td>
<td>3.15</td>
<td>11.93</td>
<td>48.46</td>
</tr>
<tr>
<td>600C #1</td>
<td>549.65</td>
<td>0.60</td>
<td>605.81</td>
<td>1.48</td>
<td>25.48</td>
<td>82.57</td>
</tr>
<tr>
<td>600C #2</td>
<td>565.11</td>
<td>0.58</td>
<td>608.91</td>
<td>1.28</td>
<td>21.80</td>
<td>80.66</td>
</tr>
<tr>
<td>750C #1</td>
<td>161.82</td>
<td>0.59</td>
<td>222.60</td>
<td>8.91</td>
<td>43.15</td>
<td>91.20</td>
</tr>
<tr>
<td>750C #2</td>
<td>167.44</td>
<td>0.63</td>
<td>224.86</td>
<td>7.54</td>
<td>72.70</td>
<td>92.27</td>
</tr>
</tbody>
</table>

Table 7.9: Mechanical properties of high temperature strained, room temperature tensile tested ARL XXX samples and base material.

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Lower YS</th>
<th>Elong. at LYP</th>
<th>Upper YS</th>
<th>Elong. at UYP</th>
<th>UTS (MPa)</th>
<th>Elong. at UTS</th>
<th>Elong. at Fracture</th>
<th>RA (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>450C 7% #1</td>
<td>1336.0</td>
<td>1.00</td>
<td>1376.4</td>
<td>0.82</td>
<td>1376.4</td>
<td>0.82</td>
<td>8.91</td>
<td>51.98</td>
</tr>
<tr>
<td>450C 7% #2</td>
<td>1341.1</td>
<td>0.99</td>
<td>1376.6</td>
<td>0.84</td>
<td>1376.6</td>
<td>0.84</td>
<td>9.51</td>
<td>51.72</td>
</tr>
<tr>
<td>600C 11% #1</td>
<td>--</td>
<td>--</td>
<td>703.11</td>
<td>0.56</td>
<td>864.79</td>
<td>6.66</td>
<td>16.34</td>
<td>64.69</td>
</tr>
<tr>
<td>600C 11% #2</td>
<td>--</td>
<td>--</td>
<td>692.96</td>
<td>0.53</td>
<td>842.84</td>
<td>5.67</td>
<td>14.30</td>
<td>65.40</td>
</tr>
<tr>
<td>750C 11% #1</td>
<td>--</td>
<td>--</td>
<td>832.86</td>
<td>1.09</td>
<td>1363.8</td>
<td>8.49</td>
<td>14.82</td>
<td>42.21</td>
</tr>
<tr>
<td>750C 11% #2</td>
<td>--</td>
<td>--</td>
<td>818.59</td>
<td>1.03</td>
<td>1372.3</td>
<td>8.71</td>
<td>14.84</td>
<td>40.54</td>
</tr>
<tr>
<td>BM</td>
<td>--</td>
<td>--</td>
<td>1192.8</td>
<td>1578.9</td>
<td>14.0</td>
<td>45.40</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Figure 7.47: ARL XXX high temperature (HT) tensile tests.
Figure 7.48: ARL XXX high temperature pre-strain data.
Figure 7.49: ARL XXX HT pre-strained, followed by room temperature tensile tests.
The results of high temperature (HT) tensile tests are similar to that of Gleeble™ high temperature straining to failure at 450 °C and 750 °C. 600 °C was selected as the third testing temperature in between A\textsubscript{1} and A\textsubscript{3}. As temperature increases the yield strength (YS) decreases from 946.6 MPa at 450 °C to 161.8 MPa at 750 °C and ultimate tensile strength (UTS) decreases from 1154.9 MPa to 222.6 MPa. On the other hand, total elongation at failure increases from 11\% up to maximum of 73\% at 750 °C. Reduction-in-area (RA) also rises from 48\% at 450 °C to 92 \% when tested above A\textsubscript{3}. Based on HT tensile tests and high temperature straining tests at Gleeble™, amount of strain applied in HT pre-strained followed by RT tensile test is 11\% at 600°C and 750°C, which is above UTS as there is no void formation. At 450°C, amount of strain applied is different because the sample fails at lower strain (less than 11\%). 7\% strain is utilized above UTS with no void formation.

The results of HT tensile tests reveal that the strain at necking is lowest at 600°C among the three testing temperatures. This is probably due to low ductility and void formation along phase boundaries in the two-phase region. Future study is needed to understand the behavior.

The results of HT pre-straining, followed by RT tensile tests are compared with RT mechanical properties without pre-straining. After 7\% pre-straining at 450 °C (below A\textsubscript{1} temperature), yield point phenomenon can be observed in the engineering stress-strain curve. The upper yield strength is the same point as UTS with magnitude of 1376.4 MPa. Its value is slightly lower compared to the strength of base material at RT, but also the closest to RT properties and highest when compared to other pre-strained samples at different temperatures. Yield/tensile ratio equals to 1 may lead to rupture at low plastic
strain. The total elongation of about 9% also indicates lower ductility than the original material.

As ARL XXX is 11% pre-strained at 600 °C in between A₁ and A₃ temperatures, a significant decrease of YS and UTS can be seen compared to RT base metal. This is likely related to straining within the two phase temperature range and to the low strain that causes necking in the mixed microstructure of overtempered martensite and austenite. It is also related to the mixed microstructure of tempered and fresh martensite that forms upon cooling from straining in the two phase region. However, the overall ductility/elongation at fracture of 14.3 to 16.3 % is not affected.

After 11% pre-straining at 750 °C (above A₃ temperature), UTS decreases to about 1363.8 MPa at similar extent as the sample pre-strained at 450 °C. Yield strength is lower than that of base metal and after straining at 450 °C. There is no adverse effect of straining at 750 °C on total elongation at failure, which is about 15 % (similar to the sample tested at 600 °C). One sample from each of the ARL XXX high temperature pre-straining tests was selected for LS metallography and SEM fractography.

Figure 7.50, 7.52 and 7.54 show LS images of the samples subjected to high temperature straining, followed by RT tensile test. These samples were etched with 5% Nital solution. The deformed microstructure of both samples tested at 450 °C and 600 °C consist of cracks and a few voids around the cracking regions. All grains are strained along the tensile direction and their original morphology is lost. It can be expected to see tempered martensite at 450°C and mixture of over-tempered martensite (ferrite) and fresh martensite after straining at 600°C. For ARL XXX at 750 °C, no crack is found and only a few voids are observed at the edge of fracture surface.
Comparing SEM fractography images in Figure 7.51, 7.53 and 7.55 for ARL XXX testing from 450 °C to 750 °C, all samples have the cup and cone fracture features. Both samples at 450 °C and 600 °C consist of radial zone at outer diameter, indicating a rapid rupture. Number of radial ridges present in sample at 450 °C is higher than that at 600 °C. Microcracks are also found on fracture surface. The cracks present in outer diameter of sample tested at 450 °C have longer length and larger amount compared to defects found in other samples. These two samples contain a mixture of ductile dimples and transgranular quasi-cleavage facets.

On the other hand, the sample tested at 750 °C does not contain radial ridges. Small and short-length microcracks can be observed around the fracture surface and ductile dimples are the only features found on surface. The low magnification image also shows that the sample pre-strained above A₃ has lowest reduction-in-area (RA), which indicates lowest ductility at room temperature.

7.7.3 Summary of Tensile Testing

Evaluation of effect of hot induction bending on room temperature (RT) mechanical properties of ARL XXX shows that bending below the A₁ temperature might favor low ductility at RT. Although the strength and ductility of material are only slightly affected, there is a significant drop of total elongation. On the other hand, pre-straining in between A₁ and A₃ results in the greatest drop of UTS among samples with some improvement of RT ductility. Therefore, hot induction bending at 750°C is most practical in terms of reproducing the base metal strength and ductility. However, ballistic properties after processing above the A₃ temperature need to be evaluated. Further evaluation of the effect
of post bending heat treatment on possible restoration of ductility after straining at 450 °C is also suggested.

Figure 7.50: Optical images of ARL XXX 450 °C 7% prestrained, RT tensile test.

Figure 7.51: SEM fractography of ARL XXX 450 °C 7% prestrained, RT tensile test.
Figure 7.52: Optical images of ARL XXX 600 °C 11% prestrained, RT tensile test.

Figure 7.53: SEM fractography of ARL XXX 600 °C 11% prestrained, RT tensile test.

Figure 7.54: Optical images of ARL XXX 750 °C 11% prestrained, RT tensile test.
7.8 Study of Heat Treatment Response

7.8.1 Development of Hollomon-Jaffe Parameter

For the purpose of studying effect of hot induction bending on properties of ARL XXX, such as hardness, heat treatment response of the material was investigated by developing a relationship between Hollomon-Jaffe parameters (HJP) and hardness similar to the analysis on Armox 440 in section 6.5. This can reduce the amount of practical work required to find a matching temperature and heat treatment time that produce equivalent hardness.

ARL XXX contains 0.22 % carbon content. Therefore, the “c” constant in HJP equation is 16.42 based on equation (6) in Chapter 4 and the parameter is generated using $T^*(c + \log t)$. Disc-shape samples were heated in light radiation furnace (LRF) at 450 °C (below $A_1$ temperature) and at 750 °C (above $A_3$ temperature). Six different heat treatment holding time were utilized in between 1 and 100 minutes. Then, samples were mounted and slightly polished for making hardness indents randomly on surface under 1 kg load. A plot of Vickers hardness versus HJP is shown in Figure 7.56. Original data of the plot with
treatment time and temperature is shown in Appendix B. Hardness of base material is also indicated in the plot, which is approximately 453 HV.

Tempering at 450 °C (below A₁ temperature) has greater effect on ARL XXX between holding time of 1 min to 10 min (drop of ~20 HV). Longer holding time does not induce additional effect on hardness. Induction bending at this temperature with dwell time up to 100 min is practical and does not lead to significant change on hardness.

Induction bending at 750 °C (above A₃ temperature) leads to significant increase of hardness (~50 HV) in range of 1 min to 1 hour holding time. Fresh martensite forms on cooling due to the high hardenability of ARLXXX. It is expected that induction bending above the A₃ temperature will result in loss of toughness. Post-bend tempering treatment is needed to temper the fresh martensite and restore the original mechanical properties.

Figure 7.56: Plot of Vickers hardness versus Hollomon-Jaffe parameters of ARL XXX.
Regression analysis was performed on two series of data at 450 °C and 750 °C. Prediction formulae of Vickers hardness in function of HJP are formed. Figure 7.57 shows the regression analysis of ARL XXX at 450 °C by drawing a second-degree polynomial trendline, which can assist the study of tempering response. The predicted hardness formula is shown below:

\[
HV = 1 \times 10^{-5} (HJP)^2 - 0.425 (HJP) + 3505.5
\]  

(10)

R-squared value is 0.99, which is close to perfect fitting. The equation is valid with HJP parameters in between 13157 to 14602. On the other hand, regression analysis of ARL steels at 750 °C is shown in Figure 7.58, which is also fitted by second-degree polynomial curve. Last data point at 100 minutes holding time is not included because of unusual sharp drop of hardness. The hardness formula is predicted as shown below:

\[
HV = 2 \times 10^{-6} (HJP)^2 - 0.0755 (HJP) + 1266.6
\]  

(11)

It consists of the lowest R-squared value at 0.78. The equation is applicable at higher range within HJP parameters of 18616 and 20661. There is a significant drop of hardness when holding at 750 °C for 100 minutes. Metallographic characterization and SS-DTA on cooling curves were performed but no conclusion can be drawn. Optical images of the above sample is compared with that tested at 750 °C for 60 minutes, as shown in Figure 7.59 and 7.60 They both consist of typical martensite structure after cooling from above A₃, but there is no significant difference in terms of features and grain morphology that can explain the reasons of sudden hardness reduction. Further study is required to identify the causes of this phenomenon.
Figure 7.57: Regression analysis of ARL XXX HJP range between 13157 and 14602.

Figure 7.58: Regression analysis of ARL XXX HJP range between 18616 and 20661.
Figure 7.59: ARL XXX LRF test at 750 °C for 1 hour.

Figure 7.60: ARL XXX LRF test at 750 °C for 100 min.
7.9 Summary of ARL XXX steel

ARL XXX high strength armor steel is a recently developed new material. A thorough study of different properties and phase transformations of base metal and brief evaluation of weldability of ARL XXX through computational modelling and metallographic characterization is described in this chapter. The methodology for developing optimal process control window of hot induction bending of high strength alloys is also applied on this new material. Gleeble™ testing, such as hot ductility tests, high temperature straining tests and development of CCT diagram through dilatometry tests are used to evaluate effect of temperature, extension rate, strain and cooling rate on microstructure, phase transformation and properties of ARL XXX after hot induction bending. High temperature straining followed by RT tensile tests were conducted to study the effect of hot induction bending on RT mechanical properties. Hollomon-Jaffe relationships were developed to evaluate the effect of heating during hot bending on hardness of the steel material.

Various methods, including SS-DTA, were applied to determine $A_1$ and $A_3$ temperatures, as well as other possible phase transformations. The critical transformation temperatures were used to set the testing temperature range for developing optimal parameters of hot induction bending. Hot ductility testing shows that ARL XXX has increasing ductility as temperature increases regardless of extension rates. Higher bending temperatures are recommended for better formability.

ARL XXX welds with different preheat temperatures were analyzed. Weld sample with highest preheat temperature ($260^\circ$C) contains no defect, while weld solidification cracks and hydrogen-induced cracks can be observed in other welds with lower preheat temperature. Metallographic characterization was used to confirm the location and nature
of defects. ThermoCalc™ simulation was applied to understand weld solidification process that leads to possible solidification cracking.

A CCT diagram was generated for ARL XXX steel. Only martensite transformation is observed over the range of cooling rates with $t_{8/5}$ from 3.1 to 46.76 seconds through Gleeble™ dilatometry testing. JMatPro™ simulations indicate the formation of small amount of bainite at slowest simulated cooling rate of 0.1 °C/s. Martensite start temperature ranges from 331 to 339 °C and martensite finish temperature is between 122 °C to 155 °C.

High temperature straining test at Gleeble™ shows that void formation is only found in samples tested to failure at 450 °C with 20.14% elongation and 750 °C with 70.20% elongation. The amount of strain applied has no significant effect on microstructure or triggers void formation in samples tested above ultimate tensile strength (UTS), at UTS, in between yield strength (YS) and UTS, and at YS.

High temperature straining followed by room temperature tensile tests of ARL XXX reveals that bending in between $A_1$ and $A_3$ leads to sharp decrease of UTS to 842.8 MPa compared to 1578.9 MPa of base metal without pre-strianing. Testing conducted at 450 °C and 750 °C lead to small reduction of strength to about 1376.4 MPa and 1363.8 MPa respectively. However, ARL XXX strained at 450 °C results in large drop of total elongation and the upper yield strength being at same point of UTS. In conclusion, hot induction bending above $A_3$ temperature is more practical as it achieves similar RT mechanical properties as ARL XXX base material.

On the other hand, response of heat treatment on hardness of the high strength steel through Hollomon-Jaffe parameters is evaluated at 450 °C and 750 °C. Tempering time at 450 °C does not have strong effect on hardness, which causes about 20 HV drop as holding
time increases from 1 minute to about 1.5 hour compared to base metal that has hardness of 453 HV. Heat treatment at 750 °C above A₃ leads to about 50 HV increase of hardness regardless of holding time due to formation of fresh martensite. Post-bending heat treatment can be applied to restore RT mechanical properties. Ballistic testing and evaluation of the effect of post bend heat treatment on restoration of base metal properties and ballistic testing should be performed as a next stage of this research to allow final selection of optimal temperature range for induction bending.
Chapter 8 : Conclusions

Temperature Distribution of Gleeble\textsuperscript{TM} sample of alloy Ti-6Al-4V

1. Hot ductility test sample with ¼” diameter is selected for performing high temperature Gleeble testing of alloy Ti-6Al-4V. It has the most uniform temperature distribution among the tested dimensions. A gauge length of about 10 mm is used for Gleeble straining tests involving extensometer measurement, as there is only 1-2 °C difference between the testing temperature and that measured at 1/6” from center.

Hot Ductility Testing Analysis of alloy Ti-6Al-4V

2. In order to evaluate the effect of temperature and extension rate on microstructure and defect formation, cross-section metallography and SEM fractography were performed on some MIL-STD 46077 Class 2 samples after hot ductility testing. It shows that formation of strain-induced pores (SIP), which are initiation sites of cracking, can be dependent on temperature. For samples tested in between the estimated start of $\alpha$ to $\beta$ transformation (795 °C) and the recrystallization temperature at 890 °C, maximum amount of voids is observed. No SIP is found if the testing temperature increases up to above the recrystallization temperature and near $\beta$-transus temperature. Ti-6Al-4V samples tested below 795 °C contain limited number of SIP. Therefore, the presence of two-phase mixture of $\alpha$ and $\beta$ phases
might increase the material’s tendency to develop void defects. Further study is required to confirm the effect and mechanism behind. Hot induction bending of alloy Ti-6Al-4V in between 795 and 890 °C is not recommended.

3. The effect of extension rates at 1 mm/s and 10 mm/s was also investigated. No significant effect is found in terms of hot ductility, microstructure, and formation of SIP. On the other hand, SEM fractography exhibits the predominant fracture mode of Ti-6Al-4V Class 2 samples is ductile dimple rupture. The dimples become shallower as temperature decreases.

_Gleeble™ High Temperature Straining Analysis of alloy Ti-6Al-4V_

4. For the purpose of studying the effect of strain at temperature on SIP formation, cross-section metallography was performed after testing the sample to various level of strain. At 650 °C, SIP begins to form as strain reaches 19.1 % (above the ultimate tensile strength). At 430 °C, porosity is only found in the failure sample with 30.7 % of strain. Thus, amount of strain has stronger effect at higher temperature for alloy Ti-6Al-4V and develops voids with lower level of strain. Hot induction bending can be utilized at 650 °C at lower than 19.1 % of strain and at 430 °C at strain up to 24.0 – 30.7 %.

_High Temperature (HT) Straining followed by Room Temperature Mechanical Testing of alloy Ti-6Al-4V_

5. In order to evaluate the effect of hot induction bending (high temperature straining) on room temperature (RT) service properties, alloy Ti-6Al-4V samples were pre-
strained at three different elevated temperatures, followed by standard RT tensile testing. 11% pre-straining at 430 °C for class 2 material leads to an increase of RT strength due to strain hardening but a reduction of total elongation compared to alloy Ti-6Al-4V without pre-straining. However, the grade 5 sample strained at 650 °C with 10 % strain leads to decrease in strength and elongation.

6. Testing at 430 °C with 10% strain was repeated with grade 5 alloy. A significant reduction of both strength and ductility is observed compared to sample without HT pre-straining.

**Temperature Distribution of Gleeble™ sample of Armox 440**

7. Hot ductility sample in ¼” diameter is utilized for Gleeble™ high temperature testing of Armox 440 high strength steel. Only 1 °C temperature difference is found at 1/6” from center when tested at 650 °C and 6 °C drop at 880 °C. Gauge length of 10 mm is applied for testing that involves strain measurement.

**Hot Ductility Testing Analysis of Armox 440**

8. Longitudinal-section metallographic characterization indicates that defect formation of Armox 440 is dependent on temperature. Above A3 temperature, tendency of void formation disappears. In between A1 and A3, maximum amount of elongated voids is observed, which are oriented along the straining direction. The presence of ferrite/austenite phase mixture is more likely to develop crack initiation sites. Therefore, hot induction bending between A1 and A3 temperature is
not recommend. Below $A_1$ temperature, defect formation is limited. Voids in average size of 25 microns are mainly located at the edge of fracture surface.

**Gleeble High Temperature Straining Analysis of Armox 440**

9. Testing at 500 °C, 600 °C and 650 °C below $A_1$ temperature shows that there is no effect in microstructure as the amount of strain reaches to the ultimate tensile strength (UTS) of Armox 440 at respective temperatures. As strain level increases to above UTS where necking occurs, ferrite grains begin to elongate and change their orientation towards the straining direction. When Armox 440 is tested to failure at these three temperatures, small and limited number of voids are found. The maximum strain recommended to avoid failure at bending at 500°C, 600 °C and 650 °C is 35.9 %, 34.7 % and 39.6 % respectively.

10. Testing at 880 °C above $A_3$ temperature reveals the best effect. No defect is formed even the sample is strained to failure. Only grain refinement and fresh martensite needles are found due to recrystallization and complete austenite transformation. Hot induction bending above $A_3$ temperature of Armox 440 is recommended based on the combined results of hot ductility testing and high temperature straining testing.

11. A relationship between local strain and void formation is developed for the sample tested to failure at 650 °C using digital image correlation technique (DIC). Six voids are observed near the fracture center with maximum true strain. Total engineering strain obtained from DIC is about 2.3, which is more than 5 times of the average value along the 10 mm gauge length in the Gleeble™.
High Temperature Straining followed by Room Temperature Tensile Testing of Armox 440

12. After pre-straining with 11% strain at 500 °C, 600 °C and 650 °C respectively, Armox 440 shows a reduction of RT strength to about 869 MPa, 790 MPa and 1016 MPa at the three temperatures, compared to that without pre-straining in range of 1446 to 1489 MPa. This is likely due to presence of over-tempered martensite when testing below A₁ temperature. Total elongation also decreases to range of 6.94 % to 9.47 %. Yield point phenomenon can be observed in the three curves indicating upper and lower yield point. Difference in mechanical properties between three temperatures can be influenced by two competing mechanisms: over-tempering and dislocation strengthening.

13. When Armox 440 is pre-strained at 880 °C with 11% strain above A₃ temperature, both UTS and total elongation reduce compared with the sample without pre-straining, but in a lesser extent. The strength decreases to 1334 MPa and the elongation at fracture to 14.2%. The effect on ductility is likely related to the formation of fresh martensite upon fast cooling from austenite.

Tempering Response of Armox 440

14. Tempering of Armox 440 steel results in a drop of at least 90 HV hardness regardless of temperature and dwell time. A relationship between hardness versus Hollomon-Jaffe parameters (HJP) is established. Regression analysis over the range of HJP generates a prediction formula, which serves as a tool for hardness estimation based on the designated tempering time and temperature.
15. At 500 °C, tempering time has no effect on hardness. At 600 °C, hardness starts to decrease as dwell time goes over 20 minutes. As tempering temperature increases to 660 °C, hardness deteriorates rapidly within the range of holding time, especially after 20 minutes that leads to more than a drop of 100 HV. ThermoCalc simulation of equilibrium phase fraction plot indicates the formation of two types of carbides close to 660 °C. Thus, long dwell time might lead to overtempering resulting in a dramatic drop of hardness.

**Phase Transformation Study of ARL XXX**

16. Solidification process of ARL XXX and Armox 440 steels are compared via ThermoCalc Scheil simulation. Both materials have a wide solidification temperature range. ARL XXX solidifies as austenite phase throughout the entire process, indicating higher potential for solidification cracking.

17. Critical phase transformation temperatures of ARL XXX were determined using various techniques. SS-DTA reveals higher A$_1$ and A$_3$ temperatures than ThermoCalc prediction. Based on the analysis, 450 °C (below A$_1$) and 750 °C (above A$_3$) are selected as the temperatures for most of the testing.

**Continuous Cooling Transformation Diagram for ARL XXX**

18. Martensite transformation is the only phase change observed over the range of cooling rates with $t_{8/5}$ from 3.1 s to 46.8 s. M$_s$ temperature is in between 331 to 339 °C. M$_f$ increases from 122 to 155 °C with decreasing cooling rate.
19. Hardness of the samples with fastest and slowest cooling rates under 1 kg load only consist of small difference, ranging from 510 HV to 484 HV.

**Characterization of ARL XXX Welds**

20. ARL XXX GMAW fillet welds contain different defects depending on the preheat temperature applied. Preheat at 17 °C leads to formation of solidification cracks in fusion zone and hydrogen-induced crack in heat-affected zone (HAZ). Both samples with 66 °C and 149 °C preheat consist of solidification cracks. Weld sample with highest preheat temperature at 260 °C is free of cracking. However, the defects observed in these samples are not repeatable along the weld length.

21. ThermoCalc™ Scheil simulation of the weld zone predicts a wide solidification range up to 130 °C, with primary ferrite solidification followed by complete austenite transformation. UCS parameter shows that diluted weld in ARL XXX steel with ER70S-6 consumable has moderate risk of cracking and base metal exhibits high risk of cracking.

**Hot Ductility of ARL XXX**

22. Ductility of ARL XXX increases steadily as temperature rises from 400 °C to 800 °C. Therefore, higher temperature results in better formability. However, the ductility of ARL XXX is lower than that of Armox 440.

23. In general, change of extension rate from 1 to 10 mm/s has no significant effect on hot ductility of ARL XXX steel. Above 500 °C, reduction-in-area of ARL XXX at 1 mm/s is always slightly higher compared to that at 10 mm/s.
**Gleeble High Temperature Straining analysis of ARL XXX**

24. Straining at 450 °C below A₁ temperature up to ultimate tensile strength (UTS) has no effect on microstructure and void formation of ARL XXX. As strain level increases to 12.48% above UTS, some grains orient towards the straining direction. Testing to failure with 20.14% strain develops small voids with less than 20 microns diameter.

25. As temperature rises to 750 °C above A₃, amount of strain does not show noticeable effect on ARL XXX microstructure or defect formation. As the sample is strained to failure with 70.20% strain, a few larger voids greater than 20 microns diameter are observed. Hot induction bending above A₃ is recommended for ARL XXX based on the analysis of temperature and strain parameters.

**High Temperature Straining followed by Room Temperature Tensile Testing of ARL XXX**

26. Pre-straining with 7% strain at 450 °C below A₁ of ARL XXX slightly decreases yield strength (YS) and UTS at room temperature. In addition, yield/tensile ratio is close to 1 and ductility declines rapidly.

27. Testing at 600 °C between A₁ and A₃ with 11% strain results in deterioration of RT strength to 843 - 865MPa compared to the sample without pre-straining, due to formation of two-phase mixture. However, ductility of material is not affected.

28. Straining at 750 °C above A₃ with 11% strain does not affect total elongation at fracture. UTS at room temperature is similar to that of sample pre-strained at 450
°C. Hot bending above A₃ is preferred in terms of retrieving room temperature mechanical properties of base material.

29. The predominant fracture mode of high temperature pre-strained samples is ductile dimple rupture. Microcracks can be found on all samples. Additional fracture features are observed in samples tested at 450 °C and 600 °C, which include a radial zone at outer diameter and transgranular quasi-cleavage facets.

**Heat Treatment Response of ARL XXX**

30. Unlike Armox 440, tempering at 450 °C does not cause huge drop of hardness on ARL XXX. After 1 minute holding time, the sample possess comparable hardness as base metal. As dwell time increases to 100 minutes, only a total drop of about 20 HV is recorded.

31. Heat treatment at 750 °C leads to increase of hardness to above 500 HV due to presence of fresh martensite. Dwell time in range of 1 minute to 1 hour does not induce significant change on hardness. But a drop of more than 50 HV is measured as heat treatment time increases to about 1.5 hour. Further investigation is necessary to identify the cause.
Chapter 9: Future Work

The research work discussed in this document develops the optimal range of parameters for hot induction bending of high strength alloys. It widens the high temperature data of the three high strength alloys and serves as a process control tool in manufacturing industry. Future work is suggested on multiple aspects to better understand the relationship between process parameters and their effects on microstructure and properties, including:

1. Development of post-bending heat treatment of high strength steels to restore room temperature mechanical properties.
2. Evaluation of the effect of hot induction bending on toughness and ballistic properties of high strength alloys.
3. Metallurgical characterization on ARL XXX hot ductility testing samples for investigation of effect of temperature and straining rate on microstructure, void formation and fracture mode.
References


4. *Are quenched and tempered (Q&T) steels readily weldable?* ; Available from: http://www.twi.co.uk/technical-knowledge/faqs/material-faqs/faq-are-quenched-and-tempered-qt-steels-readily-weldable/.


70. Showalter, D., et al., *Development and Ballistic testing of a New class of High Hardness Armor Steel* Army Research Laboratory. p. 3-6.


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### Appendix A: Armox 440

Table A.1: Data Summary of Armox 440 HJP and hardness

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Figure B.1: ThermoCalc™ Scheil simulation of ER70S-6 filler metal.
Figure B.2: ThermoCalc™ Scheil simulation of MA04-C2.

Figure B.3: ThermoCalc™ Scheil simulation of MA04-C3.
Table B.1: Data Summary of ARL XXX HJP and Hardness

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