### **Failure Analysis of Variably Sized 4340 Steel Mandrels Utilized in the Production of Seamless Superalloy Rings through the Vertical Ring Rolling Process**

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By

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### <span id="page-2-0"></span>**Abstract**

Vertical ring rolling is a forging process which forms seamless metal rings through applying a large force at elevated temperatures onto component materials stabilized by cylindrical mandrels. Carlton Forge Works, a company which produces rings of superalloy materials such as INCO718 and 718Plus, is experiencing a consistent failure of mandrels due to extreme conditions. An analysis of the lifetime (bulk material, heat treatment, and use) was performed, which aided in the identification of process variables tied to mandrel failure. Experiments were formed surrounding three variables of the mandrel heat treatment: Austenitization temperature, quenching temperature, and tempering conditions, with the goal of analyzing the degradation of mandrels prior to failure with hardness measurements. Rockwell Hardness C (HRC) was used in each of the three experiments. The first experiment assessed incomplete austenitization due to overcrowded furnaces at 1400˚F and 1550˚F. Secondly, quenching for 10 minutes at 70˚F, 100˚F and 150˚F, was performed to simulate quench vat conditions. Finally, extra tempering at 300˚F, 400˚F, 500˚F, 570˚F, 700˚F, 900˚F and 1400˚F was performed to replicate over-tempering of the mandrels during rolling. Hardness degradation below the HRC range of superalloy rings (35-40 HRC) was used to numerically fit the lifetime of components and propose solutions for mandrel failure.

**Keywords**: Ring Rolling, Vertical Ring Rolling, Forge Tooling, 4340 Steel, Failure Analysis, Over-Tempering

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### <span id="page-6-0"></span>**1.0 Introduction**

Carlton Forge Works (CFW) (Paramount, Ca) is a company which utilizes Vertical Ring Rolling to supply the aerospace industry with rings manufactured from aluminum, steel, titanium, and INCONEL alloys. CFW faces consistent component failure in their manufacturing process, with a high turnover rate of 4340 steel mandrels within their vertical ring rolling operations. The following introduction will seek to familiarize and address the background information regarding the vertical ring rolling process, the material of the mandrel component, and failure analysis terminology and techniques.

### <span id="page-6-1"></span>1.1 Ring Rolling

Producing seamless metal rings of large diameters is of vital importance to the aerospace industry. Seamless metal rings are manufactured from a variety of materials, however since the early 1900s one process has risen above the alternatives as the standard for producing such products [1]. Ring forging, or ring rolling, is a hot forging process by which seamless metal rings are formed. At its base, ring rolling is a shaping process which increases the diameter of a headed and pierced ring through reductions in wall height and thickness [2]. Ring rolling is composed of a few distinct steps.

To begin, a stock material for the seamless ring is pre-punched with a hole using a hydraulic press in a process known as upsetting. The stock is subsequently heated to a temperature above the given materials recrystallization temperature to initiate hot forging [2]. Once the ring stock has been formed, it is placed on a mandrel and raised into contact with a driving / main roll. The driving roll rises to a high velocity, and with an applied pressure plastically deforms the preformed ring to a set increased diameter [3]. The wall thickness is controlled by axial rolls off to the side of the ring which restricts deformation past the desired tolerances for thickness [4]. This process is displayed in *Figure 1*.



**Figure 1:** Horizontal ring rolling schematic with key components identified [3].

<span id="page-6-2"></span>Most ring rolling machines are classified as horizontal ring rolling, in which the ring stock, guide rolls and driving roll are oriented in an upright manner. A less common, but selectively implemented variation of the process is that of vertical ring rolling, in which the preformed ring is placed on a horizontal mandrel, which rotates in tandem with the driving roll as a force is applied vertically, shown in *Figure 2* [5].



Figure 2: Vertical ring rolling schematic with key components identified [6].

<span id="page-7-2"></span>This orientation of the process allows the pressure of the driving roll to be compounded with an applied load of specified tonnage, a feature of the vertical process which allows for greater ease in manufacturing high temperature materials such as INCONEL 718, or thicker ring geometries, due to the ability to add a set compressive load during production [7]. Vertical ring rolling (VRR) is the process by which many components at CFW are produced, and as a result will be the environment / procedure referred to primarily in the following report.

### <span id="page-7-0"></span>*1.1.1 Mandrels*

During the VRR process, the mandrel faces an added tonnage which places an additional stress onto the component. This stress is not present in horizontal ring rolling where the idyll rolls experience only the horizontal stress and the elevated temperature of contact with the ring. The mandrel below a vertically rolled component faces not only the tonnage of the driving roll, but the temperature of the ring stock, cycles of rotational velocity, and a bending moment. This combination of environmental factors is the circumstances which promote fracture of the mandrel components at CFW.

#### <span id="page-7-1"></span>*1.1.2 4340 Steel*

<span id="page-7-3"></span>The mandrel component of the CFW system, and a common material used in forging environments is AISI 4340 steel. 4340 is a steel grade classified as a low alloy with a carbon content of 0.4%. Additional compositional distribution of common alloying elements is shown in *Table I*. Sulfur and Phosphorus are known impurities within steels, whereas transition metals such as Molybdenum, Chromium, Manganese and Nickel are alloyed to improve a variety of mechanical properties and corrosion resistance. In 4340, Silicon is notably added as a deoxidizer within the steel to react away impurities which could result in intermetallic inclusions.



**Table I:** AISI 4340 Bulk Composition Ranges [8].

<span id="page-8-1"></span>4340 steel's mechanical properties are displayed in *Table II* for a specimen that has been quenched and tempered at 400°C. 4340 possesses relatively high strengths and moduli values: properties which make it an optimal choice for high performance applications in the structural, aerospace and manufacturing industries alongside a given toughness comparable to other steels. 4340 also exhibits high creep and fatigue resistance. However, the low relative cost in comparison to other high strength and high toughness steels is the primary reason for 4340's widespread implementation.

**Table II:** AISI 4340 Mechanical Properties [9].



### <span id="page-8-0"></span>*1.1.3 Tempering of 4340*

The CFW mandrel component is known to be heat treated by quenching and tempering at 200°C. Quench and tempering  $(Q&T)$  is one of the most common heat treatments utilized for steels.  $Q&T$  begins with the Austenitization of the steel between 845-950°C, well above Ac1, at which steel begins to form austenite. Once the steel is Austenitized (Austenite phase is formed through the core of the component) the steel is removed from the furnace and immediately quenched to produce Martensite. Martensite is a metastable interstitial solid solution with a body centered tetragonal crystal structure, a structure which increases the strength and hardness of the steel, while decreasing ductility relative to other microstructures such as pearlite or bainite.

The quenching can occur in a variety of solutions (commonly water or oil). However, it fundamentally serves to drop a steel's temperature past the point at which the Martensite transformation starts. This process, if achieved in a rapid manner, produces a near homogenous Martensitic phase throughout the microstructure [10]. A quick quench additionally prevents the weak Austenitic phase from being retained within the microstructure.

Quenching is implemented to take advantage of the strong Martensitic phase, with the main variables resulting in the strength of the steel being the speed of initial quench and the final temperature reached [11]. Despite this increase in strength, a major disadvantage of Martensite is its inherent brittleness. Pure Martensitic steels are practically useless, as the detrimental loss of ductility significantly decreases fracture toughness and increases the probability of failure.

The tempering process (a heat treatment procedure performed post quenching) is the most economical solution to the disadvantages of the Martensitic phase in steels. The tempering process holds a quenched steel isothermally (at a temperature below the Ac1, around 200-600°C) for a set duration of time in an environment in which Austenite cannot re-form [12]. The process of tempering initiates the formation of carbide precipitates which segregate from the solid solution of the steel, followed by a decomposition of retained austenite if it remains in the microstructure. A tempered Martensitic microstructure is shown in *Figure 3*.



**Figure 3:** Tempered martensite microstructure in 4340 Steel (Q&T at 400 °F).

#### <span id="page-9-0"></span>*1.1.3.1 Tempering Temperature*

The primary variable controlled in the tempering process is temperature, with ranges of isothermal holds sitting between 200 and 700°C. The higher the temperature at which a sample is tempered the lower the strength becomes, however greater improvements in fracture toughness and ductility are produced. Past 300°C, 4340 steel samples shift into the formation of pure cementite precipitates which spheroidize and begin to dominate the pure martensitic properties [13].

As shown in *Figure 4*, there is an inescapable tradeoff between strength and toughness in tempered steels, with decreases in the ultimate tensile and yield strength of 4340 necessary for increases in ductility, and impact and fracture toughness [14]. All 4340 samples in the data below have been tempered for relatively low times  $(-1-2)$  hours). Samples are often tempered at lower temperatures to promote high hardness and strength with some improved ductility from unquenched Martensite; while conversely samples are tempered at high temperatures to promote the greatest ductility and toughness. As a result, a compromising temperature of around 400°C is commonly implemented to reap benefits of both mechanical property regimes [13].



<span id="page-9-1"></span>**Figure 4:** Mechanical properties across tempered 4340 steels. LTT is regions of low temperature tempering, in reference to toughness and strength variables. Temper embrittlement regions are identified as TE [13].

#### *1.1.3.2 Tempering Time*

The effects of tempering time are far fewer in magnitude in relation to the property changes produced by the control of tempering temperature. Tempering time primarily changes steel properties through its relation to the kinetics of the nucleation of the carbide precipitates. After long durations of tempering times, the formation of cementite precipitation dominates, and the phenomenon of Ostwald Ripening can be observed [15]. Ostwald Ripening is the phenomenon in which the precipitates forming from a solid solution favor larger surfaces and fewer total nucleated bodies; it is a redistribution of the structure of the carbide particles in a tempered Martensitic structure. Ostwald Ripening occurs in tandem with grain coarsening, which is a similar process which favors the formation of larger grain surfaces as opposed to the nucleation of new sites in a trend towards a lower surface energy. *Figure 5* is a chart which plots the tempering time of a 4340-steel specimen (tempered at 700°C) in relation to the volume fraction of cementite precipitates and relative density, in which a critical tempering time of  $3$ -hours ( $10^4$  s) can be discerned for the start of the grain coarsening or Ostwald Ripening process [16].



<span id="page-10-1"></span>**Figure 5:** Ostwald ripening in tempered 4340 steel with critical tempering time identified between maximum growth and the propagation of coarsened grains [16].

#### <span id="page-10-0"></span>1.2 Failure Analysis

Failure modes and effects analysis (FMEA) is a common procedure used to analyze failure and fracture mechanics. FMEA takes a given system which has experienced a catastrophic failure and organizes it into smaller elements for subsequent analysis. In the case of many failed metals, the fracture surface (or the broken face of the specific component) is one region in which FMEA is focused upon. FMEA analyzes modes of failure, with emphasis on the many means by which a system has failed [17].

While FMEA deconstructs a failed system down smaller components for individual analysis, not every system which fails is composed of identifiable subsections. In the case of many engineering failures, including the problem faced by CFW, a Root Cause Failure Analysis (or RCFA) is preferential. RCFA seeks to identify a singular cause of failure at the smallest scale and provide a solution to the issue based on the observations of the root cause. RCFA begins at a large scale, analyzing environmental conditions, material conditions and process characteristics, before refining the scope of the analysis down to a fundamental cause. RCFA is vital to systems with a singular component facing mixed mode failure (MMF). MMF occurs when a singular component faces multiple discrete conditions promoting failure, as well as one which exhibits many separate modes of failure. RCFA allows for a narrowing of MMF down to a singular mechanism.

#### *1.2.1 Metallic Failure Modes*

When exposed to high performance environments, metals fail through many different failure modes. A mode is designated as a physical symptom, or the way in which a failure occurs. Variables such as temperature, stress magnitude, stress oscillation, and corrosive environments can affect the classification of the failure mode. In general, metals can fail through ductile fracture, brittle fracture, creep, corrosion, or fatigue. Fractography, or analysis of the fracture surface, allows for conclusions to be made based upon topographical features unique to the failure modes described in the section above. Shown in *Figure 6* are the two common failure modes, ductile (*a.*), and brittle (*b.*). Ductile fractures are characterized by microvoid coalescence, a phenomenon caused by ductile behavior which manifests as

small divots or pores in the surface [18]. Brittle fractures are characterized by a granular texture, evidence of clean breaks during the fracture [19]. Other common fractographic surface details include beach lines, which are evidence of fatigue and manifest as small waves imprints in the fracture surface detailing the number of fatigue cycles, and pits (small holes) in the metal surface which are indicative of corrosion effects. Brittle fractures in particular can also possess hackle or chevron lines, which are topographical features caused by fast fracture which point back to an initiation site.

<span id="page-11-2"></span>

**Figure 6:** Failure modes a.) ductile fracture surface in Q&T 4340 steel [18] b.) brittle fracture surface in Q&T 4340 steel [19].

The initiation site on a fracture surface arises as one of the most crucial details when a failure analysis is conducted. Initiation sites are not solely linked to a single cause and can be identified as the presence of a foreign particle, an inclusion, a void, or a surface crack. Initiation sites are often surrounded by a region of fast fracture in the proximity around the site. Fast fracture regions are smooth and are limited to a small region about the initiation. It is quite common however, that a fracture surface exhibits MMF. A singular surface can include both ductile and brittle features, as well as mechanical symptoms such as shear bands or shear rupture. The resulting fractography derived from analyzing failure modes is essential to analysis, the fracture surface being the key to the fundamental mechanisms and the priority of mechanisms within a failure system.

### <span id="page-11-0"></span>*1.2.2 Metallic Failure Mechanisms*

A failure mechanism is described as the means by which a failure is propagated. Mechanisms are resultants of a material or environmental condition and can widely vary. Metallic failure mechanisms vary more broadly than failure modes, and as such are more difficult to classify. A common failure mechanism in metals is known as mechanical overload. Overload failure occurs when a material property limit is exceeded within a component, whether it be yield strength, melting point, or modulus [20]. Another common failure mechanism is thermal cyclic fatigue, where a component is exposed to drastic changes in the temperatures it is exposed to in set oscillations [21]. The resulting thermal stresses can build up in concentrations which promote fracture. Other mechanisms such as impurities can prompt fracture through the formation of stress concentrations. Failure mechanisms can arise through the heat treatment process, the origin of the material, or the conditions it faces during its lifetime (to identify a few), and the aforementioned characteristics will be the basis of study for the CFW failure analysis.

### <span id="page-11-1"></span>1.3 Tempered Martensite Embrittlement

A given heat treatment process, such as tempering, relies on careful balancing of time and temperature to achieve desirable results. However, the same variables which can be tailored to achieve optimal results, when mishandled, can lead to failure inducing mechanisms. In the case of tempering, over tempering of a specimen can lead to premature failure through the same mechanisms which give tempered steel its initial increase in toughness. If a sample is held at a low temperature (200-400°C) for

an extended period of time, temper embrittlement can occur. Temper embrittlement, also known as tempered Martensite embrittlement (TME), is a failure mechanism predicated on the formation of carbides in intermediate temperature regimes. Intermediate temperatures ranges encompass 200-400°C, and that TME can occur through either tempering or slow cooling a low alloy steel through the range [22]. This drop in mechanical properties is manifested in the data in *Figure 7*.

The resulting drop in toughness is a direct result of the transformation of retained Austenite to inter-lath cementite, and the formation of larger carbides on prior Austenite grain boundaries. The formation of such carbides in specific regions act as fast fracture pathways, leading to premature failure. These microstructural symptoms are also exacerbated by long exposure times, or concentration of Carbon and Phosphorous. Increased concentrations of Phosphorous impurities can aggravate the loss of toughness. Similarly higher concentrations of Carbon also result in lowered toughness [22].



<span id="page-12-2"></span>**Figure 7:** Embrittlement phenomena a.) temper embrittlement trough observed in temperature vs fracture toughness of Q&T 4340 steel [22] b.) Hardness vs temperature in Q&T 4340 steel [23].

#### <span id="page-12-0"></span>*1.3.1 Characterizing Tempered Martensite Embrittlement*

TME manifests as abrupt changes (dips/valleys) in mechanical properties in steels tempered in the temperature range between 200 and 400°C. As such, characterizing TME is dependent on mechanical testing and analysis. Toughness testing such as Charpy Impact Tests are the most common means by which to analyze changes in the energy absorption of a sample. However, in addition to toughness measurements, other properties of the material change in the intermediate temperature regime shifting from the expected distribution. One fundamental property which changes during the TME range is hardness, which increases as a direct result of the embrittlement process. Fractography is also essential to analyzing the effects of TME, as analysis of the fracture mechanics present at the surface (such as ductile behavior) are indicative of the coarse carbides produced during the TME.

### <span id="page-12-1"></span>1.4 Microstructural Impurities and Temper Embrittlement

At elevated temperatures, high loads, and cyclic loading, microstructural defects or impurities can propagate failure at a higher rate than otherwise observed in static conditions. Defects such as inclusions and voids are also some of the most common initiation sites in metallic failure systems. While forged (relative to cast) components tend to contain lower concentrations of voids and inclusions, the presence of such microstructural defects at all is inherent in any metallic component. Inclusions can be oxide based, intermetallic, or metallic in nature, each leading to similar degradations in mechanical properties through a variety of mechanisms [22].

Inclusions and voids, possessing properties different to the bulk of a sample act as local stress concentrators for tensile or compressive forces. The formation of inclusions also promotes an increase in the surfaces and thus surface energy for fracture propagation, and as such can act as a highway for such

mechanisms once failure has been initiated. Impurities can segregate to various regions of the microstructure such as grain boundaries, but as a whole fundamentally increase the probability of failure through the formation of low resistance sites.

Inclusions in 4340 steels tend to originate from a select few common impurity elements such as Phosphorus and Sulfur [12]. All impurities are typically a result of either poor quality ore, or poor-quality control during the initial forging process [24]. However, in general, steel impurities are resultant of the sources in which the ores of iron and other alloying elements are refined. Arsenic is another (more rare) elemental impurity which forms inclusions (oxides). When present in steel, Arsenic segregates to the grain boundaries, and similar to Sulfur, increases the likelihood of intergranular cracking. *Figure 8* shows the effect of common impurity elements  $P \& S$  on the fracture toughness of 4340 steel [25].



**Figure 8:** Fracture toughness vs impurity concentration of 4340 steel tempered at two temperatures [12].

<span id="page-13-1"></span>The drop in fracture toughness in relation to impurities can also be classified as a form of Temper Embrittlement (TE) dependent on chemical factors. This process is slightly distinct from the previous failure mechanism discussed (TME), however in principle the cause and results remain similar. Given a steel specimen with high concentrations of impurity elements, if it is exposed to an intermediate temperature range (200-400°C) for an extended period of time during tempering or cooling, embrittlement can manifest through extreme drops in fracture toughness. Within the intermediate temperature range of TE, elements such as Arsenic, Antimony and Phosphorus segregate to grain boundaries and accelerate the embrittling process. Active elements such as Arsenic in quantities as small as 100 ppm or smaller can promote TE [22]. Ultimately the effects of impurity induced TE work in tandem with the effects of TME to detrimentally effect the properties of a steel tempered in the intermediate temperature range.

### <span id="page-13-0"></span>*1.4.1 Characterizing Microstructural Impurities*

While TE does not possess any resolvable microstructural changes from normal tempered steel, there are optical and chemical means by which to identify the impurities within a steel specimen. X-Ray Fluorescence (XRF) is the most common and easily accessible means by which to determine the content of a sample's bulk, and with detection limits as small as 1 ppm embrittling impurities such as Arsenic are more than readily visible. Energy Dispersive X-ray Spectroscopy (EDS) is a more complex means by which to analyze chemical composition, however, offers the extended opportunity for elemental mapping, allowing for detection of elements in tandem with topographical distribution in the microstructure. Similar to characterizing TME, fractography is also vital, particularly the initiation site which might contain an impurity characteristic of the sample.

### <span id="page-14-0"></span>1.5 Failure Solutions

A vital component of any failure analysis, especially RCFA, is the solution. Without proposal of a solution, a given failure analysis is incomplete. The solution to a given failure is dependent on many conditions, but primarily the identified root cause / mechanism and the constraints of the suggested solution. In the case of CFW, the solution for failure had to be identified without halting operations at CFW, all the while remaining both economical and feasible given the budget for the mandrels and the implementation abilities set at the CFW facility. The two primary means by which to propose a solution to a metallic component in the setting of CFW, would be either a heat treatment adjustment, or a materials selection analysis.

### <span id="page-14-1"></span>**2.0 Problem Statement**

Carlton Forge Works (CFW) faces a repeating problem with component failure. In the vertical ring rolling of seamless rings (made of INCO718, 718Plus, and various other alloys), the mandrels on which the rings are forged consistently fracture. The mandrels are made of 4340 Steel quenched and tempered at 400°F. The mandrels which break are exposed to a myriad of conditions: extreme temperature fluctuations through exposure to the Inconel rings and subsequent non-standardized water cooling, intense loading through bending moments and high tonnage, and cyclic loading through a set rotational speed. The mandrels fail through mixed mode failure. Available literature and analysis show that the mechanism of failure could be thermal cyclic fatigue, over tempering, or microstructural defects, with each failure mode correlating to a distinct treatment or prevention method. To address the issue of mandrel failure, the proposed senior project will seek to complete a full root cause failure analysis procedure: which includes not only the identification of the failure mode of multiple mandrels but the proposal of multiple viable solutions for mitigation of failure that fit within budgetary feasibility. The specific goals of the project will be to identify the modes of failure in multiple mandrels, and then once known, propose both heat treatment and materials selection solutions to address the root cause. The goals will be met through materials characterization through XRF and Rockwell Hardness C, and fractography utilizing both Optical and SEM Microscopy. Solution proposals once the failure mode is identified will seek to address probable material causes within the grade of 4340 utilized, or a heat treatment which can be implemented alongside the currently implemented procedure. All solution proposals will be investigated with strict adherence to financial means, with the final solution for failure mitigation being the most viable option in relation to cost, equipment and time when compared to the current solution CFW implements (high turnover of mandrels).

### <span id="page-14-2"></span>2.1 Constraints

The components analyzed in this project vary in size between 2 inches and 9.5 inches in diameter, and average to 20 feet in length. As the mandrels are made from 4340 steel, the sheer size of the components is far beyond the abilities of feasible analysis on a scale mimicking that of CFW's facilities. As a result, the largest constraint of the project is centered about an intrinsic disconnect between the abilities of Cal Poly (CP) facilities to imitate that of CFW's components. However, avenues of replication were uncovered over the course of the project which allow for conclusions to be made across scales, however the scope of what was accomplished was fundamentally hindered by the problem of size. Sectioning fracture surfaces and other pieces of the mandrel proved to be a bottleneck which halted extensive iteration of experiments or much of the fractography. This report will seek to accomplish as much as possible with the tools available at CP while providing suggestions for future work in the project oriented around larger scale analysis more closely replicating CFW's problem.

### <span id="page-15-0"></span>**3.0 Experimental Methods**

### <span id="page-15-1"></span>*3.0.1 Introduction to Experimental Methods*

To propose a solution to mandrel failure that was both monetarily feasible and easy to implement for CFW, an optimization of the heat treatment was selected as the primary means/consideration for the component failure and subsequent analysis. Experimentation began with investigating the current mandrel heat treatment, followed by witnessing the process on the production floor at CFW. After witnessing the heat treatment, areas of optimization were identified. While the current heat treatment accounted for the size of components, CFW possessed no current standardization for batch size during the process. This lack of batch size consideration has led to overcrowding in both the Austenitizing and Quenching of the mandrels. Due to the fact that the mandrels are forging components and are exposed to temperatures above the initial temper, over tempering also emerged as a primary cause of mandrel failure. Thus, with three stages of the heat treatment identified for optimization, scaling and analysis could commence.

### <span id="page-15-2"></span>3.1 Sectioning & Heat Treatment Scaling

Samples for all experiments were sectioned from two mandrels, one 4 inch and one 8 inch given by Carlton Forge Works. Disks from the mandrel were taken far away from the fracture surface and sectioned into 1-inch cubes. Cuts were performed on a horizontal band saw. Prior to every subsequent experiment, the cube samples were annealed at 845°C to remove prior stresses or differing microstructures developed during use. Annealing the cube samples also served to most closely replicate the condition in which CFW receives mandrel components specified by ASTM A-322 (barring Cal Poly equipment).

*Figure 9* shows the current mandrel heat treatment for CFW, which is a common quench and temper procedure used on low alloy steels. The CFW Heat Treatment produces tempered Martensite, the desirable microstructure for the mandrel application due to high strength and toughness. The heat treatment begins with a step Austenitization of 4-hrs at 1300°F 4-hrs and 1400°F and 1.5-hrs at 1550°F This is performed to ensure homogenous distribution of heat in an attempt to account for component size. Following Austenitization a 1.5-hr water quench is performed, followed by a 4-hr temper at 400°F and an air cool to room temperature.



**Figure 9:** Heat Treatment graph of the current mandrel treatment for CFW last amended in 2004*.*

<span id="page-15-3"></span>However due to the sheer size of mandrel components and the extreme time durations of the CFW Heat Treatment, a scaling factor was necessary to ensure that experiments were possible at Cal Poly while most readily mimicking conditions at CFW. This scale down was achieved through the

implementation of the 1-inch per 1 hour industry standard. The inch-per-hour standard is a common piece of scaling used in industrial heat treatments, where it is assured that 1 hour of exposure at a given temperature is sufficient to conduct said temperature through 1-inch of bulk steel material. This common practice is implemented in the CFW Heat Treatment currently, where the step-Austenitization involves exposure to 1300°F and 1400°F for 4 hours each to account for heat distributing into the center of an 8 inch mandrel (4 inches of radius = 4 hours of heating), thus it was decided that using the inch-per-hour standard was acceptable for a scale down factor.

With the new standard, a conversion of the CFW Heat Treatment was produced, shown in *Figure 10*. The major revision, aside from time, was the elimination of the step-Austenitization. As mentioned previously sections of the mandrel were cut into 1-inch cubes, which by the inch-per-hour standard, require only 30 minutes of exposure at a given temperature to be homogenously heated. These small samples would not require steps up to 1550°F and would fully austenitize with simple exposure to the highest temperature of the heat treatment. The inch-per-hour standard also reduced the austenitizing to 30 minutes, the quench to 15 minutes, and the temper to 30 minutes. To ensure that the scaling factor was sufficient, HRC testing was performed on mandrel sections given from CFW post heat treatment and compared to 1-inch cubes which underwent the Cal Poly (CP) Heat Treatment. Both samples after tempering were measured at multiple points to be between 51.1-52.4 HRC, which validated the scaling factor and proved the CP Heat Treatment's ability to mimic the CFW Treatment.



<span id="page-16-1"></span>**Figure 10:** CP Heat Treatment performed as a scaled down version of CFW Treatment using the inch-per-hour industry standard.

### <span id="page-16-0"></span>3.2 Hardness Measurements

As the primary microstructure desired for the mandrels is tempered Martensite, hardness measurements became the foremost means of mechanical analysis for the project. While microstructural analysis was used as a subsidiary, due to the lack of an optical presence of retained Austenite, the relative percent of Martensite within a tested steel sample could be most readily proven with hardness measurements. For the case of the project, Rockwell Hardness C (HRC) scaling was used, *Figure 11*, which implements a diamond tipped indenter.



**Figure 11:** Experimental methods a.) Wilson Rockwell Hardness C test machine utilized b.) 1-inch mandrel cubes polished prior to HRC tests.

<span id="page-17-0"></span>For each data point when an HRC value was collected for all of the experiments, measurements were performed evenly across the surface of the sample as depicted below. An average surface hardness was generated from five values, the range of which was used to plot the mean and error bars using the maximum and minimum values to ensure the spread of HRC values could be determined as statistically significant or insignificant. Prior to HRC measurements, the surface of the sample was ground with a sanding belt to remove oxidized layers, foreign contaminants, and surface aberrations from the sectioning process. For 4340 steels, a 100% Martensitic structure possessed an HRC of 60, while a 50% Martensitic structure possessed an HRC of 52 [26], as shown in *Figure* 12. These HRC protocols were implemented for every experiment conducted.



<span id="page-17-1"></span>**Figure 12:** Isothermal Temperature (IT) diagram of 4340 steel with HRC values scaled for various microstructures [26].

### <span id="page-18-0"></span>3.3 Austenitization Experiment

The first process variable addressed in the CFW Heat Treatment was the Austenitizing stage. The experiment performed was inspired by an observation from the operators on the floor, who noticed that due to overcrowding, some of the larger mandrels come out of the furnaces at the 1550°F Austenitizing stage not even glowing. If the mandrels are not red hot when removed from the furnaces, the steel has not reached 1550°F and the maximum incandescent temperature for 4340 steel sits around 1400°F. 1550°F is the minimum threshold for complete Austenite transformation, while 1400°F would keep the steel within the two-phase region and promote poor Martensitic formation.

To measure the effect of incomplete Austenitization, an experiment was conducted on multiple 1 inch steel cubes sectioned from the mandrel. Two sets of samples would be Austenitized and quenched, one at 1550°F and one at 1400°F the Heat Treatment schematic being shown in *Figure 13.* Following the heat treatment, HRC values were taken.



<span id="page-18-2"></span>**Figure 13:** Heat Treatment Chart of Austenitization experiment with a soak at 1400 and 1550°F followed by a water quench*.*

### <span id="page-18-1"></span>3.4 Quench Experiment

The second process variable addressed in the CFW Heat Treatment was the quenching stage. The experiment performed was inspired in tandem with the Austenitizing experiment. Whereby the lack of batch size considerations resulted in overcrowding of the stage. In the case of quench vat overcrowding, if the water quench is performed too slowly due to sheer volume of steel within the vat or the temperature of the water, cooling rates will occur too slowly to properly or fully form Martensite. Operators on the floor observe that the quenching stage is also a bottleneck for the heat treatment process as a whole, resulting in overcrowding. Due to this bottleneck, the experiment conducted sought to analyze the effect of water temperature on the mandrel steel properties rather than the volume of steel, which would be difficult to replicate. Contacts at CFW also wanted to find a maximum "safe vat range" for their components if room temperature quenching could not be obtained feasibility at the scale of the components.

In order to analyze the effects of a water temperature on Martensite formation, a second heat treatment experiment was conducted. The experiment saw three sets of mandrel steel Austenitized at 1550 $\degree$ F followed by 10-minute water quenches at three separate temperatures, 70 $\degree$ F (Room temperature), 100°F and 150°F. The heat treatment performed is visualized in *Figure 14*.



<span id="page-19-1"></span>**Figure 14:** Heat Treatment Chart of Quench Experiment with separate quenches at various temperatures following standard Austenitization at 1550°F.

### <span id="page-19-0"></span>3.5 Over-Temper Experiment

The final process variable identified to address mandrel failure was the tempering stage. As the mandrels are forging components and face temperatures above their temper during use in production, over tempering emerges as the primary cause of failure when analyzing the heat treatment and thermal lifetime of the mandrel component. *Figure 15* shows a typical thermal cycle for a mandrel component. Mandrels are air cooled after the 4-hour 400°F temper, and during active rolling the mandrel can reach up to 1400°F. 1400°F is the forging temperature of INCO718 and 718Plus, the two most common superalloy ring materials which the mandrels break under. Once the part is removed the mandrel sits at 700°F in between components. CFW practices a random water cooling, whereby operators will shower the mandrels in water between either batches or individual rolls, after the cooling the mandrels reach 500°F Once finished for a batch the mandrels are left to air cool to room temperature.



<span id="page-19-2"></span>

It should be noted that completely mimicking the thermal cycle of a mandrel during its lifetime is impossible. Mandrels at CFW are variably sized and are used on multiple ring materials which require differing forces and temperatures to forge. Mandrels also do not evenly face the temperatures of its thermal cycle on the component, as rolling is dispersed across the length of a mandrel during a set. These variances should not be ignored, however, to analyze over tempering on the 4340 steel, certain

assumptions had to be made. It is with this preface that the over tempering experiment for the project was produced.

To begin, eight temperatures from over the thermal cycle of the mandrel were selected, those values and the location in the mandrel thermal cycle are given in *Table III*. The eight values cover the widest spread of the thermal lifetime of the component, a large range with which to track the degradation of HRC and quantify over tempering.

<span id="page-20-0"></span>



To mimic mandrel components, the 1-inch cubes of sectioned mandrel steel underwent the scaled CP Heat Treatment. Following the CP Heat Treatment and directly succeeding the 400°F temper, the samples were placed into furnaces set to one of the eight chosen "use temperatures" and tempered for additional lengths of time. The time intervals at which the samples were exposed are given in *Table IV*. The longest exposure at elevated temperature was capped at 4-hours, which when scaled up by the inchper-hour standard is a 24-hour component exposure to the temperature.



<span id="page-20-1"></span>

A general schematic of the over tempering heat treatment experiment is shown in *Figure 16*. Two samples were tested for each of the five time intervals and at each of the eight temperatures. Five HRC points were taken from each sample and used to generate average hardness values.



<span id="page-21-2"></span>**Figure 16:** Heat Treatment chart of Over Tempering Experiment. An initial CFW Heat Treatment is followed by a maximum temper time of 4 hours at various temperatures in the mandrel thermal cycles*.*

### <span id="page-21-0"></span>3.6 Non-Heat Treatment Experimental Methods

Due to the time limits and constraints on the project heat treatment was selected as the primary avenue for addressing the mandrel failure. However, smaller subsidiary experiments were conducted to investigate potential further work on the project.

X-Ray Fluorescence (XRF) analysis was conducted on sections of mandrel steel on a Rigaku NEXDE in order to analyze composition and impurities. Scanning Electron Microscopy (SEM) imaging of the fracture surface was also attempted. However, sectioning of the delicate fracture surface proved to be challenging, with many details of the surface being destroyed during cutting on the horizontal band saw. Waterjet cutting was also attempted, however the high surface energy of the fracture surface caused detrimental oxidation rendering the sample undetectable under the SEM. What salvaged band saw sectioned mandrel pieces were degreased with acetone and attempted to image under the SEM, however the high surface energy, oxidation, and contaminated surface (mandrels sit around on the production floor for months contaminating fracture surface) resulted in poor images and the inability to resolve features above 1000X magnification.

Metallography was performed as a qualifying indicator of the presence of Martensite on certain samples, however not performed on all samples due to the limited supply of sectioned mandrel steel. All metallography was performed through the same process: sectioning mandrel steel on the horizontal band saw, mounting in Bakelite, rough polishing on increasing grits from 200-600, fine polishing on successive 50 micron to 5-micron pads, and etching with 2% Nital solution for 30 seconds.

### <span id="page-21-1"></span>**4.0 Results**

As mentioned previously, the primary method of analyzing mandrel failure in this project is the measurement of hardness (HRC) of mandrel steel across its lifetime. HRC has been equated to the presence of Martensite, with 55-60 HRC indicating 100% Martensite, ~50 HRC indicating either 50% Martensite (post quench) or 400°F tempered Martensite, and anything lower than 50 HRC indicating insufficient Martensite or degraded tempered Martensite.

The results of the Austenitization experiment are shown in *Figure 17*. The trend is expected, with the fully Austenitized sample (1550°F) possessing an average hardness across its surface and bulk of 55

HRC. Consequently, the incompletely Austenitized sample (1400°F) possessed an HRC of half the previous, only around 28 HRC. These results are also supported by metallography performed on the samples, the microstructures of which are shown in *Figure 18*. The fully Austenitized microstructure shows tempered Martensite, whereas the incompletely Austenitized sample shows the presence of proeutectoid ferrite and pearlite. These are microstructural artifacts from the annealing stage performed prior to the heat treatment (and the condition mandrels arrive in), a fact supported by it possessing a hardness typical of pearlite (HRC 28).



**Figure 17:** HRC of samples Austenitized at 1550 and 1400°F.

<span id="page-22-0"></span>

<span id="page-22-1"></span>**Figure 18:** Microstructure of Martensite post complete Austenitization and Quench at a.) 1550°F b.) 1400°F.

Results of the quench experiment are shown in *Figure 19*. As the temperature of the quenching vat is raised from 70°F to 100°F to 150°F, the HRC of the steel samples drops. At a proper room temperature quench the HRC is indicative of a fully Martensitic structure, while at 100°F the structure resembles the hardness of a 50% Martensitic structure. Past water temperatures of 150°F, HRC degrades below 50 which indicates the presence of large amounts of retained austenite and other slower cooling phases. Metallography was not performed on the quenched samples due to the inability to decipher retained Austenite through optical microscopy.



**Figure 19:** Safe quench vat range identified between 70°F and 100°F.

<span id="page-23-0"></span>The sponsors at CFW desired a safe vat range, or upper maximum water temperature which the quench solution could sit at to ensure proper quenching of the mandrels. That threshold was decided at 100 °F due to its HRC being that of 52. This value ensures that the HRC of the mandrel and minimum rests at the HRC of the mandrel post its initial temper. While not ideal, due to the scale of the facilities at CFW 100°F quench vat temperature is a far more reasonable threshold than true room temperature quenching. Results from the over tempering experiment are given in *Figure 20*.



<span id="page-23-1"></span>**Figure 20:** Over Tempering estimated through degradation in HRC at exposure to elevated temperatures. Heat Treatments begin with the 400°F temper followed by more tempering at separate temperatures.

The trends that emerge from the over-tempering experiment fit expected results. At low temperatures such as 300°F and 400°F prolonged exposure post the initial temper see plateaus in hardness around 52 HRC. This plateau effect is expected in low alloy steels, as the diffusion of carbon from Martensite during the tempering process is limited over time. It should be noted that the 300°F over

tempered sample emerged harder than the 400°F sample, which illuminates 4340 steel's propensity for air hardening. Once the exposure temperature is raised to 500°F, however, hardness immediately degrades, in the case of the experiment by around 10 HRC. All samples over tempered between 500-700°F plateaued in hardness at around 45 HRC. Exposure to extreme temperatures such as 900 and 1400°F (forging temperature of INCO718 and 718Plus) led to extreme degradations in hardness, reaching a low of 33 HRC. This over tempering stresses the necessity of cooling the mandrels, as over tempering is accelerated by elevated temperatures.

In addition to the hardness degradation plots, the over tempering experiment was designed to tie into an observation from the floor at CFW. Operators state that they can tell a mandrel is about to fail when it scars. Scarring in the context of this project, is defined as the point at which the HRC of the mandrel dips below the HRC of the ring material which is being rolled upon it. The relation between over tempering and scarring will be analyzed further in the discussion section, however *Figure 21* displays the hardness of common ring materials charted alongside degradation plots. These bounds are the maximum hardness of 718Plus, the most common material which breaks mandrels, and INCO718, a softer component. HRC values of the two ring materials were given by the company contact at CFW.



<span id="page-24-1"></span>**Figure 21:** Hardness Degradation for Over tempering Experiment following initial CFW Heat Treatment*.* Values for the maximum HRC of 718Plus and minimum HRC of INCO718 plotted as constants*.*

### <span id="page-24-0"></span>**5.0 Discussion**

Analysis of the results from both the Austenitization and Quench experiments is relatively straightforward. The Austenitization experiment stressed the importance of accounting for the overcrowding of the furnaces, as mandrels which are not austenitized completely possess a microstructure lacking Martensite which handicaps the properties of the component prior to further heat treating. A simple solution to the problem of overcrowded Austenitizing is raising the furnace temperature from 1550°F to 1750°F. The higher austenitization temperature will produce a buffer zone within the heat treatment and ensure that the mandrels homogenously reach 1550°F at a minimum.

Similarly, the quench experiment stressed the importance of water temperature relative to the formation of Martensite. If the vat temperature reaches above 100°F the cooling within the water will be too slow to sufficiently form Martensite, also handicapping the properties of the mandrel prior to the tempering process. A vat temperature of a maximum of 100°F has been identified as the limit for the quench vat, and whether solved through smaller amounts of mandrels in the vat or through constant circulation, control of the water is essential to allowing proper amounts of Martensite to form.

The results of the over tempering experiment, however, produce a more interesting analysis in terms of quantifying and offsetting mandrel failure. With the HRC values of INCONEL components plotted on the hardness degradation charts from the over tempering experiment, a region for scarring of the mandrels could be identified between 33-43 HRC. The degradation charts also allow for temperature and time to be correlated to mandrel failure, with elevated temperatures sending steel samples into the scarring region faster. From this scarring region, the project has identified other regions which could be equated to a mandrel's lifetime. These regions are given in *Table V*.

<span id="page-25-0"></span>

<b>HRC</b> Range	Temperature Range (°F)	Region
$>52*$	$<$ 400	<b>Safe</b>
$43 - 52$	$400 - 500$	Degradation
$33 - 43$	$550 - 700$	Scarring
<33	$900 - 1400$	Failure

**Table V:** HRC Degradation Qualified to Regions of Mandrel Lifetime

\*Maximum of safe region may exist around 57 HRC due to propensity for brittle failure

One detail which should be noted from the regions is the effectiveness of CFW's current practice of random water cooling. As the mandrels sit at 500°F post the random water cooling, the temperature range is directly above the scarring region, which means the hosing on the floor barely maintains mandrel HRC above the threshold for failure. With this knowledge, it is recommended that CFW move from random to frequent water cooling between rolling in order to improve component lifetime on the floor. These regions can also be used in the future on the floor to estimate mandrel lifetime. As these values indicate surface hardness, HRC measurements on the floor could be implemented to monitor mandrel behavior in any of the regions. A simple hardness measurement is a far more feasible means of analyzing mandrel lifetime than sectioning and metallography to detect Martensite.

With over tempering and regions of lifetime identified, the project sought to scale the experiment back up to that of CFW. Using the inch-per-hour standard (the same standard which was used to scale down CFW's treatments), the over tempering experiment can be shifted to mirror the lifetime hours of a mandrel on the floor at CFW. This scale up is designated in *Table VI*, where the tempering time has been scaled to that of even heat distribution through an 8-inch mandrel (4 inches of radius = 4 hours of heat exposure or estimated lifetime).



<span id="page-25-1"></span>**Table VI:** Mandrel Lifetime & Equivalent Over Tempering

For example, exposure to an at-temperature condition for 60 minutes in the over-tempering experiment can be equated to 8 hours of the same thermal exposure in the case of an 8-inch mandrel. This scaling can be used to estimate the entirety of a mandrel's life when joined with the qualified regions of HRC degradation. This relationship is seen in *Figure 22*, where an original estimate for the lifetime of an 8-inch mandrel exposed to 900°F can be quantified between the end of the 4-hour temper and the hour at which the mandrel's HRC dips into the failure region.



## Mandrel Lifetime at 900°F

<span id="page-26-0"></span>**Figure 22:** Estimated Lifetime of Mandrel (at 900°F) using the inch-per-hour standard. Shown in green is a reduction in initial temper increasing the overall lifetime of the component.

From the figure a means by which mandrel failure can be offset can be identified. If the CFW heat treatment is reduced from 4-hours to 2-hours, two hours of mandrel lifetime can be added. A 2-hour temper is more than sufficient to form tempered Martensite 2-inches into the bulk of a mandrel, but more importantly the offset in HRC degradation by 2-hours would give at minimum 2-hours of life back to the mandrel, which on the floor at CFW could save the company 4 to 5 batches of components.

Ultimately, after thorough analysis of the heat treatment, a revision designed to prevent mandrel failure was generated, a procedure shown in *Figure 23*, and reemphasized in the recommendations section of the report.



**Figure 23:** Suggested Heat Treatment to amend the CFW Heat Treatment with batch size adjustments.

### <span id="page-27-2"></span><span id="page-27-0"></span>5.1 Non-Heat Treatment Solution Discussion

<span id="page-27-3"></span>Outside of the primary heat treatment solution, the subsidiary experiments identified in the methods produced more elements of the mandrel failure problem. XRF analysis shown in *Table VII*, indicated the 4340-mandrel steel contained 27 ppm of Arsenic, a quantity which is embrittling and detrimental, however without knowledge of steel suppliers to CFW, and lack of EDS analysis capabilities this detail remained unfounded.





<span id="page-27-1"></span>Similarly, due to the lack of ability to resolve SEM images of the fracture surfaces, investigation into the nature of the failure mode was not achievable. Another issue the mandrels may face, as identified in the introduction is cooling through intermediate temperature ranges and the problem of Tempered Martensite Embrittlement. This phenomenon, however, is only measurable through K<sub>IC</sub> fracture toughness measurements, and without the capabilities to perform a Charpy Impact Test vs over tempering graph, this failure mode was unidentifiable. Solutions avenues tangential to the heat treatment thus were not investigated below surface level, however the details ascertained will be utilized to prompt future work suggestions for this problem.

### **6.0 Conclusions**

- 1. Mandrels must homogenously reach 1550°F for complete Austenitization.
- 2. Quenching above 150°F reduces Martensite formation due to slower cooling.
- 3. Exposure to temperatures above 700°F leads to over tempering.
- 4. Hardness can be used to indicate mandrel lifetime on the floor.

### <span id="page-28-0"></span>**7.0 Recommendations**

- 1. Austenitize at 1750°F to account for batch size.
- 2. Control quench vat temperature, ideally below 100°F.
- 3. Reduce initial temper time to 2 hours to offset degradations in hardness while maintaining correct microstructure.
- 4. Water cool mandrels more frequently during rolling and batches keeping temperature below 500°F.
- 5. Investigate a true batch size standardization for mandrel size vs number of mandrels at each stage.

### <span id="page-28-1"></span>**8.0 Future Work**

- 1. Produce a similar over-temper experiment tracking Fracture Toughness (KIC) in place of HRC in order to examine the effects of TME.
- 2. Examine fracture surfaces to conclude mode of failure and examine the presence/quantity of carbides in various portions of the mandrel bulk.
- 3. Implement EDS analysis to determine the potential for Arsenic segregation.
- 4. Investigate steels with lower concentrations of Arsenic and other impurities.

### <span id="page-29-0"></span>**References**

[1] K. Davey, M.J. Ward,A practical method for finite element ring rolling simulation using the ALE flow formulation,International Journal of Mechanical Sciences,Volume 44, Issue 1,Pages 165-190, (2002).

[2] Guo, Jun, Dongsheng Qian, and Jiadong Deng. "Grain refinement limit during hot radial ring rolling of as-cast GCr15 steel." Journal of Materials Processing Technology 231 (2016): 151-161*.*

[3] Negahban Boron, A., et al. "Prediction of work-rolls failure in hot ring rolling process." *Scientia Iranica* 29.2 (2022): 461-477.

[4] Teja, P. S., et al. "Simulation and optimization studies on the ring rolling process using steel and aluminum alloys." (2019).

[5] Costalupes, Brock. "Analysis of Failed Forging Saddling Mandrels and Process Improvements for Increased Mandrel Lifespan." (2012).

[6] W. Jia, L. Hua, H.J. Mao,The influence of surface layer microstructure evolution of M2 steel cold -ring rolling mandrel roller on fatigue crack initiation,Journal of Materials Processing Technology,Volumes 187–188, Pages 562- 565, (2007).

[7] Zhu, Xing-lin, et al. "Microstructure evolution of Inconel 718 alloy during ring rolling process." *International Journal of Precision Engineering and Manufacturing* 17.6 (2016): 775-783.

[8] MacKenzie, Scott. "Failure Analysis of Heat Treated Steel Components." *ASM International* #05113G (2008)

[9] "AISI 4340 Steel | 36CrNiMo4 | 1.6511 | EN24 | 817M40 | SNCM439." *ASTM Steel* (Accessed 2022) [https://www.astmsteel.com/product/4340-steel-ai](https://www.astmsteel.com/product/4340-steel-aisi/)si/

[10] Lee, Woei-Shyan, and Tzay-Tian Su. "Mechanical properties and microstructural features of AISI 4340 highstrength alloy steel under quenched and tempered conditions." *Journal of materials processing technology* 87.1-3 (1999): 198-206.

[11] Niazi, Najeeb et al. "Austempering Heat Treatment of AISI 4340 Steel and Comparative Analysis of Various Physical Properties at Different Parameters." World Academy of Science, Engineering and Technology, International Journal of Materials and Metallurgical Engineering 2 (2015): n. pag.

[12] Kula, Eric B., and Albert A. Anctil. *Tempered Martensite embrittlement and fracture toughness in 4340 steel*. ARMY MATERIALS RESEARCH AGENCY WATERTOWN MA, 1967.

[13] Clarke, A.J., Klemm-Toole, J., Clarke, K.D. *et al.* Perspectives on Quenching and Tempering 4340 Steel. *Metall Mater Trans A* 51, 4984–5005 (202[0\). https://doi.org/10.1007/s11661-020-059](https://doi.org/10.1007/s11661-020-05972-1)72-1

[14] Li, He-Fei et al, *The Relationship between Strength and Toughness in Tempered Steel: Trade -Off or Invariable?*, Advanced Engineering Materials Volume 21 180116, 01/04/2019, 10.1002/adem.201801116

[15] Manokaran, M., et al. "Influence of tempering in different melting routes on toughness behavior of AISI 4340 steel." *Journal of Materials Engineering and Performance* 29.10 (2020): 6748-6760.

[16] Jena, Pradipta Kumar, and Ponguru Senthil P. "Effect of tempering time on the ballistic performance of a high strength armour steel." *Journal of applied research and technology* 14.1 (2016): 47-53.

[17] Mobley, R. Keith. *Root cause failure analysis*. Butterworth-Heinemann, 1999.

[18]Lee, Woei-Shyan, and Tzay-Tian Su. "Mechanical properties and microstructural features of AISI 4340 highstrength alloy steel under quenched and tempered conditions." *Journal of materials processing technology* 87.1-3 (1999): 198-206.

[19]Voorwald, H. J. C., et al. "Increasing fatigue resistance of AISI 4340 steel by nitrogen plasma ion implantation." *Engineering Failure Analysis* 104 (2019): 490-499.

[20] Prakash, DG Leo, et al. "Crack growth micro-mechanisms in the IN718 alloy under the combined influence of fatigue, creep and oxidation." *International Journal of Fatigue* 31.11-12 (2009): 1966-1977.

[21] Nascimento, M. P., et al. "Effects of surface treatments on the fatigue strength of AISI 4340 aeronautical steel." *International Journal of Fatigue* 23.7 (2001): 607-618.

[22] Krauss, George. *Steels: processing, structure, and performance*. Asm International, 2015.

[23] Channa, Iftikhar A., et al. "Effect of Tempering Temperature on the Properties of Martensitic Stainless Steel AISI-420." *Sukkur IBA Journal of Emerging Technologies* 2.1 (2019): 51-56.

[24] Navasaitis, Jonas, Aušra SELSKIENĖ, and Gintautas Žaldarys. "The study of trace elements in bloomery iron." *Materials Science* 16.2 (2010): 113-118.

[25] Pranay Choudhary & Warren M. Garrison Jr. (2010) The Effect of Inclusion Type on the Toughness of 4340

[26] Angré, Alexander, et al. "Phase transformation under isostatic pressure in HIP." *Powder Metallurgy* 60.3 (2017): 167-174.