An Intercritical Heat Treatment Study of High Strength, Microalloyed Ferrous Open Die Forgings

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Kelly Stewart

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An Intercritical Heat Treatment Study of High Strength, Microalloyed Ferrous Open Die Forgings

Kelly Stewart
Scot Forge Company
Materials Engineering Department
California Polytechnic State University, San Luis Obispo
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Dr. Blair London	Materials Engineering, Cal Poly SLO
Patrick Nowak	Metallurgist, Scot Forge Company
Kyle Rackers	Metallurgist, Scot Forge Company
Dr. Chet VanTyne	Metallurgical Engineering, Colorado School of Mines

Abstract

High strength, low alloy steel is most commonly utilized in plate or sheet form-with a thickness under 4 in, one unconventional application however, is open die forging where cross-sectional area can be as large as 9.5 in by 11.5 in. When forging to larger section size than one would thermo-mechanically roll sheet steel, a new set of complications, such as variation in microstructure and mechanical properties, arise. This study investigates the heat treatment and processing options needed to negate the inherent microstructural irregularity and Charpy V-Notch (CVN) toughness variation. Intercritical heat treatment—normalizing and then quench and tempering above the $A_{C3,}$ 1750°F and then again between 1440°F – 1750°F, the A_{C1} and A_{C3} respectively, has lessened the CVN toughness variation, increasing the success rate of passing parts in production from 25% to 75%. By controlling cooling rate and section size in this micro-alloyed steel, lower variability was attained.

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Introduction

Company Sponsor

Scot Forge Company (Spring Grove, IL), an open die forging group, produces large-scale forgings from over 600 alloys. Scot Forge started over 100 years ago and has become one of the largest North American open die forging facilities (1). Scot Forge provides fully machined and finished forged parts for oil and gas, national defense, aerospace, and industrial applications. A large portion of their work is ferrous forgings. Scot Forge handles a variety of steels including but not limited to stainless, duplex, low alloy, and plain carbon steels.

Open Die Forging Methodology

Open die forging is the process of heating metal ingots and applying directed pressure through a shaped die and a flat anvil to achieve plastic deformation and a desired outcome shape (2). A piece starts as an ingot—an as-cast metal block, and is brought to a temperature elevated above the recrystallization temperature, typically between 1900°F and 2400°F and rough forged to the maximum diameter needed (Figure 1A). The next step involves specialized tools to score or mark the planned forging procedure (Figure 1B). The part is then pressed and “drawn” to the correct dimension of each different segment or feature of a part (Figures 1C and 1D). After the necessary dimensions and part features have been forged, a final procedure may be employed to improve the surface finish of a part (Figure 1E).

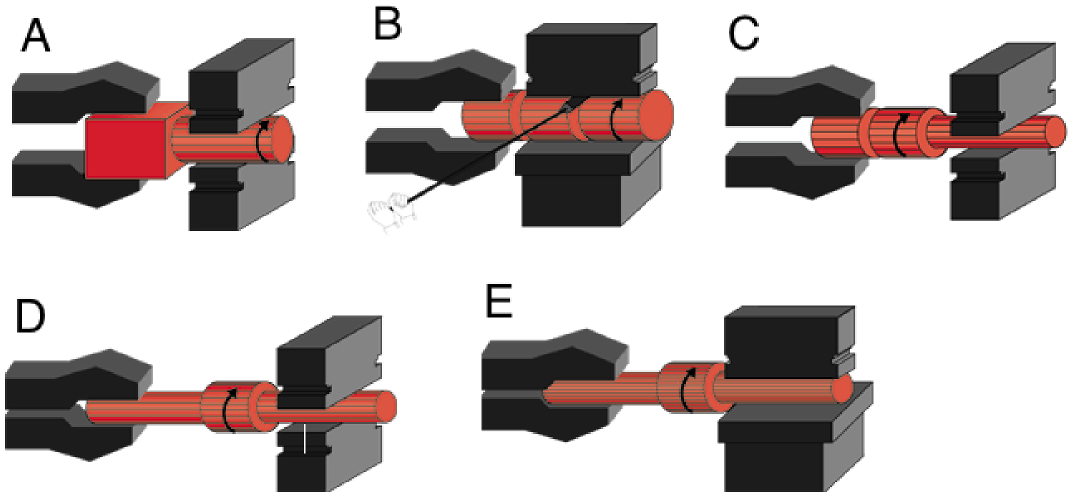


Figure 1: The basic steps of open die forging are detailed in this schematic drawing (3).

The part forged in Figure 1 is called a spindle—one of many shapes within Scot Forge Company's capabilities, such as hubs, rings, hollows, flat and round bars, as well as intricate specialty shapes. Each shape varies in dimension, as per customer needs. When open die forging, a concern worth noting is that of metal movement during the forging process. There are two main types of forging—open and closed die forging. In closed die processes, the starting material stock is completely contained within the die when force is applied, constraining metal flow.

With open die forging, the metal is being plastically deformed and drawn out with each forging pass between a shaped upper die and a flat base, meaning the heated metal flows and moves on one plane when force is applied on a perpendicular plane. As a result of this metal movement, grain flow and reduction of cross sectional area both become notable properties with respect to their effect on the overall performance of the part. As noticed in figure 2 below, forging give lamellar grain flow that is uniform throughout an

entire part, as compared to other processing mechanisms like casting—completely non-uniform grain flow and bar stock—with limited cohesive grain flow.

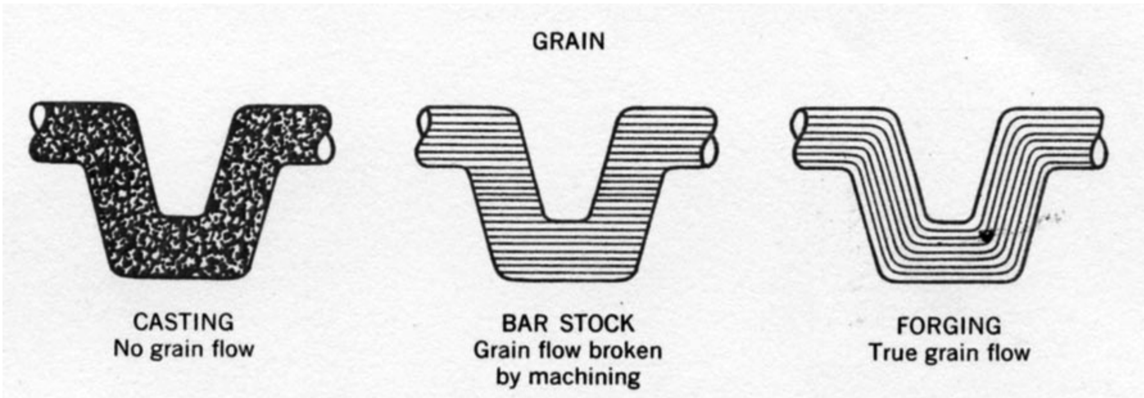


Figure 2: A schematic drawing comparing the grain structure and flow of casting, bar stock, and forging parts (4).

Forging procedures are assigned based on the application and grain flow needs for each part. The ingot size a part will be formed from is selected by the needed reduction of area ratio. The reduction ratio can be calculated by dividing the original ingot cross sectional area by the forged part's final cross sectional area. The calculation varies slightly for differing desired shapes (5). A 3:1 reduction ratio is necessary to avoid residual internal defects from the casting process, such as non-consolidation or porosity.

Ferrous Forgeability

With the basics of open die forging in mind, there is another variable that becomes relevant: the choice of metal being forged. Scot Forge Company works with nickel, copper, aluminum, titanium, and ferrous based alloy systems, along with a few specialty alloys. The majority of their work is ferrous alloys, commonly referred to as steels. Once the customer communicates their material needs, the forgeability of a material must be assessed before procedures are detailed. Temperature dependent phase

transformations, susceptibility to cracking, deformation rate, and—specifically with ferrous metals—crystallographic orientations must be taken into consideration when discussing the forgeability of an alloy (6). The steel family is temperature dependent allotropic alloys—meaning an alloy can exhibit multiple crystallographic orientations and formations, at different temperatures. As a result, some chemical composition steels are more forgeable than others (Table I). Table I gives the relative forgeability of many alloys and the heightened forgeability of steels is noticeable.

Table I: Relative Forgeability of Metals and Alloys (2)

Highest Forgeability
Aluminum Alloys
Copper Alloys
Carbon, Low Alloy Steels
Magnesium Alloys
Martensitic Stainless Steels
Maraging Steels
Austenitic Stainless Steels
Nickel Alloys
Semi-Austenitic PH Stainless Steels
Titanium Alloys
Iron-Base Superalloys
Cobalt-Base Superalloys
Columbium Alloys
Tantalum Alloys
Molybdenum Alloys
Nickel-Based Superalloys
Tungsten Alloys
Beryllium
Lowest Forgeability

Ferrous Heat Treatment

After a ferrous alloy is forged into the appropriate shape, the next step is heat treatment. Post-forging heat treatments are often utilized to improve mechanical properties such as hardness, tensile and yield strength, and Charpy V-Notch (CVN) toughness as well as to relieve stress in concentrated areas. There are a variety of heat treatment options depending upon the chemical composition and the customer's mechanical property goals. Common ferrous heat treatment methods include annealing, normalization, normalize and temper cycles, and quench and temper cycles.

Annealing means a part is heated above the A_{C1} critical temperature and then cooled in a regulated furnace. The critical temperature is the temperature at which a steel begins to change phases. Annealing would be used to produce a relatively soft forging—perhaps to increase machinability.

The next heat treatment, normalization is similar to annealing, however a part is cooled in still air, rather than inside a furnace. By cooling in still air, grain size can be refined or homogenized. Normalization is often used as a first step to homogenize the microstructure before further heat treatment.

Normalize cycles are another heat treatment option. The part is normalized by heating below the A_{C1} critical temperature, before being air-cooled. The normalizing temperature is dependent upon the desired mechanical properties for a part.

A quench and temper cycle is similar, but the steel is heated below the A_{C1} critical temperature before being rapidly cooled down in some sort of medium, like water, oil, polymer, or forced air. This cool down process is known as quenching. After the first step, the steel is then heated below the critical temperature and quenched again. By

implementing rapid cooling techniques, the microstructure changes from austenite to martensite rapidly as the temperature decreases, producing favorable, high mechanical properties in most cases.

Heat treatment holding times depend upon the chemistry of the steel in question. The time for which a part is held at those elevated temperatures is dependent upon the geometry of the part (5). An industry rule-of-thumb states that a part should be heat treated for 60 minutes per each inch of cross-sectional thickness.

Steel Chemistry

Steel, by definition, is iron with carbon added. Many steels utilize additional alloying elements to enhance different properties. Common alloying elements are manganese, nickel, nitrogen, phosphorus, silicon, chromium, molybdenum, vanadium, and niobium. Most all of these elements are fully soluble in iron, so they help enhance the properties. With more alloying elements, the hardenability of a steel increases, shifting the nose of a cooling rate diagram to the right, meaning that a more effective quench can be achieved at slower rates than those of a steel without the alloying elements.

Manganese, nickel, and nitrogen all act as austenite stabilizers, meaning they help to lower the A_{C3} critical temperature and increase the stability of the austenite formed at elevated heat-treating temperatures. Manganese is known to increase ferrite strength, but lower plasticity. Nickel strengthens and toughens ferrite. Phosphorus, generally considered a harmful inclusion in steel, can aid low carbon, low alloy steels in strength and corrosion resistance. Silicon, molybdenum, vanadium and niobium all mainly affect

the hardenability as previously discussed. Lastly, chromium is added to steels to reduce softening when tempered, as well as improve resistance to corrosion and oxidation.

Low Carbon, Low Alloy Steel (LCLA) Background

In this specific situation, the customer chose a low carbon, low alloy grade of steel (LCLA) to be forged. Low carbon, low alloy steel like this grade is actually microalloyed—meaning there are small quantities of many alloying elements. The most influential elements are listed below (Table II).

Table II: Approximate Chemical Composition of LCLA Steel

Element	Wt. %
C	0.14
Mn	1.18
P	0.008
Cr	0.07
Mo	0.03
V	0.095
Nb	0.001

This grade is categorized as a high strength, low alloy (HSLA) steel. As highlighted in Table I, LCLA steel is relatively forgeable, however the needed parts are forged as per customer requirements, to a much larger than any specifications guarantee heat-treatment cycles, to achieve the necessary mechanical properties. The parts are to be forged to a cross sectional area of 9.5 in x 11 in. ASTM specification A131 give heat treatment instructions and rules for LCLA steel in thicknesses up to 4 in (7).

The parts at the center of this study are outside of the bounds relevant to that specification and there is little to no literature pertaining to processing this grade of steel in larger section size. LCLA steel is commonly used as a plate steel, known for its high

weldability, thus this section size is historically unusual. When LCLA steel in larger section size undergoes heat treatment as detailed in plate steel specification, ASTM A131, mechanical properties goals are not met, and property results are inconsistent regardless of whether they pass or fail.

Charpy V-Notch Toughness Testing

Charpy V-Notch (CVN) toughness is a common sought after mechanical property in forged parts. High toughness is desireable in low carbon, low alloy steel. Toughness as a property is not directly related to a single processing mechanism; it is dependent upon the ductility, strength, and microstructure of a material specimen. CVN testing is conducted first by machining a small bar shaped specimen, with a v shaped notch on one flat side (Figure 3).

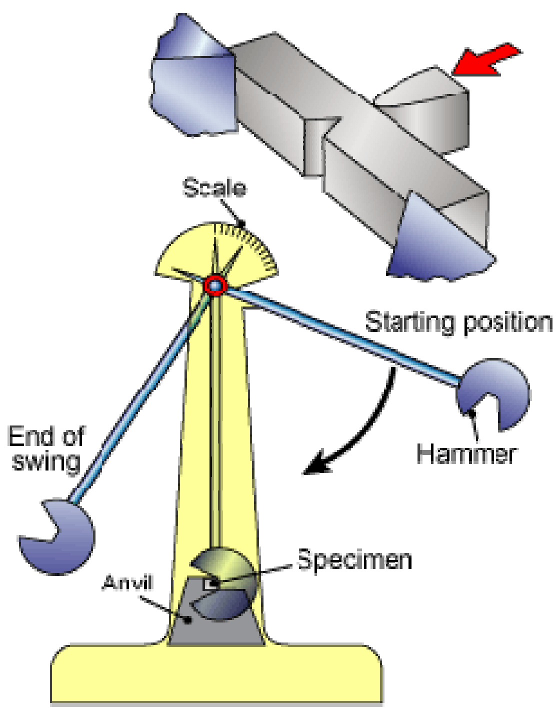


Figure 3: A schematic drawing displays the testing methods for Charpy V-Notch testing.

That specimen is cooled to a specified temperature and positioned in the above apparatus. Once secured, a pendulum-like arm hits the specimen and the force needed to break a specimen is recorded in ft-lbs.

Problem Statement

The current problem is low and/or inconsistent CVN toughness in forged LCLA steel. Prior work and ASTM specifications have shown success in smaller section, < 4 in thick plate. However, when processed in larger section size, mechanical properties, specifically CVN toughness results, were lowered and/or inconsistent. Parts being processed in this manner had a 25% success rate of meeting customer CVN property requirements. This project will conduct heat treatment research to characterize mechanical behavior after heat treatment in larger section sizes with this steel. By classifying the cause and effect of a variety of heat treatments, the best possible heat treatment procedure can be defined. The goal of the heat treatment of LCLA steel is to increase values of and lessen inconsistencies between CVN toughness results, while holding tensile and yield strength at a passing level, as per customer goals.

Heat treatment cycles will be tested first in small-scale parts, as a proof of concept venture. From those trials, the most effective treatments will be implemented in production scale parts and mechanical testing results will be observed. Test furnaces, production scale furnaces, water quench tanks, and the mechanical testing lab will all be used to realize these project goals.

Procedure

Heat treatment began on a research level scale before progressing to production scale trials. Single Sensor-Differential Thermal Analysis (SS-DTA) was completed with this alloy grade, confirming the A_{C1} to be at 1440°F and the A_{C3} to be at 1750°F. These computations helped identify the bounds of viable heat treatment.

On a research level, tensile and impact strength specimens were cut from K11852C01, heat AF166 material, for all trials. One tensile and two impact specimens became each sample set. Each set was labeled alphabetically: set A, set B, etc. All specimens were placed in a preheated furnace and austenitized at 1850°F for 1 hour, allowing for 10 minutes additional time for heat up, then immediately quenched in a 5 gallon bucket of water. The water quenchant was at various temperatures between 50-90°F, depending on the day and production quench schedule. Pieces were agitated during quench. Following austenitization, 36 varying heat treatments were tested in three Collections (Table III).

Table III: Heat Treatment Reference Key 1

Collection 1				
Austenitize all 1850°F		Temper 2		
		900°F	1050°F	1200°F
Intercritical	1500°F	A	B	C
	1550°F	D	E	G
	1600°F	H	I	J
Collection 2				
		Temper 1		
		900°F	1050°F	1200°F
Austenitize 1850°F		K	L	M
Normalize 1850°F		N	O	P
Collection 3				
Austenitize all 1850°F		Temper 2		
		900°F	1050°F	1200°F
Intercritical	1450°F	Q	R	S
	1650°F	T	U	V
	1700°F	W	X	Y

Collection 1

Three sets of samples were intercritically heat treated at 1500°F, 1550°F, and 1600°F, respectively (Table III). A thermocouple was attached to the middle tensile bar in each set of three. Throughout this study 60 minutes of tempering was assumed sufficient per 1 in thickness, thus CVN bars were heat treated for 30 minutes before an agitated water quench and tensile bars underwent intercritical treatment for 60 minutes. This time did not begin until the material reached temperature (±10°F) according to thermocouple. After three sets of intercritical treatment had been accomplished, Brinell hardness measurements were taken from three representative bars, C, G, and J.

At this point, temper cycles at 900°F, 1050°F, and 1200°F were completed for samples ADH, BEI, and CGJ, respectively. One tensile sample was thermocoupled per temper set, identifying heat up time in furnace 5F26. Every sample was Brinell hardness

tested then sent to Exova for tensile testing and CVN toughness testing. A transverse cross section of a charpy sample from each heat treatment was cut, mounted in EpoMet, ground and polished. Microscopy was completed for all samples both as-polished and etched with Picral and 2% Nital.

Collection 2

Two sets of three samples were austenitized and water quenched or normalized, following the same thermocouple and water quenching mechanisms outlined for Collection 1. These samples were then single tempered at 900°F, 1050°F, and 1200°F and quenched (Table III). After Brinell hardness testing, samples were sent to Exova for tensile and CVN testing. Microscopy was completed for all samples both as-polished and etched with Picral and 2% Nital.

Collection 3

Following the procedure outlined for Collection 1, the nine samples were intercritically heat treated at 1450°F, 1650°F, and 1700°F and quenched, then tempered at 900°F, 1050°F, and 1200°F and quenched (Table III). Samples were Brinell hardness tested then sent to Exova for mechanical testing. Microscopy was also completed on this sample collection.

Production Scale Conversion

From the research scale heat treatment trials, heat treatment J was identified as a viable heat treatment method in larger scale parts. Heat treatment J entailed

austenitization at 1850°F for 60 minutes and water quench, Intercritical treatment at 1600°F for 60 minutes/1 in thickness and quench, and 1200°F temper for 60 minutes/1 in thickness and quench. Parts were 9.5 in x 11 in x 120 in (Figure 4).

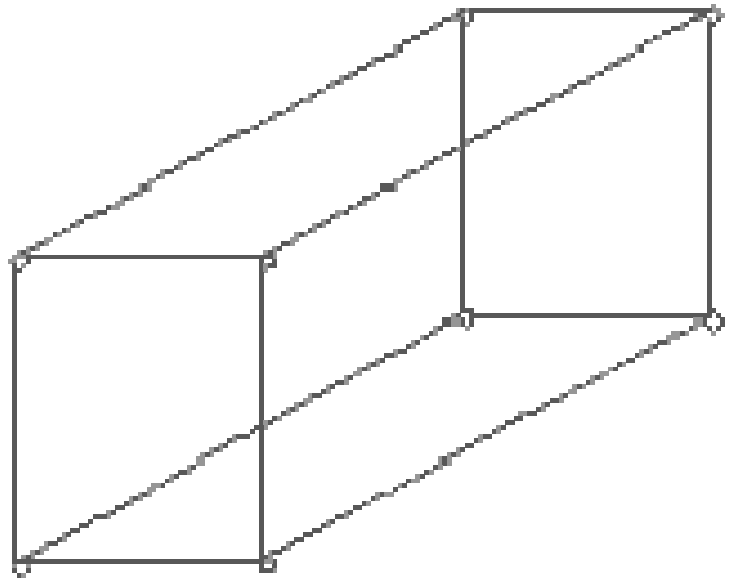


Figure 4: A basic schematic of the production parts with a cross-sectional area of 9.5 in x 11 in, with a length of roughly 120 in.

Four production scale parts underwent heat treatment J. All heat-treated parts underwent mechanical testing on longitudinal specimens from ¼ thickness. Microstructural analysis was completed on CVN specimens.

To identify microstructures as a result of depth from the quenched surface in production scale parts, CVN samples were cut at a various depths from at surface to past center (Figure 5).

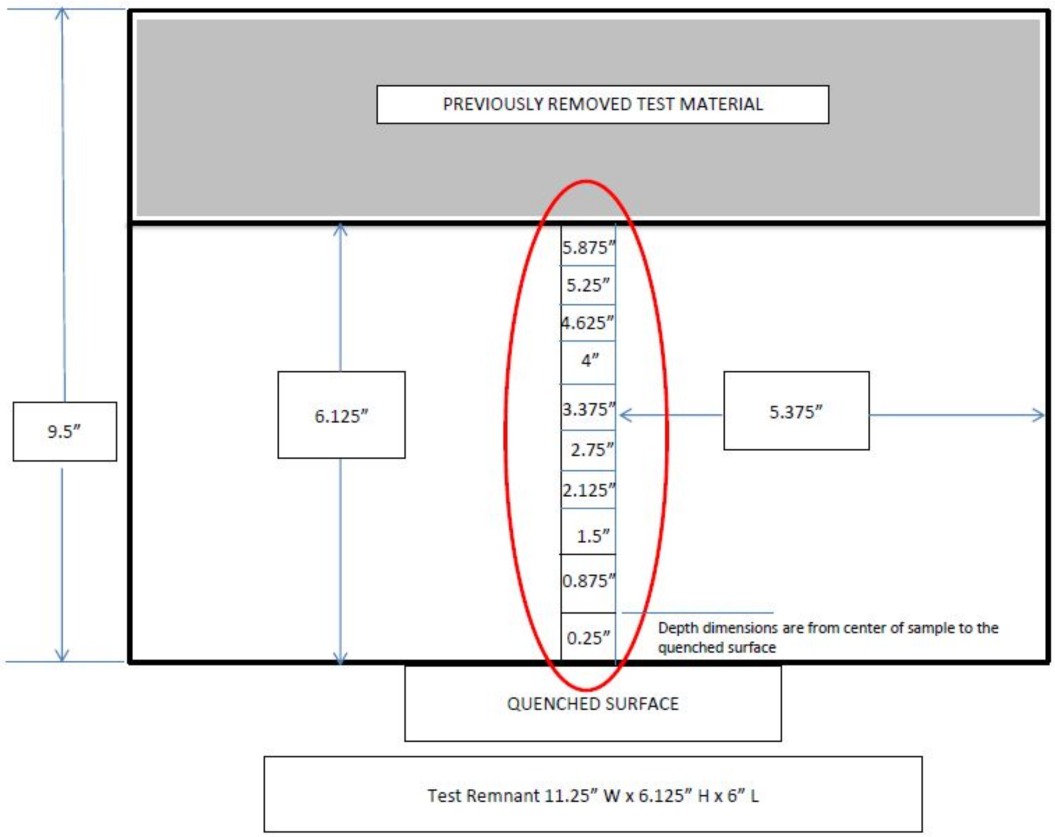


Figure 5: A cross-sectional view of the production piece prolong highlights the varying depth location CVN specimens were cut.

Specimens were cut from the prolong testing area of two production parts. A prolong is an excess of material on one end of the part that undergoes identical heat treatment. One part was heat treated with the initial problematic normalizing treatment and a second was heat treated with the proposed improved intercritical treatment J. All specimens underwent CVN impact strength testing and metallographic analysis—as polished and etched with Nital 2% and Picral. All parts thus far had been forged from ingot stock, heat treated as per experimental instruction, and lastly machined to final thickness.

Results and Discussion

In production work, parts were aiming to meet a set of mechanical testing requirements (Table IV).

Table IV: Strength Requirements

Yield	> 51 ksi
Tensile	71 - 90 ksi
Impact (Charpy)	> 37 ft-lbs

For the purpose of this study, research scale parts were compared against the same standards. The three groups of heat treatment trial samples underwent tensile and CVN testing, with a range of results (Table V).

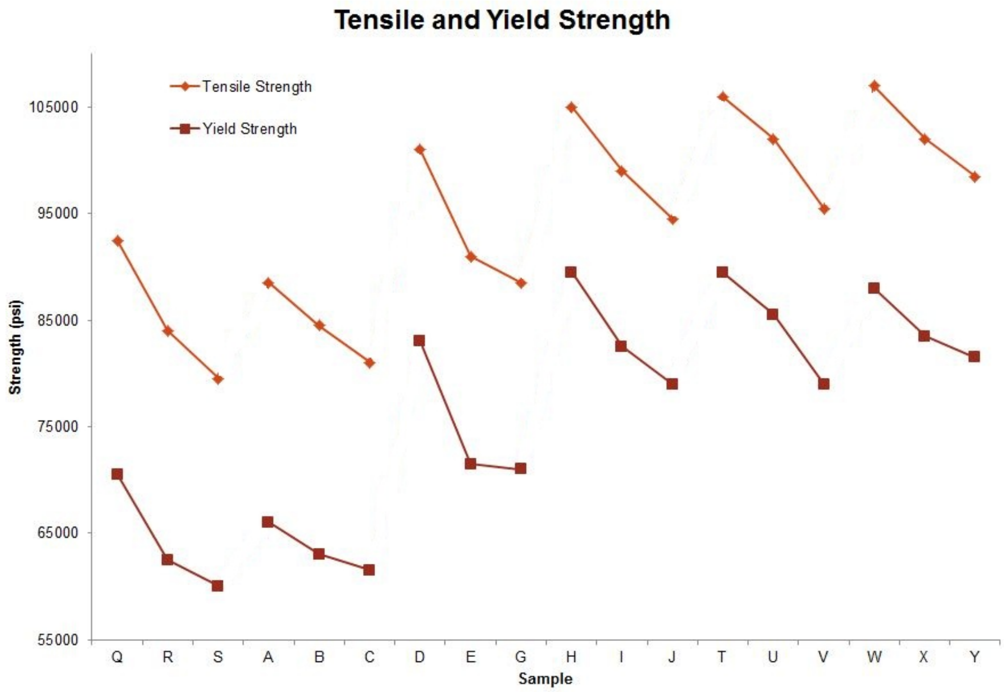
Table V: Mechanical Testing Results in Small Scale Samples*

		Strength (ksi)		Average Toughness (ft-lbs)
	Sample	Tensile	Yield	
1450 intercritical	Q	~~92.5~~	70.5	114
	R	84	62.5	173
	S	79.5	60	237
1500 intercritical	A	88.5	66	155
	B	84.5	63	191
	C	81	61.5	242
1550 intercritical	D	~~101~~	83	122
	E	~~91~~	71.5	121
	G	88.5	71	205
1600 intercritical	H	~~105~~	89.5	80
	I	~~99~~	82.5	98
	J	~~94.5~~	79	143
1650 intercritical	T	~~106~~	89.5	48
	U	~~102~~	85.5	65
	V	~~95.5~~	79	126
1700 intercritical	W	~~107~~	88	38
	X	~~102~~	83.5	44
	Y	~~98.5~~	81.5	93

*Strike through numbers did not pass customer requirements.

While the heat treatment trials were originally labeled alphabetically, they are grouped in Table V by intercritical heat treatment temperature, to better illustrate trending. Results that are struck through indicate failing results according to the requirements of Table IV. Over half of the heat treatments did not pass tensile strength requirements in research scale trials, where they performed too highly. All CVN results passed customer requirements.

With that in mind, 1 in x1 in x 6 in tensile test specimens and ½ in x ½ in x 6 in impact test specimens do not cool identically to a 9.5 in x 11 in production scale cross-section. One could infer that cooling rate would slow considerably as part cross sectional area increased. Thus, to identify the best-suited heat treatment plan for production scale parts from research scale trials, one must pinpoint relatively high strength results. If strength properties are heightened, a slower cooling rate—due to increased section size—will lower strength slightly, without rendering failed parts. Test results graphically highlight the needed balance of passing tensile strength without significantly lowered impact strength (Figure 6).



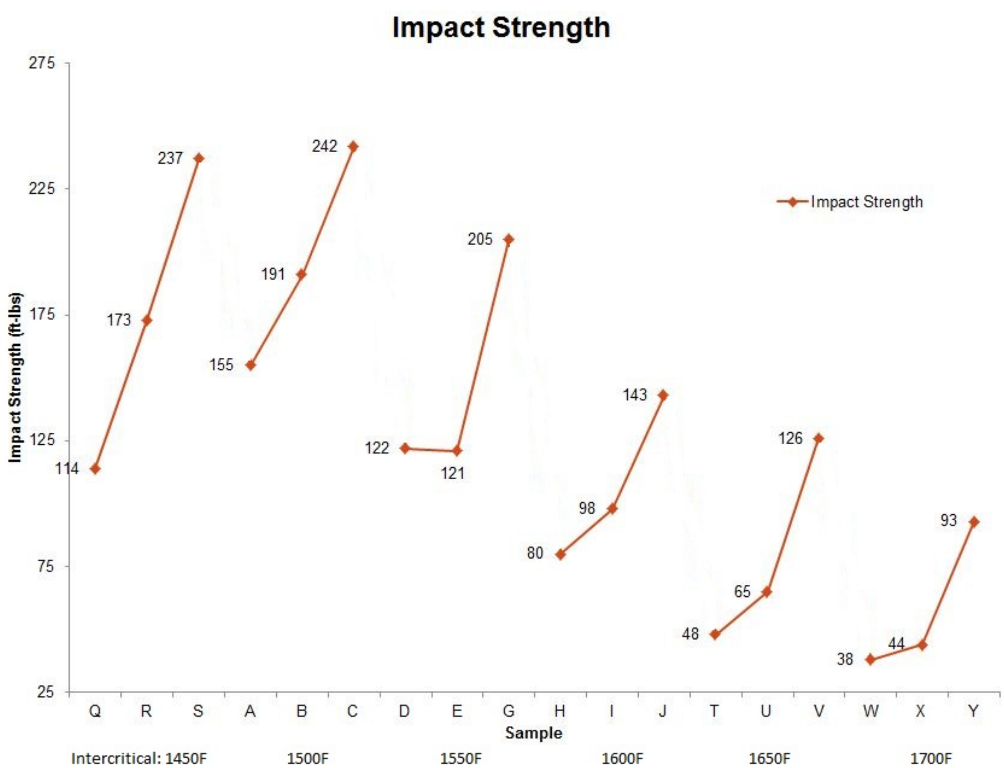


Figure 6: The tensile and yield strength (3) and impact strength (4) of all research scale intercritical heat treatment parts are documented, as grouped by intercritical treatment temperature.

An overall trend, as intercritical heat treatment temperature increased, tensile strength increased as impact strength lowered (Figure 6). However, on a smaller scale, one can observe the effects of tempering temperature on mechanical properties. Each group of three results indicates three samples with the same intercritical temperature while the tempering temperature increased from left to right. As tempering temperature increased both tensile and yield strength decreased, while impact strength increased, as expected. Visually, heat treatments H, I, and J represent an balance of highly passing tensile and yield strength, without lowered impact strength. After discussion with plant metallurgist, Patrick Nowak, heat treatment J was identified as the most viable heat treatment option for production scale parts.

When analyzing the success of various heat treatment methods in research scale testing, microstructure was studied in addition to the mechanical property results. However, due to the small size of specimens, results were less than conclusive. When performing an agitated water quench on such small specimens, the cooling rate was roughly uniform throughout the sample. When cooling rate is uniform, similar microstructures are produced throughout. As a result of inconclusive microstructural data, the decision to move forward with heat treatment J was wholly based upon the mechanical properties previously discussed. Thus testing shifted from research scale lab testing to production parts in active company orders.

Four orders were processed and tested with heat treatment J—austenitization at 1850°F for 60 minutes and water quench, intercritical treatment at 1600°F for 60 minutes/1 in thickness and quench, and 1200°F temper for 60 minutes/1 in thickness and quench. The first three orders passed all requirements, as represented by the top half of

Table VI, however the fourth failed impact strength requirements, with results shown in the bottom half of Table VI.

Table VI: Impact Strength in Production Scale Parts

	CVN Toughness (ft-lb)			
Orientation	1	2	3	Average
L	**99**	**261**	**260**	**207**
T	89	123	111	108
L	292	293	288	291
T	**266**	**112**	**263**	**214**
L	14	11	8	11
T	10	11	23	15
L	6	6	7	6
T	6	5	7	6

With these results came several questions. Most notably: how can successful orders still exhibit variation up to 150 ft-lbs in serialized samples? And how can four orders processed the same way yield three successful orders and one extreme failure?

Samples from all four orders were metallographically analyzed and it was found that all orders exhibited a ferrite-granular pearlite structure; however the unsuccessful samples contained sporadic colonies of a thermally degraded lower bainite (Figures 7, 8).

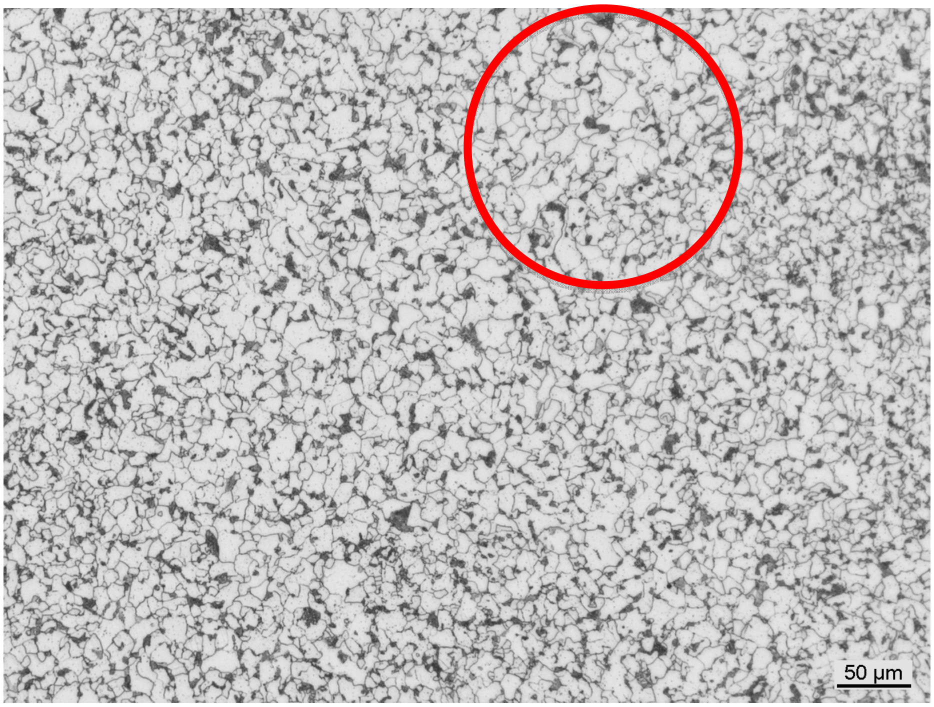


Figure 7: A successful specimen with toughness of 260 ft-lbs, machined from 1/4 thickness, displays uniform equiaxed ferrite and granular pearlite.

Figure 8: A failing specimen with toughness of 11 ft-lbs, machined from 1/4 thickness, displays equiaxed ferrite, with sporadic colonies of needle-like bainite.

The bainite colonies were the only evident cause for lowered impact strength results in the fourth order, as bainite was the only unique microstructural element in failing samples. Why would bainite form in some parts at ¼ thickness but not in others?

If the failing parts exhibited a faster cooling rate than those of the first three successful orders, bainite could be formed. If there were sections of higher hardenability sporadically throughout the material, the nose of a TTT curve could be shifted to the right, allowing a slower cooling rate to more readily form bainite as opposed to pearlite.

There could be grain growth occurring and affecting hardenability if the austenitization temperature and/or time frame were misidentified. If there was micro-segregation evident or an uneven distribution of alloying elements, hardenability would locally change as well. Lastly, another possible explanation could be that bainite formed in the successful parts as well, just at a different depth.

To assess the hypothesis of differing cooling rates as the root cause of bainite formation, records were reviewed and all four orders were heated in the same furnace loads and transfer time from furnace to water quench tank varied by a maximum of two seconds—not long enough to make a difference in parts with 9 in x 11 in cross-sections.

One can isolate cooling rate as a variable and identify the microstructural trends associated with various sample depths and their associated cooling rates. By collecting toughness testing specimens from a number of depths, microstructures and toughness results were observed as a function of depth from quenched surface. With that trend in mind, any non-trend abiding microstructures present would be a result of something other than cooling rate—such as local chemical variation and dependence as discussed.

Conclusions

Various intercritical heat treatment in research size samples exhibits a range of mechanical properties. Intercritical heat treatment J at 1600°F for 60 minutes/1 in thickness and quench, and 1200°F temper for 60 minutes/1 in thickness and quench produced the best mechanical properties. When the aforementioned heat treatment J is tightly constrained in large section size, similar to that of production scale parts, intercritical heat treatment has the potential to provide successful properties at 1/4thickness. However, with the customer's parts in mind, ¼ thickness may not be the most representative testing location in regard to demonstrating how the part will perform in service. When the problem arose, heat treatment as per ASTM A131 yielded a success rate of 25% passing CVN toughness results. When processing production scale parts with intercritical heat treatment J, a 75% success rate was attained. Thus, there is still research to be addressed, but intercritical heat treatment and processing of low carbon, low alloy steel is an improvement.

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