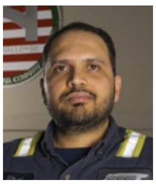
Optimizing Normalizing Cycles to Balance Microstructure and Mechanical Properties of Quenched-and-Tempered High-Strength Steel for Production Requirements



Hot-rolled microalloyed steels present heat treatment challenges due to alloy segregation and recrystallization tendencies. Normalizing can minimize these effects and promote the desired microstructure and mechanical properties. This study evaluated four normalizing strategies for round bars to optimize cycle times for the production requirements. It includes single normalization, double normalization, extended normalization and an in-line furnace pulsing approach. After normalizing, the bars were quenched and tempered, and their mechanical properties were assessed against customer specifications. The findings from this study provide guidance on selecting appropriate processing routes to achieve a balanced combination of microstructure and mechanical properties in round bars.

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Introduction

One of the major applications sectors for heat treated material is energy — typically oil and gas. The competition in this sector is like never before. With ever-increasing demand, there is also an increased expectation for the material used to perform better and last longer to reduce maintenance times. Oil drilling and completion drives equipment demand. Natural gas is a byproduct of many drilling operations. Cheap natural gas in the U.S. has driven a few key trends including power plants switching from coal to natural gas. Several multibillion-dollar liquefied natural gas (LNG) plants have come on-line in recent years. In order to grow production, oil companies must drill enough new wells to offset the decline in existing wells. Newer technologies such as horizontal drilling vs. traditional vertical drilling have greatly increased the average initial production per well and improved the long-term productivity as well. For reference, in 2014 only 60% wells were horizontal, but that number grew to 90% in 2018. Oil and gas markets and operations are divided into three major sections: (a) Upstream — exploration, drilling, completion and production; (b) Midstream — pipelines, storage tanks, tankers and rail tankers; and (c) Downstream — refining and distribution. Engineered bar products are used in upstream processing, exploration (seismic testing, computer modeling, reservoir studies), surface drilling/casing installation, intermediate and production casing and cementing, completion/perforation (perf guns), pressure pumping (frac pumps and flow control iron), production tubing installation, and wellhead/BOP final installation. For all these processes, the typical steel grades that are recommended are for drilling, drilling rigs, drill pipe (with tool joints), drill collars, mud motors and subs (connections), including 4330V, 4145 or 4140. 4330V is a high-strength, low-alloy (HSLA) steel that belongs to the AISI 4330 alloy steel family, usually containing microalloy additions of vanadium, niobium and titanium in various combinations. Even though widely used, heat treating this grade has always been a challenge. Typical test locations for large diameter (>6 inches) are 1 inch below and at mid-radius (MR), whereas mid-radius locations usually show poor ductility. Target microstructure after quenching is expected to be fully martensitic, ensuring homogeneous mechanical properties meeting mid-radius mechanical properties for bars >6 inches compared to 1-inch below indicates potential nonuniform microstructure throughout the bar cross-section. The observed difference in MR and 1-inch results suggested incomplete martensitic transformation due to alloy segregation, poor quenching or insufficient tempering. There is very limited information available on the best thermal processing cycles to follow to obtain the desired mechanical properties. An attempt was made to investigate the effect of thermal processing, in particular normalizing on the mechanical properties.

Materials and Methods

The material used in this study was AISI 4330V, continuously cast as 21.125-inch round billets and further hot rolled into various sizes. Processing of mainly 7- and 7.5-inch-round-diameter bars was reported in this study. Chemical composition of this material is reported in Table 1. Base chemistry was analyzed using optical emission spectroscopy (OES), and C, S were reported through LECO analysis.[1]

The *S&T Hardenability Workbook*[2] showed that this material has excellent hardenability and, even after heavy tempering, the hardness of 39 HRC (365 BHN) can be obtained with a mixed microstructure of at least 50% martensite and remaining bainite or ferrite/pearlite mixture. Hot-rolled microstructures clearly showed the high level of segregation, as shown in Fig. 2.

To minimize the effects of alloy segregation, most industrial heat treatments began processing with normalization. Normalization involves heating the steel above upper critical temperature and cooling in air. Normalizing helps generate new, finer austenitic grains during heating, and cooling follows, forming new, finer ferritic grains with a smaller grain size. Finer grain size improves both strength and toughness which is expected from this alloy grade and it also prepares the material for further hardening treatment. Depending upon microalloy additions, if the steel contains stand-alone V or Nb, then the normalizing temperature requirements could vary. If there is a combination of V and Nb, then much higher normalizing temperature is required to put the complex carbides, nitrides or carbonitrides formed during casting back into the solution.[3–5]

In order to predict the normalization temperatures, the FactSage® Scheil-Guliver module was used to determine the alloy segregation chemistry in the last 15% liquid, which is expected to have the highest possible alloy segregation. Similarly, average composition of the first 85% solid to form was also calculated by weight balance. Using the segregated chemistries as input, carbide

Table 1

Chemical Composition of 4330V Steel (wt. %)

Bal	C	Si	Mn	Mo	Cr	V	Ti	P	S
Fe	0.3100	0.2600	0.9000	0.4700	0.9800	0.0800	0.0016	0.0080	0.0030

Figure 1

Calculated hardenability (a) of 4330V steel and as-quenched, tempered and hardness of 50% martensite (b) using the *S&T Hardenability Workbook.*

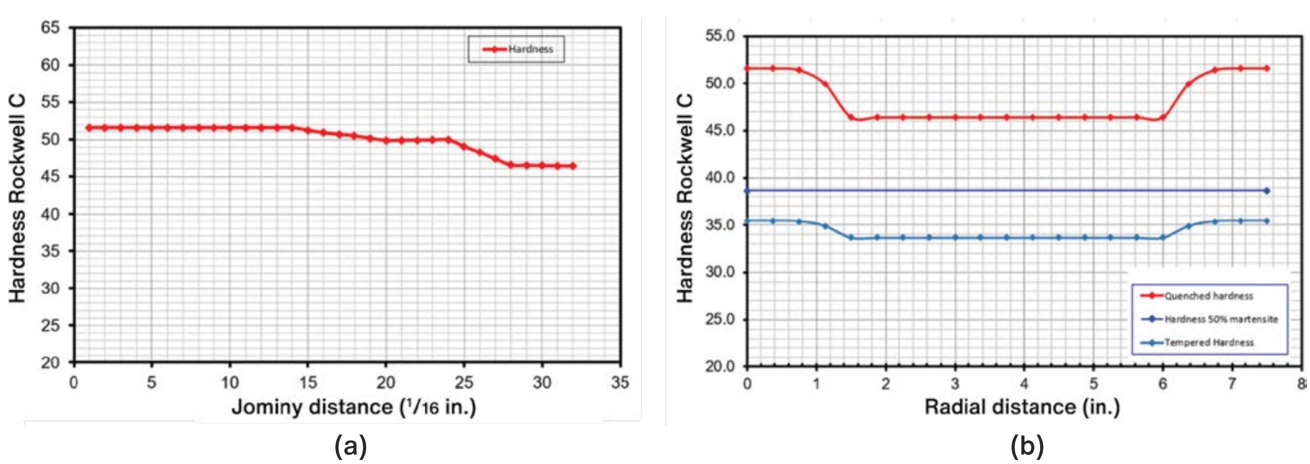
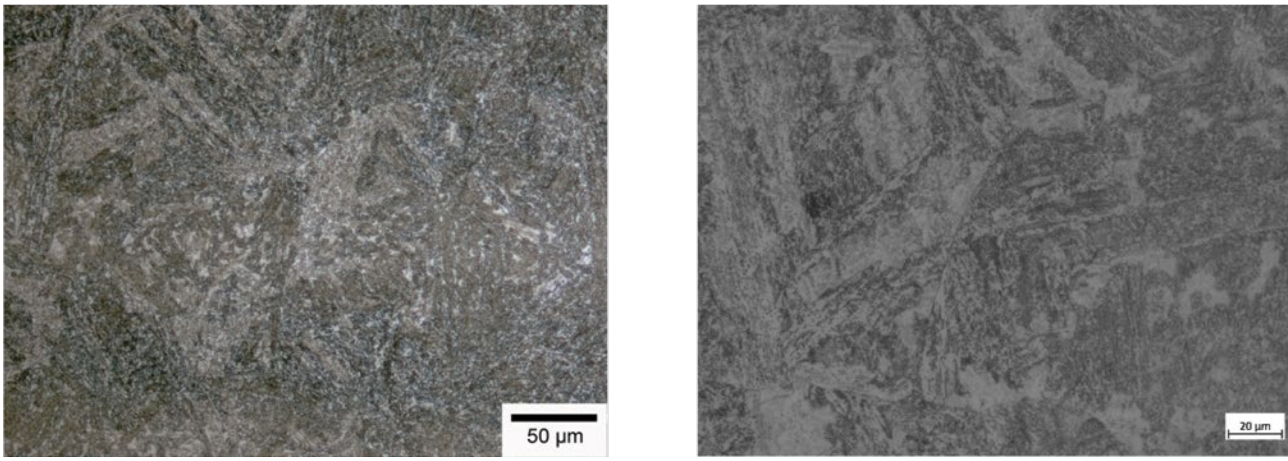


Figure 2

Optical microstructures taken from mid-radius location of a 7-inch-diameter bar showing mostly bainitic microstructure with heavy segregated regions.



(a) (b)

and nitride stability calculations were conducted, and an example is shown in Fig. 3.

It is not necessary to choose the temperature above highest carbide solvus but a reasonably high temperature, 900°C (1,650°F), was selected for the trials. Once the normalization temperatures were selected, the next step was to determine the strategy for the soak time required. Several studies have reported the effect of time and high temperature on the overall performance of this grade. This study used for four different cycles: single normalization, double normalization, extended normalization and pulse normalization. Nucor Steel Memphis Inc.'s heat treatment facility has continuous furnaces. For single, double and pulse (stop and go) normalization, these in-house furnaces were utilized. For the extended normalization, a rotary hearth furnace at third-party location was chosen. Normalization times varied but the remaining thermal processing cycle including austenitizing, quenching and tempering steps were kept constant to minimize any process variation. Typical thermal processing cycles chosen were demonstrated in Fig. 4.

Figure 3

Typical carbide-nitride stability maps for 4330V steel. For this chemistry, the highest carbide solvus temperature was 1,030°C (1,886°F) for M6C carbide.

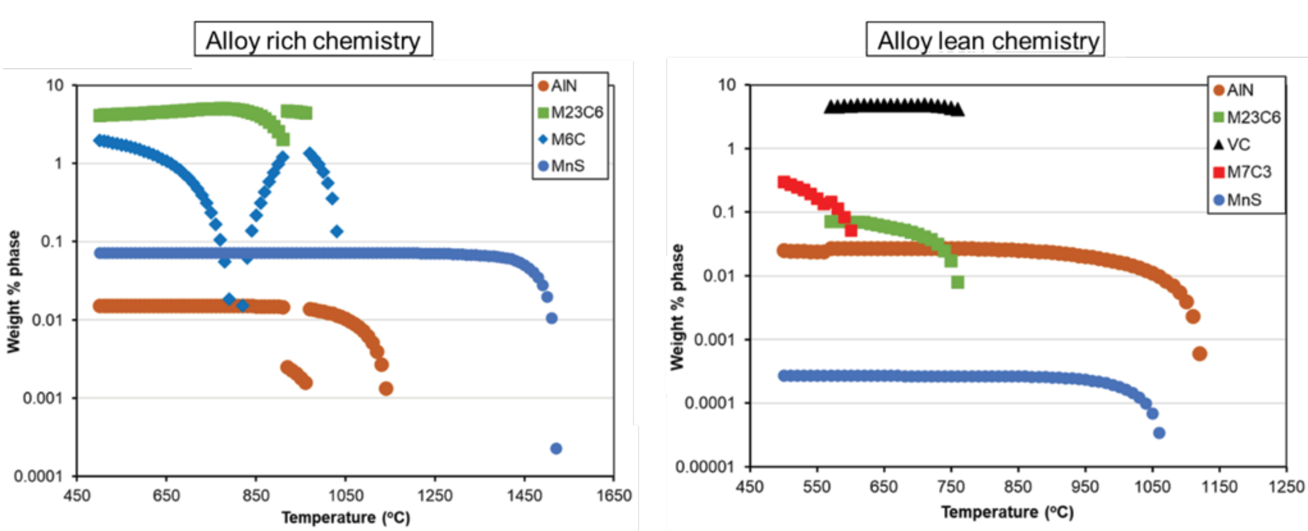
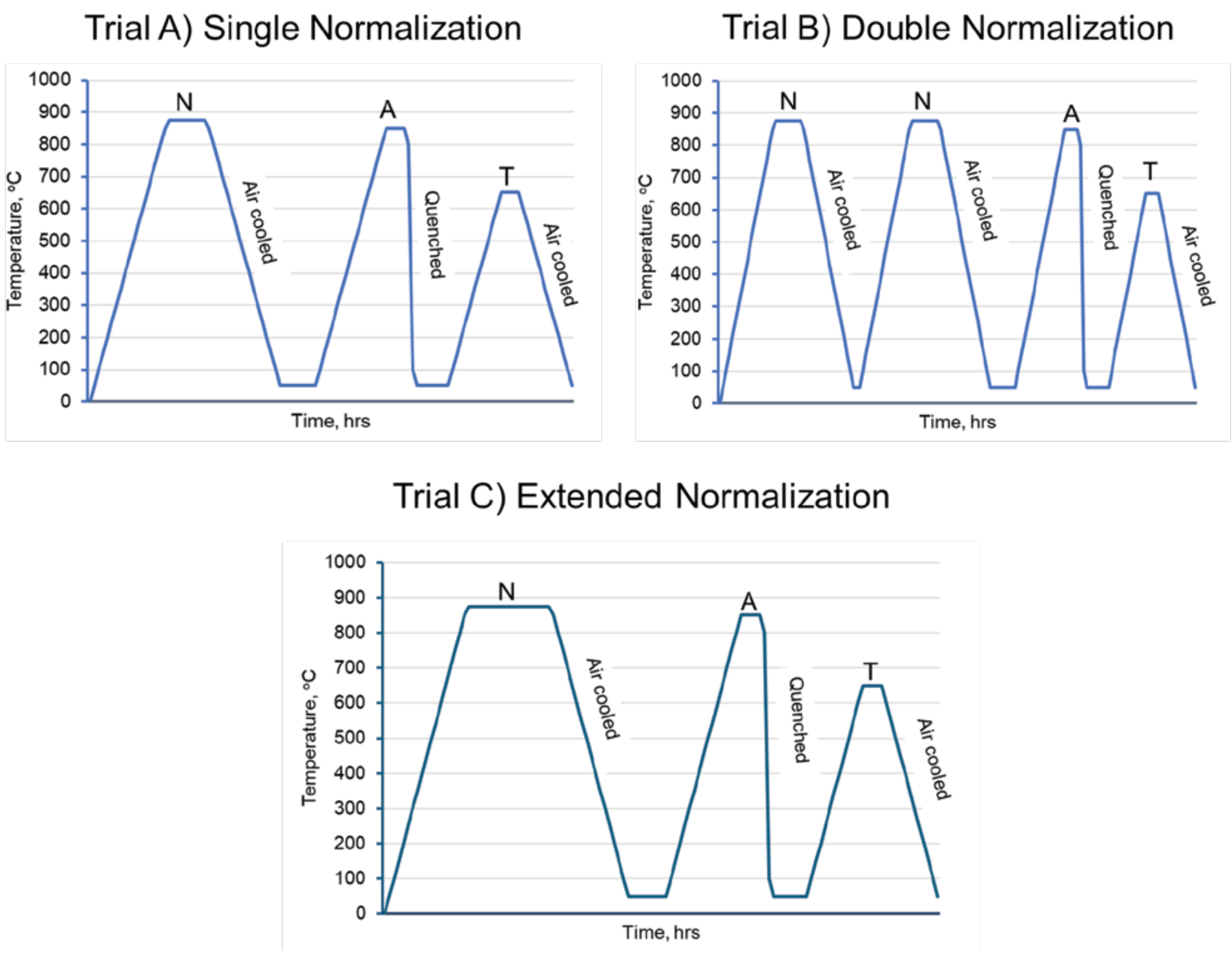


Figure 4

Thermal processing cycles selected for 4330V steel, where N = Normalizing, A = Austenitizing and T = Tempering.



Pulse normalization cycle appears similar to extended normalization hence not included here.

It was noted that the primary issue with the 4330V-NQT (normalized, quenched and tempered) steel was a shorter residence time in the furnace. Single normalization yielded about 90 minutes of residence time; hence the cycle was repeated for double normalization. But the material was cooled to room temperature before second normalization. With the rotary hearth furnace, the material was held at normalization temperature for a minimum of 300 minutes. The final trial of pulse normalization included a "stop and go" technique. This involved moving the bar forward for approximately 1 minute to achieve a 90° rotation and then holding it in place for an equal amount of time. Hardness was measured after normalization and after the finished step of tempering. Various mechanical tests were performed per ASTM standards,[6,7] including tensile testing at 1 inch below and at mid-radius locations. Charpy V-notch impact toughness tests were performed at two different temperatures, room temperature and at –4°F. Samples from mid-radius locations were machined and used for toughness verification. An average of three tests has been reported for Charpy V-notch tests.

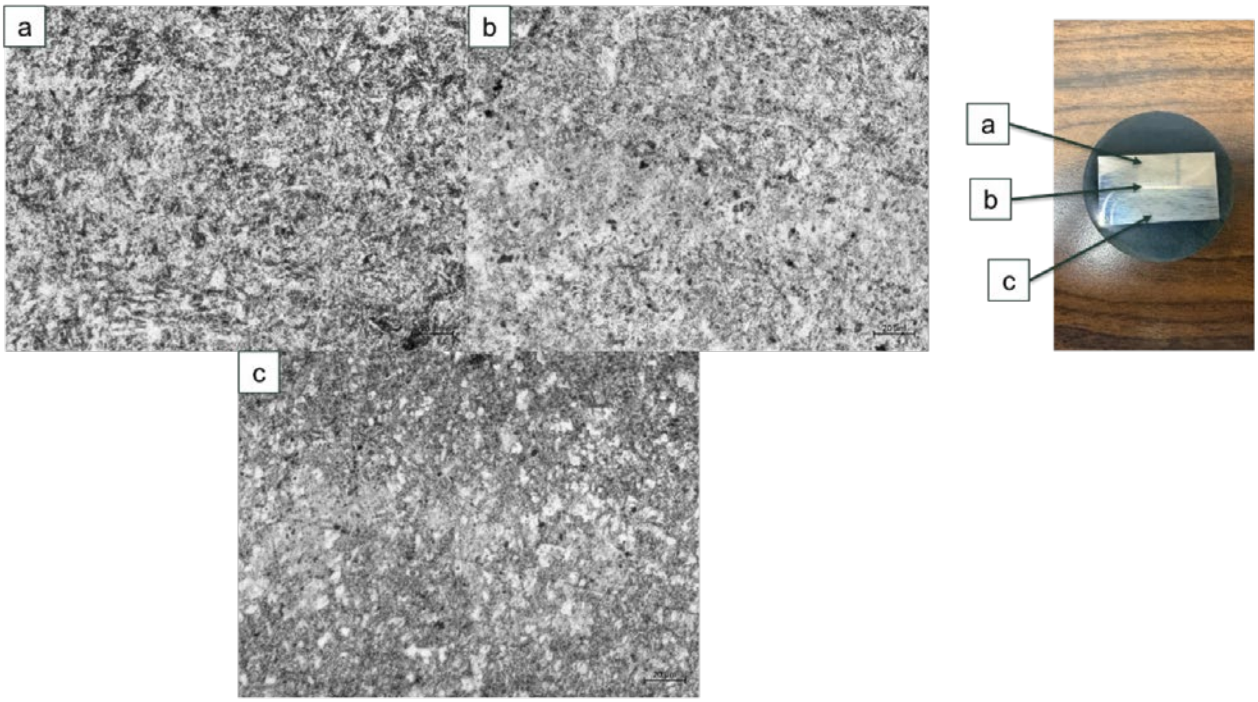
Results and Discussion

Optical microstructures were evaluated after each normalizing cycle. Single normalization was not very effective in reducing the segregation. Extended normalization and pulse normalization did show some improvement, as shown in Figs. 5 and 6, respectively. The samples were taken from the mid-radius location. Standard sample preparations steps including grinding and polishing were followed. Samples were etched with 2% Nital.

Table 2 shows the difference in hardness of normalized material after pulse normalization and single normalization. Pulse normalization yielded a slight increase at the surface level, with a gradient of hardness variations approaching the center of the material. The highest difference in hardness compared to the control sample was

Figure 5

Optical microstructures after extended normalization of 4330V taken from mid-radius location of 7.5-inch round bar and etched with 2% Nital.



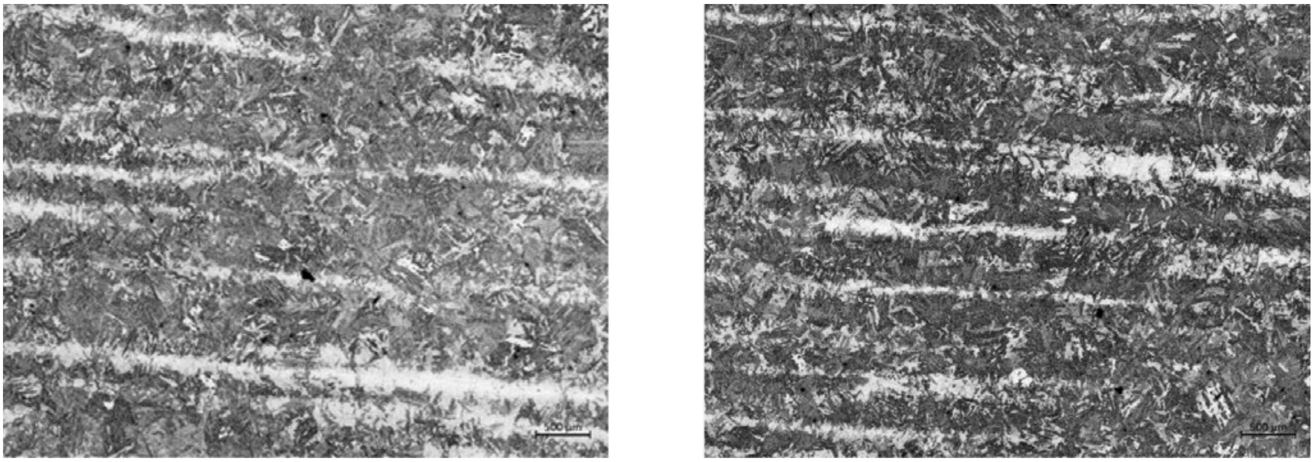
seen to be ~12 HBW, with the center only increasing by ~5 HBW after the pulse normalization.

Tempered hardness, tensile tests and Charpy V-notch test results are summarized in Tables 3–5. All trials met the target range of 321–375 BHN. Representative measured values by location are summarized in the following.

After analyzing the optical microstructures of quenched and tempered 4330V, which was given extended normalization, it was revealed that the microstructure was

Figure 6

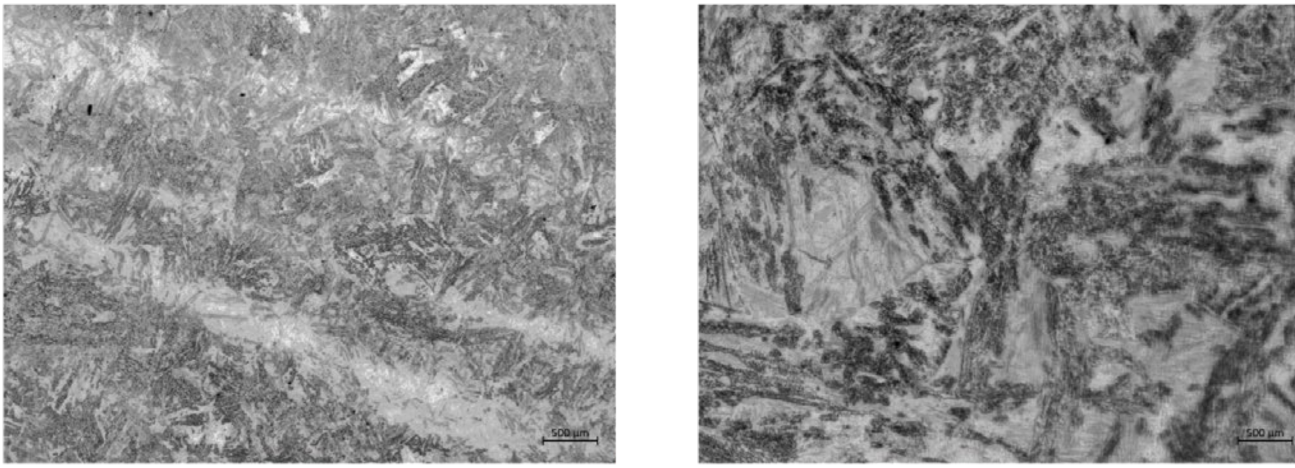
Optical microstructures of the sample after single normalization of 4330V at center (a) and mid-radius (b), indicating the level of segregation and insufficient soaking at normalizing temperature.



(a) (b)

Figure 7

Optical microstructures of the sample after pulse normalization of 4330V at center (a) and mid-radius (b), showing the effects of longer soak time and reduction in segregation.



(a) (b)

Table 2

Hardness Comparison (HBW) Between Trial and Normalized 4330V 7.5-Inch Bar Samples at Various Radial Locations. The ΔH column shows the increase in hardness resulting from the trial processing.

Location	Pulse normalized (HBW)	Single normalized (HBW)	ΔH
Surface	355	344	+ 11
Surface	351	344	+ 7
Surface	351	347	+ 4
Surface	356	344	+ 12
Mid-radius	395	389	+ 6
Mid-radius	385	380	+ 5
Mid-Radius	386	385	+ 1
Mid-Radius	394	385	+ 9
Center	368	363	+ 5

Table 3

Summarized Tempered Hardness (BHN)

Trial	Surface (S)	1 in.	Mid-radius (MR)	Center (C)
A (Single)	344–354	353–359	360–366	352
B (Double)	343–346	346–351	359–362	358
C (Extended)	336–341	342–343	349–350	332
D (Pulse)	351–355	370–375	385–396	368

Table 4

Summarized Tensile Strengths and Ductility at 1 Inch Below and at Mid-Radius Location

Trial	Tensile 1 in. (ksi)	Yield 1 in. (ksi)	Elong. 1 in. (%)	ROA 1 in. (%)	Tensile MR (ksi)	Yield MR (ksi)	Elong. MR (%)	ROA MR (%)
A (Single)	168	155	18.6	57.8	174	159	16.1	46.2
B (Double)	169	157	17.1	56.3	174	160	16.2	47.2
C (Extended)	175	163	17.0	56.0	178	168	14.0	47.0
D (Pulse)	171	157	17.7	51.6	176	160	16.6	45

uniform tempered martensite. For this analysis, the sample was taken from the tensile specimen pulled from the mid-radius location and also Charpy V-notch test specimen from −4°F test lot was selected. Fig. 8 shows the uniform microstructure.

Table 5

Summarized Impact Toughness at Room Temperature and at −4°F. No CVN testing done for Trial D (Pulse).

Trial	CVN MR @ 70°F (ft-lbs)	CVN MR @ −4°F (ft-lbs)
A (Single)	48.1	32.0
B (Double)	48.6	36.6
C (Extended)	48.0	31.0

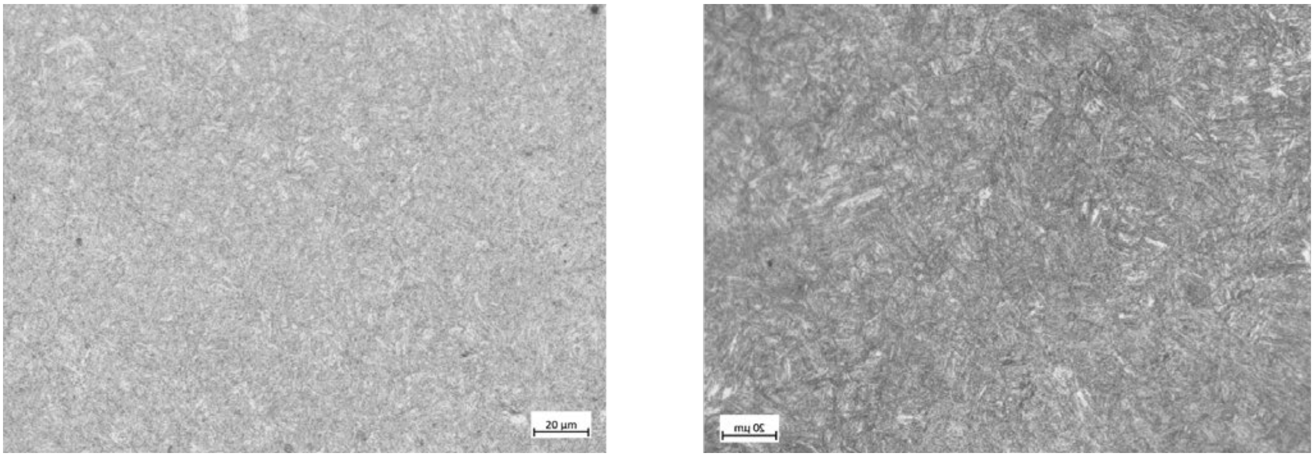
Conclusions

Based on the trials performed and by analyzing the mechanical test results, the following conclusions can be drawn:

- Extended normalization delivered the most uniform microstructure and the highest mid-radius yield strength (168 ksi).
- Double normalization provided the best low-temperature MR Charpy toughness (36.6 ft-lbs at −4°F).
- Normalization strategy should be selected based on target property balance; for strength-critical applications, extended normalization is preferred.

Figure 8

Optical microstructure of tensile specimen (a) and −4°F (b) CVN test specimen from mid-radius location after extended normalization and quench and tempered showing no signs of segregation.



(a) (b)

- Further optimization of normalization time and temper parameters is recommended to raise −4°F toughness without compromising yield strength.

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Did You Know?

Berry Metal Celebrates Its 75th Anniversary

Berry Metal Co. announced it is celebrating its 75th anniversary of innovation and service to the iron and steel industry.

Berry Metal was founded in 1951 by Walter Berry in Harmony, Pa., USA. However, its roots go all the way back to 1918, when Walter and his father, William Berry, patented a tin-free bearing bronze alloy, "Berry Metal." In the years since, the company has evolved from a furnace equipment supplier to focus on steelmaking technology.

"For three-quarters of a century, Berry Metal Company has proudly represented American engineering and manufacturing excellence," said George Boy, president and chief executive officer of Berry Metal.

The company has developed over 50 patents and delivered innovative solutions for blast furnaces, basic oxygen furnaces and electric arc furnaces over the past 75 years it's been in business. These products and solutions have helped steel producers improve safety, productivity and performance.

"We've adapted with every generation of steelmaking technology while remaining true to the values that have defined us since 1951 — innovation, reliability and putting our customers first. Those principles will continue to guide us for the next 75 years," Boy continued.