

Effects of Varying Austenitizing Temperatures on Vacuum Hardening of Type 440C Stainless Steel

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Abstract

Type 440C stainless steel is frequently used to make components that require atmospheric corrosion resistance. It is also one of the few stainless steels which can be hardened to the degree necessary to make anti-friction roller bearings. Vacuum furnace hardening is commonly used for Type 440C bearing components. In this study, the effect of hardening temperature, quenching media (oil or gas pressure quench) and tempering temperature on the final part hardness will be described.

Introduction

Due to an excellent combination of atmospheric corrosion resistance, strength and hardness, 440C stainless steel enjoys widespread use in applications from household knife blades to aerospace components. 440C is a martensitic stainless steel that can be quenched and tempered over a wide range of temperatures, permitting an optimization of hardness, corrosion resistance, and ductility for many different applications [1]. This alloy is commonly used in ball bearing applications with the typical industry standard that the bearing balls and races are heat treated to 58 Rockwell C (HRC) minimum [2]. However, experience has shown that for small parts, e.g. ball bearing components with size scales on the order of a tenth of an inch (a few millimeters), the austenitization temperature of 1925°F (1052°C) that is required by AMS 2759/5 [3], the general specification governing heat treating of stainless steel aerospace components, often results in insufficient hardness. Prior experience has shown that heating to higher austenitization temperatures can help to meet the minimum hardness requirements.

While 440C is used almost exclusively in the quenched and tempered state, which yields a microstructure of tempered martensite, there is considerable concern with the austenite phase that exists during heat treating. One concern is the

growth of austenite grains during austenitization. The presence of austenite that is retained after quenching and tempering in finished bearing components has been a concern to bearing engineers for many decades. Conversion of retained austenite to martensite in service results in dimensional changes which can cause premature bearing failure. It is generally desirable to minimize the level of retained austenite in 440C components since rolling contact fatigue endurance is frequently less important than maximizing dimensional stability.

The goal of this study is to measure the response of quenched and tempered 440C to austenitization over a range of temperatures. Ring samples were chosen that had a thin cross-section to represent typical small-scale 440C bearing rings. A great deal of attention was given to hardness testing and the ability of the specimens to be accurately tested since hardness specifications are typically the only mechanical evaluation that can be specified due to the small size of the parts of interest. The samples were also evaluated using crush and dimensional stability testing as well as metallographically to address the aforementioned retained austenite concerns.

Experimental

A single heat of vacuum arc remelted (VAR) 440C with a starting diameter of 2.875" (73mm) was obtained for this study, Table 1. Ring specimens were machined from the bar that had a thin cross-section, 0.125" (3.2 mm) thick, to represent the small size scale parts of interest, Figure 1.

Table 1. Compositions of VAR 440C used for this study.

| Element | Cr | C | Si | Mn | Mo | Ni | S | P | Cu | Fe |
|----------|------|------|-----|------|-----|------|-------|------|-----|------|
| Weight % | 16.8 | 1.02 | 0.7 | 0.44 | 0.5 | 0.17 | <.001 | 0.01 | 0.1 | Bal. |

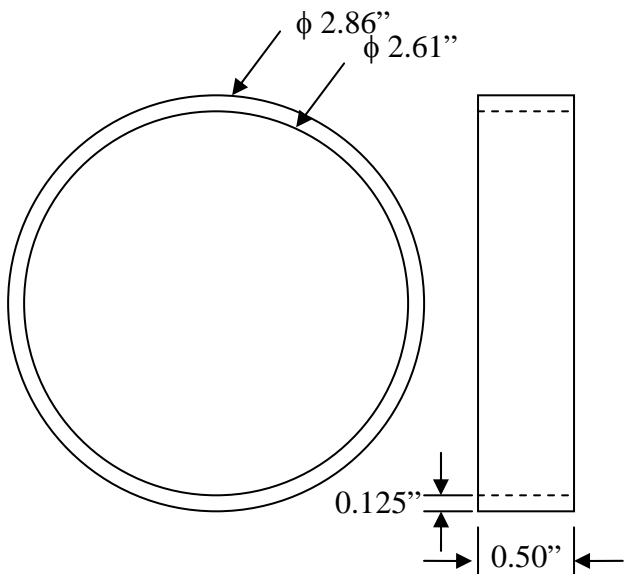


Figure 1: Dimension of ring samples machined from VAR 440C, not to scale.

The samples were heat treated in two different vacuum furnaces, one with an integral oil quench and the other with an integral gas pressure quench. The rings were heated at temperatures ranging from 1900F to 2000F (1038C to 1093C) and soaked at temperature between 30 to 60 minutes after reaching the prescribed austenitization temperature. The oil quench was performed using a 160F (71C) integral agitated oil bath while the gas quenching was done using 6 bar of forced pressure nitrogen. All samples were then double refrigerated and tempered using both the AMS 2579/5 minimum specification limits of -90F and 325F, and an alternative tempering cycle of -120F and 350F. This resulted in three different test groups that were identified as A through C, Table 2. Within these groups, there were as many as five different austenitization temperatures. Please refer to Table A1 in the Appendix for a summary of conversions from degrees F to degrees C for temperatures of interest in this study. After the samples were heat treated, the faces were lapped followed by outer diameter (OD) and inner diameter (ID) grinding.

Hardness: After grinding, Rockwell C hardness testing was performed per ASTM E-18 [4] on the inner bore surfaces of the rings to evaluate the effects of the austenitization temperatures. The hardness tests were distributed evenly around the inner circumference and a total of 2-3 hardness readings were taken per ring with 23 to 30 rings per austenitization condition tested. A large number of hardness tests were performed to enhance the confidence of the statistical analysis.

Crush Test: To provide a screening test for strength and ductility, crush tests were performed on 10 ring samples from each lot. The samples were loaded along a diameter in compression until failure by placing them between two platens

of a Sintech universal test machine. The load and deflection were monitored and recorded digitally.

Table 2: Test conditions for three groups of samples.

| | Test Group A | Test Group B | Test Group C |
|--|-------------------------------------|---|---|
| Vacuum Hardening Temperatures (°F) | 1925 (A1) 1950 (A2) 1975 (A3) | 1900 (B1) 1925 (B2) 1950 (B3) 1975 (B4) 2000 (B5) | 1900 (C1) 1925 (C2) 1950 (C3) 1975 (C4) 2000 (C5) |
| Quenching Type | Oil | Oil | 6-bar pressure quench with nitrogen |
| Temperatures and Times for Double Refrigeration and Temper Cycles | -90F/325F 2 to 2.5 hrs each | -120F/350F 2 to 2.5 hrs each | -120F/350F 2 to 2.5 hrs each |

Dimensional Stability: Thermal cycling can cause retained austenite to transform to martensite resulting in unacceptable geometric changes. To evaluate the susceptibility of the ring samples, two sample rings from each lot were evaluated using a thermal cycle test similar to requirements of MIL B-81793 rev D paragraph 4.8.10 for instrument bearings [5]. The initial sample diameters were recorded followed by thermal cycling between -100F and 300F with 2 complete cycles of 25 hours at -100F and 25 hours +300F. The diameters were then measured again and the geometrical changes were calculated. The maximum permitted size change allowed for bearings of this size is 0.0001 inch of growth per inch of bearing ring diameter or 0.000025 inch which ever is larger [5].

Grain Size: Shepherd fracture grain size measurements were performed on fractured rings to estimate the prior austenite grain size. This test involves fracturing a specimen after quenching and comparing the fracture surface with a set of standard fractures, rated from 1 to 10. This test was initially suggested by B.F. Shepherd in 1934 [6] and has been refined considerably since then [7]. For hardened steels, these results have been shown to correlate well with the micro test in ASTM E112, and the test has been added as an acceptable alternate practice in section A4 [8].

Metallography: The ring samples were sectioned, mounted, polished and etched using standard metallographic practices to reveal the microstructures. Microstructures were compared optically for changes in carbide structure with the varying austenitizing temperatures.

Results

Hardness: Figure 2 is a plot of the Group A (oil quenched with AMS 2759/5 compliant tempering) Rockwell C hardness measurements as a function of austenitizing temperature after tempering. The mean values represent between 60 and 90 hardness readings while the error bars represent ± 4 standard deviations. These standard deviation limits were chosen to be consistent with subsequent statistical process control limits that used a CPK of 1.33 (four standard deviations). It can be seen that for the material used in this study, some of the samples austenitized at and below 1925F would fail the 58 HRC minimum requirement while at 1950F and above, all of the samples would meet the minimum hardness requirement.

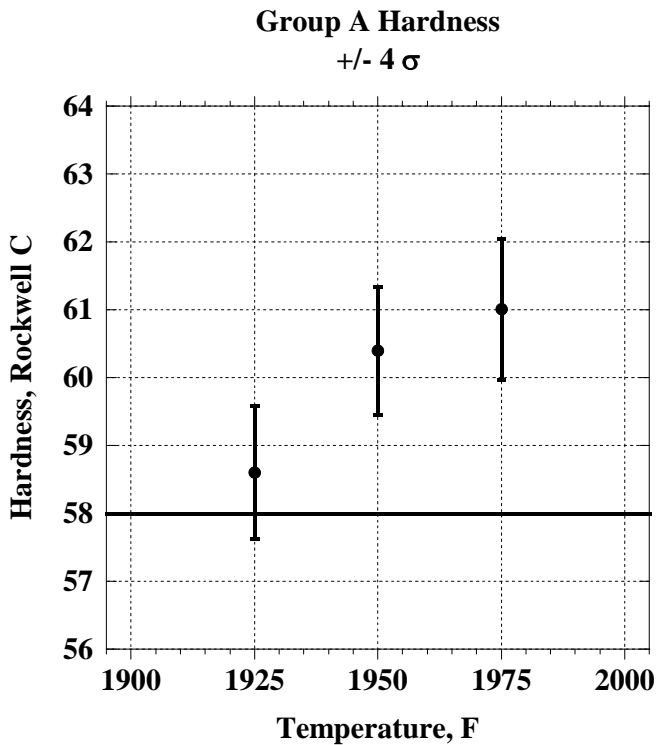


Figure 2: Average hardness results for Group A, oil quenched with AMS specified tempering, with error bars representing ± 4 standard deviations. Some of the samples at 1925F would fail the 58 HRC requirement.

Similar plots were made for Groups B, Oil quenched with Standard Tempering Treatment, Figure 3, and Group C, nitrogen gas pressure quench, Figure 4. These plots showed similar behavior in that the 1925F and below austenitizing temperatures have some samples that fail the 58 HRC requirement. In all cases the hardness positively correlated to the austenitizing temperature.

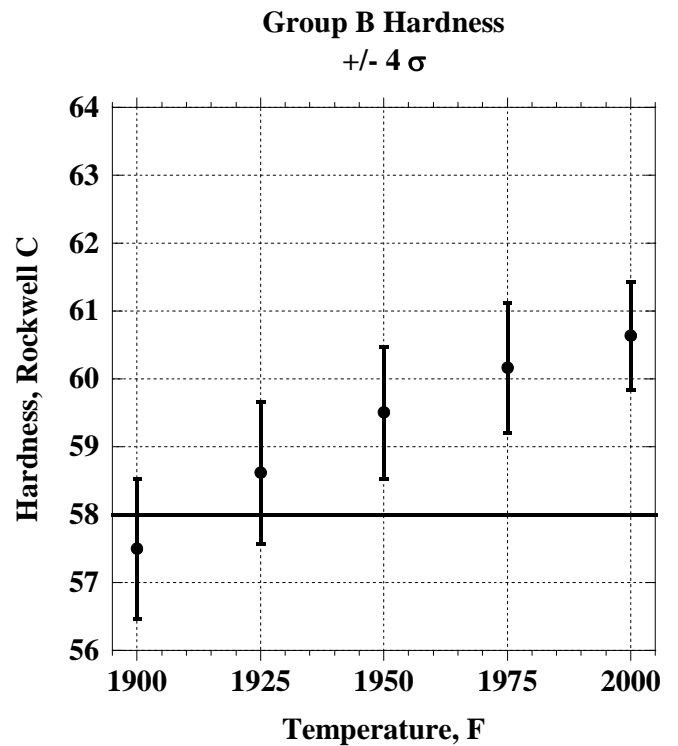


Figure 3: Average hardness results for Group B, oil quenched with standard tempering, with error bars representing ± 4 standard deviations. Some of the samples at 1900 and 1925F would fail the 58 HRC requirement.

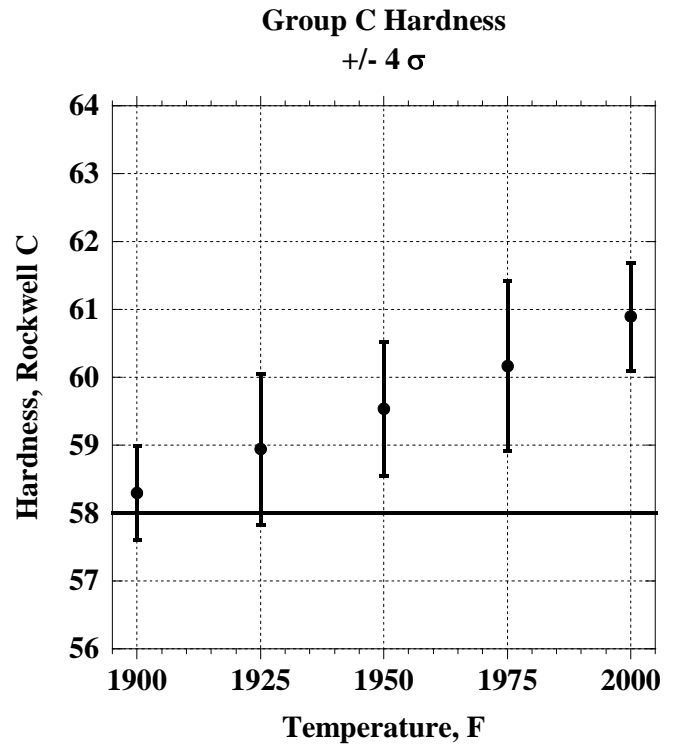


Figure 4: Average hardness results for Group C, nitrogen gas pressure quenched with error bars representing ± 4 standard deviations. Some of the samples at 1900 and 1925F would fail the 58 HRC requirement.

Crush Test: Data results for peak load, Figure 5 and displacement, Figure 6 were plotted for each test group. For both tests, all of the groups performed with similar results and no positive or negative trends were readily observed.

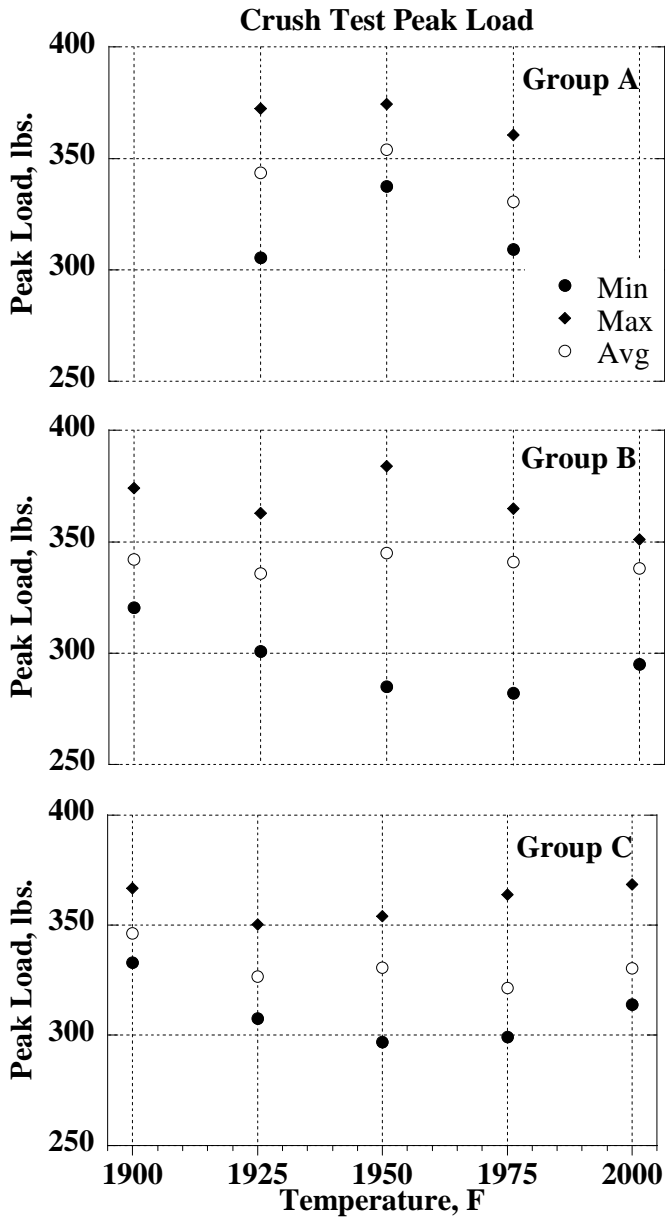


Figure 5: Peak load for crush tests for Group A (top), B (middle) and C (bottom) as a function of austenitization temperatures. Minimum, maximum and average data are shown.

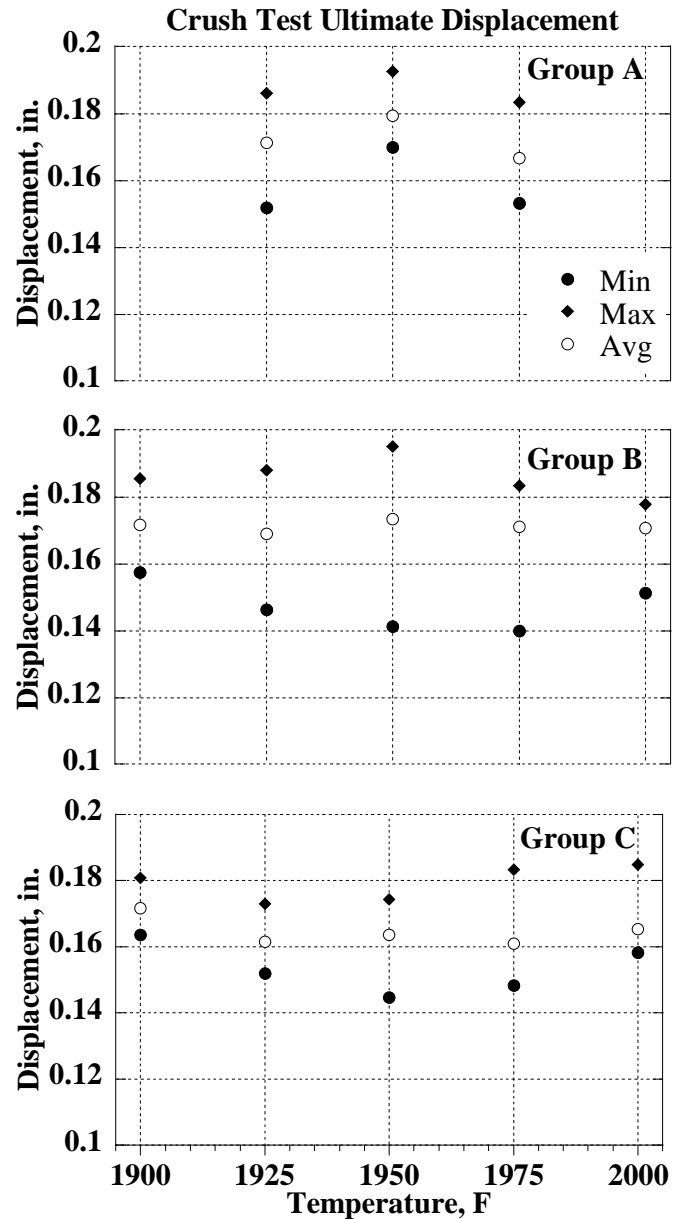


Figure 6: Crush test ultimate displacement for Group A (top), B (middle) and C (bottom) as a function of austenitization temperature. Minimum, maximum and average data are shown.

Grain Size: Shepherd grain size measurements were made on samples which represented the extremes of the hardness measurements, i.e. the minimum and maximum hardness levels, Table 3. The samples were all similar in their prior austenite grain size measurements.

Table 3: Shepherd Fracture Grain Size measurements for both quenching conditions. The prior austenite grain sizes were similar for all processing conditions.

| Test Temperature, °F | Groups A & B Oil Quench | Group C 6 Bar Nitrogen Pressure Quench |
|----------------------|-------------------------|--|
| 1900 | 8 | 8 |
| 1925 | 8 | 8 |
| 1950 | 8 | 8 |
| 1975 | 8 | 8 |
| 2000 | 8 | 7 |

Dimensional Stability: The stability measurements were made on 2 samples from each condition and averaged, Figure 7. All of the measurements were well within the specification limit of 235 microinches.

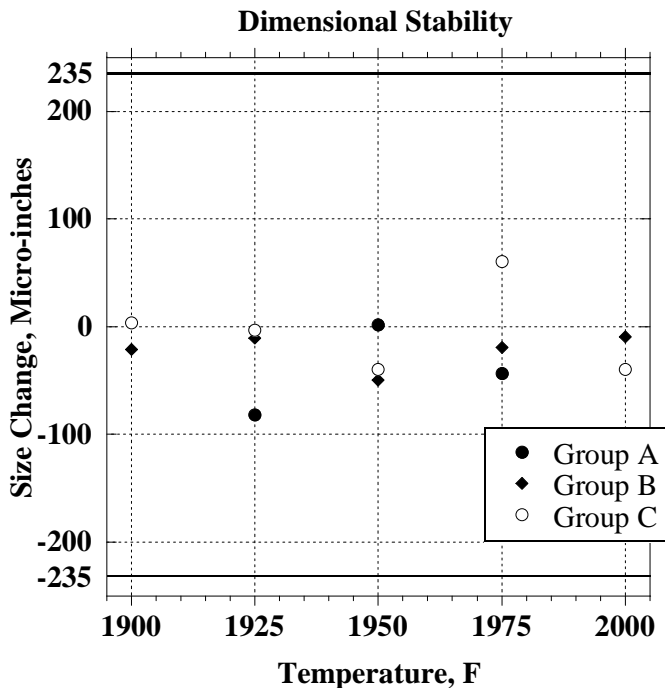


Figure 7: Dimensional stability measurements for all three processing groups. In all cases the dimensional changes are well within the control limits for a bearing of this size, ± 235 microinches.

Discussion

Hardness: As the austenitizing temperature was increased, the hardness was found to increase for all groups, Figures 2-4. Test groups B and C, which had the same tempering conditions, showed essentially the same hardness as a function of austenitizing temperature relationship. Group A, which had slightly different tempering temperatures had similar hardness at 1925F, but higher hardness at 1950F and 1975F. Years of heat treating experience with 440C has shown measurable differences in hardness for nominally identical compositions and heat treatments. Thus, it is not suggested that the numbers

measured should be considered as absolute for all possible compositional variations that are within the specification limits. However, the relative relationship between the hardness values and austenitization temperature could be used as a guide for predicting what changes could be expected for a given heat of material as the austenitizing temperature is changed.

To determine if the observed trend of increased hardness as the austenitizing temperature was increased was statistically significant, analysis was used to compare the hardness values of the samples for each test group (the null hypothesis is that there was no significant difference of hardness as the austenitizing temperature increased). With the large sample size of hardness data collected and assuming a normal distribution of the population, the effect of hardening temperature within each test group was compared. In this analysis, the “Z Test for Determining the Difference between Two Population Means” was applied using a 95% confidence interval [9]. In every case, it was determined that as the hardening temperature was increased, there was a statistically significant increase in resultant hardness. No statistically significant difference was observed between the oil quenched (group B) and the gas pressure quenched (Group C) samples that were tempered with the same conditions.

In industry today, it is typically expected to have controlled processes that have a capability index (Cpk) of 1.33 or better [3]. A Cpk of 1.33 means that the process has at least a +/- four standard deviation range relative to the average. The data from all three test groups indicate that to meet a Cpk of 1.33 with a minimum specification tolerance of 58.0 HRC, then a minimum austenitizing temperature of 1950F must be used.

Thermodynamic calculations for a nominal composition of 440C were performed using MTDATA, Figure 8. In the temperature range of interest, the equilibrium microstructure during austenitizing changes significantly. At 1900F, the $M_{23}C_6$ carbide is significantly more stable thermodynamically than the M_7C_3 carbide. As the temperature is raised to around 2000F, the $M_{23}C_6$ carbide is no longer stable, but rather the M_7C_3 carbide is now stable. At the temperatures in between, the proportion of the carbides which are stable varies. While this is a theoretical calculation, it does suggest that the microstructure is changing substantially as a function of the austenitizing temperature. The final hardness can be expected to be affected by the primary carbide stoichiometry and distribution changes. Though not obvious in the figure, another consequence suggested by this calculation is that the amount of carbon in solution that is in equilibrium increases in the temperature range of interest. This would provide more carbon during the tempering cycle to form secondary carbides. Though not explicitly proven in this work, it is reasonable to expect that both the primary carbide variations and the change in carbon available to the secondary carbides will directly affect the change in hardness. At a minimum, one would typically expect more carbon in solution to result in a higher as quenched hardness [10]. The final tempering steps complicate the analysis of the role of the two mechanisms and make it difficult to provide a definitive statement of which

effect dominates. However, it is reasonable to conclude that a relatively complex change in the distribution and stoichiometry of the primary and secondary carbides exists as the austenitizing temperature is changed.

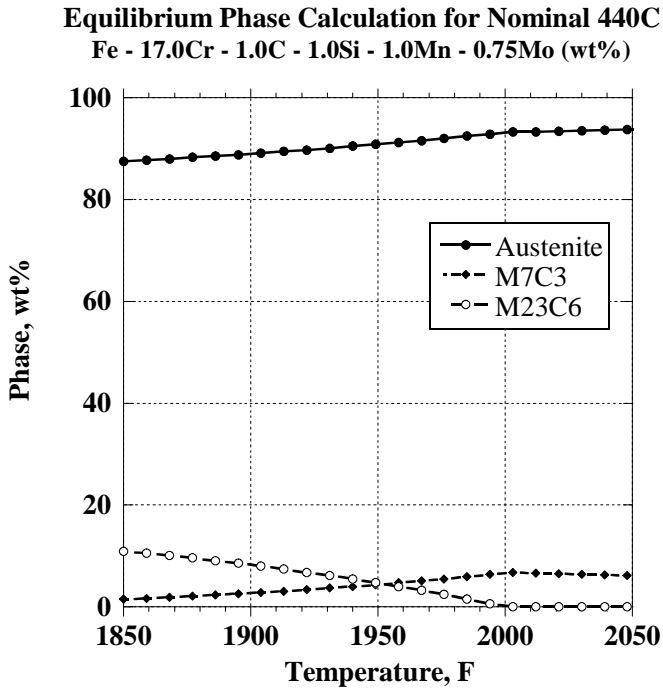


Figure 8: Calculation of equilibrium phases for a nominal composition of 440C. In the temperature range of interest, the equilibrium microstructures changes significantly.

Representative microstructures of the two temperature extremes of the oil quenched specimens are shown in Figure 9. The size of the large primary carbides qualitatively appears to change, with the 1900F austenitization temperature having larger primary carbides. This observation is consistent with the thermodynamic calculations that the amount of austenite increases and that the amount and stoichiometry of the carbides decreases with austenitizing temperature.

While one can not definitively state which of the carbon and carbide reactions dominate the change in hardness, it is reasonable to conclude that it is not due to a Hall-Petch grain size effect. All of the samples tested had essentially the same Shepherd fracture grain size of 8, Table 2. If the increase in hardness were due to changes in grain size, one would expect to see some decrease in grain size as the austenitizing temperature were raised, which was not observed.

Crush Test: Ring crush testing was performed as a screening test to determine if changing the austenitizing temperature would have a significant effect on the mechanical response. Both the peak load and displacement in compression at the ring failure point were measured and compared. The observed trends did not show any obvious differences between the test groups at each temperature, Figures 5 and 6. To insure that this observation was true, the peak load and displacement data was statistically compared. Since the sample size was relatively small, the Two-Sample t test with a 95% confidence

interval for testing differences between two means was used [3]. The specific equations can be found in the Appendix. It was determined that there was no difference between the displacement or peak-load results for all test groups A1 through A3 and B1 through B5. Comparing test groups C1 through C5 (pressure quench tests), one test group, C1 (1900F), was determined to be statistically different than the other C groups. It had higher displacement and peak-load average results. Comparing groups to each other, there was no statistical difference between groups A and B. Group C, with the exception of C1, was statistically different than group A and B (lower resultant peak-strength and displacement). Although statistical significance was calculated in some comparisons, the overall analysis showed no difference between the test groups.

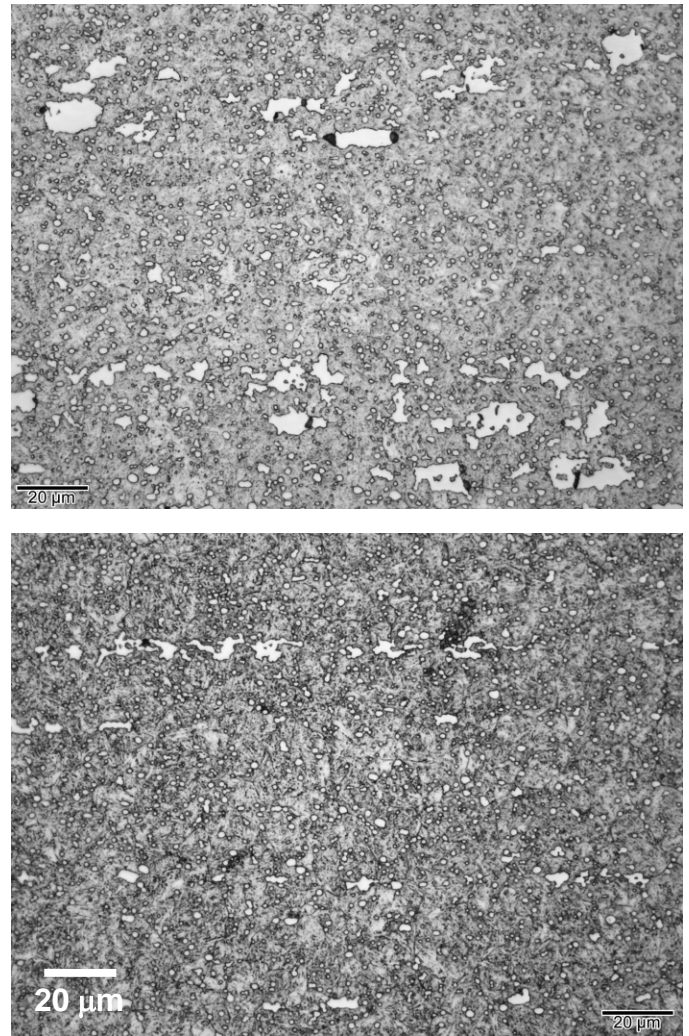


Figure 9: Representative optical micrographs of heat treated samples austenitized at 1900F (Top) and 2000F (Bottom) etched with Vilella's Reagent. The morphology of the primary carbides changed with processing temperatures, with samples austenitized at 1900F being much larger than 2000F.

Dimensional Stability: As with the crush test data, the size change data did not show any obvious systematic relationship

between size and austenitization temperature. All samples tested were well within the permissible limits after thermal cycling. There was a concern that raising the austenitizing temperature would create excessive retained austenite and subsequently dimensional stability would be jeopardized. The data, again, did not support this concern.

Summary: The primary concern addressed in this paper was to determine what temperature would be required to ensure that small section parts would meet a 58 HRC requirement. Using modern statistical process control methodologies, it was determined that 1950F would be required, while the typical industry standard of 1925F would be expected to fail some of the parts. Metallographic examination suggests that the morphology and distribution of the primary carbides changes with the austenitization temperature while thermodynamic calculations support these observations and further suggest that the stoichiometry of the primary carbides may be changing. In light of the microstructural changes observed, it must be noted that if corrosion performance is the primary driver for a particular application, then the corrosion resistance should be examined for the materials austenitized at various temperatures. However, previous work has found encouraging results for enhanced corrosion resistance as the austenitization temperature is increased [11-13].

All crush test data for both the displacement and the peak loads did not suggest any obvious changes in the mechanical properties as the austenitization temperature was varied. Given the ring crush test was considered a screening test, it is possible that more subtle changes may not have been detected given the "averaging" nature of many properties that is captured with the crush test. A final concern with changing the austenitization temperature is with the retention of said austenite after heat treatment. This could lead to problems in a contact fatigue environment, but as previously mentioned, there was no obvious degradation in the mechanical properties that were measured. Additionally, no dimensional stability effects were observed during testing that might be expected if any significant amounts of retained austenite were present after heat treatment.

Conclusions

- For all three of the test groups, hardness increased with increasing austenitization temperature.
- Thermodynamic calculations predict that the amount of, and the specific stoichiometry of the primary carbides changes with the austenitization temperature.
- The microstructures showed changes in the primary carbide morphology and distribution which higher austenitization temperatures having smaller primary carbides.
- The Shepherd fracture grain size measurements were similar for all of the test groups. Thus, the changes in hardness are not due to grain size difference, but rather carbide morphology, stoichiometry and distribution.

- The minimum austenitization set-point temperature to achieve a 58HRC minimum for all three conditions, using a Cpk of 1.33 is 1950F.
- There were no negative effects observed for either dimensional stability or crush test properties within the austenitization range covered in this study.
- The 6 bar nitrogen gas pressure quench produced specimens with mechanical properties similar to those obtained from oil quenching out of vacuum.

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Appendix

Temperature Conversions: The following table can be used to convert from English to SI units for the temperatures of interest in this study.

Table A1 Conversions from English to SI for temperatures of interest in this study.

| °F | °C |
|------|------|
| -120 | -84 |
| -90 | -68 |
| 160 | 71 |
| 325 | 163 |
| 350 | 177 |
| 1900 | 1038 |
| 1925 | 1052 |
| 1950 | 1066 |
| 1975 | 1079 |
| 2000 | 1093 |

Two sample t test: The following equations were used to analyze the data using the two sample t test.

Two sample t Test Formula

$$T = \frac{\bar{X} - \bar{Y} - (\mu_1 - \mu_2)}{S_p \sqrt{\frac{1}{m} + \frac{1}{n}}}$$

Where S_p is the pooled estimator:

$$S_p^2 = \frac{(m-1)S_1^2 + (n-1)S_2^2}{m+n-2}$$

where:

- u1 is the true mean for population 1
- u2 is the true mean for population 2
- X is the sample mean for population 1
- Y is the sample mean for population 2
- m is the sample size from population 1
- n is the sample size from population 2
- S1 is the sample standard deviation for population 1
- S2 is the sample standard deviation for population 2