THE INFLUENCE OF TITANIUM ON GRAIN SIZE IN HIGH-TEMPERATURE CARBURIZED STEELS

by:

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Proceedings of the ASM 20th Heat Treating Conference
October 10-12, 2000
St. Louis, Missouri

Introduction

High heat treating temperatures for some steels can often shorten processing times and decrease manufacturing costs (1). For applications that require case hardening, high temperature carburizing is becoming of greater commercial interest. Recent technological advances in the field of carburizing, evolving from gas carburization to vacuum and plasma carburization, have allowed carburizing cycles at temperatures up to 1093°C (2000°F) (2-3). Aside from shorter heat treatment times, other advantages of vacuum and plasma carburizing include enhanced microstructures by reducing surface oxidation, thus improving fatigue life, and a more uniform case depth (4).

Despite these benefits of vacuum and plasma carburizing, the heat treatment of parts at elevated temperatures often leads to unwanted prior austenite grain growth. It is well known that both strength and toughness of a steel can be improved through the refinement of grain size (5). Therefore, an increased grain size can have a detrimental effect on mechanical properties, compromising the benefits of carburizing.

Microalloyed steels obtain attractive strength and toughness characteristics essentially through the mechanisms of grain size refinement using carbide and nitride precipitates (6). The effectiveness of this strengthening mechanism is largely dependent upon the size of the precipitate (7-9). As a result, precipitate dispersion and size are important factors that must be considered in the design of an alloy.

The purpose of this work is to investigate the use of titanium nitride for controlling grain growth during high temperature carburizing. Specifically, grain refinement effectiveness of various Ti:N ratios and atmosphere effects during carburizing are investigated. Carburizing temperatures examined ranged from 927°C (1700°F) to 1093°C (2100°F).

Experimental Approach

This section contains a description of alloy design rationale and a brief description of the techniques used to characterize precipitates and prior austenite grain sizes. Carburizing specifications are detailed. The methodology for toughness assessment is also described.

Alloy Design Using the SAE 8620 in Table 1 steel as a base composition, four alloys were designed to investigate grain growth during high temperature carburization. Each alloy also has a nominal aluminum concentration of 0.027 weight percent.

Table 1: Steel chemistry of the base SAE 8620 steel in weight percent.

C	Mn	P	S	Si	Cr	Ni	Mo
0.20	0.85	0.013	0.014	0.23	0.57	0.42	0.20

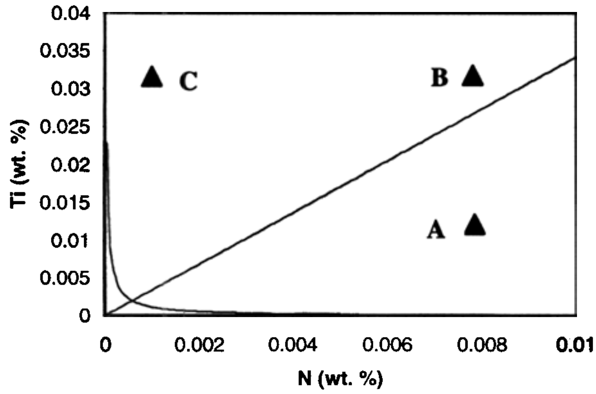
Previous research has indicated that optimal grain boundary pinning effectiveness results from a fine precipitate dispersion (7-9). Due to the stability of TiN at high temperatures and resistance to coarsening, alloying additions of Ti and N were selected in an attempt to form titanium nitride precipitates (10). The ratio of Ti:N in an alloy dictates the solute concentration of the respective elements and, subsequently, the coarsening behavior of the precipitate dispersion. Compositions that are hypostoichiometric, meaning excess (free) Ti in solution, tend to coarsen. Accordingly, a hyperstoichiometric composition, where Ti is limited, tends to coarsen less due to the unavailability of free Ti. Therefore, in order to obtain a fine precipitate dispersion for maximum grain boundary pinning effectiveness, a hyperstoichiometric ratio of Ti:N is preferred in some applications (11).

To examine the effectiveness of varying ratios of Ti:N, thus varying TiN sizes and volume fractions, three different alloys were designed based upon qualitative precipitate size and number density considerations, as shown in Table 2. Alloys designed for this work are represented on a TiN solubility curve in Figure 1. Alloy A, a hypostoichiometric alloy with regard to nitrogen concentration, was designed to provide a dispersion of fine TiN precipitates with a moderate volume fraction. A near-stoichiometric alloy, alloy B, was designed for a greater TiN volume fraction and slightly larger precipitate size. Alloy C, a hyperstoichiometric alloy, was developed to provide excess titanium in an attempt to form TiC during carburization for further grain refinement effectiveness.

Table 2: Titanium and nitrogen concentrations in weight percent for the alloys designed for this experiment. An SAE 8620 steel was used for a base composition.

Alloy	Ti	N
A	0.01	0.008
B	0.03	0.008
C	0.03	0.001

Figure 1: A solubility plot for TiN at 1150°C showing relative solubilities of Ti and N in alloys A, B, and C. A line indicating



stoichiometry is also shown.

Heat Treatments Two 22.7 kg laboratory heats were cast into square ingots for each alloy. Alloys were aluminum killed before the Ti additions were added to avoid titanium oxide formation. Cooled ingots were heated to 1232°C (2250°F), rolled into 5.8cm square bars, and then subsequently cooled, reheated to 1232°C, and then rolled again into 3.3cm square bars.

Simulated and plasma carburized heat treatments were utilized to examine grain growth control effectiveness and precipitate characteristics. Simulated carburizing cycles were conducted in the absence of a carbon-bearing atmosphere, with thermal treatments similar to the plasma carburizing treatments. For plasma-carburized specimens, a target carbon potential of 0.88% and a target case depth of 0.9 to 1.1mm were used.

Simulated and plasma carburizing heat treatments were performed over a range of temperatures to assess how the specified alloy designs will perform as a function of elevated temperatures. A review of current industrial practices indicated a maximum practical carburizing temperature near 1050°C (1922°F). Temperatures selected for this research included 927°C (1700°F), 982°C (1800°F), 1037°C (1900°F), 1093°C (2000°F), and 1150°C (2100°F) for simulated carburized specimens, and 927°C (1700°F), 1010°C (1850°F), and 1093°C (2000°F) for plasma carburized specimens.

Table 3 shows simulated and plasma carburizing temperatures and time at temperature. Time at temperature includes a plasma cleaning time of approximately 35 minutes. Times were established according to current industrial practice

Table 3: Carburizing histories for simulated and plasma carburized specimens.

Simulated or Plasma Carburizing	Temperature °C (°F)	Time at Temperature
Simulated Carburizing	927 (1700)	5 hr. 58 min.
	982 (1800)	3 hr. 26 min.
	1037 (1900)	2 hr. 10 min.
	1093 (2000)	1 hr. 31 min.
	1150 (2100)	1 hr. 10 min.
Plasma Carburizing	927 (1700)	5 hr. 58 min.
	1010 (1850)	2 hr. 24 min.
	1093 (2000)	1 hr. 17 min.

After heating, simulated and plasma carburized specimens were cooled to 843°C (1550°F) and held at temperature for approximately 1 hour. Simulated carburized samples were then oil quenched. Plasma carburized specimens were gas quenched and tempered at 177°C (350°F) to 59-62 HRC.

Precipitate Characterization Titanium-bearing precipitates were examined to characterize the particle size and to verify the basis of the alloy design. Carbon extraction replicas were examined using a Philips CM 200 scanning transmission electron microscope operating at 200kV. Precipitate compositions were determined with energy-dispersive spectrometry (EDS). Precipitates were also examined with light microscopy.

Replicas from each alloy were examined to investigate titanium-bearing precipitate size and morphology. Area fraction calculations were performed based on data collected from ten, randomly selected areas within carbon extraction replicas from each alloy. The number and size of precipitates within a given area was recorded and normalized to a common area observed between alloys.

Prior Austenite Grain Size Analysis Prior austenite grain sizes in simulated and plasma carburized bulk specimens were determined to identify possible grain refinement of the experimental alloys relative to the base composition. Prior austenite grain boundaries were delineated in mounted and polished bulk specimens using a saturated, aqueous picric acid, hydrochloric acid, and teepol etchant. Etching times varied from 15 seconds to several minutes. Prior austenite grain sizes were measured using the circle-intercept method, as detailed in ASTM standard E 112.

Toughness Characterization Previous research has shown that the use of titanium additions in some steels may lead to a significant decrease in toughness (12). For this study, toughness was evaluated in plasma carburized specimens with the Brugger impact test method using impact force measurements (13). Similar to a Charpy V-Notch impact test, the Brugger impact test consists of a specimen that is subjected to an impact force from a swinging hammer. Unlike a Charpy V-Notch test specimen, however, Brugger impact test specimens are carburized and the radius portion of the test specimen more closely resembles a gear-tooth geometry (Figure 2). Three to four replicate tests were performed per alloy. Testing was conducted at room temperature.

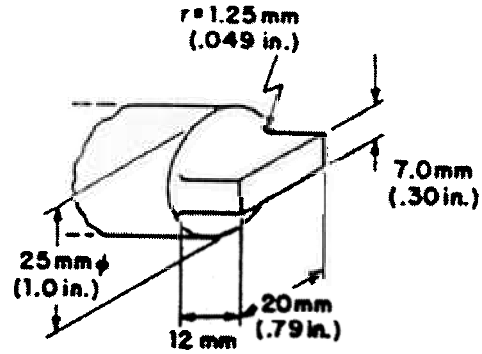


Figure 2: Schematic of one-half of a Brugger impact test specimen. Extended "tab" on cylinder of specimen impacted with hammer (14).

Results

Precipitate Characterization Micrographs of Ti-bearing precipitates in the experimental alloys in the as-received condition are presented in Figure 3.

The morphologies of the titanium bearing precipitates for alloys A and B resembled distinct cuboids and appeared orange when examined under plain light. These observations, in conjunction with EDS data, suggests the identification of these precipitates as TiN. Observations of precipitates in the replicas from alloy C show relatively indistinct, globular precipitates that contain titanium in addition to manganese, chromium, and sulfur. These particles are presumed to be complex titanium carbonitrides, possibly in combination with other distinct phase such as MnS.

Analysis of precipitate size and number count indicate differences between alloy compositions. As Table 4 indicates, the finest average precipitate size was observed in alloy A, with alloys B and C having progressively larger dispersions. Area fraction calculations, analogous to volume fractions, are also presented.

Prior Austenite Grain Size Prior austenite grain microstructures and size data for the simulated carburized specimens are presented in Figures 4 and 5, respectively.

Similar to the simulated carburized specimens, prior

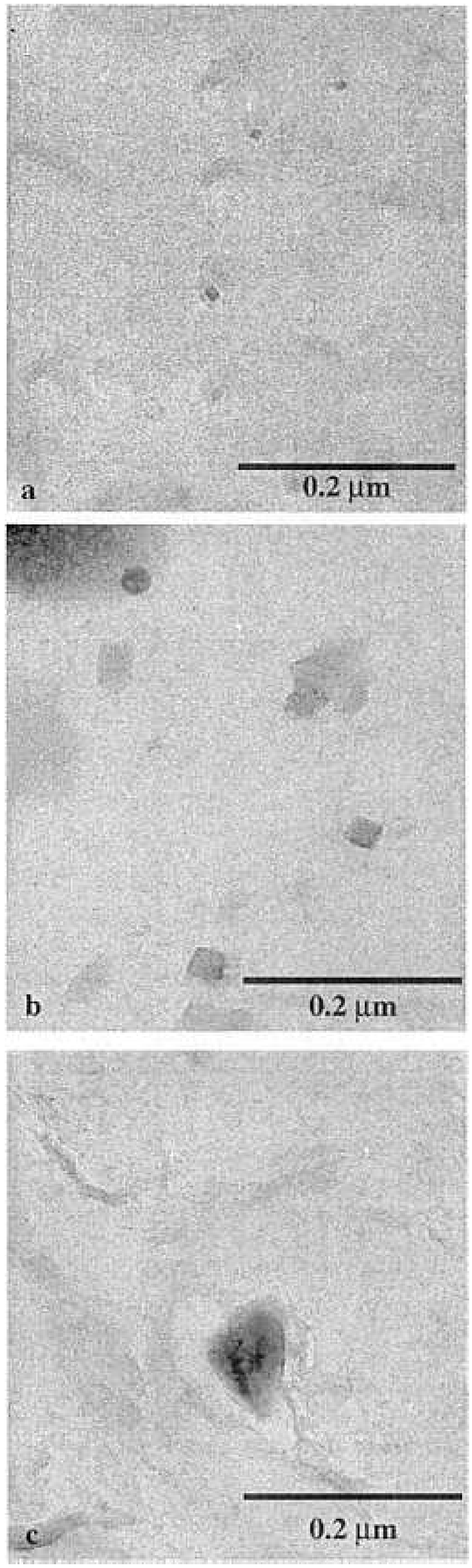


Figure 3: Ti-bearing precipitates from alloys A (a), B (b), and C (c). Note the size difference between precipitates.

austenite grain sizes for plasma carburized specimens were also evaluated. For the plasma-carburized specimens, however, both core and case grain sizes were determined. Results are presented in Figure 6.

Table 4: Ti-bearing precipitate analysis data from alloys A, B, and C.

Alloy	Average Precipitate Size (μm)	Max. Precipitate Size Observed (μm)	Min. Precipitate Size Observed (μm)	Replica Area Fraction
A	0.019	0.070	0.007	2.8
B	0.035	0.110	0.020	9.2
C	0.130	0.325	0.030	0.53

Simulated carburized specimens exhibit evidence of a decreasing prior austenite grain size in Ti-containing alloys relative to the base composition. Results show a refinement of up to 3 ASTM grain sizes for the stoichiometric alloy (alloy B) treated at 1037°C, 1093°C, and 1150°C relative to the base composition. Alloy A also has a notable grain size reduction in the 1037°C and 1093°C simulated carburized specimens, with a reduction of approximately 2.5 and 1.0 ASTM grain sizes respectively.

Grain refinement trends in the plasma carburized specimens are similar to those in the simulated carburized specimens. Grain refining effects of TiN increase with heating temperature. At 927°C to 1010°C, Ti additions have only a small apparent grain refining benefit compared to the base steel. At 1093°C, however, Ti-containing alloys appear somewhat effective for controlling grain growth. Alloy B shows a refinement of approximately 2 ASTM grain sizes in the case and core regions in specimens treated at 1093°C relative to the base composition. Alloy A also exhibited a grain refinement of approximately 1 ASTM grain size in the case and core regions in specimens treated at this temperature. Grain refinement effectiveness for alloy 'C' was not significant in plasma carburized specimens treated at each temperature.

Toughness Characterization Impact force data from Brugger impact testing is presented in Figure 7 for each of the alloys tested at the three different plasma carburizing temperatures. Data represents an averaged value with a 95% confidence interval.

As illustrated by the figure, there does not seem to be a significant difference in impact force between the experimental alloys and the base composition at any of the three plasma carburizing temperatures. A significant decrease in impact force for all alloys and the base composition is evident at the highest carburizing temperature.

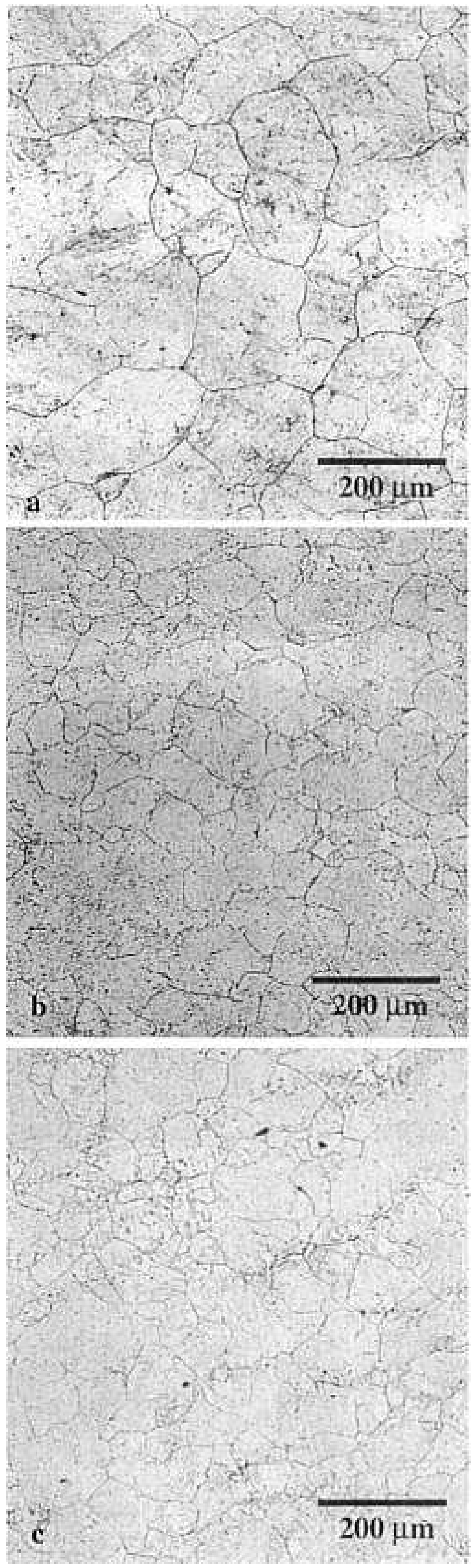


Figure 4: Photomicrographs showing prior austenite grain boundaries in alloys A (a), B (b), and C (c) in simulated carburized specimens treated at 1150°C (2100°F).

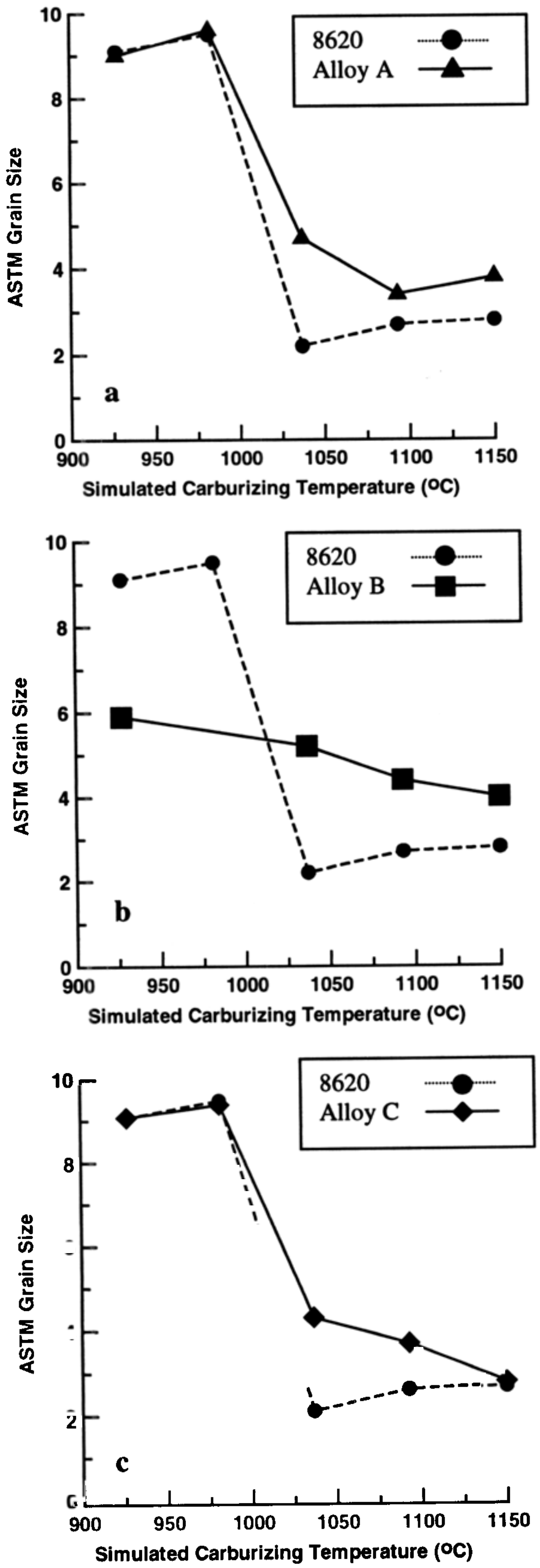


Figure 5: Prior austenite grain sizes of simulated carburized specimens at specified temperatures.

Discussion

Alloy Design and Precipitate Characteristics
Data from extraction replicas of hypostoichiometric (alloy A), stoichiometric (alloy B), and hyperstoichiometric (alloy C) alloys suggest a difference in precipitate size and number density as predicted by the alloy design rationale. Precipitates from alloy A were significantly smaller in size compared with those observed in alloys B and C. This observation is in accordance with the prediction that by limiting the amount of titanium in solution, a fine precipitate dispersion is created. By altering the concentration of titanium and nitrogen, the average titanium-bearing precipitate size varies accordingly. Precipitates evaluated in alloy B, having a near-stoichiometric Ti:N ratio, were slightly larger than the precipitates seen in hypostoichiometric alloy A. Alloy C contained the largest average precipitate size, corresponding with the substantial excess titanium level.

Consistent trends were observed for area/volume fraction calculations. Qualitative estimates of relative precipitate fractions based upon data collected directly from precipitate measurements were in agreement with the expected trends based upon alloy Ti and N concentrations. The position of alloy B with regard to a TiN solubility curve suggests a greater area fraction of precipitates compared with alloys A and C. As illustrated in Table 4, alloy B exhibits the largest area fraction, with alloy A having a slightly larger area fraction than alloy C.

The purpose of controlling the precipitate size and volume fraction is to obtain an optimal precipitate dispersion for maximum grain boundary pinning effectiveness. Precipitate characterization illustrates differences in precipitate size, morphology, and volume fraction between each alloy that agree with the alloy design rationale. Alloy A contains precipitates with the lowest average size (a fine dispersion); therefore a suitable precipitate size for grain boundary pinning is present. However, volume fraction considerations are also important. A discussion of the resulting prior austenite grain sizes, as controlled by the titanium bearing precipitates, is presented in the following section.

Prior Austenite Grain Refinement The primary focus of this project was to attempt to control grain growth during high temperature heat treatments using TiN. The previous section commented upon the presence of a suitable TiN precipitate dispersion for effective grain growth control in alloys A and B, while the relative precipitate sizes appear to be "behaving" as expected, a comparison of prior austenite grain sizes between alloys and the base composition is required to determine grain refinement effectiveness.

At lower temperatures below 1010°C, Ti additions have no apparent grain refining benefit compared to the base steel and may even be detrimental to maintaining a fine grain size according to this work. As the temperature increases, however, grain refinement was observed in the experimental alloys near 1037°C, with a decrease in prior austenite grain size of nearly 3 ASTM sizes. At these temperatures, the TiN dispersion is

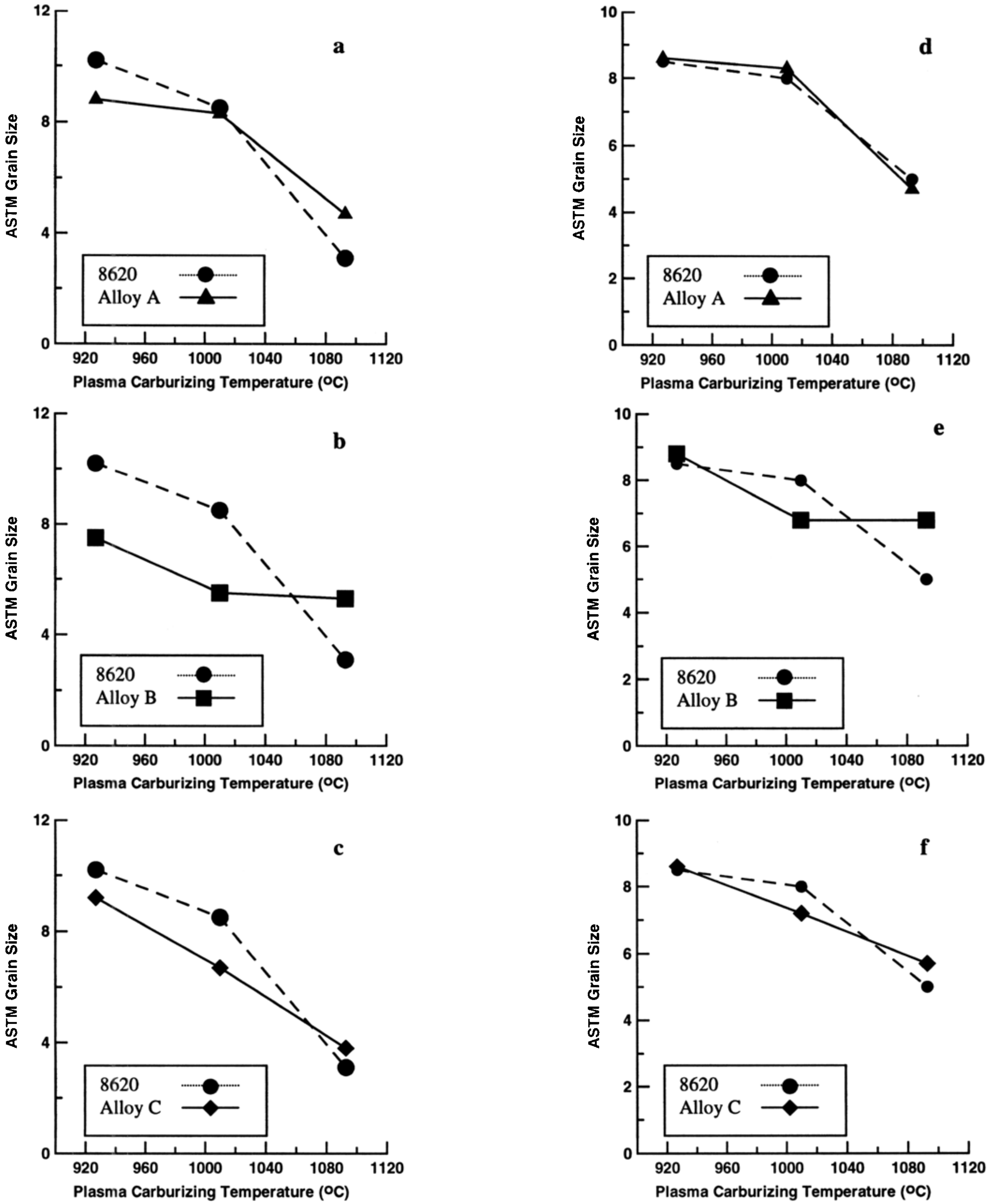


Figure 6: Prior austenite grain size data for plasma carburized specimens. Figures (a-c) represent core grain sizes, while figures (e-f) represent case grain sizes for each alloy.

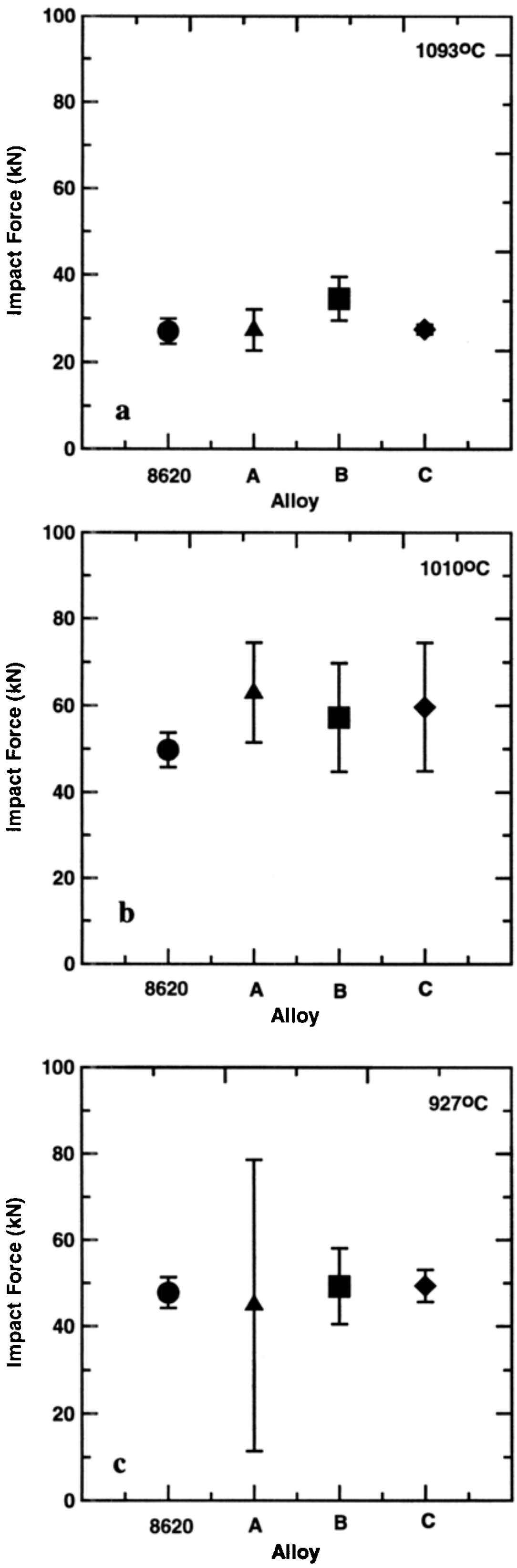


Figure 7: Impact force data for plasma carburized specimens for each temperature.

apparently influencing grain growth. It is possible that the Ti:N ratio used in alloy B is preferred compared to alloy A, due to the increase in volume fraction. Large precipitates, as demonstrated by alloy C, were not effective for controlling grain growth at any of the temperatures examined in this work.

Atmosphere effects (i.e. carburizing) observed included a prior austenite grain size reduction within the case structure of the plasma carburized specimens in alloy B of approximately 1.5 ASTM grain sizes. Further work is necessary to identify the specific effects of carbon potential on precipitates.

Toughness Impact force data from Brugger impact testing was examined to identify possible effects on toughness in the Ti-bearing alloys. As the data illustrates (Figure 7), impact force values were similar for each alloy compared with the base composition at the respective plasma carburizing temperatures. A marked drop in impact force was also observed in the 1093°C carburized specimens compared with results from the specimens treated at lower temperatures.

Further work is necessary to identify the particular parameters responsible for the drop in impact force at the highest temperature. No trends exist between grain size and impact force. It is possible that factors such as retained austenite content, carbide formation, or other microstructural features are responsible for the variances in the impact force data. Ti-bearing precipitates do not have an obvious effect on the toughness of these steels.

Conclusions

This research has examined the use of TiN precipitates for grain growth control during high temperature carburizing. The following conclusions summarize the primary findings:

Precipitate size and volume fraction correlate with a solubility-based alloy design approach through a variation in Ti:N ratio.

- Prior austenite grain refinement was observed in titanium bearing alloys; notably alloy A, the hypostoichiometric alloy, and alloy B, the stoichiometric alloy.
- Grain refinement effects appear to be significant at higher temperatures of approximately 1093°C (2000°F). No evidence of grain refinement from the presence of Ti-bearing precipitates was observed at lower temperatures.
- Impact force data does not indicate a significant effect on toughness between the Ti-modified alloys and the base 8620 steel.

Acknowledgments

This research project was made possible by support from the Advanced Steel Products and Processing Research Center, Colorado School of Mines, Golden, Colorado. Special thanks are extended to Republic Engineered Steels, MacSteel, and The Torrington Company.

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