

# Comparison Between Vanadium and Niobium Effects on the Mechanical Properties of Intercritically Heat Treated Microalloyed Cast Steels

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#### Abstract

In this study, a comparison between V- and Nb-containing low-carbon microalloyed cast steels has been made in terms of strength and impact energy after intercritical heat treatment (IHT). After austenitizing at 950 °C and quenching in water, the alloys were intercritically heated at 760, 800 or 840 °C followed by quenching and tempering at 620 °C. In comparison with conventional heat treatment, IHT increased the impact energies at the expense of some reduction in the strength due to the dual ferritic/martensitic microstructure. However, the impact energy of the alloy containing only V, especially after intercritical heating at 760 and 800 °C, was higher than that of the Nb-containing samples. The improvement in the impact energy required an optimum amount of coarse carbonitride precipitates. While, the impact fracture surface of the alloy V was composed of dimples indicating ductile fracture, cleavage facets in the fracture surface of Nb-containing samples indicated brittle fracture.

Keywords Microalloyed cast steel  $\cdot$  Intercritical heat treatment  $\cdot V \cdot Nb$ 

# 1 Introduction

Microalloyed cast steels are low- to medium-carbon steels containing Mn (1.5–2 wt%) and minor additions of strong carbide- and nitride-forming elements such as Nb, V and Mo [1–3]. These steels have been developed since mid-1970s to benefit from advantages of casting process and microalloyed wrought steels. Strengthening mechanisms in microalloyed wrought steels basically include grain refinement and precipitation hardening. Hence, it is possible to enhance the strength and toughness simultaneously. On the other hand, cast steels overcome the shortcomings of wrought products such as high production cost, restrictions in the product shape and time-consuming fabrication procedures [4–7].

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Thermo-mechanical processing plays a major role in governing the mechanical properties of wrought microalloved steels by controlling carbonitrides precipitation. These precipitates suppress austenite grain growth and promote precipitation hardening [8–11]. Controlling the mechanical properties of microalloyed cast steels only relies on heat treatment. Considerable enhancement in the strength of microalloyed cast steels have been reported after different heat treatment routes [12, 13]. The conventional heat treatment cycles applied to these steels include austenitization (900–950 °C) followed by quenching or cooling in the air, and tempering (600-700 °C) [6, 7, 14]. Microstructural evolution taking place during tempering includes two conflicting events: tempering of martensite and carbonitrides precipitation. While the former leads to softening, the latter results in hardening. Hence, Voigt [15] referred to this stage of heat treatment as "temperaging". However, compared with the wrought grades, lower impact energy has been identified as the most important disadvantage of these alloys limiting their applications. Therefore, another heat treatment route, i.e. intercritical heat treatment, was proposed to surmount the lack of toughness [14, 15].

Intercritical heat treatment (IHT) is an extra heat treatment step carried out between quenching and temperaging. This step includes heating the specimen in the " $\alpha + \gamma$ " twophase region of Fe-Fe<sub>3</sub>C phase diagram followed by quenching or cooling in the air. Heating temperature is between 730 and 860 °C for a steel containing 0.2 wt% carbon. Voigt [15] studied IHT response of a plain carbon cast steel along with two grades of microalloyed cast steels containing Mo and Mo-Nb-V. Based on the observations, IHT had an adverse effect on the mechanical properties of plain carbon and Mo-containing steels due to the slight improvement in the low-temperature impact energy at the expense of considerable decrease in the yield and ultimate tensile strength. On the contrary, the sample containing Nb and V was positively affected by IHT since its impact toughness was improved significantly without considerable decrease in the strength. Tao et al. [16] compared the effects of air cooling with quenching after IHT on the mechanical properties of a microalloyed cast steel. Based on their results, quenching after IHT provided superior impact energy due to the microstructure consisting of ferrite and tempered martensite.

Due to their solubility products, carbonitrides formed by various microalloying elements exhibit different thermal stabilities governing the extent of their dissolution and coarsening at elevated temperatures [8]. Therefore, it is expected that each microalloying element will have its own unique behavior at heating temperature used for IHT, which can affect the final properties after heat treatment. Despite beneficial studies conducted on IHT of microalloyed cast steels, the role of each microalloying element in IHT response of microalloyed cast steels needs more evaluation. Among different microalloying elements, V and Nb are the most important ones. Hence; the aim of this research was to study the influence of V and Nb, individually or in combination, on IHT response of low-carbon microalloyed cast steels.

### 2 Experimental Procedures

### 2.1 Materials

A 100 kg capacity, 125 kW, 3 kHz basic lined induction furnace was used for melting. The base composition was aimed to be a low-carbon (about 0.15 wt%) cast steel containing 1.5 wt% manganese. Graphite and ferromanganese were added to the melts to adjust carbon and manganese contents. After deoxidizing with ferrosilicon and Al shots, ferrovanadium and ferroniobium were used for adding the microalloying elements to the melts. Vanadium and niobium contents were selected to be about 0.1 wt%. Table 1 shows the chemical compositions of the heats. All heats were poured directly from the furnace into the sand molds at 1600 °C in the form of 2-inch Y-blocks prepared as per ASTM E436-84 standard [17]. After casting, the blocks were cut in the form of strips with the dimensions of  $30 \text{ cm} \times 5 \text{ cm} \times 3 \text{ cm}$ .

## 2.2 Heat Treatment

The alloys were heat treated by four different routes including three IHT cycles and one conventional heat treatment (CHT) cycle. IHT consisted of austenitizing, intercritical heating and temperaging. Austenitizing was carried out at 950 °C for 1 h followed by water quenching. Then, the samples were heated at intercritical temperature for 1 h with subsequent water quenching. Intercritical heating was conducted at three temperatures of 760, 800 and 840 °C. Finally, the samples were temperaged at 620 °C for 4 h. For CHT, intercritical heating was excluded and the alloys were austenitized, quenched and temperaged similar to intercritically heat treated samples. It should be noted that the austenitizing and temperaging temperature and time were selected according to the optimum values reported by Rassizadehghani and Voigt [14]. Figure 1 is the schematic diagram for the applied heat treatment cycles.

## 2.3 Mechanical Tests

Tensile, hardness and room temperature Charpy V-notch (CVN) tests were carried out to evaluate the mechanical properties of different alloys. Tensile specimens were prepared as per ASTM E8 standard [18] and tested in a tensile testing machine of 30 kN capacity at a crosshead speed of 4 mm min<sup>-1</sup>. Room-temperature Charpy impact test was carried out on specimens prepared according to ASTM E23 standard [19]. Three samples were tested in each heat treated condition and the average value was reported. Brinell hardness test was conducted using a 2.5 mm ball and load of 62.5 kgf. The hardness values in this paper are the average of at least five indentations.

Table 1	Chemical composition
of the al	loys (wt%)

Designation <sup>a</sup>	С	Mn	S	Р	Si	V	Nb	N
V	0.14	1.4	0.01	0.02	0.16	0.11	_	0.011
Ν	0.17	1.5	0.01	0.01	0.10	-	0.1	0.009
NV	0.14	1.4	0.01	0.02	0.16	0.11	0.09	0.011

<sup>a</sup>CHT and IHT prefixes are added to these designations to indicate the heat treatment applied to each composition



Fig. 1 Schematic representation of the thermal cycles applied for different heat treatment routes

# 2.4 Microstructural Characterization

The samples were prepared by conventional metallographic techniques and etched with 2% Nital. An Olympus PMG3 optical microscope equipped with Omnimet image analyzer was used to observe the microstructures and measure the volume fraction of martensite formed after quenching from intercritical temperatures. The volume fractions were determined based on the area fraction occupied by martensite and the reported values were the average of at least ten measured areas.

Scanning electron microscopy was carried out in a Cam-Scan microscope equipped with an Oxford instruments energy dispersive spectroscopy (EDS) analyzer to observe the microstructures and fracture surfaces of the impact specimens.

# **3** Results and Discussion

#### 3.1 Microstructure

The microstructures of all alloys in as-cast condition consisted of ferrite grains and pearlite colonies (Fig. 2).

Figure 3 shows the microstructure of the alloy V quenched from 760, 800 and 840 °C. Light and dark regions in the figure represent ferrite grains and martensitic regions, respectively. Therefore, it can be concluded that the temperature range of 760–840 °C lies between  $Ac_1$  and  $Ac_3$ . The volume fraction of martensite increased from about 55–70% in the microstructure of the samples quenched from 760 and 840 °C, respectively. The volume fraction of martensite and its variation with temperature in the alloys N and VN were approximately the same as those measured in the alloy V.



Fig. 2 Optical micrograph of the as-cast alloys: a V, b N, and c NV

Scanning electron microscopy of the quenched Nb-containing samples from intercritical temperatures revealed formation of coarse carbon-rich niobium carbonitrides in the lath and grain boundaries (Fig. 4). In comparison with the Nb-containing alloys, fewer and finer carbides were observed in the microstructure of the alloy V intercritically



Fig.3 Optical micrograph of the alloy V quenched from: a 760, b 800 and c 840  $^{\circ}\mathrm{C}$ 

quenched from 760 and 800  $^{\circ}$ C (Fig. 5). Furthermore, the carbides were barely observed in the microstructure of the alloy V quenched from 840  $^{\circ}$ C. This behavior is in accordance with the solubility products of niobium and vanadium



**Fig. 4** Back-scattered micrograph of the alloy N after quenching from: **a** 760, **b** 800 and **c** 840 °C, along with (d) EDS result from indicated precipitate by arrow

2 3 4 5 6 549 cts Cursor: 6.388 keV (549 cts)

carbides. The temperature dependence of solubility products of vanadium carbide and niobium carbide in austenite can be expressed as Eqs. (1) and (2) [20, 21]:

$$\log[V][C] = 6.72 - 9500/T \tag{1}$$

$$\log [Nb] \left| C + \frac{12}{14N} \right| = 3.97 - \frac{8800}{T}$$
(2)

where [C], [N], [V] and [Nb] are the amount of dissolved carbon, nitrogen, vanadium and niobium in austenite (wt%), respectively. Assuming [C] and [N] equal to 0.15 and 0.01, respectively; the amount of dissolved microalloying element in austenite can be estimated at different temperatures. Figure 6 exhibits the changes in dissolved V and Nb with temperature. Niobium carbide precipitation is expected due to the minor dissolution of Nb in austenite in the temperature range of 760–840 °C (0.0001–0.0007 wt%). Moreover, dissolution of 0.004 wt% niobium in austenite at 950 °C indicates that some precipitates remain from austenitizing temperature. On the other hand, the dissolved V in austenite



Fig. 5 Back-scattered micrograph of the alloy V after quenching from: **a** 760 and **b** 800 °C, along with **c** EDS result from indicated precipitate by arrow



Fig. 6 The variation of dissolved vanadium and niobium in austenite with temperature

at 840 °C, being about 0.1 wt%, justifies the insignificant vanadium carbide precipitation in the microstructure of alloy V quenched from 840 °C. Furthermore, the volume fraction of vanadium carbide precipitates is less than niobium

carbide precipitates due to the fact that the solubility of V is approximately an order of magnitude greater than that of Nb in the temperature of 760–840 °C.

The microstructure of the alloy V after different heat treatments are shown in Fig. 7. Ferrite grains and tempered martensite were observed in the microstructure of the IHT samples. The CHT sample only consisted of tempered martensite. It should be noted that the carbonitride precipitates formed during temperaging stage were not detectable by SEM due to their small size (less than 20 nm). Hence, in the following section, they are referred as "fine carbonitrides" to differentiate them from coarse precipitates formed during intercritical heating.

#### 3.2 Mechanical Properties

The yield strengths of the heat treated samples are shown in Fig. 8. Among the samples that were conventionally heat treated, CHT-N sample had the lowest yield strength. Addition of vanadium has led to a significant increase in the strength of CHT-V and CHT-NV samples. The reason for this difference can be related to the different stability of the carbonitrides formed from V and Nb. As described in the previous section, niobium carbonitrides are not



Fig. 7 Optical micrograph of the temperaged alloy V after: a IHT at 760 °C, b IHT at 800 °C, c IHT at 840 °C and d CHT



Fig. 8 The yield strength of the heat treated samples

completely dissolved even at austenitizing temperature (950 °C). On the other hand, vanadium is completely soluble at austenitizing temperature. Thus, the potential for fine carbonitrides precipitation during temperaging stage of CHT-V and CHT- NV samples is greater than that of the alloy N. As depicted in Fig. 9, hardness results also confirm this trend. The hardness of the CHT-N sample is less than the hardness of the CHT-V and CHT-NV samples.

Performing IHT reduced the yield strength of the alloys N and NV compared to those conventionally heat treated. The average reduction in the yield strength of the samples IHT-N and IHT-NV were 8 and 19%, respectively. The reason for the loss of strength can be related to two factors: the formation of ferrite at intercritical temperatures and the coarsening of the carbonitride precipitates. The precipitates formed at intercritical temperatures not only have a marginal contribution to strengthening due to their coarseness and incoherency, but also decrease the amount of dissolved microalloying elements which can enhance the strength after temperaging stage.

As shown in Fig. 8, the yield strength of the alloy V intercritically heated at 760 and 800 °C, like IHT-N and IHT-NV



■ IHT-V ● IHT-NV

144

**4**72

800

Intercritical Temperature (°C)

Fig. 10 Impact energy of the heat treated samples

42

28 39

180

160

140

120

100

80

60

40

mpact Energy (J)

158

760

Fig.9 Yield stress (YS) to tensile strength (UTS) ratio of the heat treated samples

samples, were decreased. However, the yield strength of the sample heated at 840 °C was similar to the conventionally heat treated sample. As stated in Sect. 3.1, unlike IHT-N and IHT-NV samples, coarse precipitates were not observed in the microstructure of the sample IHT-V heated at 840 °C. Therefore, vanadium contributes more effectively to strengthening during the temperaging stage. As a result, the precipitation hardening compensates for the softening due to the formation of the small amount of ferrite.

The yield stress to tensile strength ratio (YS/UTS) is used as a criterion for the reduction in the yield stress after IHT [13, 15]. The decrease in this ratio indicates more reduction in the yield stress after IHT. The values of YS/UTS ratio for the alloys after different heat treatments are shown in Fig. 9. Although, compared to the CHT samples, this ratio decreased after IHT; its reduction is less than 0.03. Therefore, it can be concluded that performing IHT has not had an adverse effect on the strength. In addition, this feature implies the feasibility of improvement in the toughness without significant reduction in the strength.

Figure 10 shows the impact energy of the alloys after various heat treatments. As seen in this figure, the IHTs have improved the impact energy of samples. During initial austenitizing, vanadium (and to a certain extent niobium carbonitrides) is dissolved in austenite. After quenching, these carbonitrides remain in the supersaturated solid solution which leads to nucleation and growth of carbonitrides at intercritical temperatures. Overaging is quite possible due to the remarkable growth and decreasing coherency of the precipitates at intercritical temperatures [15]. Austenitic regions transform to martensite after quenching from the intercritical temperatures. During the temperaging, martensite is tempered while ferrite grains remain unchanged. At this stage, the precipitation hardening resulted from fine carbonitrides formed in ferrite grains and tempered martensite is not significant as a result of coarse carbonitrides formed at the intercritical temperatures. Hence, the dual ferritic/martensitic microstructure containing a relatively coarse carbonitrides distribution in both phases increases the strength and toughness after IHT. On the other hand, the nucleation and growth of carbonitrides in CHT only takes place at temperaging stage resulting in a finer and more uniform distribution of carbonitrides. Consequently, the strength is enhanced at the expense of deterioration in the toughness.

As shown in Fig. 10, the influence of the intercritical temperature on the impact energy of IHT-V was different from that of other IHT samples. While the impact energy of Nb-containing samples did not change remarkably with the intercritical temperature, the impact energy of IHT-V samples intercritically heated at 760 and 800 °C improved significantly. The reason for this difference can be attributed to the size and amount of the coarse precipitates in the microstructure of the alloy V. As described in Sect. 3.1, heating at temperatures of 760 and 800 °C resulted in a smaller coarse carbonitrides compared to Nb-containing samples. In other words, more volume fraction of coarse carbonitrides, part of which remain from austenitizing temperature, in the microstructure of Nb-containing samples has led to the lower impact energy values. However, the reduction in the impact energy of IHT-V specimen intercritically heated at 840 °C indicated that the complete removal of coarse carbonitrides did not result in the impact energy improvement. As a result, in order to improve the

IHT-N CHT-V

CHT-N\

CHT-N

• 87

₹75 €69

840



Fig. 11 Variation of hardness with impact energy of the heat treated samples

impact energy, an optimum amount of coarse carbonitride precipitates was required.

The variation of hardness with impact energy for different heat treatment routs are shown in Fig. 11. The points obtained from CHTs are separated from those obtained from IHTs. In comparison with IHT points, CHT points have higher hardness but lower impact energy. In IHT region, the hardness decreases with impact energy. However, at the same level of hardness, the alloy V shows superior impact energy.

Scanning electron microscopy images of the impact fracture surfaces of IHT-V, IHT-N, and IHT-NV samples intercritically heated at 800 °C are shown in Fig. 12. The fracture surface of the sample V was composed of dimples indicating ductile fracture. The presence of transgranular cleavage facets in the fracture surface of IHT-N and IHT-NV samples indicated brittle fracture. On the other hand, the fracture surfaces of all conventionally heat treated samples, even the alloy V, were dominated by cleavage facets demonstrating brittle fracture (Fig. 13).

# 4 Conclusions

The influence of V and Nb addition, individually or in combination, on IHT response of low-carbon microalloyed cast steels was studied and the resulted mechanical properties were compared to those obtained from CHT. Based on the observations:

(1) Coarse carbon-rich niobium carbonitrides were observed in the lath and grain boundaries of the Nb-



Fig. 12 Fracture surface of the alloy: a IHT-V, b IHT-N and c IHT-NV intercritically heated at 800  $^{\circ}$ C

containing samples quenched from all intercritical temperatures. Fewer and finer carbides were observed in the microstructure of the alloy V intercritically quenched from 760 and 800 °C. Furthermore, the carbides were barely observed in the microstructure of the alloy V quenched from 840 °C.



Fig. 13 Fracture surface of the sample CHT-V

- (2) In comparison with CHT, IHT reduced the yield strength of the alloys N and NV. The reason for the loss of strength was the formation of ferrite at intercritical temperatures and the coarsening of the carbonitride precipitates. However, the yield strength of the alloy V intercritically heated at 840 °C was similar to that of the conventionally heat treated sample due to the coarse carbonitrides dissolution preserving vanadium to contribute to precipitation hardening during the temperaging stage.
- (3) IHT enhanced the impact energies of the alloys. However, the impact energy of Nb-containing samples did not change remarkably with the intercritical temperature. On the other hand, the impact energy of the alloy V intercritically heated at 760 and 800 °C improved significantly.
- (4) The impact fracture surface of the intercritically heat treated sample of the alloy V was composed of dimples indicating ductile fracture. The presence of transgranular cleavage facets in the fracture surface of N and NV samples indicated brittle fracture. However, the fracture surfaces of all conventionally heat treated samples, even the alloy V, were dominated by cleavage facets demonstrating brittle fracture.

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