Influence of Reheating Temperature and Vanadium Content on Transformation Behavior and Mechanical Properties of Medium Carbon Forging Steels

Dragomir GLIŠIĆ,[1] Nenad RADOVIĆ,[1] Ankica KOPRIVICA,[2] Abdunnaser FADEL[1] and Djordje DROBNJAK[1]

1) University of Belgrade, Faculty of Technology and Metallurgy, Department of Metallurgical Engineering, Karnegijeva 4, 11120 Belgrade, Serbia.   2) Institute of Ferrous Metallurgy, Vuka Karadžića bb, 81400 Nikšić, Montenegro.

The influence of vanadium and nitrogen on microstructure and mechanical properties of medium-carbon steels has been studied by means of metallography and mechanical testing. Vanadium addition to the low nitrogen steel suppresses the formation of ferrite–pearlite following the low reheating temperatures and microstructure consists of bainitic sheaves. Increasing nitrogen at the same vanadium level promotes the acicular ferrite formation. For high reheating temperatures, dominantly acicular ferrite structure in both the low nitrogen and the high nitrogen vanadium steels is obtained. The results suggest that vanadium in solid solution promotes the formation of bainite. The effect of nitrogen is related to the precipitation of VN particles in austenite with high potency for intragranular nucleation of acicular ferrite and to the precipitation of V(C, N) particles in ferrite with high potency for precipitation strengthening. Addition of both vanadium and nitrogen considerably increases the strength level, while CVN_{20} impact energy increases on changing the microstructure from bainitic ferrite to the fine ferrite–pearlite and acicular ferrite.

KEY WORDS: V-microalloyed steels; medium-carbon steels; bainite; acicular ferrite.

1. Introduction

The main purpose of vanadium addition to high-strength microalloyed steels is the precipitation strengthening by V(C, N) particles. It is commonly accepted that particles precipitated in ferrite on continuous cooling from the austenitization temperatures during and after the austenite/ferrite transformation are the most effective. However, at high nitrogen contents precipitation of VN particles can occur already in the austenite.[1–3] Those VN particles are proved to be highly potent for intragranular nucleation of acicular ferrite, which could effectively improve toughness of high strength steels.[1] In this manner vanadium addition indirectly enhances the formation of acicular ferrite structure and improves toughness. The effect of nitrogen on microstructure and mechanical properties is primarily related to the interaction with vanadium, as with other alloying elements, such as titanium and niobium.

However, there is some disagreement in literature regarding the effect of dissolved vanadium on transformation behavior of microalloyed steels. It is generally believed that vanadium atoms segregate to austenite grain boundaries rendering them less effective as ferrite nucleation sites. Although many authors suggest that vanadium increases hardenability by preventing grain boundary nucleation, thus suppressing ferrite–pearlite formation,[4,5] there are opposite opinions, that vanadium actually does not suppress grain boundary reaction and even decreases hardenability.[6,7]

The aim of this work was to examine the influence of vanadium and nitrogen contents on microstructure and properties of medium-carbon steels, in attempt to clarify the effect of vanadium on transformation behavior and mechanical properties.

2. Experimental Procedure

Three medium carbon forging steels with varied content of nitrogen and vanadium were melted in a laboratory vacuum furnace and cast into 60 kg tapered ingots. Chemical compositions of the steels are given in **Table 1**, where inscriptions "High-N", "Low-N" and "V-free" indicate vanadium and nitrogen content. The as-cast ingots were reheated to 1 200°C and press forged into 80×80 mm bars. The bars were subsequently reheated to 1 150°C, hot rolled

Table 1. Chemical compositions of experimental steels [wt%].

Steel	C	Si	Mn	P	S	Cr	Ni	Mo	V	Ti	Al	N
High-N	0.26	0.30	1.48	0.0080	0.0080	0.29	0.16	0.03	0.12	0.01	0.02	0.0166
Low-N	0.25	0.34	1.47	0.0060	0.0080	0.29	ND[*]	0.01	0.12	0.01	0.02	0.0057
V-free	0.25	0.32	1.46	0.0070	0.0080	0.28	0.16	0.03	-	0.01	0.02	0.0059

* ND – Not determined

to 22 mm-diameter rods and cooled in still air from finishing rolling temperature ranging between 880–960°C.

The 180 mm long test pieces were cut from the rods, homogenized at 1 150°C and oil quenched. The test pieces were subsequently reheated to different austenitizing temperatures, ranging from 950 to 1 300°C for 30 min, followed by cooling in still air. The temperature was measured by thermocouple placed in the center of the cylindrical specimen and the cooling rate was estimated to be between 1.15 and 1.35°C/s, depending upon the reheating temperature. In all thermal treatments argon as a protective atmosphere was used.

In order to examine the influence of soaking time on transformation behavior, additional samples of High-N steel were reheated to 950°C in dilatometer and held for 10, 180 or 960 min, and cooled at the rate of 60°C/min, so as to simulate cooling conditions in still air.

Room temperature mechanical properties are evaluated from round tensile specimens (l_0=40 mm, d_0=8 mm) and standard Charpy V-notch longitudinal specimens, which were machined from the center of the heat treated test pieces.

Microstructure was examined using conventional metallographic techniques. The transverse specimens were polished and etched in a 2% nital solution.

To determine prior austenite grain size separate set of specimens 15 mm long were reheated to the austenitization temperatures for 15 min, water quenched and subsequently tempered at 450°C for 24 h. Prior austenite grain boundaries were revealed by etching in saturated aqueous solution of picric acid. Austenite grain size was measured by linear intercept method using the network of segregations along the grain boundaries.

3. Results

3.1. Microstructure

Microstructure of the High-N steel air cooled from 950–1 050°C (**Fig. 1**(a)) consists of polygonal ferrite idiomorphs, nucleated at grain boundaries or within former austenite grains, surrounded by pearlite and some acicular ferrite. With increasing the austenitization temperature to 1 150–1 300°C, the fraction of acicular ferrite increases and becomes dominant morphology, alongside with grain boundary idiomorphs surrounded by some pearlite (Fig. 1(b)).

The Low-N steel reheated to 950–1 150°C is characterized by bainitic sheaves nucleated at grain boundaries (**Fig. 2**(a)) and by the absence of primary grain boundary ferrite. For the high austenitizing temperatures (1 300°C), microstructure consists of acicular ferrite, grain boundary ferrite and small fraction of pearlite (Fig. 2(b)).

The ferrite–pearlite structure is predominant in the V-free steel after austenitization at 950°C (**Fig. 3**(a)). Individual plates of intragranulary nucleated Widmanstätten ferrite could also be observed in this microstructure. For the high austenitizing temperature (1 250°C) microstructure changes to Widmanstätten ferrite and some acicular ferrite, while former austenite grain boundaries are decorated with allotriomorphs or idiomorphs, surrounded with pearlite (Fig. 3(b)).
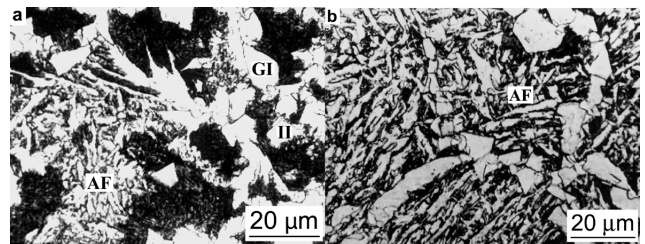

Fig. 1. Microstructure of the High-N steel air-cooled from austenitization temperature of: a) 950°C, b) 1 300°C (GI, grain boundary ferrite idiomorphs; II, intragranular idiomorphs; AF, acicular ferrite).
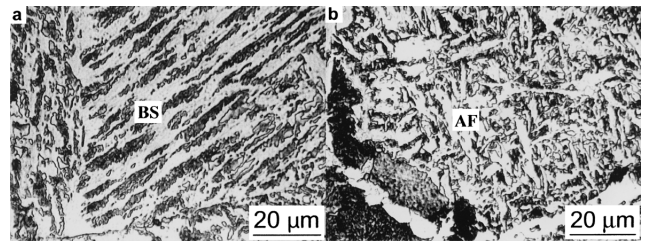

Fig. 2. Microstructure of the Low-N steel air-cooled from austenitization temperature of: a) 1 050°C, b) 1 300°C (BS, bainitic sheaves; AF, acicular ferrite).
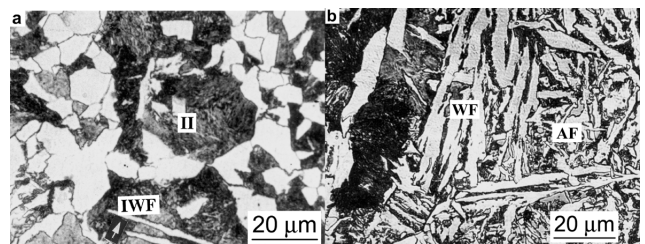

Fig. 3. Microstructure of the V-free steel air-cooled from austenitization temperature of: a) 950°C, b) 1 250°C (II, intragranular ferrite idiomorph; IWF, intragranular Widmanstätten ferrite; WF, Widmanstätten ferrite; AF, acicular ferrite).
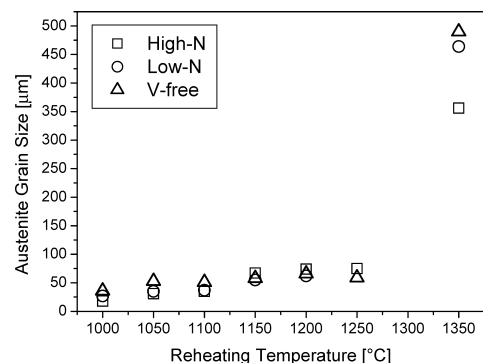

Fig. 4. Austenite grain size as a function of austenitization temperature.

The prior austenite grain size change with reheating temperature is shown in **Fig. 4**. There is a slight increase of austenite grain size up to 1 250°C and abnormal grain growth is evident at 1 350°C.

The microstructures of the High-N steel dilatometer probes are shown in **Fig. 5**. After 10 min the microstructure
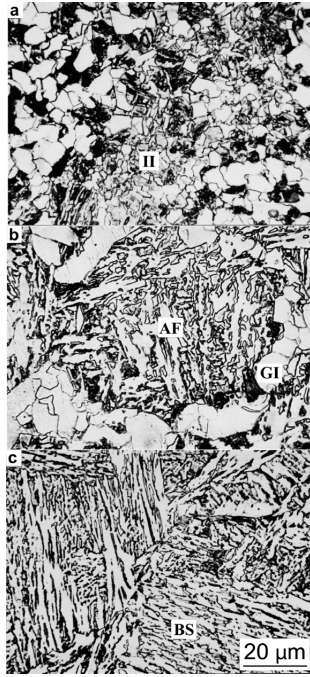
Fig. 5. Microstructures of the High-N steel samples held at 950°C for: a) 10 min, b) 180 min, c) 960 min, and cooled at rate of 60°C/s in dilatometer.
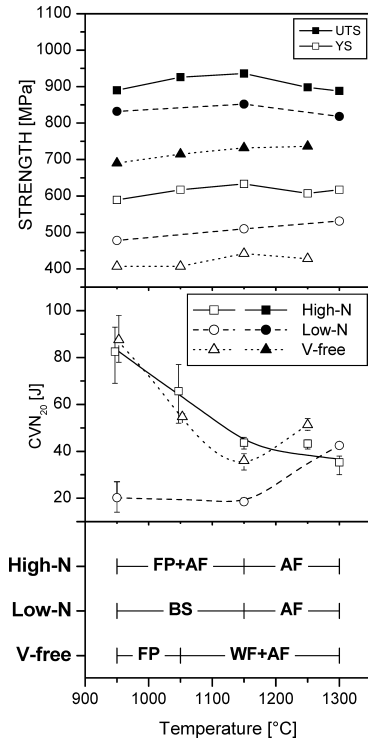

Fig. 6. Effect of austenitizing temperature on strength, Charpy V-notch impact energy at 20°C and structure of three experimental steels: High-N (166 ppm N), Low-N (57 ppm N) and V-free steel (FP, ferrite–pearlite; AF, acicular ferrite; BS, bainitic sheaves; WF, Widmanstätten ferrite).

Table 2. Distribution of alloying elements and temperature for complete dissolution of VN and VC.

	$[V]$ wt %	$[N]$ ppm	$[N]_{TiN}$ ppm	$[N]_{free}$ ppm	$[V]_{VN}$ wt %	$[V]_{free}$ wt %	T_{VN} °C	T_{VC} °C
High-N	0.12	166	29	137	0.05	0.07	1077.3	881.7
Low-N	0.12	57	29	28	0.01	0.11	933.0	879.4

consists mainly of polygonal ferrite idiomorphs nucleated at grain boundaries (GI) or intragranulary (II), surrounded by some pearlite and very small portion of acicular ferrite (Fig. 5(a)). It is similar to the microstructure of the still air cooled sample held for 30 min at 950°C (Fig. 1(a)), but with increased fraction of acicular ferrite. With increasing time to 180 min the fraction of acicular ferrite increases and becomes dominant morphology, alongside with grain boundary idiomorphs (Fig. 5(b)). After 960 min the microstructure consists predominantly of bainitic sheaves (BS), as shown in Fig. 5(c). The prior austenite grain size increased from 10 to 14 μm after 960 min at 950°C.

3.2. Mechanical Properties

Room temperature tensile properties and Charpy V-notch (CVN$_{20}$) toughness are summarized in **Fig. 6** alongside with main microstructural features which change with increasing austenitization temperature from 950 to 1 300°C.

A low strength level in the V-free steel (59 ppm N) is considerably increased by addition of 0.12 % of vanadium in the Low-N steel (57 ppm N). YS and UTS are increased by approx. 70 MPa (from 407–442 MPa to 478–531 MPa) and 120 MPa (from 690–736 MPa to 818–852 MPa), respectively. In the High-N steel (0.12 % V, 166 ppm N) there is further increase in YS by approximately 100 MPa (to 589–633 MPa) and UTS by approx. 70 MPa (to 888–936 MPa).

The Low-N steel has as low CVN$_{20}$ impact energy values as 20 J for austenitization temperatures below 1 150°C, while for 1 300°C impact energy reaches 42 J. The V-free and the High-N steels exhibit an impact energy level of 69–93 J and 78–102 J, respectively, for austenitization temperature of 950°C. With increasing reheating temperature to 1 150°C CVN$_{20}$ impact toughness steeply decreases to approx. 40 J for the High-N steel, and to somewhat lower

values for the V-free steel. For austenitization temperature of 1 300°C the High-N steel impact energy drops to 30–38 J, while the V-free steel exhibits an increase to about 50 J.

4. Discussion

4.1. Austenite Composition

Transformation behavior is discussed in terms of austenite composition at the reheating temperature. Taking the solubility product of alloying elements into consideration the distribution of vanadium and nitrogen in austenite can be rationalized in terms of the temperature for complete dissolution of VN and VC[8]:

$$\log[V][N] = -7\,840/T_{VN} + 3.02 \quad\ldots\ldots\ldots\ldots(1)$$

$$\log[V][C] = -9\,500/T_{VC} + 6.72\ldots\ldots\ldots\ldots(2)$$

It is assumed that the total amount of Ti is within TiN particles, which are insoluble at the reheating temperatures. Using stochiometric ratio Ti : N = 3.4 it could be calculated that titanium ties 29 ppm of nitrogen. The amount of nitrogen left in solid solution, alongside with brief summary of alloying elements distribution is given in **Table 2**. The temperatures calculated using Eqs. (1) and (2) are also given in

the table, depending on the vanadium and nitrogen content.

4.2. The Effect of Vanadium on Microstructure

Microstructures after reheating at low temperatures (950–1 150°C) show that vanadium suppresses the formation of ferrite–pearlite. Formation of hard phases such as bainite in the V-containing Low-N steel (Fig. 2(a)), in contrast to predominantly ferrite–pearlite structure in the V-free steel (Fig. 3(a)), indicates that vanadium increases hardenability. There is a possibility of strong effect of vanadium in solid solution on transformation behavior.

It could be assumed that vanadium segregated toward austenite grain boundaries increasing its surface energy and suppressing formation of grain boundary ferrite that renders grain boundaries inert for bainite nucleation.[9] Similar effect of boron was reported earlier.[10] Segregation of vanadium toward austenite grain boundaries also was confirmed.[7] However, it has been considered by some authors that vanadium actually suppresses bainite formation by preventing the nucleation of coarse grain boundary plate structure.[11] Current results do not support that view.

For high reheating temperatures (1 250–1 300°C) transformation behavior changes in both the V-free and the V-containing (Low-N) steel. It could be expected that, in addition to a larger austenite grain size, the VN particles are completely dissolved at these temperatures. The presence of grain boundary ferrite in the Low-N steel (Fig. 2(b)) imply that the effect of vanadium segregation to grain boundaries and consequently hardenability are somehow diminished. It can be assumed that at high reheating temperatures, due to the thermal dispersion, vanadium is more evenly distributed throughout the austenite grain interior,[12] so it's concentration at grain boundaries is diluted and therefore an inhibiting effect on grain boundary ferrite nucleation is precluded. The increase of austenite grain size at high austenitization temperatures, although relatively modest (Fig. 4), acts in favor of the acicular ferrite formation, since intragranular nucleation is promoted by a greater number density of inclusions than austenite grain surface nucleation sites.[13]

The V-containing steel yet shows higher retarding effect on transformation than the V-free steel, in which dominates the Widmanstätten ferrite (Fig. 3(b)). Due the role of VN particles in intragranular nucleation of acicular ferrite,[1,14,15] it is assumed that acicular ferrite nucleation in the V-free steel is less probable. In this regard, TiN particles shouldn't be omitted, but it seems that they are less potent as nucleation sites, since crystal lattices misfit ratio with ferrite (3.8%) is higher than in the case of VN (1.3%).[10] This consideration and present results support earlier findings that VN particles are preferential sites for intragranular nucleation of acicular ferrite. In fact, VN precipitates on the manganese sulfide particles which then act as sites for the intragranular nucleation of acicular ferrite.[1,14,15] On the other hand, He and Edmonds[7] offer an alternative mechanism for intragranular acicular ferrite nucleation. They suggest that vanadium segregates to austenite grain interiors and form vanadium rich regions or Fe–V clusters that could be energetically favorable sites for acicular ferrite nucleation.

Microstructures of the High-N steel obtained after differ-ent holding times at 950°C support previous assumption that vanadium suppresses the grain boundary ferrite nucleation and therefore promotes bainite formation. The austenitization temperature of 950°C is below the temperature for complete dissolution of VN in the High-N steel (T_{VN}=1 077°C), therefore some vanadium as VN precipitates should be expected. Concentration of vanadium in solid solution increases with soaking time as VN particles dissolve, reaching the equilibrium value in austenite for the temperature given. Different microstructures achieved for different soaking times clearly reflect compositional changes of austenite. After 10 min (Fig. 5(a)) it seems that austenite condition is just slightly different from the air-cooled sample after 30 min at 950°C shown in Fig. 1(a), where besides ferrite–pearlite, also small islands of acicular ferrite are present. Increasing the time to 180 min, fraction of acicular ferrite increases. Continuous network of grain boundary ferrite implies that grain boundary nucleation sites were not neutralized by vanadium segregation, while it seems that the number density of VN particles was high enough to favor the intragranular nucleation of acicular ferrite (Fig. 5(b)). The absence of grain boundary ferrite after 960 min (Fig. 5(c)) leads to the conclusion that concentration of the vanadium in solid solution was sufficient to segregate to the grain boundaries, rendering them ineffective as ferrite nucleation sites. It shouldn't be omitted that for an effective grain boundary segregation effect low grain boundary mobility is required.[16] This condition could be considered as fulfilled, since the average grain size increase from 10 to 14 μm after 960 min at 950°C is negligible.

4.3. The Effect of Nitrogen on Microstructure

Comparing microstructures of the High-N and the Low-N steels for lower reheating temperatures (950–1 150°C), it seems that higher nitrogen content suppresses the formation of bainite (Fig. 1(a)). In the High-N steel great deal of vanadium is assumed to be in VN at 950°C (temperature for complete dissolution of VN is 1 077°C, Table 2), implying that less vanadium is available to segregate to grain boundaries. Therefore, grain boundary ferrite formation is not suppressed and intragranular nucleation of acicular ferrite is promoted (Fig. 1(a)). In the Low-N steel all of the vanadium is available to segregate to grain boundaries (T_{VN}=933°C) and reduce its potential for grain boundary ferrite nucleation, promoting bainite formation (Fig. 2(a)). The presence of acicular ferrite in the High-N steel microstructure (Fig. 1(a)), in contrast to bainitic structure in the Low-N steel (Fig. 2(a)), as observed previously,[1] indicates an effect of nitrogen. High nitrogen creates the more favorable condition for VN precipitation on MnS inclusions which then serve as the preferred sites for acicular ferrite nucleation.[1]

For high austenitization temperatures (1 150–1 300°C), microstructures of the High-N and the Low-N steels are practically the same—they consist predominantly of acicular ferrite structure (which is favored on transformation from a large austenite grain size[13]) with grain boundary ferrite (Figs. 1(b) and 2(b)). The fact that the microstructures of both the High-N and the Low-N steels are similar indicates that 28 ppm of N in the Low-N steel seems to be sufficient to promote the precipitation of VN on MnS parti-

cles, which then serve as the nucleation sites for the formation of acicular ferrite. Recent results of Garcia-Mateo *et al.* show that VN particles formed in austenite are particularly effective as acicular ferrite nucleation sites.[14] In that respect, it could be assumed that even at low nitrogen content VN particles precipitate in austenite when cooled from high austenitizing temperatures since there would be enough vanadium available in the grains interior due to thermal dispersion.

4.4. Strength

The strength increase with increasing vanadium and nitrogen contents, shown in Fig. 4, is principally due to precipitation strengthening by V-carbonitride second phase particles.[5,17] As the nitrogen content is increased the precipitation of $V(C,N)$ in basically acicular ferrite and bainitic structure is enhanced and particles become more dispersed.[5] The strength is therefore highest for the High-N and lowest for the V-free steel. The effect of microstructure, which changes with reheating temperature, seems to be small what reflect the small sensitivity of strength to structure.

There is a relatively high increase in strength level in the Low-N vanadium containing steel in respect to the V-free steel, in particular for samples reheated to low temperatures. The temperature for complete dissolution of VN particles in the Low-N steel is 933°C, so that all of the vanadium and nitrogen are in solid solution at 950°C. This implies a possibility for significant precipitation of $V(C,N)$ particles to occur. However, considering bainitic structure of the Low-N steel reheated to low temperatures and given that carbonitride precipitation in bainite is still uncertain,[18] observed precipitation strengthening seems unexpected. It is commonly accepted that considerable strengthening effect arises from particles which nucleate in ferrite, during or after the austenite–ferrite transformation, and that particles precipitated in austenite have weak strengthening effect due the inevitable coherency loss during phase transformation. However, it has been shown recently by thorough TEM examination that actually all carbonitride particles in commercial microalloyed steels are incoherent.[19] Therefore, it is now suggested that carbonitride particles formed in austenite may have significant strengthening effect.[20,21] These results lead to the assumption that the observed strength increase in Low-N bainitic steel could be ascribed to the dispersion strengthening of all carbonitride particles regardless of their origin.

On the other hand, according to the thermodynamic calculations chemical driving force for $V(C,N)$ precipitation in undeformed austenite is generally small and also decreases with decreasing nitrogen (and carbon) content.[5,18] Furthermore, at low nitrogen content extensive precipitation of $V(C,N)$ is delayed to somewhat lower temperatures then calculated by Eqs. (1) and (2).[14] These findings together with a recent hint that there is a possibility of abundant precipitation of nanoscale nitrides and carbides in the bainite[18] offers an alternative explanation for observed strengthening effect in bainitic structure of the Low-N steel.

4.5. Toughness

Toughness is known as a strong structure sensitive property. Therefore, the change of CVN_{20} energy in Fig. 4 reflects microstructural changes. The lowest toughness exhibits the Low-N steel with bainitic structure, in agreement with previous findings.[1] In this type of structure CVN_{20} energy values would depend mainly on size distribution of relatively coarse carbides, serving as a fracture initiation sites.[22] Once critical-sized microcrack is formed by fracturing of particle, crack readily advances through the sheaves of bainite plates with similar crystalographic orientation. Marked toughness increase in the Low-N steel after high reheating temperatures coincides with microstructural change from bainite to acicular ferrite. Fine interlocking acicular ferrite plates/needles without crystallographic parallelism increase energy required for crack propagation through matrix/matrix boundaries.[23]

The highest toughness is exhibited by the fine ferrite–pearlite structure present alongside with the acicular ferrite in the High-N and ferrite–pearlite structure in the V-free steels reheated in the low temperature range. The CVN_{20} energy is decreased with increasing reheating temperature as the fraction of ferrite–pearlite structure is decreased in favor of acicular ferrite in the High-N steel, or Widmanstätten ferrite in the V-free steel. It should be emphasized, however, that these two steels exhibit a highly different strength level and that a high reheating temperature is more relevant to the hot forging process. So a combination of UTS of about 900 MPa and CVN_{20} of about 40 J after reheating at 1 300°C in the High-N steel with acicular ferrite structure seems to be very promising. Moreover, a combination of 1150 MPa (UTS) and either 25–27 J at −50°C or 33–37 J at room temperature (Charpy U-notch impact energy) in steels reheated to 1 200°C with predominantly acicular structure has been reported previously by Ishikawa and Takahashi.[1]

5. Conclusions

(1) Vanadium addition suppresses the formation of ferrite–pearlite and promotes the formation of bainite following low reheating temperatures (950–1 150°C).

(2) Increasing nitrogen enhances the formation of acicular ferrite structure due to precipitation of VN particles, which are preferred sites for intragranular nucleation.

(3) For high reheating temperatures (1 250–1 300°C) acicular ferrite becomes dominant structure in both the low nitrogen and the high nitrogen vanadium containing steels, indicating more favorable conditions for VN particles precipitation and intragranular nucleation.

(4) Strength increases with addition of vanadium and nitrogen due to precipitation strengthening effect of $V(C,N)$ particles.

(5) Impact toughness increases as microstructures change in the following sequence: bainite sheaves, acicular ferrite, and fine ferrite–pearlite.

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