Development and Applications of V-N Microalloying Technology in China

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Abstract: This paper reviews the recent development of V-N microalloying technologies and its applications in HSLA steels in China. Enhanced-nitrogen in V-containing steels promotes precipitation of fine V(C,N) particles, and improves markedly precipitation strengthening effectiveness of V(C,N), therefore, there is a significant saving of V addition in a given strength requirement. V-N microalloying can be used effectively for ferrite grain refinement as well by the nucleation of intra-granular ferrite promoted by VN precipitates in Austenite in V-N steels. Compared with Nb-Ti steels, V-Ti-N steels show superior weldability under large heat input welding due to the ideal ferrite-based microstructure with relatively fine ferrite grain size in CGHAZ. V-N microalloying process is a cost-effective way which has been widely used for high strength rebars, section steels, forging steels, seamless pipes, and CSP strip steels in China.

Keywords: V-N microalloyed steels, precipitation strengthening, intra-granular ferrite nucleation, ferrite grain refinement, weldability

INTRODUCTION

V, Ti and Nb microalloying technology, which has been developed since 1960's, has achieved more and more application in worldwide range due to its significant technical and economical advantages. Microalloying technology has represented great promotion of development of iron & steel industry, which is acknowledged as one of the greatest physical metallurgical achievements in the 20th century. On the basis of research and development for more than half century, microalloying technology, including its alloy design principle, production process, application area, etc, has been developed and improved greatly. Microalloyed steel has become a kind of indispensable structural steel.

Among the three microalloying elements of V, Ti and Nb, V is generally regarded to improve the strength of steel through precipitation strengthening. According to relevant researches during the past two decades, it has been found that increasing of nitrogen in V-containing steel is very necessary to improve the precipitation strengthening of vanadium[1-3]. The concept that nitrogen should be as an important alloy element in V microalloyed steel have been widely accepted. Further researches in recent years also demonstrate that V-N microalloying can not only improve precipitation strengthening effect of V, but also effectively refine ferrite grain size through promoting nucleation of intragranular ferrite [4-7]. The most updated researches [8-9] indicate that increasing of nitrogen content improves the welding adaptability of V-Ti steels. These new research results have founded the application base of V-N microalloying technology.

This paper reviews briefly the metallurgical principles of N effects in V-containing steels and the most updated research results in the field of V-N microalloying technology. The applications of V-N microalloying technology have been introduced in high strength rebars, high strength heavy section steels, microalloyed forging steels, CSP strip steels and heavy plate for offshore platform.

ROLES OF NITROGEN IN V-CONTAINING STEELS

The influence of nitrogen on precipitation of V(C, N)

As it is well known that all the carbides /nitrides of V, Nb and Ti have NaCl cubic crystal structure, the similarity of their crystal structure enables them to show continuous or extended mutual solubility. According to the published solubility data[3] of VC, VN, the solubility of VN is about two orders of magnitude lower than that of VC, either in austenite or in ferrite, thus the increasing of nitrogen content in V-containing steels would produce the important impact on precipitation of V(C,N). Thermodynamic calculation results indicates that, increasing of N content in V steel can improve the driving force of V(C, N) and raise the precipitation temperature of V(C, N), as shown in Figure 1. Under the condition of low N content, the precipitated phase mainly shows vanadium carbide; while as the N content increases, it mainly shows vanadium carbonitride; and when N content reaches 200ppm, the precipitate might be VN or V(C,N) with rich N within the whole precipitation temperature range.

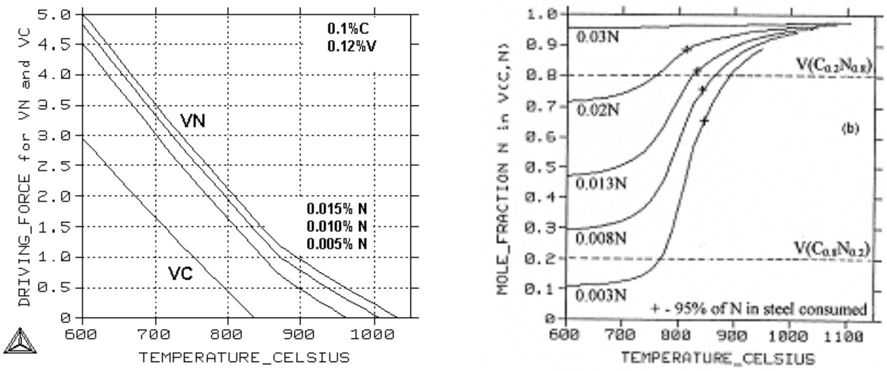


Figure 1 *Example of thermodynamic calculation on the precipitation of nitrides, carbonitrides and carbides* (a)Chemical driving force for precipitation of VC and VN in 0.1%C-0.12% V steel; (b) Mole fraction N in V(C,N) precipitation phase as a function of N content in 0.10% V steels[3]

The influence of N content on the distribution of V in steel is shown in Figure 2 [7]. It can be seen from the figure that there is a significant difference in vanadium distribution between V-steel and V-N steel. In V-steel with low content of N, nearly 60% of total V is solid-soluted in the matrix, and only about 35% of total V precipitated in the form of V(C, N). However, the situation is changed in V-N steel with high content of N: 70% of total V is precipitated in the form of V(C, N) and only 20% of V is solid-soluted in the matrix. This experimental results indicate that when there is insufficient N in V-containing steel, most of V fails to play its effect of precipitation strengthening and is wasted; and after increasing of N, V that is previously in the solid solution state changes into the precipitation state, so as to fully play its effect of precipitation strengthening.

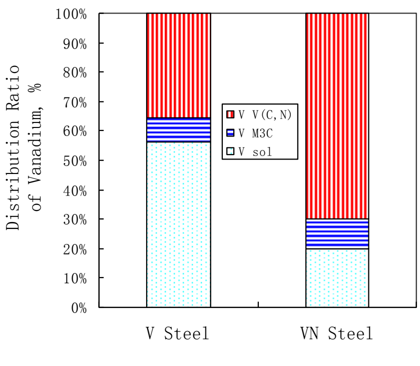


Figure 2 Comparison of vanadium distribution in V and V-N as-rolled rebars (0.20%C- 0.11% V)[10]

Effect of N on precipitation strengthening of vanadium steels

As above-mentioned, increasing of N content promotes the precipitation of V in the steel, resulting in ~70% of total V in form of V(C,N). Furthermore, as shown in Figure 3, the size distribution of precipitated V(C, N) particle shows that increasing of N content promotes the increasing in mass fraction of fine particles less than 10nm, which are main contributor to precipitation strengthening effect.

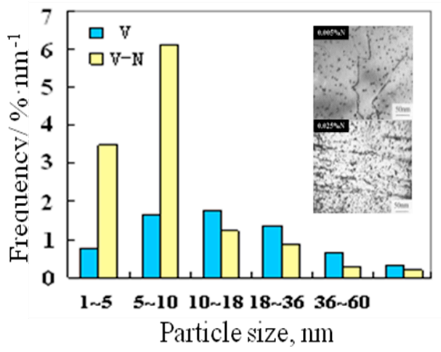


Figure 3 The size distribution of V(C,N) particles in V steel and V-N steel (0.20%C- 0.11% V) [10]

Zajac et al's results indicated that the precipitation strengthening effect of V(C,N) is linearly increased along with the increasing of N content in various C content V steels, as shown in Figure 4. For V containing steels, the strength is increased by more than 6MPa when N is increased by 10ppm. According to Figure 4(b), it can also be seen that C content in steel has significant influences on precipitation strengthening of V. With the increasing of C content, precipitation strengthening effect of V is also increased linearly. Increasing of 0.01% C in V containing steel may contribute to 5MPa of precipitation strengthening.

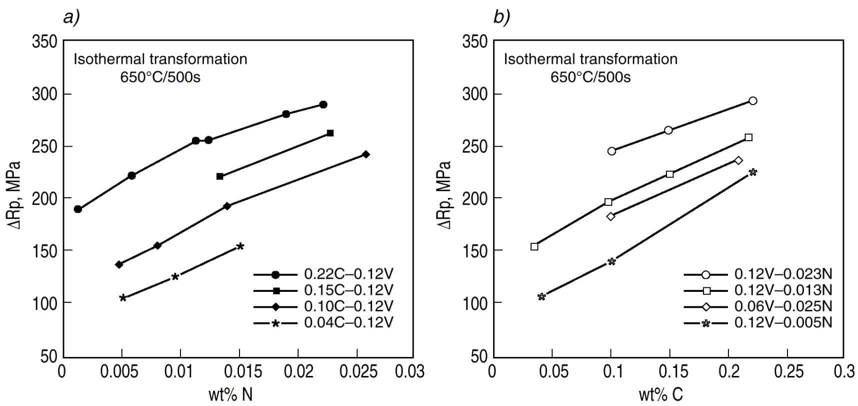


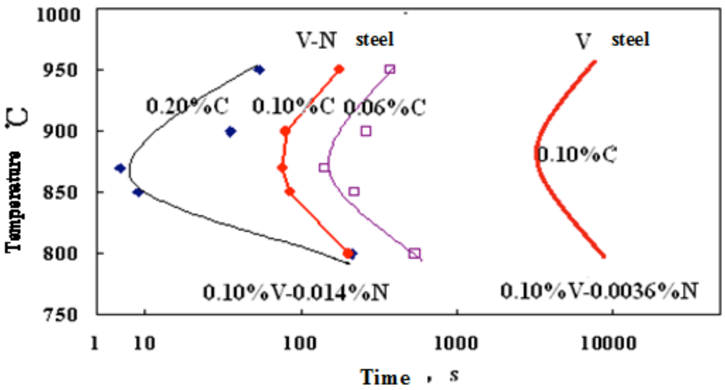
Figure 4 Deduced values of precipitation strengthening for isothermally transformed V-steels (650°C/500s). (a) as a function of N-content (b) as a function of C-content[2]

Effect of N on grain refinement of V-containing steels

Intra-granular ferrite (IGF) technology is used to refine the ferrite grain size, which has become an important method of grain refinement. According to the misfit theory, the ferrite nucleation potency of particles is determined by the lattice coherency between particles and ferrite at the interface. Recent experimental results clearly reveal that VN and TiN particles have the strong ferrite nucleation potential, which promote the nucleation of intra-granular ferrite plates. The increasing of N content in V-N steel raises the driving force of V(C,N) precipitation, thus promotes the precipitation of V(C,N) in austenite, which creates proper condition for IGF nucleation.

(1) Kinetics of V(C,N) precipitation in austenite in V-N steel

Figure 5 shows the precipitation-temperature-time curves of V(C,N) precipitation in austenite region of low carbon V steel and V-N steels. It can be seen clearly from the figure that the precipitation of V(C,N) in austenite is very slow in 0.10%C-0.10%V-0.0036%N low N steel, and the start time of V(C,N) precipitation is 390s at the nose temperature of 850-870℃. however, after increasing of N content, the kinetics condition for precipitation of V(C,N) in austenite is greatly improved, and precipitation start time at the nose temperature is reduced to 76s in 0.10%C-0.10%V-0.014%N high N steel. It can also be seen from Fig.5 that C content in V-N steels has significant influences on V(C,N) precipitation in austenite region, and the precipitation start time at the nose temperature can be reduced to less than 10s in 0.20%C V-N steel.



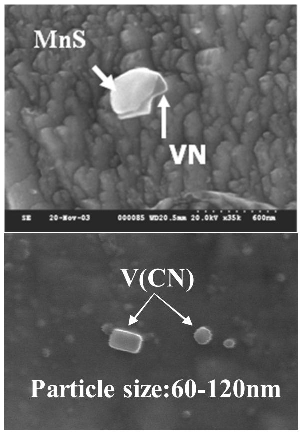


Figure 5 Precipitation-temperature-time diagram of V(C,N) in low carbon V-steel and V-N steel

(2) Nucleation of intragranular ferrite on VN particles

V(C,N) particles precipitated in austenite of V-N steel as shown in Fig.5, which will become the core of intra-granular ferrite nucleation, contribute to the ferrite grain refinement. The experimental results in Figure 6 clearly indicated that there is a significant ferrite grain refinement in hot-rolled V-N steel due to the nucleation of intra-granular ferrite on VN particles compared with the V-steel and C-Mn steel.

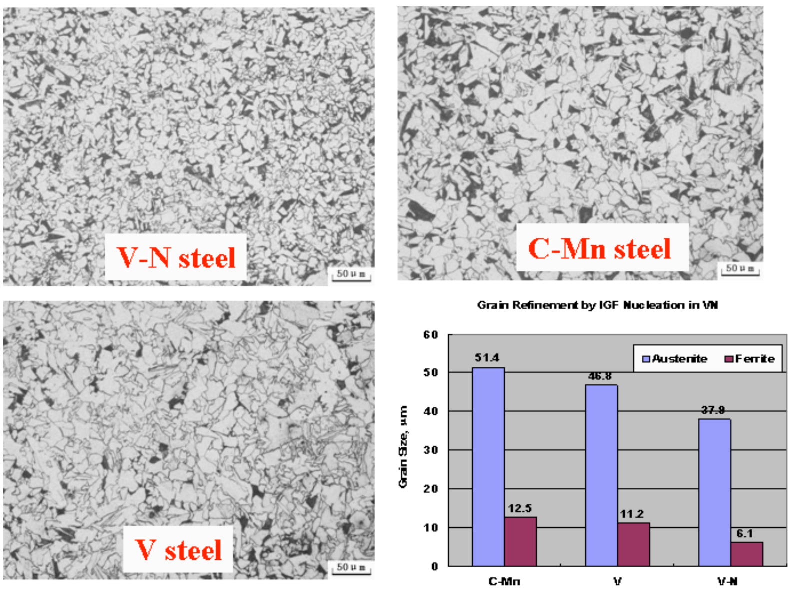


Figure 6 Intragranular ferrite nucleus in V-steel with high N content [12]

On the basis of intra-granular ferrite formed on VN particles, obvious ferrite grain refinement is realized in V-N microalloying steel. These new research results have broadened the understanding of V microalloying technology, i.e. Vanadium can use not only for obvious precipitation strengthening, but also by effectively for ferrite grain refinement.

(3) new TMCP technology- VCN-PCRP

With the combination of the technologies of the intragranular ferrite (IGF) on VN particles and the recrystallization controlled rolling (RCR), a new controlled rolling technology has been developed, which is named as **V(CN) Precipitation Controlled Rolling Process (VCN-PCRP)**. The diagram of the physical metallurgical principle for this technology is illustrated in Figure 7. V-N muilt-microalloying design is the key to guarantee the precipitation of VN particles could occur in austenite. And the high solubility of V(C,N) creates the condition for the relatively low reheating temperature and benefit to control the growth of coarse initial austenite grain during reheating. The rolling process may be divided into two stages: in the first stage, the rolling is implemented during high temperature recrystallization area so as to reduce the austenite grain size through the repeated recrystallization of austenite; in the second stage, the rolling deformation is within the range of VN precipitation temperature to induce VN precipitation in austenite, so as to provide the cores for IGF nucleation during ferrite phase transformation, and finally realize ferrite grain refinement.

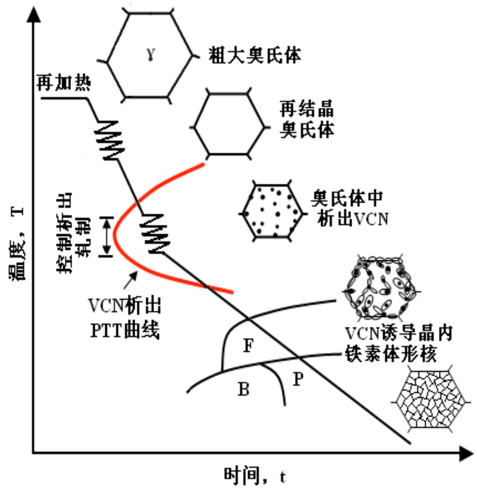


Figure 7 Schematic illustration of VCN-PCRP process

Effect of N on weldability of V-Ti steel

V-Ti microalloying design is widely used in various steel products with attractive strength and toughness match through suitable controlled rolling and cooling process. Recently, the effect of proper increasing of N content in V-Ti steel on weldability were investigated. The results show that increasing N content on the basis of V-Ti steel make the impact energy of CGHAZ improved. As shown in Figure 8, the CGHAZ impact values of V-N-Ti steel are apparently higher than V-Ti steel and Nb-Ti steel at different $t_{8/5}$ times, in particularly, V-N-Ti steel shows superior impact values of CGHAZ to Nb-Ti steel when $t_{8/5}$ time longer than 100s. Figure 9 shows the microstructure of CGHAZs at $t_{8/5}$=180s. There is significant difference in CGHAZ microstructure among three kinds of steel. For V-N-Ti steel, the ferrite is dominant constituent and its volume fraction is more than 80%, while for Nb-Ti and V-Ti steel, the bainitic microstructure is main constituent. It means that the increasing of N content of V-Ti steel changes the microstructure constituent of the CGHAZ. Obviously, the polygonal ferrite is the soft phase in microstructure and facilitates to improve the impact toughness of the CGHAZ. Further studies indicate that the increasing of N content in V-Ti steel refines the austenite grain size before phase transformation, raises the start temperature of $\gamma \rightarrow \alpha$ phase transformation and promote the IGF nucleation. All these factors lead to the increasing of ferrite volume fraction in CGHAZ of V-N-Ti steel.

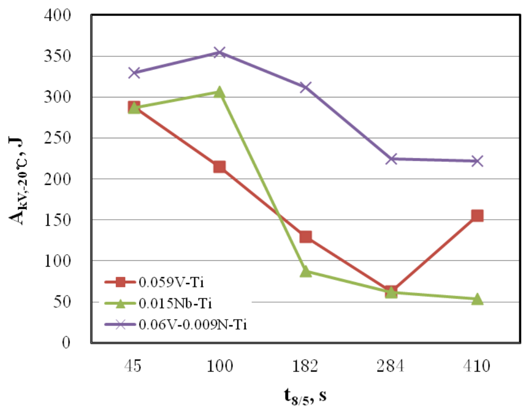
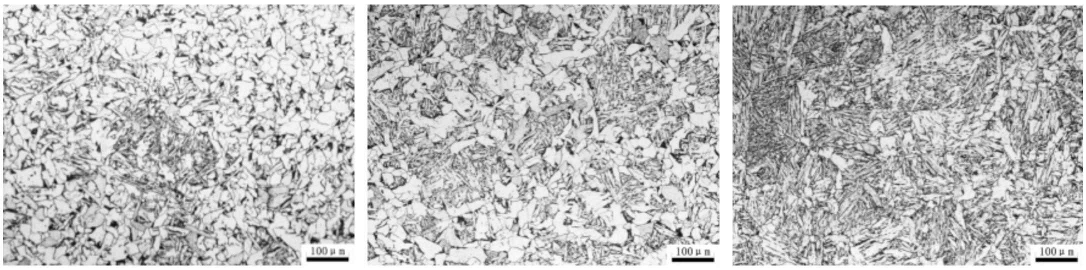


Figure 8 Comparison of impact energy of simulated coarse grained HAZ between V-N-Ti, V-Ti and Nb-Ti steels (0.08%C-1.2Mn-0.06%V)



V-N-Ti steel V-Ti steel Nb-Ti steel

Figure 9: Optical microstructures of the CGHAZ at $t_{8/5}$ =180s

APPLICATION OF V-N MICROALLOYING TECHNOLOGY

High-strength rebar

Reinforcing bars, as the biggest steel products in China, take about 1/4 of the total steel production. As shown in Figure 10, China consumed huge number of rebars each year, and the total rebars production reached 206 million tons in 2013. In order to satisfy the requirements of rapid growth of the construction industry, as the output of rebars is increased, the government is all the time trying to push the upgrading of rebar in last 15 years. It can also be seen from Fig.10, the output of Grade 3 rebars in China was only about 200,000 tons in 2000, less than 1% of the total rebar output, while in 2013, the output of Grade 3 rebars reached 124 million tons, covering 60% of the total rebars output. The rapid growth of high strength rebar market has forcefully pushed the application of V microalloying technique in China steel industry.

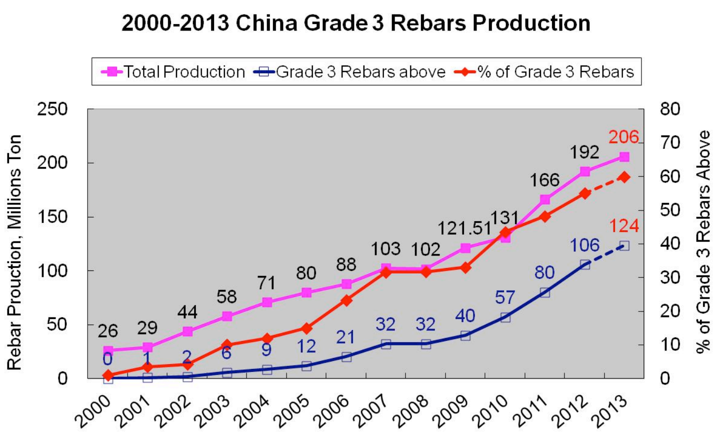


Figure 10 China rebars Production

As we known, the characteristics of rebars production process are always with high rolling speed and high finish rolling temperature, and it is quite suitable to apply vanadium microalloy technique in rebar alloy design. However, V addition will markedly raise the production cost of rebars, and it will be very sensitive to rebars products. A lot of research results indicated that nitrogen is a cost-effective alloy element in vanadium microalloyed steels, and V-N microalloyed process will be a cost-effective way for high strength rebars production. As shown in Figure 11, for a given strength requirement, the V content in V-N microalloyed rebars steel can be markedly saved compared with only V rebars steel, and there is a significant cost saving in V addition.

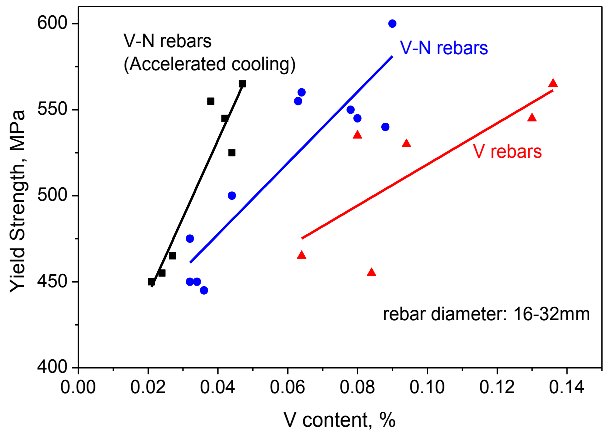


Figure11 Effects of V, N and ACC process on the strength of rebars

On the basis of research works in laboratory and mill scale trials, we developed low cost V-N microalloyed high strength hot-rolled rebars produced by converter + continuous casting process in China, and the typical chemical compositions of grade 400MPa and 500MPa are listed in table Ⅰ. In V-N microalloyed rebar, V content of grade 400MPa rebars is reduced to 0.02-0.04%, which is only half of that V-Fe microalloyed rebar. The regression results on the strength data of the mill trial rebars indicated that the yield strength of V steel and V-N steel can be expressed as following formula:

V-Steel: $Rel = Rel_{(20MnSi)} + 1056\times[V\%]$

V-N Steel: $Rel = Rel_{(20MnSi)} + 1994\times[V\%]$

It can be seen clearly from regression results that the strengthening effect of vanadium in V-N rebars is almost double compared with that in V rebars.

Table Ⅰ Typical chemical compositions of grade 400MPa and 500MPa hot-rolled rebar in China (wt%)

Grade	Alloy	C	Si	Mn	P, S	V	N	Gauge, mm
400MPa	V-N	0.20-0.24	0.45-0.60	1.25-1.45	<0.035	0.02-0.03	0.008-0.012	Φ6-Φ16
						0.03-0.04		Φ16-Φ40
	V-Fe	0.20-0.24	0.45-0.60	1.25-1.45	<0.035	0.05-0.07	0.003-0.006	Φ10-Φ16
						0.07-0.09		Φ16-Φ40
500MPa	V-N	0.20-0.24	0.45-0.60	1.40-1.55	<0.035	0.05~0.07	0.010-0.015	Φ16-Φ32
						0.07-0.09	0.012-0.018	Φ32-Φ40
	V-Fe	0.20-0.24	0.45-0.60	1.40-1.55	<0.035	0.07~0.12	0.003-0.006	Φ16-Φ32

The experimental results demonstrated that V-N rebars are provided with high stability of mechanical properties, which strength fluctuates within 75MPa, and good weldability and Anti-Seismic Performance, can satisfy Grade 1 requirement of earthquake resistance.

Section steels

Section steels are widely used in railways, bridges, shipbuilding, civil engineering and so on. With regards to production of section steels, the majority of section steels is produced under the condition of hot rolling by groove rolling plus air cooling. Its rolling process is of the similar characteristics to those of rebar rolling. For section steels with heavy section size and complicated section shape, microstructure and property imhomogeneity along cross-section may occur due to non-uniformity of deformation and cooling pattern. At present, these steels are mainly of C-Mn steels with yield strength levels 235 MPa and 345 MPa and with quality levels A and B (Grade A do not require the impact energy and Grade B requires the impact energy at 20℃). With the development of relevant industries, the demands for high strength, high toughness and large gauge products of section steels are increasing. Here are two examples showing the application of V-N microalloying technology in section steels is very promising.

The angle steels are used for construction of electricity power tower. With the increase of transmission voltage for high voltage transmission about 500KV for AC or DC power transmission and ultra-high voltage transmission (1000kV for AC power transmission or 800kV for DC power transmission), the strength level of angel steels is needed to improve from Q235MPa to Q345MPa and Q420/Q460MPa, meanwhile, the quality grade is raised from grade A and B to grade C and D (The impact energy at 0℃, -20℃ is required). Table Ⅱ compares to the chemical composition and mechanical prosperity of V-N and V microalloyed angle steels of Q420C and Figure 12 shows the comparison of impact energy at 0℃ of two kinds of steels. It can be seen that V-N microalloyed steel is featured with improved toughness, fully meet the requirement of Q420C. Recent results show that, the vanadium addition can be reduced by half when applying V-N microalloying producing Q420 angle steel, thus significantly reduced the production cost.

Table Ⅱ Chemical composition and mechanical property of angle steels of Grade Q420

Alloying	Grade	Chemical composition，wt%						Mechanical properties		
	Q420	C	Mn	Si	V	N	S,P	Rm/mm^2	Rel/mm^2	A，%
V-N	Max.~Min	0.17-0.13	1.35-1.20	0.40–0.30	0.08 ~0.06	0.015-0.020	<0.020	610~570	470~450	27 ~ 25
V	Max.~Min	0.20-0.15	1.50-1.30	0.40–0.30	0.10 ~0.15	0.005	<0.020	650~590	480~440	23 ~ 20

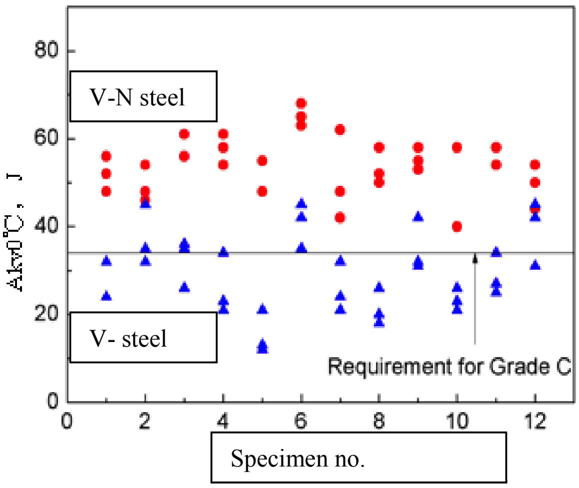


Figure 12 The impact energy at 0℃of V-N steel and V-steel of Grade Q420C

Bulb-flat steel is a kind of section steel used for stiffeners in ship hull structures. High strength, high toughness, good weldability and large gauge are the developing trend of bulb-flats for shipbuiding. According to China national standard GB/T9945-2001 "hot rolled bulb flats" executed in the production of bulb-flats, the maximum size of bulb-flats is 430mm(b) × 20mm(h), the highest strength level is D/E40 with a yield strength 390MPa, D and E represent the quality level, which need to meet the requirement of -20□ and -40□ low temperature impact toughness, respectively.

The previous D40 bulb-flat steel took the alloy design of Nb-V muilt-microalloying, whose chemical composition is similar to typical one of E36 plate (Table Ⅲ). This kind of steel shows the lower yield strength and low-temperature toughness at bulb position (Table Ⅳ). By using V-N microalloying design, especially controlling the ratio of V/N in steel, the uniformity of microstructure and properties at different positions of bulb flat is remarkably improved (Table Ⅳ). The outstanding advantage of V-N microlloying bulb flat is its significant increase in low-temperature toughness, shown in Figure 14, fully meeting toughness requirement (≥47J) of E40 bulb flat. It is concluded that V-N microalloying is the best choice in alloy design for large-gauged D/F40 bulb flats.

Table Ⅲ Chemical composition of D40 bulb–flat steel

alloy	C	Mn	Si	S	P	V	Nb	Als	Ti	Ni	Cu	N
V-N	0.079	1.38	0.32	0.0015	0.011	0.088	/	0.026	0.012	0.24	0.24	160ppm
V-Nb	0.08	1.40	0.29	0.00	0.02	0.07	0.02	0.03	0.010	0.24	0.26	47ppm

Table Ⅳ Mechanical properties of D40 bulb–flat steel

Steel	Specimen position	R_m/MPa	R_{eL}/MPa	A/%	Hardeness/HV	$A_{kv,-20□}$/J
V-N	Bulb center	510	420	30	160	209
	Flat	520	425	26.5	173	223
V-Nb	Bulb center	545	400	28	152	17
	Flat	560	445	25	172	32

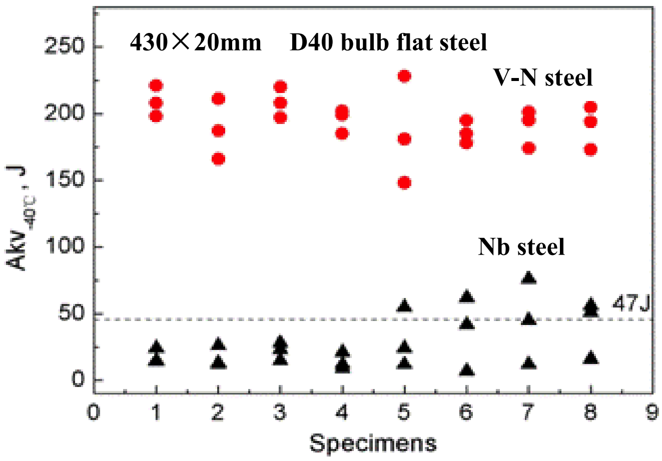


Figure 13 Comparison of impact energy at -40℃of V-N and Nb-V bulb-flat steels

Microalloyed Forging Steels

By adding small amount of microalloying element V in the medium-carbon steel, the precipitation strengthening of fine V(CN) particles can improve the strength of the ferrite-pearlite steel to achieve the level of traditional QT steel [12-14]. This is the basic design principle for microalloyed forging steels. The typical V addition is within the range of 0.06-0.20% according to the different requirements of strength.

It is necessary to enhance N content in vanadium microalloyed forging steels. The recent study results indicated that enhancing N content to 150-200 ppm in microalloyed forging steel will have a beneficial effect in three areas: (1)to improve the stability of dispersed TiN [15-16]; (2) to contribute to the microstructure refinement and increasing of ferrite volume fraction by refining prior austenite grain and promoting the nucleation of intragranular ferrite (IGF) induced by VN precipitated in austenite; (3) to increase the precipitation strengthening of V(CN).

Table Ⅴ Effect of nitrogen on microstructure and properties of steel 38MnVS6

N ppm	prior austenite grain D_γ /μm	Pearlite colony /μm	Ferrite Volume /%	VCN Precipitates，%		Strength，MPa		Impact energy
				Total	<18nm	R_m	$R_{p0.2}$	A_{KU2}/J
36	91.0	13.9	4.0	0.671	0.0158	897.5	600.0	53.0
98	49.8	12.5	11.8	0.693	0.0165	829.5	528.0	81.7
190	34.5	11.5	15.8	0.936	0.0375	905.5	636.5	70.0
260	50.8	10.8	22.4	-	-	886.0	632.5	55.3

High strength CSP strip steels

The metallurgical characteristics for thin slab continuous casting and rolling (TSCR or CSP) technology, such as near net shape con-casting, fast solidification, low soaking temperature, heavy pass deformation, etc., bring the challenge for the alloy design and production of HSLA CSP strip steels compared with the traditional hot rolling strip. Firstly, the traditional HSLA steels designed with the compositions within peritectic range (C content of 0.07%-0.15%) are difficult to produce; Secondly, Nb microalloying technology is facing with problems of cracks and mixed-grain on the process, especially for EAF steel with high normal N content. Taking into consideration of the above mentioned features, the alloying design basis with V-N microalloying technology has brought an effective method for development of high strength product on the thin slab continuous casting and rolling [17-19]. Nucor Steel Company developed a series of high strength CSP strip steels with yield strength of 350 -550 MPa and applied the design of low carbon (＜0.07%) and V-N microalloying technology.

In China, we successfully developed 550-600MPa high-strength CSP strip steels with improved formability, which applying V-N microalloying design, as shown in Table Ⅵ. The microstructure of industrially trialed strips with different thickness contain ferrite and a small amount of pearlite and is relatively uniform along the thickness direction. The strips less than 6mm in thickness get ultrafine grain structure with ferrite grain size smaller than 5μm.

Table Ⅵ Chemical composition and mechanical properties of industrialized strips in China

	Chemical composition, %						Mechanical property (Trans.)				
Thickness mm	C	Si	Mn	V	N	Als	R_m MPa	$R_{p0.2}$ MPa	A %	$A_{kv,-20℃}$ J	Ferrite grain size, μm
4.5	0.067	0.19	1.55	0.16	0.020	0.027	700	632.5	24.3	/	3.7
6							700	607.5	25.0	56.0	4.2
8							680	595	24.3	31.3	5.9
12							702.5	607.5	25.0	32.5	7.6

Charpy impact specimen: 5*10*55,7.5*110*55 and 10*10*55 are for strips of 6mm, 8mm and12mm, respectively

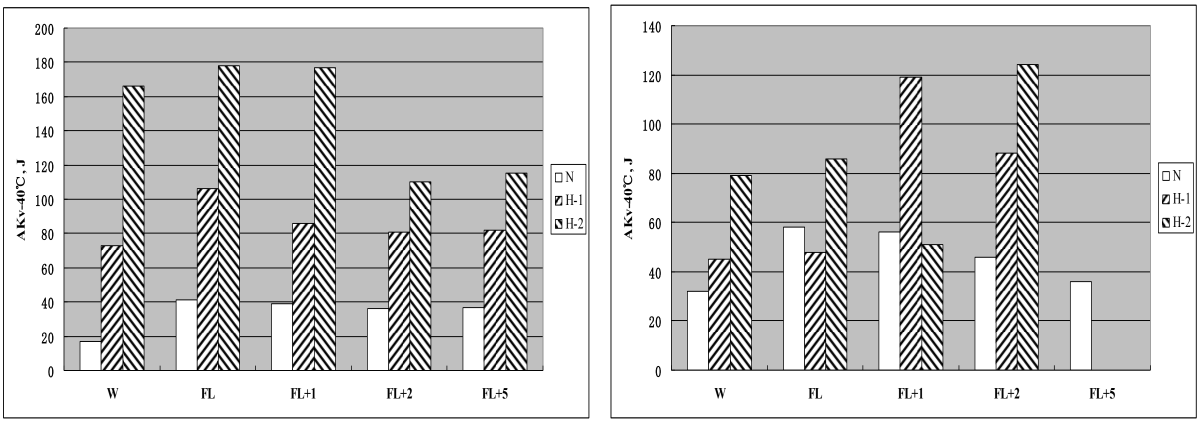
Heavy plate for offshore platform application with high heat input welding

Table Ⅶ lists the chemical composition of three kinds of plates used for offshore platform. Steel N-E36 is Nb-V microalloyed steel used currently for offshore platform; steels H-1 and H-2 are the new developed of V-N microalloyed steels. These three kinds of plates have the same microstructure of ferrite plus pearlite under normalizing condition and all meet the requirements of strength, low-temperature toughness and Z-direction performance as well processing performance.

Figure 14 shows the impact energy at -40□ at different positions of welded joints with welding heat inputs of 50kJ/cm and 100kJ/cm, respectively. It is seen that the new developed V-N microalloyed steels H-1 and H-2 shows improved values of impact energy in HAZ compared with V-Nb microalloyed steel.

Table Ⅶ Chemical composition of the experimental steels

Steel	C	Si	Mn	Ni	Cu	Als	Ti	V	Nb	N	Ceq
N	0.16	0.16	1.60	0.34	--	0.027	0.008	0.043	0.036	0.004	0.46
H-1	0.15	0.28	1.55	0.005	--	0.036	0.011	0.074	--	0.009	0.42
H-2	0.13	0.32	1.48	0.24	0.25	0.035	0.012	0.05	--	0.009	0.43



50kJ/cm 100 kJ/cm

Figure 14 Impact energy at -40℃ of welded joint

CONCLUSIONS

N is a cost-effective alloying element for V-containing steels. V-N microalloying technology is to use cheap N element to optimize precipitation of V, so as to better extend the grain refinement and precipitation strengthening effects and significantly improve strength of steel. V-N microalloying technology can realize high strength with low content of V, therefore less V is needed to achieve the desired strength and save cost for steel production.

In V-containing steel with high content of N, precipitation of VN in austenite offers effective cores for the intragranular ferrite nucleation, and on the basis of VN induced intragranular ferrite nucleation, V can realize refinement of ferrite grain. Therefore, in V steel with high content of N, V not only plays the traditional effect of precipitation strengthening, but also use by effectively for ferrite grain refinement.

V-N microalloying technology has been widely used for the development of high strength rebars, high strength section steels, microalloyed forging steels, high strength CSP strip steels etc, and forcefully pushed the application of V microalloyed steels in China.

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