La Metallurgia Italiana
International Journal of the Italian Association for Metallurgy

Heterogeneous intragranular nucleation of ferrite in high strength low alloy steels

V. Colla, R. Valentini, M. De Sanctis, R.A. Ishak Nakhla, M. Castagnini

In the production of High Strength Low Alloy (HSLA) steels with low carbon content markedly high yield strength (>600 MPa) can be achieved thanks to the possibility of finely control the microstructure resulting from appropriate thermomechanical cycles, as small additions (<0.1%) of strong carbide/nitride formers (Nb, Ti, V) is used to attain higher mechanical properties. In the final controlled rolling stage, the precipitation of carbides/nitrides arrests the recrystallisation of the deformed austenite, therefore, multiplies the preferential ferrite nucleation sites during successive cooling. However, this process is limited to small sheet thickness due to the limited rolling capacity of the industrial rolling mills. This work aims at evaluating the grain refining possibility in long heavy profiles taking advantage of a different strengthening mechanism defined as precipitation hardening. This mechanism introduced new preferential ferrite nucleation sites inside the austenite grains initiating at the vanadium carbide/nitride precipitates at high temperature (intragranular ferrite nucleation). Samples of an industrial commercial carbon steel with different vanadium content were casted in two different profiles at two different rolling finish temperature. The profiles were metallographically characterized to determine the volumetric fraction of ferrite and perlite microconstituents, and the mean grain size, and the perlite inter-lamellar distance was measured. The preferential intragranular ferrite nucleation sites were analysed through Transmission Electron Microscopy. A model derived from literature and based on semi-empirical equations was adopted to estimate the mechanical properties as a function of the microstructure and to explain the effect of V on such final properties.

KEYWORDS: HIGH STRENGTH LOW-ALLOY STEELS, NUCLEATION, MODELLING

INTRODUCTION

High Strength Low Alloy (HSLA) steels combine high strength, low ductile-brittle transition temperature and excellent weldability, and are suitable for a wide range of applications. These properties are achieved by combining small additions of some alloying elements (Ti, Nb, V) and careful control of the time-strain-temperature sequence during controlled rolling.

Thermomechanical Control Process (TMCP) is a very powerful technique for increasing strength, toughness and weldability of HSLA steels through austenite conditioning to produce an as fine as possible ferrite by controlling temperature and deformation conditions during hot rolling. The method was initially adopted for thick sheet steels and perfectioned to achieve excellent mechanical properties. The adaptation of TMCP to steel profiles was implemented by upscaling results from Yonei et al. [1] However, the main issue with TMCP for thick profiles arises from local differences in thickness. In

V. Colla
Scuola Superiore Sant'Anna, TeCIP Institute, Pisa, Italy
valentina.colla@santannapisa.it

R. Valentini, M. De Sanctis, R.A. Ishak Nakhla, M. Castagnini
Università di Pisa, Department of Civil and Industrial Engineering, Italy

renzo.valentini@unipi.it, massimo.desanctis@unipi.it, r anda.ishak@unipi.it

the case of "H" shaped profiles, the maximum thickness ratio between flanges and web can rise up to 3, which generates a large difference in conditions such as the rolling temperature or the reduction ratio between web, flanges and fillet during hot rolling. For high product thickness, other well-known issues arise, e.g. excessive rolling load, too long residence times, low finishing temperatures and cooling rates. To overcome these problems, grain refinement by nucleation of intragranular ferrite on precipitates can be applied. Grain refining is the most useful steels strengthening mechanism, holding the unique capability to increases both strength and toughness [2].

Several literature works suggest that VN or VC particles precipitated into the austenitic grains during or after hot rolling can help intragranular ferrite nucleation [3,4] provided that the rolling and subsequent cooling are suitably controlled. Therefore, TCMP plays a fundamental role in the achievement of a fine intragranular microstructure of ferrite or acicular ferrite in V micro-alloyed steels, being this strongly dependent on size and number of nucleated particles as well as on the hot rolling temperature cooling rate.

The present work investigates how the mechanism of precipitation hardening can be applied in the production of long heavy profiles through TCMP by adopting a model based on semi-empirical equations to assess the benefits of V addition in terms of final mechanical properties based on the obtained microstructure.

MATERIAL AND METHODS

Samples of 3 industrial commercial carbon steels with different V content (0% V, 0.03% V, 0.07 V) were casted in 2 different profiles, H-profile (IPN 100) and U-profile (UPN 180), at two different rolling finish temperature. Table 1 shows the metallurgy of the 3 considered grades, while Table 2 shows the mechanical characterization of the considered products in terms of Ultimate Tensile Strength (UTS), Yield Strength (YS), percentage elongation ($El_{\%}$) and resilience measured at different temperature values using a V-shaped specimen (KV).

Tab.1 - Metallurgy of the considered steel grades.

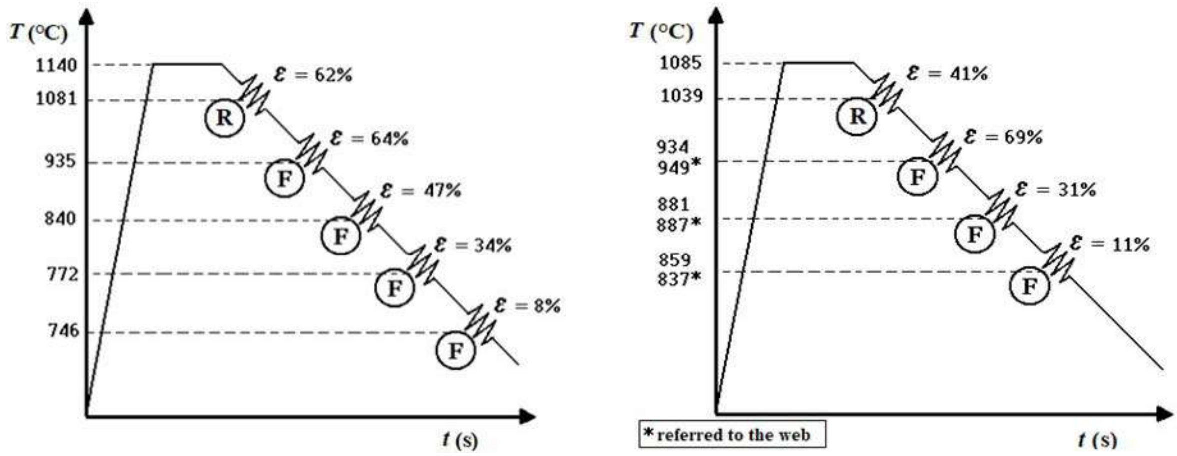
Steel grade	**Metallurgy (wt%)**													
	C	Mn	Si	P	S	Cr	Ni	Mo	Cu	Sn	Al	V	N	Ti
G_a	0.19	1.35	0.32	0.019	0.013	0.11	0.06	0.01	0.17	0.008	0.005	-	0.009	0.012
G_b	0.19	1.34	0.32	0.010	0.023	0.06	0.05	0.01	0.16	0.007	0.003	0.03	0.0107	0.002
G_c	0.185	1.33	0.32	0.012	0.029	0.09	0.07	0.01	0.19	0.008	0.002	0.07	0.0112	0.002

Tab.2 - Mechanical characterization of the considered products.

Profile	**Steel grade**	**UTS (MPa)**	**YS (MPa)**	**El%**	**K_V (J)**			
					0°	**-20°**	**-40°**	**-60°**
IPN 100	G_a	580	450	29.1	79-77-84	75-76-75	76-72-70	41-72-45
	G_b	607	470	31.5	62-72-63	65-58-88	61-85-95	41-33-29
UPN 180	G^a	568	384	33.3	76-74-105	79-86-99	47-67-29	48-26-49
	G_b	607	443	29.3	51-86-83	53-50-42	52-29-37	11-15-10
	G_c	661	516	21.9	25-37-28	19-20-23	11-12-8	5-5-6

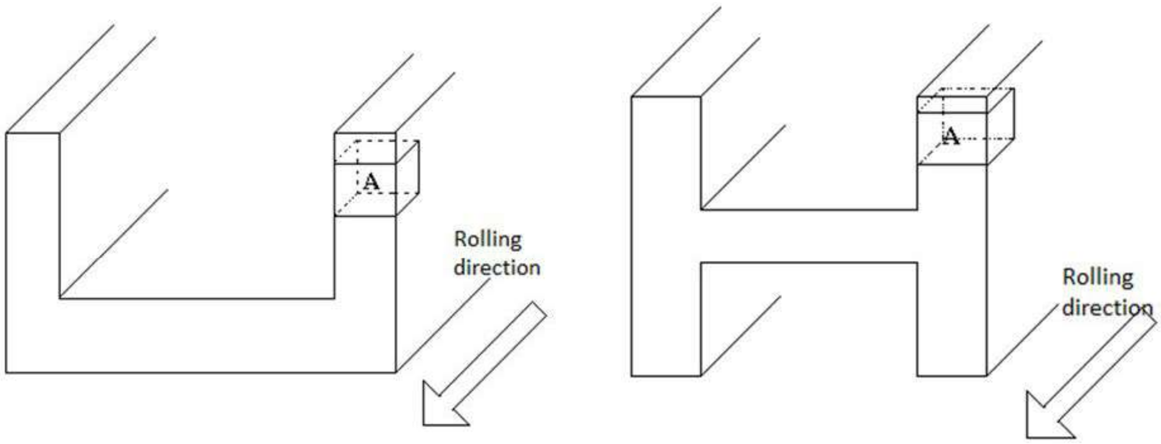
To characterize the thermal history of the considered products, temperatures at the exit of the different stages of roughing and finishing mills were recorded through pyrometers and are schematically represented in Figure 1 together with the profile deformation per pass (ε).

Samples of the obtained profiles underwent metallographic characterization through Optical Microscopy (OM) and Transmission Electron Microscopy (TEM) to determine the volumetric fraction of ferrite and perlite microconstituents, and the mean grain size as well as to measure the pearlite inter-lamellar distance. The preferential intragranular ferrite nucleation sites were also analyzed through TEM. Raw cubic specimens were taken as depicted in Figure 2 and underwent standard metallographic preparation.



(a) (b)

Fig.1 - Overview of thermomechanical cycle for (a) IPN 100; (b) UPN 180 steel grade A.



(a) (b)

Fig.2 - Raw specimen position for (a) IPN100; (b) UPN 180 steel grade A.

RESULTS OF THE EXPERIMENTAL CHARACTERIZATION

The main difference between the thermomechanical cycles of IPN100 and UPN 180 lies in the temperature field in the γ field at the finishing mill. For both profiles, significant plastic deformations in the low-temperature range are carried out in last four rolling cages: but for IPN 100 temperature at significantly lower. Table 2 shows that for steel grades G_a and G_b IPN 100 shows higher YS values compared to UPN 180, and for G_a also UTS is higher. Focusing of UPN 180 only, V addition increases both UTS and YS, but decreases ductility ($El_{\%}$). Finishing temperature and of V content also affect resilience at low temperature and brittle-ductile transition temperature (BDTT): for G_a BDTT<-60 °C, for G_b -60 °C<BDTT<-40 °C, for G_c BDTT~0 °C.

Although for IPN 100 the high-V grade was not tested, it is worth noting that for the medium-V grade no significant resilience decrease is observed with respect to grade G_a.

The results of the microstructural analysis carried out via OM and TEM are summarized in Table 3.

Tab.3 - Mechanical characterization of the considered products.

Profile	Steel grade	Ferritic phase f_α (%)	Pearlitic phase f_P (%)	Grain size d (μm)	Pearlite interlamellar distance S_0 (nm)
IPN 100	G_a	32,5	67,5	7,9	200
	G_b	27,9	72,1	6,1	150
UPN 180	G^a	33,2	66,8	10,1	210
	G_b	25,6	74,4	9,5	160
	G_c	31	69	7,1	120

Such as shown in Figure 3, the microstructure consists in all cases of a mixture of polygonal ferrite (A) and pearlite colonies (B), but a finer grain size is obtained via reduction of the temperature of the final rolling stages and via V addition (see Table 3).

The refinement of ferritic grain is primarily due to an increase in ferrite nucleation sites during laminate cooling and passage through the inter-critical field. In the finishing stages, plastic deformations are imposed at relatively low temperatures, at which the dynamic and static recrystallization of the austenitic structure is slower. During phase transformation at the cooling stage, ferritic grains tend to form on the heavily defective regions of grain boundaries. The cumulated plastic deformation within the austenitic grains allows to significantly increase the ferrite preferential nucleation sites, by introducing dislocations and deformation bands in the austenitic grains. It is also well known that V reduces the austenite recrystallization rate, as a result of both a slowing down of the grain boundaries motion due by inducing micro-deformations in the crystalline structure of the solid solution (the so-called solute drag), and a pinning force on the grain boundaries due to possible precipitation of V (C,N), which is highly favoured if plastic deformations are imposed at sufficiently low temperatures (the so-called strain-induced precipitation).

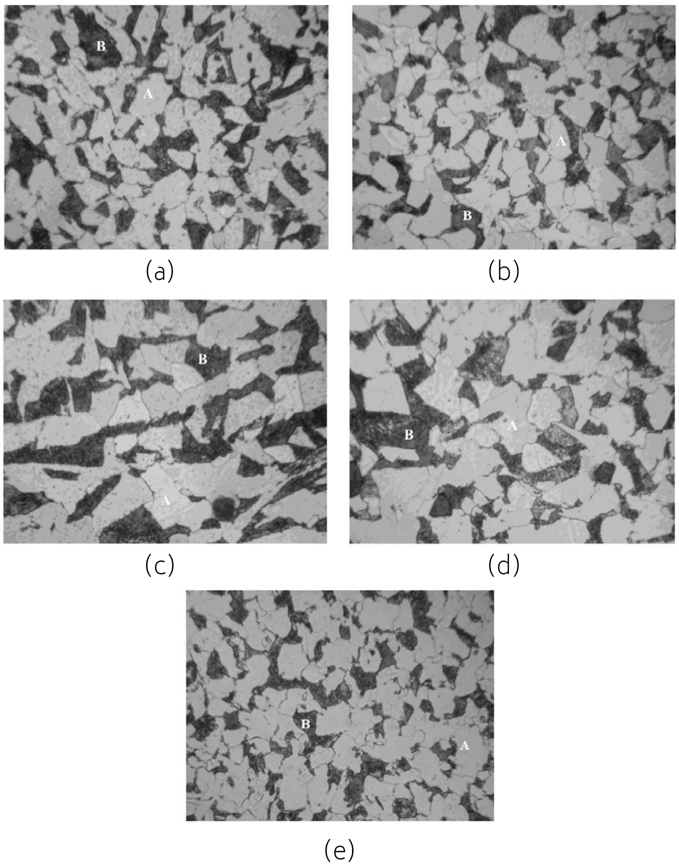


Fig.3 - Optical micrography (1000x) for: a) IPN 100 G_a; b) IPN 100 G_b; c) UPN180 G_a; d) UPN180 G_b; e) UPN180 G_c.

DISCUSSION AND CONCLUSION

Literature results show that the maximum temperature at which recrystallization is stopped by carbide/ carbonitride precipitation decreases as the ε increases [5]. Considering the high deformation rates imposed in the rolling mill, it can be assumed that recrystallization stops at T~900 °C in the considered V-alloyed steels. Therefore, only for IPN 100 the temperature at the finishing stages strongly favour the precipitation of V (C,N), while for UPN 180 this effect appears significantly reduced. This explains the finer ferritic grain (see Table 3) obtained IPN 100 with a medium-V grade G_b, which is also finer than those obtained with the high-V grade G_c used for UPN 180. However, the relevant but not exceptional refinement of the ferritic grain does not fully justify the drastic strength increases obtained via V addition and the drop in resilience observed in the high-V grade for UPN 180.

In order to explain the obtained experimental results, considering the obtained structure is mixture of polygonal ferrite and pearlite colonies, literature models were used to compute UTS and YS based on the steel metallurgy and to the ferrite and pearlite grain size for C-Mn steels that do not contain V. In particular, the following two sets of empirical equations were used [6,7]:

$$UTS = f_\alpha^{\frac{1}{3}} \cdot \left(246 + 1140\sqrt{N_f} + 18.2d^{-\frac{1}{2}}\right) + \left(1 - f_\alpha^{\frac{1}{3}}\right) \cdot \left(720 + 3.5S_0^{-\frac{1}{2}}\right) + 97Si \qquad (\pm 45\ MPa) \tag{3}$$

$$YS = f_\alpha^{\frac{1}{3}} \cdot \left(35 + 58Mn + 17.4d^{-\frac{1}{2}}\right) + \left(1 - f_\alpha^{\frac{1}{3}}\right) \cdot \left(178 + 3.8S_0^{-\frac{1}{2}}\right) + 63Si + 42\sqrt{N_f} \qquad (\pm 45\ MPa) \tag{4}$$

$$UTS = 175.2 + 634.7C + 53.6Mn + 99.7Si + 651.9P + 472.6Ni + 3339.4N + 33.6Al + 11d^{-\frac{1}{2}} \tag{1}$$

$$YS = 21.8 + 26.1Mn + 60.2Si + 759P + 212.9Cu + 3286N + 58.7Al + 19.7d^{-\frac{1}{2}} \tag{2}$$

Table 4 compares experimental values of UTS and YS and their estimates obtained through Eq.s (1)-(4).

Tab.4- Experimental vs. estimated values of UTS and YS.

Profile	Steel grade	UTS (MPa)			YS (MPa)		
		experimental	Eq. (1)	Eq. (3)	experimental	Eq. (2)	Eq. (4)
IPN 100	G_a	32,5	67,5	7,9	200	200	200
	G_b	27,9	72,1	6,1	150	150	150
UPN 180	G^a	33,2	66,8	10,1	210	210	210
	G_b	25,6	74,4	9,5	160	160	160
	G_c	31	69	7,1	120	120	120

As expected, especially Eq.s (3) and (4) provide acceptable results for G_a, as this steel grade does not contain V, while the discrepancies are relevant for G_b and very significant for G_c for both profiles. The presence of micro-alloying elements, even if in very low percentages, certainly does not involve significant contributions in terms of hardening by solid solution, but it can lead to significant contributions in case of precipitation of carbides/carbonitrides coherent with the ferritic matrix, and this is the reason why V is used in some HSLA steels to increase YS of the final product [8]. The precipitation of these particles in the ferritic phase can provide and increase of about 80-100 MPa to YS thanks to the very small size of the precipitates (1-2 nm), to their volumetric fraction, and to their coherence with the ferritic matrix. However, it is well known that precipitation of coherent phases in ferrite heavily decreases ductility and resilience [8]. This explains the drastic increase in mechanical properties observed for UPN 180 in high-V steel grade (G_c) accompanied by a strong decrease in ductility at low temperatures. UPN 180 underwent the finishing rolling stages at relatively high temperatures (T~900 °C) that are very close to the maximum temperature of recrystallization stop. Therefore, plastic deformation contributed only to a limited extent to the precipitation of

V nitrides/carbonitrides. In other words, it is likely that for the high-V steel grade G_c rolled at the high temperature, the inter-critical field was crossed while austenite was significantly supersaturated with V. The solubility drop of V nitrides/carbonitrides in the austenite-ferrite transition and the pre-existing supersaturation of austenite has therefore led to a high precipitation potential of fine and coherent particles in ferrite. The result is, therefore, a significant increase in strength coupled to a decrease in ductility and toughness. This result is confirmed by micro-harness Vickers HV0.02 measurements carried out on single ferritic grains and shown in Table 5. A remarkable harness increase is observed in the ferritic grain for V-alloyed steel grades, with the highest value observed for G_c and UPN 180.

TEM analysis aimed at further highlighting the capability of particles of V (C,N) formed inside the previous austenitic grain to work as intergranular nucleation sites for ferrite.

TEM analyses were conducted as follows:

1) a ferritic grain was selected and oriented in correspondence with a main crystallographic pole.
2) Particles showing a strong image contrast that, thus, could present an orientation aligned with the ferritic matrix were selected.
3) The sample was tilted so as to align the beam with a second main crystallographic direction of the matrix, checking whether the particle under examination was also aligned with this second direction.

Limited to the examined particles, no crystallographic relationships were found between these small particles and the ferritic grain.

Tab.5 - Micro-harness Vickers $HV_{0.02}$ measurements carried out on single ferritic grains.

	IPN 100		UPN 180		
	G_a	G_b	G_a	G_b	G_c
Hardness ($HV_{0.02}$)	239-221-246 239-227-216 205-227-195 210-221-205 216-221-200	216-221-210 246-216-253 227-239-246 210-227-207 216-200-216	186-195-210 221-210-216 239-233-221 216-221-233 191-216-205	267-246-239 186-205-239 239-200-233 233-253-221 210-227-216	221-246-233 216-239-233 221-195-233 233-227-221 221-260-221
Mean hardness ($HV_{0.02}$)	219	225	214	227	228

The electron diffraction analysis was also extended to the nitrides possibly formed on the surfaces of the steel inclusions, considering the strong propensity for ferrite nucleation on V nitride/carbonitride particles. Such as shown in Figure 4.a, on the surfaces of the metal oxides distributed within the structure crystallization nuclei are formed first of titanium nitride particles and on these of V nitride/carbonitride particles; the latter capable of significantly favouring the nucleation speed of the ferrite during the crossing of the intercritical field and therefore of refining the final crystalline grain.

(a) (b) (c)

Fig.4 - a) Mechanism of generation of ferrite from an active oxide; b) IPN 100 G_b; c) UPN180 G_a.

REFERENCES

[1] T. Yonei, S. Suzuki, M. Oda, Y. Hida, K. Sugita e S. Mizoguchi, Development of Controlled Rolling Process for Low Temperature Wide-Flange Beams. Nippon Steel Tech.l Report, 1980, 16, 120-141.

[2] T. Kimura, Effect of Thermo-mechanical Control Process and Intragranular Ferrite Formation on Ferrite Grain Refinement of HSLA steels. CAMP-ISIJ, 1996, 9,1136.

[3] A. Ohmori, K. Oi, F. Kawabata and K. Amano, Effect of VN Precipitates on Grain Boundary and Intragranular Ferrite in a high N and V Bearing Steel. ISIJ International, 1998, 84, 797-803.

[4] F. Ishikawa, T. Takahashi and T. Ochi, Intragranular Ferrite Nucleation in Medium Carbon Vanadium Steels. Met.Trans., 1994, 25A, 929-936.

[5] S.F. Medina and A. Quisque, Influence of Strain Rate on Ricrystallisation-precipitation Interaction in V,Nb and V-Ti microalloyed steels. Mat.Sc.Tech., 2000, 16, 635-642.

[6] EC Directorat General of R&I Gutiérrez, I., Parker, S., Wadsworth, J., Property models for mixed microstructures: final report, Publications Office, 2003 ISBN: 92-894-6255-8,

[7] D.V. Doane e J.S. Kirkaldy, Hardenability concepts with applications to steel. AIME, 1978, 183.

[8] Annual Book ASTM, Properties and Selection: Irons, Steels & High-Performance Alloys. HSLA steels, 1995, 1.

[9] H. Satoh, S. Yoshida, Thermo-Mechanical Control Process for H-shapes and Its Characteristics. Technical Development Bureau Nippon Steel Corporation, 1990, 926.

TORNA ALL'INDICE >